ANALYTICAL CHEMISTRY.

On the Estimation of Boracic Acid in Borosilicates. C. Bodewig.

The estimation of boracid acid as such, or in simple borates, is of no particular difficulty, provided the re-agents, (especially hydrofluoric acid) are pure. The estimation becomes much more difficult in the presence of $Si\,O_2$.

In order to separate both acids, the mineral is fused with 4 times its weight of a carbonate, then extracted, first with water, afterwards with water to which a little ammonium carbonate has been added. It may then be washed, and the $Si\,O_2$ precipitated, by the addition of ammonium sesquicarbonate, or auminic chloride. The ammonium sesquicarbonate is added in portions of 2½ pts., until the carbonates are converted into bicarbonates. The ammonium carbonate thus obtained, is mostly expelled by heating on a water-bath, and the $Si\,O_2$, washed with water. By this means a certain amount of $Si\,O_2$ remains dissolved in the alkaline fluid, which, after expelling all the ammonium carbonate, may be precipitated, by adding zinc oxide, dissolved in ammonia. (Of course the ammonia must first be driven off by evaporation.)

(*Fres. Zeit. 1884. 113.*)

J. H. S., Jr.

On the Quantitative Estimation of Quartz in Rocks and Clays. J. Hazard.

The method depends upon the decomposition of silicates on being treated with dilute sulphuric acid, under high pressure.

(*Fres. Zeit. 1884. 158.*)

J. H. S., Jr.

On the Estimation of Nitric Acid, in the Presence of Other Acids, which mask its Reactions. Antonio Longi.

The estimation of nitrates in the presence of iodides, bromides, chlorates, bromates, etc., is not only difficult, but at times apt to produce grave errors. The author has now found a method, which depends upon the reduction of the oxygen acids, and the elimination of bromine, but more especially of iodine. The reduction of the oxygen acids, is effected with SO_2. The bromine and iodine are eliminated by G. Vortmann's reaction. In solutions containing besides the oxygen acids above mentioned, also ammonium salts, a part of the $N\,H_3$ becomes oxidized to nitric acid. In such cases, reduction must take place in the cold.

The solution to be examined, if acid, is first neutralized with Na_2CO_3, and then treated with SO_2, until it smells strongly of the latter. When reduction is complete, the solution is heated to expel excess of SO_2. Then add Na_2CO_3 to slight alkaline reaction and heat to boiling, until chromium, and the other heavy metals, have been precipitated. Filter, if a precipitate is formed, and acidify with acetic acid, to the solution now add a little acetic acid and lead peroxide, and boil till further addition of $HC_2H_3O_2$ and lead peroxide produces no fumes which color starch paper. The solution is then allowed to cool and filtered to separate the excess of lead peroxide. Any lead in solution is precipitated with sodium sulphate, and filtered. The filtrate is evaporated to dryness, the residue is dissolved in water, filtered if necessary, and then tested for nitric acid, with the usual reagents.

(*Fres. Zeit. 1884. 149.*)

J. H. S., Jr.

On a Simple Quantitative Method of Estimating Nitric Acid. E. Wildt & A. Scheibe.

This method is a modification of Schloesing's process, *i.e.*, reduction of the nitric acid, to nitric oxide, which is collected in a glass bell jar, and afterwards oxidized back to nitric acid by adding oxygen. The author uses an apparatus of peculiar construction.

The liquid to be examined is first heated to drive off air and after 15 or 20 minutes boiling a solution of ferrous chloride is run into the flask. After a few minutes the contents of the flask will turn brown, indicating reduction. The liquid is now heated to gentle boiling, and the nitric oxide generated, is freed from $H\,Cl$ by passing through a flask containing KOH. It then passes into a receiver, or gas holder, where it is oxidized back to nitric acid by the action of the oxygen of the air, and steam. The HNO_3, is then estimated in the usual way by titration.

(*Fres. Zeit. 1844. 151.*)

J. H. S., Jr.

Estimation of Phosphoric Acid.

A report of the sub-committee of the Chemical Trade Section of the London Chamber of Commerce gives the following results of analyses of a sample of Charleston phosphate submitted to five different analysts:

Phosphoric acid	26.55	26.20	26.48	26.60	26.00
Equal to phosph. lime	57.56	57.19	57.82	58.06	56.75

Four of the results were obtained by the magnesium method, the fifth is a mean of two determinations, one by the magnesium, one by the molybdate method. The report recommends that samples, finely ground by the manufacturer, shall be furnished for analysis and additional samples of the same, coarsely crushed, for determination of water, and that in case of a difference of more than 1½% of calcium phosphate in commercial analyses made by two different chemists, the results shall be submitted to both of them for their opinion as to the cause of difference. The report is signed by five members. (*Chem. News, XLIX.* 230.) A. A. B.

On the Estimation of Phosphoric Acid in Fertilizers.

D. Lindo.

A comparison of the oxalic and molybdate methods. The difficulty of removing silica entirely introduces an error which is greater in the oxalic method, but not entirely absent in the molybdate method. Presence of Al and Fe cause precipitation of SiO_2. Aluminium silicate is soluble to a small extent in ammonium citrate, but not in presence of magnesia mixture. In the oxalic method a trace of lime is dissolved from the oxalate by oxalic acid. Reprecipitations after solution in acetic acid, as generally recommended, may be avoided, however, by adding an excess (1.5 to 2 grms.) of citric acid at the proper stage of the process and filtering after 3 to 4 hours with sufficient excess of Mg. mixture. $P_2 O_5$ in presence of ammonium citrate and oxalic acid is precipitated as fully in two hours as in ten. (*Chem. News XLIX.* 247.) A. A. B.

On the Qualitative Examination, and the Quantitative Estimation of As, S, P, and Other Metals in Native Copper. O. Kuhn.

(*Fres. Zeit. 1884. 165.*)

J. H. S., Jr.

Estimation of Manganese in Cast Iron and Spiegel. C. L. Bloxam.

The method, which is particularly adapted to estimation of small quantities of iron, depends upon precipitation of iron as phosphate in presence of acetic acid. This precipitate is then dissolved in H Cl, evaporated to separate Si O_2, and oxidized with K ClO_3 ; excess of acetic acid is added, then ammonia and excess of Na_2 H PO_4. The precipitate is filtered out, redissolved and again thrown down with acetic acid and ammonia ; the combined filtrates are boiled to expel NH_3, and now precipitated as crystalline phosphate. The wet filter and precipitate are at once ignited over a Bunsen lamp. Results good.

(*Chem. News L.* 112.)

A. A. B.

Notes on the Volumetric Estimation of Iron. R. W. Atkinson.

An attempt to account for discrepancies in commercial analyses of iron ores made by different chemists. A series of results is tabulated, showing the errors which arise in standardizing a bichromate solution, when iron or steel of known composition is dissolved in sulphuric acid and titrated directly. The results of this method, which, according to the author, is in very general use, do not agree with those obtained by standardizing with ammonio-ferrous sulphate, nor by solutions of the same steel in nitric acid and subsequent reduction of the iron after evaporation with HCl ; the last two methods, however, give concordant results. The difficulty is ascribed to the action of hydrocarbons, produced during solution, upon the solution of dichromate. The results of analyses in the latter case (direct titration) are too low. (*Chem. News, XLIX.*, 117.) A. A. B.

On the use of Rosolic Acid, Methyl Orange, Phenacetoline and Phenol-Phthalein, as Indicators. Part III. R. T. Thomson.

A continuation of previous papers. The determination of alkaline hydrates in presence of large proportions of carbonates is accomplished by an improved process depending on the insensibility of phenolphthalein to precipitated barium carbonate. A solution of barium chloride, neutral to phenolphthalein, and of known strength, is added to the alkaline solution and the hydrate present is titrated directly *in the cold*, the latter point being of the utmost importance. Calcium chloride cannot replace the barium salt in this process. Directions are also given for estimation of hydrates in presence of normal sulphates and phosphates of the alkaline metals, for the estimation of bicarbonates in presence of normal carbonates, and for the estimation of alkaline arsenates and arsenites. (*Chem. News*, *XLIX.*, 119.) A. A. B.

Researches on Spectrum Photography in Relation to New Methods of Quantitative Chemical Analysis. Part II. W. N. HARTLEY.

An abstract of a paper read before the Royal Society. An account is given of the lengths and strength of metallic lines in solutions of definite strength. The sensitiveness of the spectrum reaction obtained by the writer is almost incredible. $\frac{1}{10000000}$ mg. is easily detected, and the delicacy of the reaction is practically unlimited. The spectra of the more strongly basic element exhibits the most persistent lines. Examples are given to show the application of the tables and other data presented, to quantitative work. (*Chem. News*, *XLIX.*, 128.) A. A. B.

Examination of some Musts of 1883, for Acids and Sugar. WM. SCHÄFER.

The acid was estimated with $\frac{1}{10}$ normal caustic soda solution, using litmus as indicator, and calculating the result as tartaric acid.

The sugar was estimated with Fehling's solution. For this purpose the must was heated to 80° C, and filtered, and then treated with Fehling's solution.

(*Fres. Zeit. 1884. 172.*)

J. H. S., JR.