

INORGANIC ANALYSIS.

The Gas-Volumetric Estimation of Hydrogen. A. Colson. (*Ac. des Sciences*, 1900, cxxx., 330; through *Journ. Pharm. Chim.*, 1900, xi., 335.)—This method is based upon the fact that hydrogen is absorbed by silver hydroxide. The reaction takes place slowly in the cold, but at 100° C. is rapid, and furnishes a means of separating hydrogen from a saturated hydrocarbon or from oxygen.

The mixed gases are introduced into a eudiometer, provided near the top with a horizontal side-tubulure, in which has previously been placed 1 to 2 grammes of silver hydroxide. On heating the side-tubulure the mercury rises in the eudiometer, and the absorption is complete in two and a half to three hours. In calculating the true volume of the residual gas, allowance must be made for the tension of the water-vapour formed during the reaction.

Hydrogen is completely absorbed, even at a very low pressure, whilst, as the author shows in his test estimations, no absorption of ethane, methane or oxygen takes place.

C. A. M.

The Estimation and Separation of Copper by means of Hydrazine Sulphate or Hydrazine Hydrochloride. P. Jannasch and K. Biedermann. (*Berichte*, 1900, xxxiii., 631-636.)

Estimation of Copper.—The hot solution of the copper salt is poured, with constant stirring, into a porcelain basin containing a solution of 5 grammes of pure sodium hydroxide in 50 c.c. of water. The resulting precipitate is reduced to cuprous hydroxide by adding 1 to 2 c.c. of a boiling 3 per cent. solution of hydrazine sulphate, and slowly heating the basin on the water-bath while its contents are continually stirred. On the further addition of 3 c.c. of the hydrazine sulphate solution the copper is reduced to the metallic state. After cooling, or diluting the liquid with boiled water, the precipitate is collected on a filter, washed with hot water, dried at 90° C., the paper ignited apart from the copper, and the latter oxidized over a Bunsen flame in a current of oxygen and weighed.

The results thus obtained in test experiments with copper sulphate were satisfactory.

Separation of Copper from Zinc.—In the authors' experiments the solution of the mixed sulphates was introduced drop by drop into a basin containing 50 c.c. of 10 per cent. sodium hydroxide solution. Five c.c. of a 3 per cent. solution of hydrazine sulphate were then added to the cold liquid containing a large excess of sodium hydroxide, and the whole slowly heated on an asbestos board. After about five minutes there was a complete precipitation of the copper, which was collected and oxidized as described above.

The filtrate containing the zinc was treated with hydrochloric acid, until the precipitate, which formed after some time, again dissolved, and the liquid was distinctly acid. The zinc was then precipitated as carbonate, which was ignited and weighed in the usual manner. Sometimes the zinc oxide obtained was not completely soluble in dilute acetic acid owing to the presence of traces of silica.

In this way the following results were obtained :

IN SOLUTIONS USED PER CENT.		FOUND PER CENT.	
Copper.	Zinc.	Copper.	Zinc.
16.77	7.74	16.73	7.68
15.69	8.68	15.61	8.68

Separation of Copper from Arsenic.—A solution of copper sulphate and arsenic acid was introduced drop by drop into 40 to 50 c.c. of 10 per cent. sodium hydroxide solution, the liquid warmed after the introduction of 4 to 5 c.c. of hydrazine sulphate solution, and the precipitated copper estimated as before.

The solution containing the arsenic was acidified with hydrochloric acid, a little nitric acid being added with it towards the end of the neutralization, and the whole evaporated to about 80 c.c., care being taken to have some nitric acid continually present. The clear liquid when cold was treated with ammonium hydroxide in excess, and the arsenic precipitated with freshly-prepared magnesia mixture, and estimated in the usual way. Results :

TAKEN PER CENT.		FOUND PER CENT.	
Copper.	Arsenic.	Copper.	Arsenic.
17.53	23.41	17.46	23.37
13.68	35.29	13.56	35.24

Separation of Copper from Tin.—Mixtures of pure copper and tin were dissolved in *aqua regia*, the solutions diluted with an equal volume of water, and added drop by drop to a hot solution of sodium hydroxide containing fifteen times more alkali than the weight of the dissolved metals. After the addition of 2 to 3 grammes of hydrazine hydrochloride the liquid was heated, and the heating continued for some time, in order to dissolve as sodium stannate any tin carried down with the copper. In the absence of sulphates any tin readily dissolved. The copper was collected on a double filter, washed with boiling water, and if necessary with dilute sodium hydroxide solution and again with water, dried, oxidized and weighed.

The filtrate was rendered slightly acid with strong hydrochloric acid, and the tin precipitated with ammonium hydroxide. The precipitate was dissolved by adding ammonium sulphide, and the liquid heated for a time on the water-bath and then slightly acidified with hydrochloric acid. After being heated for about an hour on the water-bath, the resulting precipitate of tin sulphide subsided and could be easily filtered. It was washed with warm hydrogen sulphide solution, ignited in a current of oxygen and weighed. The following results were obtained :

TAKEN PER CENT.		FOUND PER CENT.	
Copper.	Tin.	Copper.	Tin.
25.81	74.18	25.72	74.06
31.28	68.61	31.28	68.48

C. A. M.

The Analysis of Molybdenum Alloys. H. Bornträger. (*Zeit. anal. Chem.*, 1900, xxxix., 91.)—As a supplement to his method of analysing molybdenum glance

(ANALYST, xxiii., 332), the author describes the following process for determining molybdenum in its alloys :

About 1 gramme of the sample is dissolved in 50 c.c. of *aqua regia* on the water-bath, the solution evaporated in a porcelain basin, and the nitric acid expelled by means of hydrochloric acid.

The residue is taken up with 50 per cent. alcohol, and the liquid filtered. The residue left on the filter will now contain the bulk of the molybdic acid.

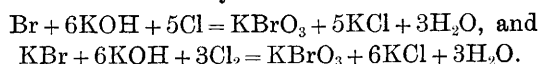
The filtrate is evaporated to dryness, and the residue once more taken up with alcohol, after which treatment the remainder of the molybdic acid will be left as a residue.

The molybdic acid in the two residues can then be weighed directly, whilst the metal in the solution can be estimated in the usual way as oxide.

From an alloy known to contain 66 per cent. of molybdenum and 44 per cent. of iron, the author obtained by this method 65.6 per cent. of molybdenum. C. A. M.

The Detection of Boric Acid in the Form of Borates. H. Bornträger. (*Zeit. anal. Chem.*, 1900, xxxix., 92.)—When borates are heated with hydrochloric, nitric, or sulphuric acid, a Bunsen flame is not coloured green. On the other hand, when heated with hydrofluoric acid alone, or with ammonium nitrate and ammonium chloride, or with sulphuric acid and hydrochloric acid, or sulphuric acid and nitric acid, or hydrochloric acid and nitric acid, borates impart a bright green colour to the flame ; and this coloration is more intense, and appears sooner, than that obtained on heating the salts with alcohol and sulphuric acid. C. A. M.

A New Method of estimating Bromides in the Presence of Chlorides and Iodides. J. v. Weszelszky. (*Zeit. anal. Chem.*, 1900, xxxix., 81-91.)—Bromine can be determined in the presence of chlorine by taking advantage of the fact that in an alkaline solution it is oxidized by chlorine to bromic acid, as in the equations :



The excess of chlorine is converted into chloride and chlorate ; but as the solution of the latter salt is too weak to act upon iodides in the cold, the amount of bromate formed may be determined by adding a solution of potassium iodide, acidifying, and titrating the liberated iodine.

For the separation of bromine and chlorine from iodine, the author makes use of Winkler's method, which is based upon the fact that chlorine oxidizes iodine or iodides in acid solution to iodic acid, whilst the bromine is only liberated. Thus, on acidifying a solution of the mixed halides, adding an excess of chlorine water, and boiling the liquid, the resulting iodic acid remains behind, while the bromine and excess of chlorine distil over. On adding potassium iodide to the cooled liquid in the flask, and titrating with $\frac{N}{10}$ thiosulphate, a sixth part of the iodine found corresponds to the iodine originally present.

In applying these reactions, if the solution contains no iodine, about 1 gramme

of potassium carbonate is introduced, together with a sufficient quantity of chlorine water, and the liquid cautiously evaporated to dryness over a naked flame. The residue, when cold, is dissolved in 100 to 150 c.c. of water, potassium iodide added, and the liquid acidified and titrated with $\frac{N}{10}$ thiosulphate. The number of c.c. used, multiplied by 0.001333, gives the quantity of bromine.

When iodine is present, the liquid is placed in a distilling flask, similar to that used by Bunsen and Fresenius in the analysis of manganese peroxide (Fresenius, "Quantit. Analyse," 6th edit., i. 382). This is connected with an absorption vessel, containing 0.5 to 1.0 gramme of potassium hydroxide dissolved in water. After the addition of a sufficient quantity of chlorine water, the flask is warmed, and when the greater part of the chlorine and bromine has passed over, a current of carbon dioxide is passed through the apparatus until the end of the distillation.

The bromine in the distillate and the iodine in the flask are determined by titration with thiosulphate, as described above.

When the solution of the halogen contains iron, the latter should be separated by precipitation with potassium carbonate; arsenic and antimony when present must also be removed.

The author gives tables of the results of test analyses of experimental mixtures of bromides, iodides, and chlorides, from which it appears that the method is extremely accurate, even when only minute quantities of the halogens are present.

C. A. M.

The Determination of Clay in Soils. F. Poquillon. (*Bull. Soc. Chim.*, 1900, xxiii., 115, 116.)—The author recommends the following method as being much more rapid than the usual process, requiring at the outside only two or three days.

Ten grammes of the soil are triturated with about 25 c.c. of water added drop by drop, and 100 to 120 c.c. of a solution of ammonium chloride (1 gramme per litre) added to the mixture. The mass is stirred with a glass rod, and after standing for five minutes the supernatant liquid is transferred to a litre flask. The residue is again treated with 100 to 125 c.c. of the ammonium chloride solution, and the liquid again decanted after five minutes, this process being repeated until the washings are clear. About six or eight washings are required in the case of heavy clay soils. The residue is then treated with dilute hydrochloric acid, washed with water, dried, and weighed, the weight giving the amount of total sand.

The liquid in the flask is treated with a few drops of hydrochloric acid to dissolve carbonates and to coagulate the clay, after which it is left until the supernatant liquid is clear. This usually takes two or three hours. The deposited clay is then collected on a weighed filter, washed with water, dried, and weighed.

The following results were obtained by this method, and by the ordinary method, which takes from eight to ten days:

Clay in the Soil.—Parts per Thousand.

Old method	... 206.0	60.2	198.0	218.0	122.0	129.3
New method	... 205.8	60.5	198.0	217.1	121.9	129.5

C. A. M.

A New Indicator: Alizarin Green B. J. Formanek. (*Zeit. anal. Chem.*, 1900, xxxix., 99-103.)—Alizarin Green B, which is manufactured by Dahl and Co., Barmen, is obtained by the action of β -naphtho-quinone-sulphonic acid on (2)-amido-(1)-naphthol-(4)-sulphonic acid.

It is a greenish-black powder, which dissolves fairly readily in water, forming a dirty-green solution. It is somewhat less soluble in alcohol, the colour of the solution being flesh-red. On adding a dilute acid to the alcoholic solution the colour becomes carmine red, whilst alkalies change it to a pure green. Like litmus, it is sensitive to carbonic acid.

The changes in the colour reactions are exceedingly sharp, and are as perceptible with an artificial light as in daylight.

C. A. M.