Analytical Chemistry.

Continuous-Observation Polarimeter Tube. JOHN SIMPSON FORD (*Analyst*, 1904, **29**, 281—282).—The 200 mm. tube described is made entirely of metal with glass end-plates secured by screw-caps as usual. At one end of the tube is fixed a conical funnel of about 65 c.c. capacity, and at the other a discharge pipe. The solution to be observed is poured into the funnel to fill the tube. After taking the reading, a second solution may be poured into the funnel and so on. Each successive solution quickly washes the previous one out of the tube, even when they are of different specific gravities.

W. P. S.

The Material and Shape of the Rotating Cathode. HERBERT E. MEDWAY (*Amer. J. Sci.*, 1904, [iv], **18**, 180—182. Compare Abstr., 1903, ii, 613).—A silver crucible may be employed as a rotating cathode for the deposition of metals, the results obtained being as accurate as when platinum is employed. Deposited copper is removed by rubbing and finally by treatment with boiling hydrochloric acid. A nickel cathode is attacked, both during the process and in the subsequent removal of the deposit, causing inaccurate results. No coherent deposit can be obtained on aluminium.

The disc form of cathode is inferior to the crucible form in retaining the deposit.   C. H. D.

Bunsen Burners and Combustion Apparatus without Gas. HARRY DRAKE GIBBS (*J. Amer. Chem. Soc.*, 1904, **26**, 760—764).—An arrangement for burning the vapour of alcohol (or other liquid combustibles) in Bunsen burners. For details, the original paper and drawings should be consulted.   L. DE K.

Mixed Indicators. MAX SCHOLTZ (*Zeit. Elektrochem.*, 1904, **10**, 549—553).—If a solution of *p*-nitrophenol and phenolphthalein in hydrochloric acid is neutralised by potassium hydroxide solution, the yellow colour of the nitrophenol ion first appears and then the pink colour of the phenolphthalein. The nitrophenol is therefore a stronger acid than the phenolphthalein. In a similar way, all the indicators may be compared. The following is the order found by the author, beginning with the strongest acid: (1) alizarinsulphonic acid, iodoeosin; (2) *p*-nitrophenol; (3) luteol; (4) hæmatoxylin, rosolic acid; (5) litmus; (6) turmeric; (7) phenolphthalein. Some other indicators which give less sharp colour changes are the following.

Alizarin lies between *p*-nitrophenol and litmus, fluorescein lies between iodoeosin and litmus, and tropæolin 000 between luteol and litmus.

Cochineal and phenacetolin are stronger than litmus, gallein is stronger than *p*-nitrophenol, lacmoid is stronger than turmeric, and brazilin and α-naphtholbenzein are weaker than luteol and litmus respectively. Among the basic indicators, cyanine is a stronger base than methyl-orange or the tropæolins. T. E.

Application of Potassium Tetraoxalate in Titration. GEORG LUNGE (*Chem. Zeit.*, 1904, **28**, 701—702).—A reply to Kühling's criticisms (*ibid.*, 596, 612) of a previous paper (this vol., ii, 289). The author has never been able to prepare a potassium tetraoxalate in which the water of crystallisation corresponds accurately with the formula $C_2O_4HK,C_2O_4H_2,2H_2O$; on the other hand, sodium carbonate and sodium tetraoxalate giving an analytical error of less than 0·1 per cent. can readily be obtained. W. A. D.

Use of Potassium Iodate for the Detection and Estimation of Iodides, Bromides, and Chlorides. STANLEY BENEDICT and J. F. SNELL (*Chem. Zeit.*, 1904, **28**, 729).—In reply to criticisms, the authors state that they were unaware of the process of Ditz and Margosches (this vol., ii, 145) published previously, and also that their method differs in several particulars from that of Bugarszky, as the use of potassium iodate permits of the estimation of chlorides, bromides, and iodides in the presence of each other. W. P. S.

Estimation of Iodine in the presence of Bromine and Chlorine. E. THILO (*Chem. Zeit.*, 1904, **28**, 866).—The process is based on the fact that when silver nitrate is gradually added to a solution containing iodides, bromides, and chlorides, the iodine is precipitated first, followed by the bromine and then by the chlorine.

The solution is therefore titrated with $N/10$ silver nitrate until a drop of the liquid no longer gives a dark spot on paper impregnated with a weak solution of palladium chloride. The filtrate may be titrated for total bromine and chlorine as usual, using potassium chromate as indicator. If bromine only is to be estimated, it is sufficient to add enough $N/10$ silver nitrate to convert all the bromine with a portion of the chloride into the silver salts, which are then

weighed in the usual manner. As the amount of metallic silver contained therein is of course known from the number of c.c. of silver solution added, the amount of silver bromide may be calculated easily.

Commercial iodine and cuprous iodide may be converted into soluble zinc iodide by trituration with zinc dust and water. If the silver iodide does not separate readily, a little salt may be added.

L. DE K.

Estimation of Fluorine in Martin Slag. L. FRICKE (*Chem. Centr.*, 1904, ii, 728—729 ; from *Stahl u. Eisen*, 24, 889—890).—In order to determine the quantity of fluorine in Martin slag, 5 grams of the powdered slag mixed with 5—6 times the quantity of potassium carbonate are kept molten in a platinum crucible at a good heat for $\frac{1}{2}$—1 hour. After extracting the cold mass with hot water and filtering, the silica and alumina are precipitated by prolonged boiling with ammonium carbonate. The filtrate is heated with a solution of zinc oxide in ammonia, evaporated to dryness, and heated to 120°. The residue is taken up with cold water and the zinc phosphate, carbonate, and silicate filtered off and washed with a 2 per cent. solution of potassium carbonate. To the filtrate, 1—1·5 grams of calcium chloride are added, it is then acidified with acetic acid, and the carbon dioxide removed by warming the solution. The solution is made alkaline with potassium hydroxide solution, and again faintly acid with acetic acid. The precipitate, which consists of calcium fluoride together with some calcium phosphate, is collected, washed with cold water until no more calcium can be detected in the washings, and finally ignited and weighed. It is then warmed with concentrated sulphuric acid in the platinum crucible, diluted, ammonia added until only faintly acid, and the phosphoric acid precipitated by molybdic acid. If only a small quantity of precipitate is formed, the phosphoric acid is estimated by titrating with lead acetate, but if the precipitate is larger it is dissolved and precipitated with magnesia mixture. The percentage of fluorine x may be calculated from the formula $x = 48·72/s.(a - 1·392d)$, in which a = weight of calcium fluoride and phosphate, d = weight of magnesium pyrophosphate, and s = weight of substance taken. The result may be checked by making the filtrate from the phosphomolybdic acid precipitate up to 250 or 500 c.c. and determining the quantity of calcium in 100 or 200 c.c. respectively. If c = weight of CaO, then $x = 67·857/s.(c - 0·7543d)$.   E. W. W.

Estimation of Water in Substances which afterwards are to be extracted with Volatile Solvents. ROBERT M. BIRD (*J. Amer. Chem. Soc.*, 1904, 26, 818—826).—An arrangement chiefly devised for the estimation of water and fat in butter. The sample is introduced into a weighed Gooch crucible and dried in a current of hot air for 20—30 minutes ; the loss in weight represents the water. The fat is then removed by extracting with ether for about half an hour, and the crucible is reweighed ; the loss represents the fat. For full particulars of the apparatus, which admits of five estimations being conducted simultaneously, the original paper and drawings should be consulted.

L. DE K.

Application of Hydrazine Sulphate in the Determination of Oxidising Substances. U. ROBERTO and F. RONCALI (*Chem. Centr.*, 1904, ii, 616; from *L'Ind. Chim.*, 6, 178—179).—Hydrazine sulphate acts with substances which liberate oxygen in precisely the same way as it does with chlorine (compare *ibid.*, 93), being decomposed by the oxygen and yielding sulphuric acid, water, and nitrogen. When potassium permanganate is gently heated with hydrazine sulphate, the solution is decolorised and nitrogen is evolved, 5 mols. of nitrogen being liberated by 4 of permanganate. In a similar way, 2 mols. of potassium dichromate yield 3 mols. of nitrogen. By measuring the volume of nitrogen liberated, the quantity of oxidising substance may be estimated. This method is also applicable to peroxides, chlorates, &c. E. W. W.

Forms in which Sulphur exists in Coal; their Effects on the Heating Power. E. E. SOMERMEIER (*J. Amer. Chem. Soc.*, 1904, 26, 764—780).—The author has studied the influence of the sulphur in coals on their heating powers as calculated by Dulong's formula, and arrived at the following conclusions.

For sulphur present as ferrous sulphate, there is a loss of 10 cal. for each per cent. of sulphur. When the sulphur is present as iron pyrites, the result is too high by about 9·6 cal. for each per cent. on uncorrected ultimate analyses. On corrected analyses (where a correction has been made for oxygen in the ash), the results are too low by 6·6 cal. When the sulphur exists in an organic combination, there is a loss of about 4·5 cal. for each per cent. so present. L. DE K.

Photometric Estimation of Sulphur in Coal. SAMUEL W. PARR and C. H. McCLURE (*J. Amer. Chem. Soc.*, 1904, 26, 1139—1143).—A description of an improved photometer used for the determination of small quantities of sulphates by means of barium chloride (compare Jackson, Abstr., 1902, ii, 172). For particulars, the illustration and table of the original paper should be consulted. L. DE K.

Estimation of Sulphur in Iron. ALLEN P. FORD and OGDEN G. WILLEY (*J. Amer. Chem. Soc.*, 1904, 26, 801—808).—The following process, originally proposed by Bamber, is recommended as being particularly suitable for analysts who have but limited experience in sulphur estimations. Two grams of the drillings are dissolved in nitric acid contained in a platinum dish covered with a watch-glass. When solution is complete, 1 gram of nitre is added, the liquid is evaporated to dryness, and the residue heated to redness for 3 or 4 minutes. The mass is now boiled with 50 c.c. of a 1 per cent. solution of sodium carbonate and the residue washed with the same hot liquid. The filtrate is freed from nitric acid by evaporating with hydrochloric acid, and the dry residue dissolved in 50 c.c. of water and 2 c.c. of hydrochloric acid. The sulphuric acid is then estimated as usual with barium chloride. L. DE K.

Estimation of Sulphur in Pig-iron and Steel by Titration with Iodine and Thiosulphate Solution. L. FRICKE (*Chem. Centr.*, 1904, ii, 727—728; from *Stahl u. Eisen.*, 24, 890—891).—In order to estimate the quantity of sulphur in pig-iron or steel, 100 c.c. of water and 75 of concentrated hydrochloric acid are added, by means of a separating funnel, to 10 grams of steel or 5 of iron contained in a ½—1 litre flask which is fitted with a condenser and receiver. The mixture is heated moderately. The water and hydrochloric acid vapours are mostly condensed, whilst the hydrogen sulphide passes through a dry vessel and then successively into three vessels of which the last two contain a solution of 25 grams of cadmium acetate in 200 c.c. of glacial acetic acid. The gas is finally expelled from the apparatus by carbon dioxide and the contents of the receiver are diluted to about 150 c.c. with the cadmium solution and titrated. For this purpose, iodine and sodium thiosulphate solutions are used, of which 1 c.c. corresponds with 1 mg. of sulphur (these solutions contain respectively 7·928 grams of iodine and 25 of potassium iodide, and 15·526 of thiosulphate and 2 of ammonium carbonate per litre). Excess of iodine solution is added to the cadmium sulphide together with 75 c.c. of a solution of 300 c.c. of concentrated hydrochloric acid in 850 c.c. of water (sp. gr. 1·124), and the excess of iodine titrated with sodium thiosulphate after adding 2 c.c. of an iodine-zinc-starch solution. The difference between the amounts of iodine and thiosulphate solution used gives the percentage of sulphur.   E. W. W.

Estimation of Sulphur and Phosphoric Acid in Foods, Fæces, and Urine. J. A. LE CLERC and WILBUR L. DUBOIS (*J. Amer. Chem. Soc.*, 1904, 26, 1108—1113).—The authors have used the calorimetric processes of Berthelot and Parr, and also the sodium peroxide process recommended by Osborne (Abstr., 1902, ii, 223), and are in favour of the latter method.

Tables are given showing the amount of sulphur in various foods and dried fæces. In the case of urine, the direct titration of the phosphoric acid in the diluted sample with uranium acetate is preferred to the combustion processes.   L. DE K.

Estimation of Sulphuric Acid in Urine by Alcoholic Strontium Chloride. ROLAND VON LENGYEL (*Pflüger's Archiv*, 1904, 104, 514—518).—The estimation of sulphuric acid in the urine by an alcoholic solution of strontium chloride instead of barium salts is recommended. The new method is correct and rapidly carried out.
            W. D. H.

Estimation of Sulphuric Acid in presence of Iron. FRIEDRICH W. KÜSTER (*Zeit. angew. Chem.*, 1904, 17, 1180).—Silberberger concludes that sulphuric acid cannot be estimated in presence of iron by Lunge's method, and, in support of his contention, quotes Küster and Thiel (Abstr., 1899, ii, 247); Küster on the contrary holds that Lunge's method gives accurate results.   A. McK.

Estimation of the Active Oxygen in Organic Persulphates.
A. WOLFF and RICHARD WOLFFENSTEIN (*Ber.*, 1904, **37**, 3213—3214).
—The method consists in adding sulphurous acid to the solution of
the organic persulphate, whereby the sulphurous acid is oxidised to
sulphuric acid, which is then estimated as barium sulphate.

E. F. A.

**Comparison of the Gravimetric Methods for Estimating
Selenium.** ALEXANDER GUTBIER, G. METZNER, and JUL. LOHMANN
(*Zeit. anorg. Chem.*, 1904, **41**, 291—304).—The majority of the
gravimetric methods for estimating selenium are based on the use of
reducing agents. The methods described were conducted with selenium
dioxide, which was specially purified. Details are given of the reduction
of selenium dioxide by hydrazine hydrate, hydrazine hydrochloride,
hydrazine sulphate, sulphurous acid, sodium sulphite, ammonium
sulphite, hydroxylamine hydrochloride, hydriodic acid, and hypo-
phosphorous acid respectively. These various methods all yield good
results. The methods recommended are those with hydrazine hydrate,
hydrazine sulphate, hydrazine hydrochloride, and sulphurous acid.

A. McK.

Estimation of Tellurium by Electrolysis. GIOVANNI PELLINI
(*Atti R. Accad. Lincei*, 1904, [v], **13**, ii, 275—277).—The author has
further tested the method devised by him for the estimation of
tellurium (this vol., ii, 147), by dissolving a known weight of pure
redistilled tellurium in nitric acid, evaporating the nitric acid, adding
concentrated sulphuric acid and ammonium hydrogen tartrate, and
electrolysing as previously described. The results obtained have errors
varying from about 0·08 to 0·16 per cent. It is found that quantities
of tellurium much greater than 1 gram can be deposited, so that the
method may be used for the purification of this element. T. H. P.

Estimation of Organic Nitrogen by Sodium Peroxide. FRITZ
VON KONEK and ARTHUR ZÖHLS (*Zeit. angew. Chem.*, 1904, **17**,
1093—1095).—Organic substances are not, as a rule, quantitatively
converted into nitric acid by sodium peroxide. The method gives
good results, however, when applied to the estimation of nitrogen in
flour.

The flour, dried at 100—102°, is mixed in a steel cylinder with an
excess of dry sodium peroxide, to which a mixture of potassium
persulphate and tartaric acid had been added. The cylinder is kept
cool in water, and when the vigorous oxidation, which is started by
means of a red-hot iron wire, is complete the whole is dissolved in
water. The nitrate is then reduced to ammonia by the addition
of pumice, alcohol, and Devarda's alloy of aluminium and copper,
and the ammonia estimated as in the Kjeldahl process. Results
obtained from various specimens of flour by this method and by
Kjeldahl's are in close agreement. A. McK.

Use of Sodium Peroxide in Analysis. HANS H. PRINGSHEIM
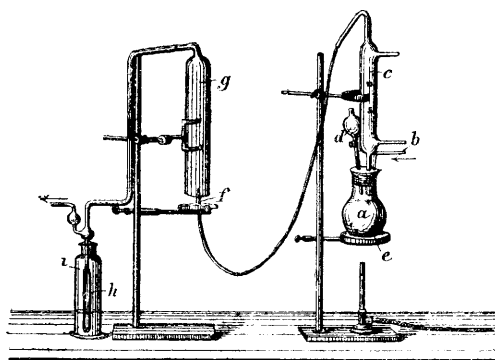(*Zeit. angew. Chem.*, 1904, **17**, 1454—1455).—A reply to von Konek

(preceding abstract). As regards the nitrogen, up to 90 per cent. may be liberated as such during the combustion. L. DE K.

Estimation of Phosphorus in Solutions. ANASTASIOS C. CHRISTOMANOS (*Zeit. anorg. Chem.*, 1904, **41**, 305—314. Compare this vol., ii, 614).—When a solution of yellow phosphorus in ether or in benzene is agitated with copper nitrate solution for a few seconds, a black precipitate is formed, which consists, not of the compound Cu_3P_2, but of mixtures of Cu_3P_2 and Cu_6P_2, approximating to the composition Cu_5P_2. The composition is, however, very variable and the precipitate is often highly oxidised.

In the method of estimation described, an ethereal solution of phosphorus is heated for several minutes with a 10 per cent. solution of copper nitrate, of which so much is taken that the solution remains blue after the black precipitate has subsided. The ether is then expelled and the product oxidised by bromine, when the actions represented by the equations : $Cu_6P_2 + Br_{12} = 3Cu_2Br_2 + 2PBr_3$ and $Cu_6P_2 + Br_{10} = Cu_2 + 2Cu_2Br_2 + 2PBr_3$, take place. After removal of the excess of bromine, the copper is separated as sulphide and the phosphorus determined by the addition of magnesia mixture in the usual manner.
A. McK.

Estimation of Phosphorus in Calcium Carbide. HJ. LIDHOLM (*Zeit. angew. Chem.*, 1904, **17**, 1452).—Ten grams of the powdered sample are put into a crucible, which is then placed into the 500 c.c. flask, *a*. A current of hydrogen is passed through the tube *b*, and when the air has been expelled the burner, *f*, is lit and the pump, attached to the exit tube of the wash-bottle *h*, put into action. After fitting on the condenser, 30 c.c. of absolute alcohol are introduced drop by drop

through the funnel, *d*, and the same volume of water is then cautiously added. Any hydrogen phosphide formed will burn together with the hydrogen, and acetylene and the phosphoric acid will deposit partly on the sides of the cylinder, *g*, and partly find its way into the wash-bottle, *h i*. When evolution of acetylene has ceased, hydrochloric acid is added to dissolve the calcium hydroxide, and the liquid is heated to boiling so as to expel the last traces of phosphorus compounds.

The cylinder, wash-bottle, &c., are then rinsed with dilute ammonia and the phosphoric acid is precipitated as usual with magnesia-mixture.
L. DE K.

Estimation of Phosphoric Acid in Aqueous Extracts of Soils and Plants. OSWALD SCHREINER (*J. Amer. Chem. Soc.*, 1904, **26**, 808—813).—The colorimetric process for the estimation of phosphoric acid in the presence of silica (this vol., ii, 85) has been found useful for the estimation of small quantities of phosphoric acid in aqueous solutions of soils and plants. If the solutions are not quite colourless, the colour should be removed by treatment with carbon black.

L. DE K.

Official Tests for Arsenic. WYNDHAM R. DUNSTAN and HENRY H. ROBINSON (*Pharm. J.*, 1904, [iv], **19**, 381—382, 405—407, 426—427, 448—450).—Instructions are given for applying Mayençon and Bergeret's test (Abstr., 1874, 1008), which depends on the yellow stain produced by arsenic hydride on mercuric chloride paper, to the drugs of the B. P. The stain given by 0·012 mg. of arsenic is used as a standard for comparison. When 4 grams of the drug are employed in the test, this stain represents 3 parts of arsenic per million. It is considered that for most drugs which are administered in small doses, the amount of arsenic present should be less than 3 parts per million, whilst in the case of tartaric and citric acids the quantity present should be less than 1 part per million, for the principal mineral acids, 3/10 part per million, and for solution of ammonia, 1/10 part per million. Special directions are given for the application of the test to a number of drugs which offer particular difficulties. E. G.

Electrolytic Methods for the Detection and Approximate Estimation of Minute Quantities of Arsenic in Beer, Malt, and Food-stuffs, &c. WILLIAM THOMSON (*Mem. Proc. Manchr. Lit. Phil. Soc.*, 1904, **48**, xvii, 1—14).—The author has experimented on the use of various cathodes in the electrolytic estimation of arsenic. With lead, smaller quantities of arsenic could be detected than with platinum, but the results were irregular; with aluminium and plumbago, 50 c.c. of a solution containing 1/500 grain per gallon gave no mirror, whilst tin was not procured sufficiently free from arsenic.

A form of apparatus in which pure zinc serves as a cathode is recommended and described in the paper. A comparison between it and the apparatus recommended by the Royal Commission is given, in respect to the influence of amyl alcohol and invert sugar on the production of the mirror. The preliminary reduction of arsenic to arsenious compounds is not necessary. G. D. L.

Estimation of Boric Acid in Cider, Fruits, &c. ALFRED H. ALLEN and ARNOLD R. TANKARD (*Pharm. J.*, 1904, [iv], **19**, 242—244). —The presence of boric acid in cider and fruits can be detected by the following method. The ash obtained by evaporating 20 c.c. of cider or apple juice to dryness and igniting the residue, or by directly igniting 25 grams of the fruit, is acidified with dilute hydrochloric acid; a piece of turmeric paper is immersed in the liquid and the mixture is evaporated to dryness. If boric acid is present, the turmeric paper assumes a brownish-red colour, which is changed to a

variety of colours, chiefly green and purple, when moistened with solution of sodium hydroxide.

The following method is recommended for estimating the boric acid. About 100 c.c. of cider or fruit juice are treated with a few c.c. of 10 per cent. calcium chloride solution and evaporated to dryness, or a quantity of the fruit (about 50 grams) is cut into small pieces and a solution of calcium chloride poured over the mass, which is afterwards dried. The dry residue is well charred and then boiled with 150 c.c. of water and the liquid filtered. The carbonaceous residue is incinerated at a moderate temperature and afterwards boiled with 150 c.c. of water and left for a few hours. The liquid is then filtered and the filtrate added to the first extract. The mixed filtrates are evaporated to 25—30 c.c. and, when cold, are neutralised with $N/10$ acid, methyl-orange being used as indicator. An equal volume of glycerol is now added and the liquid is titrated with $N/20$ sodium hydroxide in presence of phenolphthalein. A further quantity of glycerol (about 10 c.c.) is added, and the titration continued until a permanent red coloration is produced. Each c.c. of $N/20$ sodium hydroxide required represents 0.0031 gram of boric acid.

An alternative method for the estimation of boric acid is given. The substance is treated with calcium chloride in the manner already described, and is afterwards charred and extracted with about 50 c.c. of water. The aqueous extract is transferred to a 100 c.c. flask and evaporated nearly to dryness. The charred residue is ignited, the ash is moistened with 2 c.c. of strong sulphuric acid, and the mixture warmed ; when the greater part of the hydrogen chloride has been expelled, the residue is added to the concentrated liquid in the distilling flask. The last portions are washed into the flask with 10 c.c. of methyl alcohol and the liquid is distilled nearly to dryness. Successive quantities of 10 c.c. of methyl alcohol are added to the cold residue, the distillation being repeated after each addition ; six such treatments are usually sufficient to ensure the volatilisation of the whole of the boric acid. The alcoholic distillate is passed into 25 c.c. of water in a flask. When the distillation is finished, the liquid is evaporated until free from alcohol ; the methyl borate is hydrolysed by this treatment and a solution of boric acid remains. This solution is diluted with a little water and rendered neutral to methyl-orange. An equal volume of glycerol is added, and the liquid is titrated with sodium hydroxide in presence of phenolphthalein just as in the preceding method.

The results of the estimation of boric acid in various samples of apples, pears, quinces, pomegranates, grapes, cider, and apple juice are appended.   E. G.

Analysis of Coal.   HENRI PELLET (*Chem. Centr.*, 1904, ii, 791—792 ; from *Bull. Assoc. Chim. Sucr. Dist.*, 21, 1203—1223).— Most of the ordinary methods of analysis of fuels are described and compared in the original paper, with special reference to the sugar industry. The samples of coal should pass through an 80 or 100 sieve. The moisture is determined by heating 1—2 grams at 100—105° and weighing for the first time in 10—15 minutes ;

weights varying by 2 mg. or less are considered constant. The ash is estimated by gradually heating 0·5—1 gram in a flat platinum dish to a red heat in a muffle; under favourable conditions, the weight is constant in 15—30 minutes. The volatile matter is determined by heating 1—2 grams in a covered platinum crucible at a red heat; the crucible is heated for 3 minutes after gas has ceased to burn at the lid. The sulphur is estimated from the sulphuric acid formed in combustion in a calorimetric bomb, or by heating 1 gram of powdered coal with a mixture of 20 grams of sodium carbonate and 10 of potassium nitrate, and precipitating as barium sulphate. If iron sulphide is present in coal, it is converted on burning into ferric oxide and sulphuric acid, and the quantity of sulphate in the coal is not increased. Calcium carbonate is decomposed at a dark red heat in presence of silica.

Heraeus' electric furnace is recommended for the determination of the ash and volatile matters at 1000°, and for other operations which require a constant temperature. E. W. W.

Estimation of Carbon and Sulphur in Iron and Steel. A. MÜLLER (*Chem. Zeit.*, 1904, **8**, 795—796; *Zeit. angew. Chem.*, 1904, **17**, 1453—1454).—A compact form of apparatus is described for the combustion of the evolved gases. The whole is mounted on one stand and consists of a capillary platinum ∪-tube for the actual combustion, a drying tube, and a soda-lime absorption tube. The drying tube is a combination of a spiral and a ∪-tube. The spiral contains a mixture of phosphoric and sulphuric acids, whilst the limb of the ∪-tube next the combustion tube is filled with phosphoric oxide held in position by wads of glass-wool. The evolution flask is provided with a condenser reaching down the inside of its neck, the outlet for the gases being at the upper part of the neck (Corleis' pattern). In the estimation of sulphur, the platinum combustion tube is replaced by a quartz-glass tube and the hydrogen sulphide is absorbed by means of cadmium acetate. W. P. S.

An Improved Geissler Apparatus for the Estimation of Carbon Dioxide. ENGELBERT KETTLER (*Zeit. angew. Chem.*, 1904, **17**, 1097—1098).—The apparatus described is a modified form of Schrötter's for the estimation of carbon dioxide in carbonates. It is provided with four openings; the first, for the tube containing the dilute acid; the second, for the introduction of the carbonate; the third is kept stoppered until the carbon dioxide evolved is practically all expelled from the apparatus, when a calcium chloride tube is attached to this opening and the removal of carbon dioxide then completed; the fourth contains the sulphuric acid for drying. The apparatus yields good results. A. McK.

Analysis of Soluble Glass (Sodium Silicate). PAUL HEERMANN (*Chem. Zeit.*, 1904, **28**, 879—880, 883—884).—A paper dealing principally with the estimation of free alkali in commercial sodium silicate solution.

Ten grams of the sample are diluted with 100 c.c of water and mixed with 100 c.c. *N*-barium chloride. The mixture is diluted to 250 c.c.

and at once passed through a dry filter ; the first 20—30 c.c. are rejected. One hundred c.c. of the filtrate are now titrated with $N/10$ hydrochloric acid, using phenolphthalein as indicator.   L. DE K.

Gravimetric Estimation of Calcium.   ENGELBERT KETTLER (*Zeit. angew. Chem.*, 1904, **17**, 1488—1489.   Compare this vol., ii, 517).—A reply to Brück (this vol., ii, 681). The author still thinks that weighing the calcium as sulphate is more convenient than its conversion into carbonate.   L. DE K.

Results obtained in Electrochemical Analysis by the use of a Mercury Cathode.   RALPH E. MYERS (*J. Amer. Chem. Soc.*, 1904, **26**, 1124—1135).—A lengthy paper unsuitable for adequate abstraction, setting forth the advantages of the use of a mercury cathode in the electrolytic separation of a number of metals.   By its means even chromium may be quantitatively separated from aluminium.

L. DE K.

Volumetric Estimation of Lead.   ERIC JOHN ERICSON (*J. Amer. Chem. Soc.*, 1904, **26**, 1135—1139).—A modification of Walters and Affelder's method (Abstr., 1903, ii, 614) applied to brasses and bronzes.   The alloy is dissolved in nitric acid, and to the filtrate is added excess of ammonia and then some ammonium persulphate.   On boiling, the lead is precipitated as dioxide and washed first with dilute ammonia and then with hot water.   The precipitate is treated with nitric acid and a known volume of hydrogen peroxide, the excess of which is then estimated with standard permanganate solution. From the result, the amount of lead is readily calculated.   The permanganate should preferably be checked against lead nitrate.

L. DE K.

New Method for the Volumetric Estimation of Copper, and its Application to the Testing of Copper Sulphate and Commercial Copper Sulphide.   GIOACHINO GRIGGI (*Chem. Centr.*, 1904, ii, 367 ; from *Boll. Chim. Farm.*, **43**, 392—394).—The reaction on which the method is based is the reduction of copper sulphate by hydroxylamine, according to the equation : $(NH_2 \cdot OH)_2, 2HCl + 4CuSO_4 + 10KOH = 2Cu_2O + N_2O + 2KCl + 4K_2SO_4 + 9H_2O$.   To a solution of 1·39 grams of hydroxylamine hydrochloride in water, 5·6 grams of potassium hydroxide are added, and the solution is made up to 1 litre.   This hydroxylamine solution is then added gradually to the copper salt solution until reduction is complete ; heat may be required. The sulphates of iron, zinc, sodium, magnesium, and other metals interfere with the straightforward course of the reaction.   J. C. P.

Electrolytic Assay of Copper containing Arsenic, Antimony, Selenium, and Tellurium.   GEORGE L. HEATH (*J. Amer. Chem. Soc.*, 1904, **26**, 1120—1124).—If the metal contains arsenic and antimony, but less than 0·01 per cent. of joint nickel, cobalt, and zinc, it is dissolved in nitric acid, evaporated with sulphuric acid, and finally

deposited electrolytically from an ammoniacal solution. For details of the electrolysis, the original paper should be consulted. If only traces of antimony, and less than 0·02 per cent. of arsenic, but large amounts of tellurium or selenium are present, the latter are removed by treating the boiling solution of the sulphate with a current of sulphur dioxide ; the filtrate is then electrolysed. If the sample contains more than 0·01 per cent. of antimony and other elements, these may be removed by precipitation with ammonia in the presence of ferric nitrate.

The author has also introduced another process which is based on the fact that under certain conditions the electrolytic co-precipitation of any arsenic present is prevented by addition of solid ammonium nitrate. L. DE K.

Analysis of Bauxite. TAUREL (*Ann. Chim. anal.*, 1904, 9, 323—327).—An aliquot part of the solution, free from silica, obtained by the usual process, is precipitated with ammonia, and, after purifying the washed precipitate, by redissolving in hydrochloric acid and reprecipitating with ammonia ; it is finally ignited and weighed. It represents aluminium and ferric oxides, and possibly also titanic acid. Another portion is then neutralised with ammonia, any precipitate is redissolved in formic acid, and some 5 c.c. of this is added in excess. After reducing the iron by means of a current of sulphur dioxide, the whole is boiled for an hour, and an excess of solution of ammonium citrate is added, followed by ammonia and ammonium sulphide. The precipitate so obtained, after washing and ignition, represents iron oxide and titanic acid.

If it is desired to weigh the titanic acid separately, an aliquot part of the solution is precipitated with ammonia, some 10 c.c. in excess of formic acid are added, the iron oxide is reduced completely with sulphurous acid, and the whole is very gently boiled for an hour. The precipitate is allowed to settle for a few hours, and the supernatant liquid is, so far as possible, siphoned off. The precipitate is then collected on a filter and washed with boiling water (it generally runs through the filter at first). It is finally ignited and weighed.

The filtrate may, of course, be used for a repeated iron determination. L. DE K.

Electrolytic Estimation of Manganese. J. KÖSTER (*Zeit. Elektrochem.*, 1904, 10, 553—554).—A platinum-iridium basin and a platinum anode making 600—700 revolutions per minute are used. The solution contains 5 to 10 grams of ammonium acetate, 3 grams of chrome alum, and a few c.c. of alcohol in addition to the manganese salt, which must not be the chloride. A current of 0·04 to 0·045 ampere per sq. cm. may be used, which requires about 7 volts. The liquid is heated to 75° before beginning the electrolysis ; the temperature should not be allowed to rise above 85°. Alcohol (about 10 c.c.) may be used instead of the chrome alum with almost equally good results. The quantity of manganese present should not exceed 0·3 gram, otherwise the precipitate is apt to peel off. The precipitation is complete in 20 to 25 minutes. T. E.

Quantitative Precipitations and Separations by means of Ozone. PAUL JANNASCH and WILLY GOTTSCHALK (*Ber.*, 1904, **37,** 3111).—On passing ozonised oxygen through a solution of manganese ammonium sulphate acidified with acetic acid, the manganese is quantitatively precipitated as manganese tetroxide.   W. A. D.

Estimation of Manganese in Drinking Water. GEORG BAUMERT and PAUL HOLDEFLEISS (*Zeit. Nahr. Genussm.*, 1904, **8,** 177—181).—From 250 to 1000 c.c. of the water, according to the amount of manganese present, are acidified with 1 c.c. of hydrochloric acid and evaporated to a volume of less than 100 c.c. Towards the end of the evaporation, a little zinc oxide or barium carbonate is added to precipitate any iron present. The whole is then filtered and the precipitate washed until the filtrate amounts to 100 c.c. Five c.c. of a 10 per cent. solution of sodium hydroxide are added to the latter, which should be contained in a stoppered flask, and the mixture shaken for 5 minutes, the stopper being frequently lifted to admit air. Five c.c. of a 10 per cent. potassium iodide solution are now introduced, together with sufficient hydrochloric acid, drop by drop, to dissolve the brown precipitate. After the addition of starch solution, the liberated iodine is titrated with sodium thiosulphate solution (about 0·3 gram per litre) which has been standardised against a solution of pure manganous chloride.

To avoid the evaporation of a large volume of the water, the manganese may be separated by Knorre's method (Abstr., 1903, ii, 760) and then estimated by the above method.   W. P. S.

Chemico-toxicological Detection of Potassium Permanganate. DIOSCORIDE VITALI (*Chem. Centr.*, 1904, ii, 794—795 ; from *Boll. Chim. Farm.*, **43,** 493—504).—When potassium permanganate is taken into the organism, since no free acid is present except in the stomach, the hydroxide of manganese dioxide is formed. This change takes place so quickly that the administration of an antidote such as an acid solution of hydrogen peroxide is scarcely possible. When finely chopped meat is treated with the permanganate, the surface becomes brown ; the presence of manganese can be detected in the ash by the violet coloration which is formed when it is treated with excess of concentrated sulphuric acid and potassium bromate added. The meat also gives the following reactions. By the action of concentrated hydrochloric acid, an intensely brown coloration is formed which becomes blue on the addition of water and potassium iodide. A similar coloration is also obtained by adding water and a few drops of guaiacum tincture. When the meat is treated with 10—20 times its quantity of concentrated hydrochloric acid and aloin then added, a red coloration is formed. A violet colour is obtained by adding water, an ethereal solution of phthalin, hydrochloric acid, and finally potassium hydroxide until alkaline, and shaking. By agitating the meat with dilute hydrochloric acid and some drops of a solution of aniline sulphate, a blue or bluish-green coloration is formed.

The hydroxide of manganese dioxide acts on the albumin, forming an albuminate. Since the permanganate only attacks the external

portions of the tissue, putrefaction of the internal portion is not prevented, and the ammonium sulphide which is formed reduces the manganese dioxide to oxide, and finally converts it into sulphide, the brown colour being of course destroyed; in such cases, a test for manganese can only be applied. Pugliese has found that in the case of guinea-pigs a dose of about 0·2 gram, and for men about 10 grams, of permanganate per kilogram is fatal. A tissue which has been treated with potassium permanganate decomposes hydrogen peroxide with great rapidity, liberating oxygen; the fibrin of blood has also this property, but to a much less degree. E. W. W.

New Reaction for Iron in Copper. ED. CROUZEL (*J. Pharm. Chim.*, 1904, [vi], 20, 203—205).—Equal volumes of ten per cent. solutions of copper sulphate and sodium thiosulphate are mixed, when, after twenty-four hours, a greenish-yellow precipitate, passing into the yellow, crystalline double cuprous sodium thiosulphate, is formed, but if iron is present the colour is altered by the formation of an ochre-coloured basic ferric sulphite.

Should an excess of thiosulphate have been employed, the iron can be detected by means of potassium ferrocyanide. G. D. L.

Estimation of Tungsten. LOYS DESVERGNES (*Ann. Chim. anal.*, 1904, 9, 321—323).—A criticism of various methods already proposed, The following method is said to give fairly correct results. Twenty-five c.c. of the alkali tungstate, obtained in due course, are neutralised with dilute nitric acid, using litmus solution as indicator; while boiling, a solution of mercurous nitrate containing precipitated mercuric oxide in suspension is added, and the mercurous tungstate so obtained, after being washed on a filter with a weak solution of mercurous nitrate, is dried and ignited. The residue consists of tungstic acid, which is then weighed as such. It is as well to repeat the analysis once or twice. L. DE K.

Estimation of Uranium. F. GIOLITTI (*Gazzetta*, 1904, 34, ii, 166—170).—In a solution containing uranium in the form UX_4, the addition of a slight excess of hydrofluoric acid causes the formation of a heavy, pulverulent, green precipitate, which is possibly UF_4; this can be readily washed both by decantation and on the filter with water rendered faintly acid with hydrofluoric acid. If the uranium is present in the liquid as a uranyl salt of the type UX_6, it is neces-sary to reduce it to the form UX_4 before precipitating with hydro-fluoric acid. On igniting the precipitate, together with the filter, at a bright red heat in an open crucible, it becomes completely converted into the oxide U_3O_8, which can be weighed. If this oxide is further ignited in a current of dry hydrogen until it becomes constant in weight, it is reduced to the dioxide UO_2, the weight of which acts as a check on that of the U_3O_8. This method is given by the author for the estimation of uranium, and is found to yield good results.

Another, and in some ways more convenient, method of procedure with uranyl salts is to place the solution, together with 4—5 c.c. of concentrated hydrofluoric acid, in a smooth Classen electrolytic

capsule, the volume of liquid being then made up to 100—150 c.c. with water. Making the capsule the negative electrode and using a current density of about 0·8 ampere per sq. cm., the green, insoluble fluoride becomes loosely deposited on the capsule, from which it can be readily transferred to the filter. The ignition is then carried on as before. This method, in which the reduction is carried out without the introduction into the solution of foreign substances such as stannous chloride, also gives satisfactory results.   T. H. P.

Estimation of Tin, Antimony, and [Arsenic] in Ores and Alloys. HENRI ANGENOT (*Zeit. angew. Chem.*, 1904, **17**, 1274—1275). —The process is based on the use of sodium peroxide. On heating about half a gram of an ore or alloy containing tin, antimony, and perhaps arsenic and other metals, with 7 grams of sodium peroxide in an iron crucible, the three metals are rapidly oxidised and converted into sodium salts, which are then treated with water [or in the case of antimony with dilute alcohol (1:2)] and diluted to 250 c.c. The solution contains the tin and in some cases also arsenic or lead. On adding dilute sulphuric acid to 200 c.c. of the filtrate, the tin is precipitated as metastannic acid, and may be freed from any lead by heating with nitric acid; from arsenic, it may be freed by the ammonium chloride method (*Zeit. anal. Chem.*, 1895, **34**, 557).

The antimony which is left undissolved as sodium pyroantimoniate is separated from any other insoluble metallic oxides by digesting in hydrochloric and tartaric acids, then treating the solution with sodium hydroxide and sodium sulphide. After filtering off any undissolved sulphides, the antimony is recovered by precipitation with dilute sulphuric acid, or the solution is submitted to electrolysis and the antimony weighed as metal.   L. DE K.

Comparative Experiments on the Gravimetric Estimation of Antimony as Trisulphide and Tetroxide respectively. ALEXANDER GUTBIER and G. BRUNNER (*Zeit. angew. Chem.*, 1904, **17**, 1137—1143).—Quite as good results can be obtained when antimony is estimated as tetroxide as when it is estimated as trisulphide, if the manipulation in the former case is conducted according to Brunck's method (Abstr., 1895, ii, 372).   A. McK.

Coloration of Borax Beads by Colloidal Dissolved Noble Metals. JULIUS DONAU (*Monatsh.*, 1904, **25**, 913—918. Compare this vol., ii, 684).—If a borax bead is moistened with a solution of a gold salt and then fused, it becomes ruby-coloured, on long heating, blue and finally colourless unless much gold is present, when, after long heating, the bead is liver-coloured by reflected, blue by transmitted light. A solution containing 0·7 mg. of gold per 30 c.c. colours the bead red. The ruby colour is replaced by violet, and the reaction is less delicate if much sodium chloride is present. The reaction does not take place in presence of free sulphuric acid or halogens. The gold reaction is masked by the presence of more than 6 per cent. of platinum.

The borax bead is coloured yellow by silver. The reaction will

detect 0·00018 mg. of silver. The influence of other substances is the same as in the case of gold.

Platinum colours the borax bead reddish-brown by transmitted light ; the reaction detects 0·00005 mg. of platinum, Iridium and osmium give coloured borax beads similar to the platinum bead. Rhodium gives a brown, and palladium and ruthenium black beads. G. Y.

Action of Soap on Calcium and Magnesium Solutions. V. Hugo Gottschalk and H. A. Roesler (*J. Amer. Chem. Soc.*, 1904, **26**, 851—856).—A paper illustrated by curves showing peculiarities in the titration of calcium and magnesium salts with soap solution.

L. de K.

New Method for destroying Organic Matter in Toxicological Analyses. Oreste Gasparini (*Atti R. Accad. Lincei*, 1904, [v], **13**, ii, 94—100).—The author's method for destroying organic matter is as follows. The substance, after being freed as far as possible from alcohol, is covered with concentrated nitric acid in a beaker over which is placed a clock-glass provided with holes for admitting two glass tubes containing platinum wires connected with platinum electrodes. After the acid has been allowed to act for some time, a current of 4—6 amperes at a voltage of 8 is passed through the solution. The organic matter is destroyed either in a few hours or in a few days, according to the nature and quantity present and to the current intensity. In this way, the nitric acid is split up into ions, the hydrogen ion then acting on more nitric acid, forming water and nitrous acid, which latter then becomes resolved into water, nitric oxide, and nitrogen peroxide. The anion also probably reacts, thus : $2NO_3' + H_2O = HNO_3 + O$ and $NO_3' = NO_2 + O$. The nitrogen oxides and the nascent oxygen formed in this way readily oxidise the proteid substances. The solution is then filtered in the cold and the filtrate and wash-water evaporated as far as possible on the water-bath, the residue being then taken up in 35—50 c.c. of nitric acid and subjected once more to electrolysis for five or six hours. It is then evaporated to dryness, the residue dissolved in water, and analysed in the ordinary way. The insoluble portion, which may consist of fat, stannic oxide, antimonic oxide, silver chloride, and insoluble sulphates, is intimately mixed with a mixture of four parts of potassium nitrate with one of anhydrous sodium carbonate, and the whole gradually added to a porcelain crucible containing a little potassium nitrate. The mass is kept in a fused state for some time, then allowed to become nearly cold, and the crucible placed in a porcelain basin containing cold water. The remaining operations are conducted as usual.

With meat, eggs, &c., to which arsenic trioxide, mercuric chloride, silver, barium chloride, lead nitrate, zinc sulphate, or copper sulphate had been added, this method gave good results.

Sulphuric acid may be employed instead of nitric, but the oxidation is then slower and there is a tendency for the .proteid substances to froth unpleasantly ; further, some of the elements, arsenic for example, then give rise to volatile products. T. H. P.

Estimation of Benzene Vapour in Coal-gas.   OTTO PFEIFFER
(*Chem. Zeit.*, 1904, **28**, 884—885).—A half-litre separating funnel is
filled with the sample of gas, and when all the air has been displaced,
2 c.c. of Harbeck and Lunge's reagent (equal vols. of sulphuric and
fuming nitric acid) are introduced through the stem and left in con-
tact with the gas for half an hour, the acid being allowed to spread
over the sides of the funnel.   Solution of sodium hydroxide is now
introduced until the colour changes to orange-red, and hydrochloric
acid is then added until the liquid just turns a pale sherry colour.
The liquid is then shaken twice in succession with 50 c.c. of ether for
5 minutes, and the ethereal solution placed in a flask over 1 gram
of dry potassium carbonate and 0·5 gram of blood charcoal.   After a
few hours, the liquid is filtered and the charcoal well washed with
ether.   The filtrate is heated in a flask on a water-bath until the
ether has evaporated, and the residual dinitrobenzene is then dissolved
in alcohol and estimated, volumetrically by Limpricht's process (Abstr.,
1878, 335), titrating with stannous chloride and iodine.   L. DE K.

Contribution to the Analysis of Rose Oils.   PAUL JEANCARD
and C. SATIE (*Bull. Soc. chim.*, 1904, [iii], **31**, 934—937).—The
authors consider that the percentages of stearopten and of citron-
ellol in rose oil are the most important criteria of its quality.   The
former is determined by dissolving 10 grams of the oil in 50 c.c. of
acetone, cooling this mixture to −10°, and filtering through a tared
paper placed in a well-cooled funnel. The stearopten so separated
is dried over sulphuric acid under reduced pressure.   The filtrate,
which should not yield a further deposit when again cooled to −10°,
is used, after the removal of the acetone by distillation, for the deter-
mination of citronellol and "total alcohols."   The acid and saponifica-
tion numbers are determined on the original oil.   The paper gives
tables of the principal constants of (1) oils distilled from Provence
roses under various conditions, and (2) of the range of variation shown
recently by Bulgarian rose oil.   The results obtained with Provence
oil are analogous to those found by Schimmel and Co. for Saxony oil.
T. A. H.

Estimation of Methyl Alcohol in Formaldehyde.   HEINRICH
BAMBERGER (*Zeit. angew. Chem.*, 1904, **17**, 1246—1247).—When the
sulphanilic acid method for estimating methyl alcohol in presence of
formaldehyde according to Gnehm and Kaufler (this vol., ii, 520)
is employed, a slight decomposition of the condensation product during
the distillation cannot be avoided, and the slight amount of aldehyde
which passes into the distillate appreciably influences the result.

The author's method is conducted as follows.   The formaldehyde
solution is mixed with sodium hydrogen sulphite and the condensation to
sodium hydroxymethylenesulphonate conducted at the ordinary tempera-
ture for several hours in a closed vessel.   The product is then exactly
neutralised by sodium hydroxide and distilled ; the amount of alcohol
in the distillate is estimated by density determinations.   Sodium
hydroxymethylenesulphonate remains practically undecomposed when

heated in neutral solution. The results obtained by this method are higher than those obtained by Gnehm and Kaufler's method.

A. McK.

Two New Methods for the Estimation of Sugar. H. P. T. OERUM (*Zeit. anal. Chem.*, 1904, **43**, 356—371).—(I) By the employment of Meissling's colorimeter (this vol., ii, 440) for the colorimetric estimation of the copper reduced by sugar from Fehling's solution, satisfactory results can be obtained. The cuprous oxide is collected on an asbestos filter (not on paper), and after washing is dissolved in 10 per cent. nitric acid and the colour compared with a standard solution of copper sulphate. As the amount of copper reduced is not strictly proportional to the sugar present, it is better to calculate the amount of the former from the colorimetric readings and to obtain the corresponding amount of sugar from a table such as Allihn's. The method has been found very serviceable for sugar estimations in urine.

(II) The mercury reduced by sugar from Sachsse's solution may be collected on a filter paper, washed first with warm 1 per cent. hydrochloric acid, and then thoroughly with water, dissolved in boiling nitric acid, and then titrated by $N/10$ ammonium thiocyanate with iron alum as indicator, exactly as in Volhard's method for silver estimation. It is well to standardise the solutions by a known amount of grape sugar. The rapidity and convenience of the method render it very suitable for clinical work.

M. J. S.

The Molisch-Udránszky α-Naphthol Sulphuric Acid Reaction. B. REINBOLD (*Pfluger's Archiv*, 1904, **103**, 581—617).—The best conditions under which this reaction can be obtained are described. By the gentle action of sulphuric acid on dextrose, no furfuraldehyde is formed, but another volatile compound, which gives a bluish-violet colour with α-naphthol and sulphuric acid ; its spectrum shows an ill-defined band between λ594 and 582. Besides this there are two other volatile substances formed, one of which is acid and the other neutral; the last gives the magenta-sulphuric acid reaction, but neither reduces ammoniacal silver solutions.

W. D. H.

Detection of Abrastol in Wine. EMILIO GABUTTI (*Chem. Centr.*, 1904, ii, 370 ; from *Staz. sper. agrar. ital.*, **37**, 234—236).—The wine (100 c.c.) is treated with a few drops of ammonium hydroxide, extracted with amyl alcohol (10—15 c.c.), filtered, and evaporated to dryness. The residue is heated with some strong phosphoric acid, again heated after addition of 1—2 drops of a concentrated form-aldehyde solution, and filtered. The filtrate shows a green fluorescence in presence of abrastol.

N. H. J. M.

Estimation of Cellulose in Foods and in Fæces. OSCAR SIMON and HANS LOHRISCH (*Zeit. physiol. Chem.*, 1904, **42**, 55—58).—Ten grams of the substance (dried at 100°) are heated at 100° for 1 hour with 50 per cent. potassium hydroxide solution. When cold, 3—4 c.c. of hydrogen peroxide solution are added and, if necessary, the mixture is

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again heated until it is colourless or only pale yellow. It is allowed to cool and then mixed with half its volume of 96 per cent. alcohol ; if the solutions will not mix, a little concentrated acetic acid is added.   The precipitated cellulose is allowed to settle, removed to a hardened filter paper, washed thoroughly with water, dilute acetic acid, alcohol, and ether.   The precipitate usually contains a small amount of nitrogen (under 1 per cent.), and when obtained from fæces is extremely rich in ash (20—40 per cent.).   J. J. S.

Estimation of the Total Acidity in Proteid Substances·
SIRO GRIMALDI (*Chem. Centr.*, 1904, ii, 854—855 ; from *Staz. sper. agrar. ital.*, **37**, 463—475).—It has been shown (*ibid.*, **35**, 706) that the proteids, especially those of milk, combine with mineral acids to form compounds which are neutral towards the ordinary indicators.   In order to determine the total acidity, an excess of normal sodium hydroxide solution is added to the milk which has been previously treated with a known quantity of sulphuric acid. The excess of alkali is titrated ; the difference corresponds with the amount of acid added to the milk, and the natural acidity of the milk.   A second method consists in determining the quantity of sulphuric acid which is necessary to cause coagulation of the milk on warming, adding normal sodium hydroxide solution until the coagulum is dissolved, and titrating the excess of alkali with decinormal sulphuric acid.   The coagulum formed by rennet differs from that obtained by means of sulphuric acid in its behaviour towards dyes such as aromatic sulphonic acids.   The methods described above are not, however, affected by the presence of organic acids.   The results obtained are generally about twice as great as those found by the usual methods.   E. W. W.

Toxicological Detection of Hydrogen Cyanide. DOMENICO GANASSINI (*Chem. Centr.*, 1904, ii, 718—791 ; from *Bull. soc. med.-chir. Pavia.*   See this vol., ii, 758).

Estimation of Saturated Fatty Acids. WILHELM FAHRION (*Zeit. angew. Chem.*, 1904, **17**, 1482—1488).—An adverse criticism of the new process introduced by Partheil and Ferié (this vol., i, 5).
                                            L. DE K.

The Lithium Method of Separating Saturated Fatty Acids. K. FARNSTEINER (*Zeit. Nahr. Genussm.*, 1904, **8**, 129—136).—Partheil and Ferié have described a method for the separation of the saturated fatty acids of various fats and oils (this vol., i, 5), according to which 1 gram of the fat is saponified and precipitated from an alcoholic solution with lithium acetate.   The solution is warmed to dissolve the precipitate, and, on cooling, lithium stearate, palmitate, and myristate (partially) are said to crystallise out, whilst lithium laurate and the remainder of the myristate remain in solution.   The laurate is converted into the lead salt and separated, and the insoluble lithium salts separated by fractional crystallisation.   The author has investigated this method, and comes to the conclusion that it is quite

untrustworthy, and that the percentages of palmitic, stearic, myristic, and lauric acids found by Partheil and Ferié in butter, margarine, and lard are incorrect. Apart from the experimental data given, it is also shown that the presence of lauric and myristic acids in lard would considerably influence the saponification value of the latter. The lard examined by Partheil and Ferié had a normal saponification value. W. P. S.

Rapid Analysis of Cream of Tartar and Tartaric Acid Baking Powders. R. O. BROOKS (*J. Amer. Chem. Soc.*, 1904, 26, 813—818).—The total tartaric acid is estimated by Kenricks' polarimetric molybdate method (Abstr., 1903, ii, 112). The amount of combined tartaric acid (as potassium hydrogen tartrate) is found by deducting the amount of carbon dioxide in the original powder from the quantity corresponding with the alkalinity of the white ash, and calculating the difference to its equivalent amount of potassium hydrogen tartrate. The difference between the total and combined tartaric acid then gives the free acid present.

Starch is best estimated by treating the powder with cold 3 per cent. hydrochloric acid. The undissolved starch is collected in a Gooch crucible, washed first with cold water, then with alcohol, and finally with ether. After drying at 110° for two hours, it is weighed, then ignited, and reweighed, when the loss will represent the starch.

L. DE K.

Rapid Method for Estimating Fat in Milk. J. VAN HAARST (*Zeit. angew. Chem.*, 1904, 17, 1212—1213).—Polemical. A reply to Siegfeld (Abstr., 1903, ii, 458. Compare van Haarst, Abstr., 1903, ii, 516). A. McK.

Indirect Estimation of Milk Fat by means of Fleischmann's Formula. ALBERT STEINMANN (*Ann. Chim. anal.*, 1904, 9, 348—350).—A controversy with Pierre on the above subject.

L. DE K.

Comparison of the Halogen Absorption of Oils by the Hübl, Wijs, Hanuš, and McIlhiney Methods. LUCIUS M. TOLMAN (*J. Amer. Chem. Soc.*, 1904, 26, 826—827. Compare Abstr., 1903, ii, 458).—The original Hübl process is unsatisfactory. The process given by Hanuš (Abstr., 1902, ii, 112) gives very good results provided the reagent is added from 60—70 per cent. in excess. Wijs's process (Abstr., 1902, ii, 586) gives slightly higher results, but it is sufficient to add the reagent in excess to the extent of 35 per cent. In both these processes, 30 minutes' contact is sufficient to complete the reaction.

If it is desired to determine also the extent of the substitution, a solution of iodine chloride in carbon tetrachloride is recommended as being preferable to a solution of bromine in the same solvent (McIlhiney's reagent). L. DE K.

Colour Reactions of Fatty Oils. Part III. HANS KREIS (*Chem. Zeit.*, 1904, **28**, 956—957. Compare Abstr., 1903, ii, 114).—A *résumé* of the two earlier papers. Further particulars are given as to sesamol prepared by first agitating sesamé oil with hydrochloric acid and then so often with alcohol that the oil no longer gives the characteristic furfuraldehyde reaction. The alcoholic liquid is evaporated and the residue freed from fatty acids by treatment with dilute ammonia and calcium chloride. The filtrate then yields the sesamol to ether. It cannot be distilled without decomposition. In sulphuric acid, it dissolves with a green colour. Its ethereal solution, shaken with seed oils and nitric acid, gives a passing green coloration. The author thinks that sesamol is a phenol of high molecular weight with at least two hydroxyl groups in the meta-position and probably allied to naphtharesorcinol. If a piece of pine-wood is immersed in sesamol and then in hydrochloric acid of sp. gr. 1·19, it turns a dark green (a new lignin reaction).   L. DE K.

Betaine Periodide and the Estimation of Betaine by a Solution of Iodine in Potassium Iodide. VL. STANĚK (*Zeit. Zuckerind. Böhm.*, 1904, **28**, 578—583).—The addition of a solution of iodine in potassium iodide solution to an aqueous solution of a betaine salt produces in the cold a brownish-red precipitate, rapidly changing into green crystals with metallic lustre, and in the hot to a green, metallic-looking oil which crystallises on cooling. The same precipitate is obtained by adding a solution of iodine in hydriodic acid to one of betaine or its hydrochloride. It is only slightly soluble in water, but on boiling with the latter, iodine is evolved and betaine hydriodide goes into solution; it is readily dissolved by alcohol, hydriodic acid, or aqueous potassium iodide. It melts at $58—61°$ with copious evolution of iodine vapours. Its composition varies with the conditions of formation and approximates to that of the hexa-iodide,

$$C_5H_{11}O_2N,HI,I_5.$$
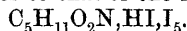
From very concentrated solutions of betaine, a solution of iodine in hydriodic acid precipitates a substance which has nearly the composition of betaine tri-iodide and which becomes green and assumes a metallic lustre when treated with water.

The betaine in a 1—3 per cent. solution of its hydrochloride is almost quantitatively precipitated by potassium tri-iodide (100 grams of potassium iodide and 153 grams of cyanogen-free iodine dissolved in 200 c.c. of water), when the solution is saturated with sodium chloride and the precipitate washed with saturated sodium chloride solution; the betaine in the precipitate is estimated by determining the nitrogen by Kjeldahl's method. When the betaine solution is very dilute or the precipitant is used in large excess, the results obtained are low. The betaine may also be precipitated from solutions containing 44 per cent. of sulphuric acid in place of the sodium chloride.

In this way, betaine may be separated from glycine, asparagine, tyrosine, glutamic acid, and ammonium salts. The precipitation is not affected by acetic or lactic acid, or by the constituents of the ash of molasses. Carbamide is partially precipitated with the betaine and a

marked precipitation occurs with the substances contained in Liebig's meat extract, probably peptones and purine bases.

In an acidified dilute solution of molasses, potassium tri-iodide produces a copious black, and to some extent crystalline, deposit containing 1·64 per cent. of nitrogen ; 65 parts of this nitrogen consist of betaine-nitrogen.   T. H. P.

Choline in Cerebrospinal Fluid. JULIUS DONATH (*Zeit. physiol. Chem.*, 1904, **42**, 563—567).—Polemical against Mansfeld (this vol., ii, 623). The criticisms passed on the methods for detecting choline are shown to be fallacious. A combination of the author's method with that originally introduced by Halliburton and Mott excludes any possible confusion with inorganic chlorides, even although traces of the latter may be soluble in absolute alcohol. Various observers are quoted who have recently confirmed the value of the choline test. [No reference is, however, made to the results obtained by Allen (this vol., ii, 623) by the iodine test.]   W. D. H.

Reactions for Brucine and Strychnine. I. Brucine. C. REICHARD (*Chem. Zeit.*, 1904, **28**, 912—914).—If a solution of brucine or its sulphate is evaporated on the water-bath with addition of silver nitrate, the silver is partly reduced and a black powder remains covered with a varnish-like substance. If the latter is now moistened with a drop of solution of stannous chloride, a very characteristic deep red colour is obtained.

If a trace of brucine or its sulphate is triturated with a drop of a 10 per cent. formaldehyde and then evaporated to dryness, a residue is obtained which, on being moistened with a drop of stannous chloride, turns blue and changes to a yellowish-green on warming.   L. DE K.

Estimation of Codeine in Opium. CHARLES E. CASPARI (*Pharm. Rev.*, 1904, **22**, 348—352).—Fifty grams of opium are extracted with water and the aqueous extract evaporated on the water-bath to a volume of about 250 c.c. Five grams of barium acetate are added and the solution diluted to 700 c.c. The precipitate formed, consisting of the meconic acid and much of the resin, is collected on a filter and washed with cold water. The filtrate is again concentrated, treated with barium acetate, diluted, and filtered. These operations are repeated until the addition of barium acetate, followed by dilution, produces no further precipitate. The solution is then concentrated and a slight excess of 10 per cent. sodium hydroxide solution added. This precipitates the thebaine and narcotine, which are collected on a filter, the morphine, codeine, and narceine remaining in solution in the filtrate. The latter is acidified with dilute hydrochloric acid, concentrated, and an excess of 2 per cent. ammonia solution added. Most of the morphine is precipitated, and is collected on a filter and washed. The filtrate is again acidified, concentrated, treated with ammonia, and filtered if necessary. After acidifying and evaporating the filtrate to a volume of 75 c.c., it is rendered alkaline with ammonia and thoroughly extracted with benzene, which dissolves the codeine, but not the narceine. The

residue obtained on distilling off the benzene is treated with an excess of $N/10$ sulphuric acid, and the excess titrated with $N/10$ sodium hydroxide.   A sample of Smyrna opium yielded 1·12 and 1·33 per cent. of codeine as the results of two estimations.

The author considers that there are two serious objections to Van der Wielen's method (Abstr., 1903, ii, 519).   In the first place, the quantity of opium taken for the estimation is too small ; and secondly, the use of an aliquot part of the ethereal solution does not conduce to accuracy.   W. P. S.

Estimation of Nicotine in presence of Pyridine.   JAMES A. EMERY (*J. Amer. Chem. Soc.*, 1904, **26**, 1113—1119).—The method is based on the fact that nicotine rotates the plane of polarisation, whilst pyridine does not.   The liquid to be tested is compared polariscopically with a solution of pure nicotine of about 1 per cent. strength, which has been carefully checked against $N/5$ hydrochloric acid.   The readings are expressed in degrees of the sugar scale.   After deducting the alkalinity due to the nicotine from the total alkalinity, the amount of pyridine may be calculated with fair accuracy.   In applying the process to the analysis of tobacco extracts, &c., the mixed bases are isolated by Kissling's process (extraction with ether in presence of sodium hydroxide, distillation of the ethereal residue, after adding some more alkali, in a current of steam, and titration with acid, using methyl-orange as indicator).   If nicotine only is wanted, the tedious preliminary extraction with ether may be omitted.   L. DE K.

Alkaloid Reactions.   C. REICHARD (*Chem. Centr.*, 1904, ii, 369 ; from *Pharm. Zeit.*, **49**, 523—524.   Compare Abstr., 1903, ii, 458, and this vol., ii, 374).—A trace of morphine, when added to a mixture of formaldoxime and concentrated sulphuric acid and heated, produces an intense bluish-violet, opaque liquid.   The colour remains when a small amount of water is added, but dilution with much water almost decolorises the solution.   Sodium hydroxide at once destroys the colour, ammonia less quickly.

Atropine when similarly treated, yields a brownish-black coloration, which disappears when a few drops of water are added.   No coloration is produced with cocaine, strychnine, or brucine.   N. H. J. M.

Detection of Quinine by J. J. André's Reaction.   P. GUIGUES (*J. Pharm. Chim.*, 1904, **20**, [vi], 55—57).—The colour reaction of quinine with chlorine water and ammonia is prevented when tincture of orange is present.   G. D. L.

Some Colour Reactions.   J. B. BALLANDIER (*J. Pharm. Chim.*, 1904, [vi], **20**, 151—152).—On allowing bromine vapour to be absorbed by feebly acid solutions of quinine and quinidine, a pale yellow colour is produced.   One drop of copper sulphate solution, followed by the gradual addition of ammonia, then gives a pink colour, changing to violet and finally green.   Mineral acids, with the green solution, give blue or violet according to the acid used (the

green colour being restored by alkalis), the pink and violet solutions similarly yielding green colours with acids.

The green colour produced with quinine by bromine and ammonia is changed by copper sulphate to blue, which is unaltered by acids.

Chelidonine and narceine give the same green tint with sulphuric acid and the tannin of gall nuts, but chelidonine alone gives a pure carmine colour with sulphuric acid and guaiacol.　　　　G. D. L.

The Xanthine Bases in Meat, Yeast, and other Extracts. II. In Yeast Extracts. KARL MICKO (*Zeit. Nahr. Genussm.*, 1904, **8**, 225—237).—Methods are given for the further purification of the various fractions containing the xanthine bases of yeast extract (this vol., ii, 458), the processes being similar to those employed in the case of meat extract (Abstr., 1902, ii, 369).

With regard to the occurrence of carnine in meat extracts, the author in no instance detected the presence of this base, and considers that it is either only occasionally present or that it is of a transitory nature.

Hypoxanthine was found to crystallise in two modifications. One form consisted of needles containing water of crystallisation, which spontaneously and readily lost their water, yielding anhydrous octahedra.　　　　W. P. S.

Chemical and Physiological Assay of Digitalis Tinctures. GEORGE BARGER and W. VERNON SHAW (*Pharm. J.*, 1904, [iv], **19**, 249—254).—A comparative study of the chemical and physiological methods of estimating the digitoxin in tincture of digitalis has shown that only the latter method is trustworthy. The amount of digitoxin found by Keller's method (Abstr., 1898, ii, 267) is less than half the quantity actually present, and the digitoxin prepared in this way contains 33 per cent. of impurity. The results of the examination of several samples of digitalis tincture by both methods are tabulated.

　　　　　　E. G.

Estimation of Tannin and Gallic Acid. W. PORTER DREAPER (*Chem. News*, 1904, **90**, 111, 112).—For the method proposed, the following solutions are required : (1) copper sulphate solution of such strength that each c.c. corresponds with 0·05 gram of cupric oxide ; (2) 50 grams of ammonium carbonate and 50 grams of sodium sulphite in 1 litre ; and (3) 20 grams of lead acetate and 60 c.c. of glacial acetic acid in 1 litre. Fifty c.c. of the tannin solution, containing from 10 to 15 grams of the tannin material per litre, are titrated with the copper solution, after heating with excess of calcium carbonate and cooling. Potassium ferrocyanide is used as indicator. The result represents the total tannin and gallic acid expressed as cupric oxide. A second quantity of 50 c.c. of the tannin solution is then treated with 25 c.c. of the sulphite solution (2) and titrated with the copper solution. In this titration, the indicator must contain an excess of acetic acid. The result represents the tannin alone. The relation of cupric oxide to tannin may be ascertained by collecting the precipitate on a filter, drying at 105°, and weighing. The amount of copper

oxide present being known, the tannin may be calculated.   Fifty c.c.
of the tannin solution are again taken and treated with 10 c.c. of the
lead acetate solution in the presence of barium sulphate. After shaking,
the precipitate is collected on a dry filter and a small amount of
anhydrous sodium sulphate added to the filtrate.   The lead sulphate
is filtered off and 40 c.c. of the filtrate heated with calcium carbonate
and titrated with the copper solution.   The result gives the gallic acid
in the filtrate, and, by difference, the total tannin.   The latter may or
may not agree with the amount found in the second titration, any
difference being due to the tannin (soluble or insoluble) in the
ammoniacal solution.   This classification of the tannins is a recognised
one, and the method forms a means of quantitatively separating the
two groups.   W. P. S.

Volumetric Estimation of Methylene-blue.   LOUIS PELET and
V. GARUTI (*Bull. Soc. chim.*, 1904, [iii], 31, 1094—1097).—The
authors, utilising the suggestion of Seyewetz (Abstr., 1900, i, 356,
522, 614, 645) that the formation of insoluble compounds between
basic and acidic dyes might be applied to the estimation of these sub-
stances, have carried out some experiments on the estimation of
methylene-blue by means of crystalline ponceau, "carmine," pyra-
mine-orange, and cotton-brown.   Of these, the first is considered
to give the best results.
    To a known volume of the methylene-blue solution (0·5 per cent. in
water) is added, drop by drop, a similar solution of the acid dye until
a drop of the liquid placed on filter paper shows the tint of the acid
dye used as a precipitant.   The precipitate formed when crystalline
ponceau is added to methylene-blue consists of 2 mols. of the former
to 1 mol. of the latter; with "carmine," the compound produced con-
tains 1 mol. of each dye.   The pyramine-orange and cotton-
brown used were impure and the composition of the precipitates
formed with these was not determined.   T. A. H.

Rapid Method of Distinguishing between Rosaniline and
Pararosaniline.   RUDOLF LAMBRECHT and HUGO WEIL (*Ber.*, 1904,
37, 3031).—If 1 part of commercial rosaniline is dissolved by heating
with 20 parts of 30 per cent. hydrochloric acid, no crystallisation
takes place on cooling, or even after some days.   If pararosaniline is
treated in the same way, the sparingly soluble hydrochloride crystal-
lises out immediately the solution is cooled. This difference can be used
to detect the presence of 0·5 per cent. of pararosaniline in rosaniline, the
rust-coloured pararosaniline hydrochloride crystallising out after 12
hours ; the presence of larger proportions of pararosaniline (5—10 per
cent.) is shown by immediate crystallisation on cooling.
    If rosaniline is dissolved in too small a volume of hydrochloric
acid, the polyhydrochloride slowly separates in crystals.   G. Y.

Estimation of Lecithin in Plants.   ERNST SCHULZE (*Chem. Zeit.*,
1904, 28, 751, 752).—In the estimation of lecithin in plants and

seeds, the absolute alcohol used for the extraction does not dissolve
out any phosphorus-containing compound other than lecithin. With
regard to the decomposition of lecithin at temperatures exceeding 60°,
the author considers that, for safety, this temperature should be
adhered to. Whether the lecithin in combination with albumin is
completely extracted at 60° still remains to be proved. The factor
employed for calculating the phosphorus found into lecithin is only
approximate, as lecithin has been obtained from rye and barley corns
containing only 2·2 to 2·3 per cent. of phosphorus, whilst the usually
occurring distearyl-lecithin contains 3·84 per cent. of phosphorus.

W. P. S.

The Ferricyanide Method of Estimating Oxygen in Blood.
FRANZ MÜLLER (*Pfluger's Archiv*, 1904, 103, 541—580).—Some modi-
fications of the Haldane-Barcroft apparatus are proposed, and the
method is found to be highly satisfactory. Some investigations on
the dissociation curve of dog's blood are described, and a number of
researches on the question in health and disease are propounded for
future work. W. D. H.

Estimation of the Alkalinity of Blood. SERGEI SALASKIN and
Z. PUPKIN (*Zeit. physiol. Chem.*, 1904, 42, 195—199).—Further experi-
ments by the Salkowski-Salaskin method have been carried out. The
process consists in heating the blood with ammonium sulphate under
reduced pressure and in a Nencki-Zaleski apparatus, the temperature
of the water-bath being kept at 40°. The operation lasts two hours.
Good results can be obtained with 10 c.c. of blood. The values are
quite different when water is added.

Under similar conditions, namely, reduced pressure at 40° and
evaporation to dryness, sodium hydroxide, carbonate, and hydrogen
carbonate evolve the equivalent amount of ammonia. In a 15 per cent.
solution, disodium hydrogen phosphate drives out ammonia until it is
half transformed into monosodium salt. The monosodium phosphate
does not evolve ammonia. Similarly, proteids evolve practically no
ammonia. J. J. S.

Simple Clinical Method of Estimating the Different
Proteids in Urine. ADOLF OSWALD (*Chem. Centr.*, 1904, ii,
858—859 ; from *Münch. med. Woch.*, 51, 1514—1515).—The amount
of the different proteids in urine may be estimated by means of
Esbach albuminometers in the following way. In the first, the total
quantity of albuminous substances is estimated by means of
Esbach's reagent. Three more are filled with urine from a
burette to the mark U. To the second is added a quantity of
a saturated solution of ammonium sulphate, sufficient to give a ratio
of salt to urine of 28 : 7·2 ; the concentration in the mixture corre-
sponds with 28 per cent. saturation and is equal to the limit of
precipitation of fibrinogen. The third is the euglobulin fraction and
in this a ratio of 3·6 : 6·4 and a saturation of 36 per cent. are obtained,
whilst in the fourth or ψ-globulin fraction the ratio is made 5 : 5 and

the saturation 5) per cent. After shaking, the albuminometers are allowed to remain for several hours. The clear liquid is then removed by a siphon or by filtration, and the precipitate dissolved in water to which, preferably, a little sodium carbonate has been added. The albumin is then estimated in each case by means of Esbach's reagent in the usual way, a few drops of acetic acid being added if sodium carbonate has been previously used. Albuminometer 2 gives the quantity of fibrinogen or fibrinoglobulin. The quantity of albumin found in the precipitate from 3, less that obtained from the precipitate in 2, is equal to the quantity of euglobulin, and, similarly, 4—3 corresponds with the ψ-globulin and 1—4 the albumin.

The following results, expressed in percentages, have been obtained in cases of nephritis :

	Total albumin.	Fibrinogen.	Euglobulin.	ψ-Globulin.	Albumin.
Acute Scarlet Nephritis...	10	0	2·5	1·5	6
Acute Nephritis (advanced)	12	0	0	0·5	11·5
Acute Nephritis	5	0	0·25	1·25	3·5
Chronic Nephritis	4—8	0	trace	1—2	3—6

E. W. W.

Detection and Estimation of Albumin in Urines. H. BELLOCQ (*Ann. Chim. anal.*, 1904, 9, 384—385).—One hundred c.c. of the clear urine are mixed with one gram of calcium acetate, neutralised with ammonia, and thoroughly boiled with constant shaking until the froth rapidly disappears when the source of heat is removed. The precipitate is at once collected, and, without washing, introduced into a 40 c.c. tube, where it is treated with 3 c.c. of nitric acid. The phosphates and oxalates dissolve, and the urates are also gradually decomposed with effervescence, whilst the albumin is not dissolved. The tube is now filled with strong alcohol and shaken. Any turbidity or deposit shows the presence of albumin. If the amount is to be estimated, the precipitate is collected, washed with alcohol slightly acidified with nitric acid, and then dried at a very moderate heat.

L. DE K.

Volumetric Estimation of Humus in Soil by means of Potassium Permanganate. W. ISTSCHEREKOFF (*Chem. Centr.*, 1904, ii, 559 ; from *J. exper. Landw.*, 5, 55—66).—The ground soil is boiled for 40—50 minutes with titrated permanganate solution, sulphuric acid and water in a 250—300 c.c. flask, and titrated with oxalic acid. It is sometimes necessary to boil with an excess of oxalic acid, to dissolve the manganese peroxide, and to titrate back with permanganate. The results agree well with those obtained by Gustavson's method.

The humus may also be estimated by weighing the carbon dioxide produced.   N. H. J. M.