

INORGANIC ANALYSIS

Determination of the Conductivity of Electrolytes. A. H. W. Aten. (*Chem. Weekblad*, 1921, 18, 51-52.)—The conductivity of electrolytes has been determined by a method the essential feature of which is the employment of a differential galvanometer and a variable resistance, and in which certain disadvantages of the Kohlrausch method are overcome. Errors arising from polarisation and the heating effect of the current may be reduced by increasing the surface of the electrodes and the volume of the electrolyte, and become smaller with higher resistance. Comparison of this method with Kohlrausch's method indicates that with a resistance not below 100 ohms an accuracy of 0.3 per cent. is obtainable. The method has a special application in certain technical analyses, such as estimation of the ash in sugar and of chlorides in drinking water, the determination of carbon dioxide in chimney gases, and the selection of the end point in the evaporation of condensed milk.
W. J. W.

Catalysis in Permanganate Titrations. P. H. Segnitz. (*J. Ind. Eng. Chem.*, 1920, 12, 1196-1197.)—Results of experiments indicate that the addition of

manganous sulphate to a solution to be titrated with permanganate solution accelerates the reaction velocity, so that the time of the peroxide-permanganate titration can be reduced from about forty-five minutes to eight seconds, and that of the cold oxalate-permanganate titration from over an hour to six minutes. The use of the catalyst does not interfere with the end point of the titration. W. P. S.

Preparation of Anhydrous Stannic Chloride. C. van Loon. (*Chem. Weekblad*, 1920, **17**, 664.)—In the preparation of anhydrous stannic chloride (*Chem. Weekblad*, 1920, **17**, 610) in small amounts, quantitative results may be obtained by using a round-bottomed flask instead of the wide tube employed by Lorenz (*Zeitsch. anorg. Chem.*, 1895, **10**, 44). W. J. W.

Solubility of Metals in Acids containing Formaldehyde. R. C. Griffin. (*J. Ind. Eng. Chem.*, 1920, **12**, 1158-1160.)—The presence of 1 per cent. of formaldehyde in dilute sulphuric acid (sp. gr. 1.075) and in dilute hydrochloric acid (1:1) considerably decreases the solvent action of these acids on wrought iron, cast iron, and steel, the corrosion loss being diminished 50 to 95 per cent. The effect in the case of other metals (manganese, bronze, tin, brass, nickel, etc.) is much less, and it is also less with 10 per cent. nitric acid, possibly on account of secondary reactions. W. P. S.

Gasometric Estimation of Nitrogen. R. L. Stehle. (*J. Biol. Chem.*, 1920, **45**, 223-228.)—The sample is digested with a small quantity of sulphuric acid and a crystal of copper sulphate, and on cooling the solution is diluted to 100 c.c. Ten c.c. are run into a nitrometer containing mercury, and connected at the lower end by a long indiarubber tube to an adjustable mercury reservoir, and dissolved air is liberated by reducing the pressure and expelled from the apparatus. A strong solution of alkali is run in to neutralise the acid, and 2 c.c. of a solution prepared by mixing equal volumes of 28 per cent. sodium hydroxide and a solution containing 12.5 per cent. of sodium bromide and 12.5 per cent. of bromine, the mixture being diluted with three volumes of water, are added. The pressure in the reaction chamber is reduced, and the nitrogen is completely evolved by shaking the nitrometer for a minute. Since a small amount of oxygen is liable to be set free, this gas is absorbed by running in 1 c.c. of sodium pyrogallate solution. The mercury is adjusted until the volume of nitrogen is exactly 1 c.c., and the weight of nitrogen present is calculated from the observed temperature and pressure. The method yields excellent results with substances of known nitrogen content, but a maximum error of about 8 per cent. is shown by comparison of this method with the usual Kjeldahl estimation when estimating the non-protein nitrogen in blood. T. J. W.

Properties of Pure Hydrogen Peroxide. O. Maass and W. H. Hatcher. (*J. Amer. Chem. Soc.*, 1920, **42**, 2548-2569.)—Pure (99.9 per cent.) hydrogen peroxide was prepared from an impure 3 per cent. solution by concentration in a sulphuric acid vacuum concentrator (*ANALYST*, 1920, 68), distillation, and

fractional crystallisation. It is almost insoluble in anhydrous ether, dissolves various anhydrous salts readily, and, on heating, is decomposed in contact with glass. Most metals cause decomposition of the pure substance, but not explosively. Pure aqueous solutions in a suitable container keep indefinitely, but are decomposed by foreign substances gaining access to the solution. Strong solutions are more stable than dilute, as the former more readily destroy impurities, and thus prevent further decomposition. Investigation of so-called "anti-catalysers" showed that in all cases these substances acted by destroying others, which caused decomposition, and that spontaneous decomposition of pure hydrogen peroxide did not occur (cf. ANALYST, 1896, 20, 38).

T. J. W.

Micro-chemical Identification of Gaseous Ammonia as Hexamethylenetetramine Picrate. C. Kollo and V. Teodossiu. (*Bull. Soc. Chim. România*, 1920, 11, 100-102.)—When a drop of a solution of formaldehyde (commercial formalin), together with a drop of a 1 per cent. solution of picric acid, is exposed to ammonia, hexamethylenetetramine is produced, and combines immediately with the picric acid to form characteristic crystals of the picrate. The test is not sufficiently delicate when carried out in this way, as ammonia picrate is formed in the presence of much ammonia, whilst in the presence of small amounts of ammonia the formation of hexamethylenetetramine picrate only takes place on rubbing, and the crystals break up and lose their characteristic form. A highly sensitive reagent is prepared by saturating a solution of formaldehyde in the cold with picric acid, and then saturating this with hexamethylenetetramine picrate. The ammonia liberated from 1 mgrm. of ammonium chloride may be detected by exposing one drop of the reagent for five to six minutes to the action of the gas under examination.

R. G. P.

New Method for the Separation and Estimation of Iron and Manganese. C. Kollo. (*Bull. Soc. Chim. România*, 1920, 11, 89-95.)—The method of separation is based on the precipitation of ferric hydroxide when hexamethylenetetramine is added to a slightly acid solution of an iron salt, the manganese forming soluble double salts of the type $\text{Mn}(\text{CH}_2)_6\text{N}_4\text{2H}_2\text{SO}_4$, manganese oxide only being precipitated in the presence of hexamethylenetetramine from a neutral solution on boiling. To carry out the separation the solution of ferric and manganese salts is slightly acidified, and a 10 per cent. solution of hexamethylenetetramine is added until the supernatant liquid does not change the colour of Congo-red paper, after which the liquid is warmed gently on a water-bath to cause the ferric hydroxide to flocculate. After removal of the manganese salts by washing on a filter, the iron is determined gravimetrically as usual, or by dissolving the ferric hydroxide in hydrochloric acid, adding excess of potassium iodide and titrating the liberated iodine with standard thiosulphate solution. The manganese in the filtrate may be determined as manganese sulphate after evaporation and ignition, or by Volhard's method after the destruction of organic matter by evaporation with sulphuric acid. The following results were obtained by these methods:

Salts.	Weight taken.	Weight found—	
		Gravimetrically.	Volumetrically.
	Grm.	Grm.	Grm.
$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	0.1298	0.1300	0.1284
$\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$	0.1406	0.1374	0.1412
$\text{FeCl}_3 \cdot 12\text{H}_2\text{O}$	0.4950	0.5139	0.5101
$\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$	0.2200	0.2300	0.2285

R. G. P.

The Use of Phenolphthaleïn and Diphenylamine in the Estimation of Manganese by the Persulphate Method. D. H. Wester. (*Rec. Trav. Chim. Pays-Bas*, 1920, **39**, 600-602.)—The substitution of an alkaline phenolphthaleïn solution for permanganate (Tillmans and Mildner, *J. Gasbeleucht. u. Wasserversorg.*, 1914) in the estimation of manganese gives inaccurate results, probably owing to variation in the alcoholic strength of the phenolphthaleïn; in extreme cases this may be 2 per cent. to 25 per cent. With constant alkaline concentration, the violet coloration is affected more by the content of water than of phenolphthaleïn in the solutions, and increase of the phenolphthaleïn does not, therefore, give a proportionately deeper intensity of colour. No better results are obtained with diphenylamine. The blue coloration disappears rapidly, and there is no relation between its intensity and the amount of manganese.

W. J. W.

Micro-chemical Identification of Potassium and Sodium in Presence of Magnesium. E. Ludwig and H. Spirescu. (*Bull. Soc. Chim. România*, 1920, **11**, 78-82.)—Calcium, strontium, and barium are first removed by precipitation with ammonium carbonate or with ammonium oxalate and sulphuric acid, the filtrate evaporated, and the residue ignited until free from ammonium salts and used for the detection of potassium and sodium. The complex nitrite $\text{K}_2\text{CuPb}(\text{NO}_2)_6$ is employed as a reagent for potassium, the test for which is carried out by placing a drop of sodium nitrite solution (20 grms. in 75 c.c. of water), and a drop of copper and lead acetate solution (copper acetate 9.1 grms., lead acetate 16.2 grms., acetic acid 2 c.c., water 75 c.c.) on the slide and adding a fragment of the residue under examination. In the presence of potassium the complex nitrite is precipitated in the form of cubic black crystals, even in the presence of large amounts of magnesium, whilst the reaction is more delicate than that with platonic chloride. For the detection of sodium by means of potassium pyroantimoniate, the soluble magnesium salts (which interfere with the reaction) are converted into insoluble basic magnesium carbonate by adding a drop of concentrated potassium carbonate or bicarbonate to a fragment of the residue and evaporating the mixture to dryness on the slide. On adding a drop of potassium pyroantimoniate solution, a characteristic precipitate of sodium hydrogen antimoniate ($\text{Na}_2\text{H}_2\text{Sb}_2\text{O}_7$) is formed.

R. G. P.