Analytical Chemistry.

Acidimetry of Coloured Solutions. An Application of the Pocket Spectroscope. Alfred Tingle (*J. Soc. Chem. Ind.*, 1918, **37**, 117; *J. Amer. Chem. Soc.*, 1918, **40**, 873—879).—A method is described whereby highly coloured acid solutions may be accurately titrated. The process depends on the fact that the absorption spectra of indicators are different in acid and in alkaline solutions. To make a determination, two similar vessels are taken, one of which contains the solution to be titrated and the other an equal volume of distilled water. To the latter, one drop of standard alkali is added, and then the indicator is slowly added from a burette until the characteristic absorption band shows a sufficiently sharp edge. The position of this edge is noted. Then the same volume of indicator is added to the solution to be estimated, and alkali added from a burette until the characteristic band is observed in the same position. This gives the end-point of the titration. The change does not involve the appearance of a new absorption band, but rather the shifting of a band already present. The method was tested on solutions of sulphuric acid of known concentration, which were coloured by the addition of neutral tea extract or liquorice. The results are quite as accurate as those obtained for colourless solutions by the ordinary method. The amount of indicator used is rather larger than that generally employed; the exact amount necessary must be found experimentally. In the present experiments, 1 c.c. of methyl-orange and 2·5 c.c. of cochineal extract were used. J. F. S.

Mercuric Oxide as a Standard for Volumetric Analysis. L. Rosenthaler (*Zeitsch. anal. Chem.*, 1918, **57**, 98).—Incze has recommended the use of yellow mercuric oxide as a standard in volumetric analysis (A., 1917, ii, 327), but the author points out that he and Abelmann had previously used mercuric oxide for the purpose (A., 1913, ii, 786). W. P. S.

Detection of Anions. Fritz Feigl (*Zeitsch. anal. Chem*, 1918, **57**, 135—138).—The substance is boiled with concentrated sodium

carbonate solution or fused with a mixture of sodium and potassium carbonates; after filtration, the solution is nearly neutralised with nitric acid and warmed with the addition of an excess of solid zinc nitrate. The mixture is filtered; the precipitate contains zinc sulphide, sulphite, phosphate, borate, fluoride, ferrocyanide, ferricyanide, and cyanide, and also molybdate, vanadate, and tungstate, whilst the filtrate contains zinc thiocyanate, chloride, bromide, iodide, sulphate, thiosulphate, and sulphite. These substances are then identified by drop reactions without further separation [See also *J. Soc. Chem. Ind.*, July.] W. P. S.

Estimation of Chlorine in Organic Substances (Gastric Juice, Blood, Milk, etc.). Sirot and Joret (*Ann. Chim. anal.*, 1918, **23**, 109—113).—The total chlorine in gastric juice is estimated by Volhard's method after the sample has been treated with Esbach's reagent (10 grams of picric acid and 25 grams of acetic acid per litre of water) and filtered. Chlorine in organic and inorganic combination is estimated in the same way after the sample has been evaporated to expel free hydrochloric acid, whilst chlorine in inorganic combination is obtained by titrating the residue left after evaporation and incineration. In the case of blood, sodium metaphosphate is recommended as a clarifier; 20 c.c. of the blood are mixed with 75 c.c. of water, 10 drops of nitric acid, 20 c.c. of 5% sodium metaphosphate solution, and 1·5 c.c. of acetic acid. The mixture is then diluted to 200 c.c., filtered, and the chlorine titrated in the filtrate. The acetic acid–picric acid solution may be used for precipitating the casein, etc., in milk previous to the estimation of the chlorine present. W. P. S.

Gravimetric Analysis. V. [Chlorides, Bromides, and Iodides.] L. W. Winkler (*Zeitsch. angew. Chem.*, 1918, **31**, i, 101—103).—Chlorides, bromides, and iodides are precipitated by a small excess of *N*-silver nitrate in 100 c.c. of the cold solution to which has been added 5 c.c. of *N*-nitric acid, or, in presence of ferric salts, 10—20 c.c. In the case of chlorides and bromides, the mixture is left for one hour and then boiled; in the case of iodides, the silver is added first, the nitric acid after half an hour, and the mixture is boiled after another half-hour. Twenty-four hours later, the precipitate is collected on a plug of cotton wool in a Kelch funnel and dried at 132°. It is washed with 50 c.c. of water acidified with nitric acid, and later with 50 c.c. acidified with acetic acid. Correction values amounting to a few tenths of a mg., according to the weight of the precipitate, are used to improve the accuracy of the results. Iodides may also be precipitated in the presence of hydrochloric acid as palladium iodide. 0·5 Gram of palladium is dissolved in nitric acid and the solution evaporated to dryness several times with hydrochloric acid; the residue is taken up with 10 c.c. of 10% hydrochloric acid, 1 c.c. of alcohol is added to remove any free chlorine, and the solution made up to 100 c.c. In absence of chlorides, the palladium iodide

remains in colloidal solution; when precipitated cold, it is flocculent, and becomes granular on heating. With a preponderating quantity of iodide, the neutral solution is diluted so that 100 c.c. will give about 0·1 gram of precipitate; 1·0 gram of sodium chloride is added, and 10 c.c. of palladium chloride solution, with agitation. The liquid is heated until the precipitate becomes granular, and the latter is collected on the cotton filter after twenty-four hours, washed with 100 c.c. of cold water, and dried at 132°. With small quantities of iodide, 100 c.c. of the liquid are acidified with hydrochloric acid and precipitated with 1 c.c. of the palladium solution in the cold. The precipitate is allowed to remain for twenty-four or forty-eight hours, according to its quantity, and is collected in the flocculent condition. The palladium iodide is somewhat soluble in presence of alkali bromides; in presence of large quantities of chlorides, a small correction is applied. J. F. B.

Titration Method for Chlorine, Bromine, Cyanogen, and Mercury. Emil Votoček (*Chem. Zeit.*, 1918, **42**, 257—260).—Chlorides may be titrated with standardised mercuric nitrate solution in the presence of a small quantity of nitric acid; 0·06 gram of crystallised sodium nitroprusside is used as the indicator, and the volume of the solution should be about 250 c.c. Sulphates, phosphates, and chlorates do not interfere, but sulphites and nitrites must not be present. The method is trustworthy and more accurate than Volhard's method. [See further, *J. Soc. Chem. Ind.*, July.] W. P. S.

The Estimation and Distribution of Bromine in the Organs and in the Blood after Dosing with Sodium Bromide. W. Autenrieth (*Münch. med. Woch.*, 1918, **65**, 33—35; from *Chem. Zentr.*, 1918, i, 472—473).—Bromine in bromides of the alkali metals can be estimated colorimetrically by treating the acidified aqueous solution with potassium hydrogen sulphate and potassium permanganate, the liberated bromine being extracted with chloroform and the extract compared with a standard bromine solution, using the Autenrieth-Königsberger colorimeter. The method is not affected by the presence of chlorine and is especially suitable for small quantities. Organs such as liver, kidneys, brain, etc., are heated in a nickel crucible with pure sodium hydroxide and a little potassium nitrate, and the acidified solution treated in the manner described. Sodium bromide is retained tenaciously by the human organism, and only very slowly eliminated by the kidneys, its retention being favoured by a diet poor in chlorine. The brain shows no specific attraction for bromine. D. F. T.

Gravimetric and Volumetric Estimation of Fluorine Precipitated as Thorium Fluoride. F. A. Gooch and Matsusuke Kobayashi (*Amer. J. Sci.*, 1918, [iv], **45**, 370—376).—Investigation of the method described by Pisani (A., 1916, ii, 393)

showed that the acidity of the solution and the excess of precipitant are important factors in the estimation of fluorine as thorium fluoride. The acidity of the solution (as free acetic acid) should be from $N/50$ to $N/5$, and the quantity of thorium added should not exceed by more than 50% the amount required for the precipitation. The thorium fluoride may be collected and ignited to oxide, the latter being taken as a measure of the thorium fluoride, $ThF_4,4H_2O$, or the excess of thorium, after filtration, may be precipitated as oxalate and this titrated with permanganate solution (compare this vol., ii, 177). [See, further, *J. Soc. Chem. Ind.*, 391A.] W. P. S.

Time as a Factor in Gravimetric Analysis. I. Precipitation of Sulphuric Acid. Z. KARAOGLANOW (*Zeitsch. anal. Chem.*, 1918, **57**, 77—98).—In the precipitation of sulphuric acid as barium sulphate, the rate at which the barium chloride solution is added has a considerable influence; the most trustworthy results are obtained when the addition is extended over a period of not less than 1·5 minutes. The concentration of the solutions, stirring, concentration of hydrochloric acid, etc., also have an influence, but temperature has little effect. The presence of potassium chloride decreases the amount of barium sulphate found, and to some extent counterbalances the effect of rapid precipitation, but this compensation depends on definite conditions of experiment. W. P. S.

The Estimation of Sulphates in Urine. A. L. FLOHR (*Arch. Néerland. physiol.*, 1918, **2**, 346—351).—The benzidine method of Rosenheim and Drummond for estimating inorganic and ethereal sulphates gives satisfactory results. If the liquid becomes coloured after hydrolysis of the ethereal sulphates by hydrochloric acid, and the colour interferes with the titration, it can be removed sufficiently by treating the liquid with animal charcoal. S. B. S.

Estimation of Non-protein Nitrogen in Blood. ISIDOR GREENWALD (*J. Biol. Chem.*, 1918, **34**, 97—101).—A full account of work previously published (A., 1917, ii, 523). H. W. B.

New Method for the Direct Nesslerisation of Ammonia in Urine. JAMES B. SUMNER (*J. Biol. Chem.*, 1918, **34**, 37—41).—In the Folin and Denis direct Nesslerisation method (A., 1916, ii, 574), the Merck's blood charcoal may be replaced by copper sulphate. The urine is treated with a practically saturated solution of copper sulphate (298 grams of the crystallised salt per litre). Copper hydroxide is then precipitated by adding a 2·03N-sodium hydroxide solution until the neutral point is almost reached, when about 90% of the creatinine is also precipitated. The small amount of creatinine remaining in solution is not sufficient to interfere with the subsequent Nesslerisation.

For rough comparative tests, standard colours similar to those

obtained by Nesslerisation are prepared by dilution of a solution containing 6% of crystallised ferric chloride and 2·5% of crystallised cobalt nitrate. H. W. B.

Apparatus for the Estimation of Nitric Acid by the Schulze-Tiemann Method. Karl Leuchs (*Chem. Zeit.*, 1918, **42**, 235).—The decomposition flask is closed with a glass stopper provided with a tapped funnel and a delivery tube, and the stopper is surrounded by a water-seal. The delivery tube, which is bent downwards and under the lower end of the gas-collecting burette, is provided with a glass non-return valve. The whole apparatus is constructed of glass. W. P. S.

Gasometric Estimation of Nitrates. C. A. Hill (*Analyst*, 1918, **43**, 215—216).—When an external reaction bottle is used in the estimation of nitrates by shaking the latter with sulphuric acid and mercury, it is necessary to fill the bottle previously with a gas inert towards nitric oxide. Carbon monoxide may be used for this purpose, and is prepared by heating a mixture of sodium formate and concentrated sulphuric acid. [See, further, *J. Soc. Chem. Ind.*, July.] W. P. S.

New Volumetric Method for the Estimation of Phosphates in Urines. Argeo Angiolani (*Giorn. Farm. Chim.*, 1917, **66**, 251—252; from *Chem. Zentr.*, 1918, i, 571).—Twenty-five c.c. of the urine are treated with 1 c.c. of 20% hydrochloric acid, 1 gram of ammonium chloride, and 10 c.c. of a citric acid–magnesium solution (a solution of 35 grams of magnesium oxide in 260 grams of citric acid, the total bulk being 500 c.c., which is then treated with 400 c.c. of 10% ammonia solution and kept for two hours). The precipitate is collected, washed with very dilute ammonia solution, dried at 30—40°, and then dissolved in 50 c.c. of $N/10$-sulphuric acid, of which the excess is then titrated with $N/10$-sodium hydroxide solution, using methyl-orange as indicator. One c.c. of $N/10$-acid is equivalent to 3·55 mg. P_2O_5. D. F. T.

Marsh's Apparatus. W. Kirkby (*Pharm. J.*, 1918, **100**, 286).—A tube loosely packed with cotton wool is interposed between the generating flask and the hydrogen jet with the object of preventing any risk of explosion. C. A. M.

Simple Process for the Estimation of Small Quantities of Arsenic in Corpses. H. Führer (*Ber. Deut. Pharm. Ges.*, 1918, **28**, 221—229).—The process consists in the destruction of the animal matter by permanganate and sulphuric acid, the distillation of the solution with sodium chloride, and the estimation of the arsenic in the distillate by the Gutzeit method, using mercuric bromide paper. [See *J. Soc. Chem. Ind.*, July.] J. H. J.

Estimation of Carbon Dioxide in Carbonates by Dittrich's Method. Béla von Horvath (*Chem. Zeit.*, 1918, **42**, 121).—Carbon dioxide may be estimated in sodium carbonate or barium carbonate by heating the same at dull redness with borax which has been heated previously at 1000°; the carbonate is decomposed readily, and the loss in weight gives the amount of carbon dioxide present. [See, further, *J. Soc. Chem. Ind.*, 369A.] W. P. S.

Filtration of Silica. P. Nicolardot and J. Koenig (*Ann. Chim. anal.*, 1918, **23**, 104—109).—The fact that a minute quantity of silica passes into the filtrate when hydrated silica is evaporated to dryness and then collected on a filter does not appear to be due to solubility of the silica; the effect of successive evaporations and heating at 110° is to agglomerate the silica so that the whole of it is retained by a good filter. It is recommended that the silica be twice evaporated with hydrochloric acid and heated at 110°, but without intervening filtration, before it is collected; the filtrate may be passed once more through the filter. [See, further, *J. Soc. Chem. Ind.*, July.] W. P. S.

Estimation of Strontium. L. W. Winkler (*Zeitsch. angew. Chem.*, 1918, **31**, i, 80 and 83—84).—*As Sulphate.*—One hundred c.c. of a neutral solution containing 0·5 gram of strontium salt are acidified with 1 c.c. of acetic acid, heated to the boiling point, and 10 c.c. of a 10% solution of sodium sulphate are added. Heating is continued until the precipitate is powdery, when it is left overnight. It is transferred to a Gooch crucible, washed with 50 c.c. of saturated strontium sulphate solution, and weighed after drying at 132°. If the filtrate is required further, alcohol is used as the washing agent. The presence of other salts, especially magnesium chloride and hydrochloric and nitric acids, leads to low results.

As Carbonate.—1·0 Gram of potassium nitrate and 10 c.c. of 10% sodium carbonate solution are added to a boiling solution of not more than 0·5 gram of strontium salt in 100 c.c. of solution. Next day the precipitate is washed with 50 c.c. of saturated strontium carbonate solution and weighed as $SrCO_3$ after drying at 132°. Owing to incomplete loss of carbon dioxide on ignition, the precipitate cannot be weighed as oxide.

As Oxalate.—The precipitation is made with 10% solution of potassium oxalate, and resembles that of the sulphate. After remaining overnight, the precipitate is washed with saturated strontium oxalate solution. It is dried at 100° for two hours and weighed as SrC_2O_4,H_2O, or at 132° for six hours and weighed as SrC_2O_4. Other salts, especially magnesium chloride, interfere. This is the most convenient and exact method of estimating strontium. H. J. H.

Time as a Factor in Gravimetric Analysis. Precipitation of Barium Chloride with Sulphuric Acid. Z. Karaoglanow (*Zeitsch. anal. Chem.*, 1918, **57**, 113—121).—In the gravi-

metric estimation of barium as barium sulphate, sulphuric acid should be used for the precipitation; alkali sulphates must not be used. The acid should be added rapidly; if it is added slowly, the results obtained are too low. [See, further, *J. Soc. Chem. Ind.*, 352A.] W. P. S.

Volumetric Estimation of Lead by means of Ammonium Molybdate. LINDT (*Zeitsch. anal. Chem.*, 1918, **57**, 71—76).—In this process, it is essential that an excess of ammonium acetate should be avoided in dissolving the lead sulphate; the results obtained are too high in the presence of such excess, but are trustworthy when the lead sulphate is dissolved in the minimum requisite quantity of the acetate solution. [See, further, *J. Soc. Chem. Ind.*, 352A.] W. P. S.

Estimation of Copper as Copper Oxide after previous Precipitation as Thiocyanate. G. FENNER and J. FORSCHMANN (*Chem. Zeit.*, 1918, **42**, 205—206).—The inconvenient drying of the cuprous thiocyanate precipitate to constant weight is unnecessary, and may be avoided by conversion of the precipitate into cupric oxide by roasting in a muffle at a temperature near 800°. [See also *J. Soc. Chem. Ind.*, 391A.] D. F. T.

Analysis of White Metal. F. KUREK and A. FLATH (*Chem. Zeit.*, 1918, **42**, 133—134).—Tin is estimated by dissolving the alloy in hydrochloric acid with the addition of ferric chloride, reducing the tin with metallic iron, separating the precipitated antimony and copper, and titrating the filtrate with ferric chloride solution. The antimony and copper are then dissolved in hydrochloric acid to which potassium chlorate is added, excess of free chlorine is expelled by boiling the solution, the two metals are separated as their sulphides, the antimony sulphide is dissolved in sodium sulphide solution, again precipitated in the presence of an excess of oxalic acid, dissolved in hydrochloric acid, the solution boiled until the antimony is reduced, and then titrated with potassium bromate solution. Suitable methods are also described for the estimation of lead, copper, iron, aluminium, nickel, and zinc in the alloy. [See, further, *J. Soc. Chem. Ind.*, 877A.) W. P. S.

Use of Metallic Silver as a Reducing Agent in the Volumetric Estimation of Iron. GRAHAM EDGAR and A. R. KEMP (*J. Amer. Chem. Soc.*, 1918, **40**, 777—784).—The reaction between metallic silver and solutions of ferric sulphate in the presence of sulphuric acid and a soluble thiocyanate has been examined. The results obtained show that silver may be employed to effect the complete reduction of the ferric salt provided the dissolved silver is precipitated by thiocyanate. The resulting ferrous solution is filtered, treated with an excess of silver nitrate, and titrated with potassium permanganate. An alternative

method consists in titrating the excess of silver nitrate with standard thiocyanate.

The thiocyanate serves to show when the reduction is complete, and further advantages of the method are that silver is usually quite free from iron, that it does not reduce titanium at all, and that it reduces vanadium quantitatively to the quadrivalent condition. [Compare *J. Soc. Chem. Ind.*, 391A.] H. M. D.

Estimation of Iron in Lactic Acid. A. Harvey (*J. Soc. Leather Trades' Chem.*, 1918, **2**, 37—38).—Iron in lactic acid can be estimated very exactly by a colorimetric method in which the colour developed with potassium ferrocyanide is matched against the colour produced by standard iron solution. Potassium thiocyanate is useless F. C. T.

Quantitative Separation of Iron from the Cerite Metals in the presence of Calcium. A. Wöber (*Zeitsch. landw. Versuchsw. Oesterr.*, 1917, **20**, 500—501; from *Chem. Zentr.*, 1918, i, 476).—A weighed sample is dissolved by prolonged treatment with 2% hydrochloric acid, and to an aliquot portion of the solution there is added tartaric acid in the proportion of approximately four grams to one of the substance. On saturating the solution with hydrogen sulphide and adding aqueous ammonia until a pure black precipitate of iron sulphide is obtained, the transiently precipitated hydroxides of the cerite metals are redissolved. The iron sulphide is treated in the usual manner, whilst the estimation of the cerite metals is effected by the method of Hauser and Wirth (A., 1908, ii, 778). D. F. T.

Estimation of Nickel with α-Benzildioxime. R. Strebinger (*Chem. Zeit.*, 1918, **42**, 242—243).—The author agrees with Grossmann and Mannheim (A., 1917, ii, 391) that Atack's method of estimating nickel by precipitation with α-benzildioxime is trustworthy for small quantities of the metal. When, however, the quantity of nickel exceeds 0·025 gram, the precipitate contains a certain amount of occluded α-benzildioxime, and the results obtained are too high. In such cases, the precipitate should be ignited and the resulting nickel oxide weighed. W. P. S.

Estimation of Chromium in Chromium Salts, Chrome Liquors, Leather Ashes, and Chromium Residues. Karl Schorlemmer (*Collegium*, 1917, 345 and 371; from *Chem. Zentr.*, 1918, i, 377—378).—The solution of the chromium salt is treated cautiously with approximately *N*-sodium hydroxide until the precipitate has redissolved. Aqueous hydrogen peroxide of approximately 3% concentration is then added, and the solution is boiled until no more oxygen is liberated. The resulting solution is acidified with sulphuric acid, and the amount of chromate estimated by one of the usual volumetric methods. Leather ash or dry

chromium residues should be mixed with anhydrous sodium carbonate and magnesium oxide and roasted until yellow, the aqueous extract then being titrated after acidification. For the oxidation of solutions of very impure chromium salts, it is better to use alkaline potassium permanganate solution. The presence of iron in the ash of chrome leather may interfere with the chromium estimation. D. F. T.

Estimation of Molybdenum. O. Binder (*Chem. Zeit.*, 1918, **42**, 255).—When molybdenum is precipitated as sulphide and the latter then ignited to oxide, the oxidation is not complete unless the substance, after preliminary ignition, is treated with nitric acid, evaporated, dissolved in ammonia, reprecipitated with nitric acid, evaporated, and ignited. A correction must be made for any traces of matter which remain insoluble when the ignited oxide is dissolved in ammonia. [See, further, *J. Soc. Chem. Ind.*, July.] W. P. S.

The Estimation of Molybdenum as Lead Molybdate. Robert Strebinger (*Oesterr. Chem. Zeit.*, 1917, [ii], **20**, 226—228; from *Chem. Zentr.*, 1918, i, 378).—For the estimation of molybdenum in ferro-molybdenum, 0·5—1 gram is fused with 10 grams of sodium peroxide in an iron crucible. The product is extracted with 500 c.c. of water, and 100 c.c. are taken for the test; after the removal of any iron by the addition of nitric acid and then ammonia solution, the solution is neutralised with acetic acid, boiled, and treated successively with solutions of lead acetate (2—5 grams) with acetic acid (2 c.c.) in 30 c.c. of water, and ammonium acetate (10 grams) in 50 c.c. of water. After boiling for a short time, the precipitate is allowed to settle for six hours. The precipitate is removed by filtration, washed with dilute ammonium acetate solution, dissolved in diluted nitric acid, and reprecipitated by the addition of a solution of ammonium acetate (10 grams) in 50 c.c. of very dilute acetic acid. After twelve hours, the lead molybdate is again separated, washed, dried, and ignited at a moderate temperature before final weighing as $PbMoO_4$. D. F. T.

A Colour Reaction of Thorium and Zirconium with Pyrogallolaldehyde. H. Kaserer (*Chem. Zeit.*, 1918, **42**, 170).—On the addition of an aqueous solution of pyrogallolaldehyde to one of a thorium compound, a yellow colour is formed, and, after a time, a dirty yellow precipitate is produced, which when filtered off leaves a colourless filtrate. Zirconium compounds, after boiling or after the addition of hydrogen peroxide, give a similar colour and precipitate with cerium compounds; the yellow colour remains after boiling. In the presence of nitric, sulphuric, or hydrochloric acid, a colourless solution and no precipitate are produced. A solution containing only 0·1 mg. of thorium nitrate per 100 c.c. shows the colour clearly. Pyrogallol, pyrogallolcarboxylic acid, and protocatechualdehyde do not give this reaction.

The pyrogallolaldehyde is prepared by dissolving 38 grams of pyrogallol and 36·3 grams of formanilide in absolute ether, condensing this with 15·2 grams of phosphoryl chloride, and filtering after twelve hours. The residue is dissolved in alcohol and precipitated by sodium chloride. The crystals are treated with warm sodium hydroxide, a current of hydrogen is passed through the solution, after which it is acidified and the aldehyde extracted with ether and purified by conversion into the bisulphite compound.

A. B. S.

Graphic Methods of Analysis. Hans Gradenwitz (*Chem. Zeit.*, 1918, **42**, 221).—The composition of such mixtures as formaldehyde, methyl alcohol, and water, and ethyl acetate, alcohol, and water, may be found from the graphs given, the data to be determined being, in the first case, the specific gravity and the formaldehyde content, and in the second, the specific gravity and the ethyl acetate content. [See, further, *J. Soc. Chem. Ind.*, 392A.]

W. P. S.

Method for Detecting Small Quantities of Chloretone (Trichloro-*tert.*-butyl Alcohol) in Aqueous Solutions. T. B. Aldrich (*J. Biol. Chem.*, 1918, **34**, 263—267).—The solution containing the chloretone is subjected to steam distillation. If a large amount of chloretone is present, it crystallises in the cooler part of the condenser in needles. When only small amounts of chloretone are present, crystallisation may not occur, but if the distillate is placed in a small flask fitted with a reflux condenser and boiled for half an hour, needle crystals are then obtained in the condenser when the amount of chloretone exceeds 0·25 mg. If protein is present, it should be digested with pepsin and hydrochloric acid before the steam distillation is carried out. The presence of other organic solvents prevents the crystallisation, and thus interferes with the recognition of chloretone by this method.

H. W. B.

Estimation of Cholesterol in Blood. L. Kast, V. C. Myers, and Emma L. Wardell (*Proc. Soc. Exp. Biol. Med.*, 1917, **15**, 1—2; from *Physiol. Abstr.*, 1918, **3**, 31).—One c.c. of blood is extracted with chloroform, and in the extract the cholesterol is estimated colorimetrically by the Liebermann-Burchard reaction (compare *Physiol. Abstr.*, 1917, **2**, 675). The values obtained are lower than those of Bloor, but are believed to be more accurate.

W. G.

Cammidge's Method for the Estimation of [Reducing] Sugar in Urine. R. W. Garrow (*Pharm. J.*, 1918, **100**, 148—149).—In estimating sugar by this method (A., 1917, ii, 276), it is noticed in titrating back the excess of iodine with thiosulphate that up to the point where the blue starch iodide is discharged the solution is transparent, but immediately after the first end-point is reached a slight opalescence begins to appear, in-

creasing to a white precipitate of cuprous iodide, and the blue colour returns. The first discharge of the blue colour should be taken as the end-point, and the titration should be done as rapidly as possible. [See, further, *J. Soc. Chem. Ind.*, 276A.]

J. F. B.

Polarimetric Estimation of Dextrose in Urine. G. FRERICHS and E. MANNHEIM (*Apoth. Zeit.*, **33**, 34; from *Chem. Zentr.*, 1918, i, 380. Compare A., 1917, ii, 393).—A 100 c.c. flask containing 5 c.c. of lead acetate solution is filled to the mark with the urine and shaken well; the liquid is then filtered and examined in the polarimeter in a 2-dcm. tube, the rotation giving the content of anhydrous dextrose in grams per 100 c.c. of urine. The lead acetate solution should contain 10 grams of the salt and 5 grams of 30% acetic acid in 20 grams of water. If the lead acetate treatment fails to decolorise the urine sufficiently, the latter may be decolorised with charcoal, either at the same time as the lead acetate treatment or subsequently. As the charcoal absorbs a certain proportion of the dextrose, a correction becomes necessary, for which empirical values are given. D. F. T.

Colorimetric Estimation of Dextrose in Urine. V. I. ISAACSON (*J. Lab. and Clin. Med., St. Louis*, 1918, **3**, 289—294; from *Physiol. Abstr.*, 1918, **3**, 120).—A copper sulphate method, in which the unreduced copper is estimated after adding ammonia in a colorimeter against a standard. S. B. S.

Estimation of Dextrose in Urine. C. H. HUGENHOLTZ (*Pharm. Weekblad*, 1918, **55**, 609—614).—A comparison of the iodometric, polarimetric, and fermentation methods of estimating dextrose in urine. The first method is very accurate, the second gives slightly low results, and the values derived from the third method are extremely erratic. A. J. W.

Estimation of Sugar in Normal Urine. STANLEY R. BENEDICT and EMIL OSTERBERG (*J. Biol. Chem.*, 1918, **34**, 195—201).—In this method, which permits of the estimation of traces of sugar accurately to within a few thousandths of 1%, the creatinine and polyphenols, and most of the total nitrogen and the glycuronic acid, are first removed from the urine by precipitation with a mercuric nitrate reagent, and the dextrose then estimated colorimetrically after treatment with picric acid. The necessary special reagents are prepared as follows. Mercuric nitrate solution, by adding slowly 220 grams of mercuric oxide to 160 c.c. of concentrated nitric acid until it has dissolved, then boiling, cooling, and adding 60 c.c. of 5% sodium hydroxide solution. It is made up to 1 litre and filtered. Picrate–picric acid solution, by adding 36 grams of picric acid and 400 c.c. of hot water to 500 c.c. of 1% sodium hydroxide solution and shaking until the picric acid has dissolved. It is cooled and made up to 1 litre.

To perform the estimation, 15 or 20 c.c. of the urine are placed in a 500 c.c. beaker, together with an equal volume of the mercuric nitrate solution, and, after mixing, solid sodium hydrogen carbonate is added until frothing ceases and an alkaline reaction to litmus paper is obtained. After filtering, the excess of mercury is removed by adding a pinch of zinc dust and a drop or two of concentrated hydrochloric acid. From 1 to 4 c.c. of the final filtrate (containing about 1 mg. of dextrose) are measured into a large test-tube graduated to indicate 12·5 and 25 c.c. Water is added if required to bring the volume to 4 c.c., and 1 c.c. of 20% sodium carbonate solution is run in, followed by 4 c.c. of the picrate–picric acid solution. The mixture is heated in boiling water for ten minutes, cooled, diluted to the mark, and compared in a colorimeter with a standard solution similarly prepared from 1 mg. of dextrose in 4 c.c. of water or with a permanent standard of picramic acid or potassium dichromate solution. The former is prepared by adding 0·5 c.c. of 20% sodium carbonate solution and 15 c.c. of the picrate–picric acid solution to 105 c.c. of exactly 0·01% picramic acid solution in 0·02% sodium carbonate solution and then diluting to 300 c.c. with water. To prepare the dichromate standard, dissolve 0·536 gram of potassium dichromate in 1 litre of water.

To estimate the fermentable sugar, a second estimation in the urine after fermentation is necessary. About 20 mg. of dextrose and a one-quarter cake of yeast are added to 25 c.c. of the urine. After mixing, it is allowed to remain in an incubator at 35—38° for eighteen to twenty hours. The clear urine is then decanted and the estimation of dextrose carried out as above. The difference between the two estimations gives the fermentable sugar.

H. W. B.

Modification of the Lewis-Benedict Method for the Estimation of Dextrose in the Blood. Stanley R. Benedict (*J. Biol. Chem.*, 1918, **34**, 203—207. Compare A., 1915, ii, 111).—The modification consists in adding the solution of picric acid in sodium picrate, employed in the estimation of dextrose in urine (see preceding abstract), instead of picric acid to the laked blood, which renders the subsequent evaporation unnecessary.

H. W. B.

Sources of Error in the Estimation of Dextrose by the Colorimetric Picrate Method. T. Addis and A. E. Shevky (*Proc. Soc. Exp. Biol. Med. New York*, 1918, **15**, 79).—The reddish-brown colour produced on heating dextrose, picric acid, and sodium carbonate varies with the temperature, duration of heating, and amount of carbonate present.

G. B.

Inversion and Estimation of Sucrose. A. R. Rose (*Proc. Soc. Exp. Biol. Med. New York*, 1917, **15**, 16—17).—Heating for ten minutes at 100° with 2 volumes of saturated picric acid inverts the sucrose; then 1 vol. of 20% sodium carbonate

is added, and after a further twenty minutes' heating the total dextrose + lævulose is estimated colorimetrically according to Lewis-Benedict. The amount originally present is estimated in a similar tube, in which the sodium carbonate was added before heating. The difference between the two tubes represents invert-sugar (compare preceding abstract). G. B.

Estimation of Furfuroids (Furfurosans) in the Different Products of Beet Sugar Factories. I. R. Gillet (*Bull. Assoc. chim. Sucr. Dest.*, 1917, **35**, 53—62).—It is known that other substances besides pentoses and pentosans yield more or less furfuraldehyde under the well-known conditions of distillation with hydrochloric acid. Chalmot has stated that sucrose yields not more than 0·2%, but the author, operating on 5—20 grams of pure sucrose, has obtained from 0·38 to 0·75%. of furfuraldehyde under the Tollens-Councler conditions of working. The method of procedure is described fully, and attention is directed to certain points which are of importance in securing uniform results. To prevent superheating, the distillation flask should not be immersed in the bath below the level of the liquid in the flask. The temperature of the bath should be such as to produce uniform distillation at the rate of 30 c.c. in twelve to fourteen minutes; when operating on 10 grams of sucrose, the author found it necessary to heat the bath to 155—160°, since at lower temperatures distillation was slow and the distillate was often cloudy, owing to the presence of an unknown, yellow substance in suspension. Great importance is attached to strict adherence to the prescribed method of replenishing the acid during distillation, exactly 30 c.c. being added as soon as 30 c.c. has distilled. In spite of attention to these and other details of procedure, it was found impossible to secure absolutely uniform yields of furfuraldehyde from sucrose. J. H. L.

Colour Reaction for Ground Wood Pulp or the Incrusting Matters of Wood with Phenylhydrazine Hydrochloride. S. Jentsch (*Zeitsch. angew. Chem.*, 1918, **31**, 72).—An aqueous solution of phenylhydrazine hydrochloride gives an intense orange-yellow coloration with raw wood fibre, which subsequently changes to a characteristic bright green on drying in presence of air; the appearance of the green colour is accelerated by suitable heating. Cotton and other pure cellulose fibres are stained only to a pale yellow, which changes to a characteristic light brown after drying. The above test for lignocellulose is stated to be sharper and more definite than the phloroglucinol–hydrochloric acid test. [See also *J. Soc. Chem. Ind.*, 365A.] J. F. B.

New Reaction of Formic Acid and Hyposulphites. E. Comanducci (*Boll. chim. farm.*, 1918, **57**, 101—102).—The presence of formic acid in a liquid may be detected by heating the latter gently with concentrated sodium hydrogen sulphite solution until gas bubbles begin to escape, the liquid being then cooled and

fresh, dilute sodium nitroprusside solution poured carefully on to its surface; a green or blue ring is thus formed, hydrogen cyanide being liberated at the same time. The blue precipitate, $Na_4Fe_2(CN)_9$, results from the interaction of the nitroprusside and sodium hyposulphite, the latter being formed by the action of the formic acid on the sodium hydrogen sulphite (compare A., 1904, ii, 845). T. H. P.

Estimation of Lactic Anhydrides in Lactic Acid. F. C. Thompson and Kyohei Suzuki (*J. Soc. Leather Trades' Chem.*, 1918, **2**, 115—121).—Lactide reacts completely in the cold with alkali hydroxide in ten minutes if the alkali is in considerable excess. No heating is therefore necessary in the analysis of lactic acid. Furthermore, the proportion of lactide present in lactic acid depends on the dilution and time of keeping, so that analytical results do not indicate the amount of lactide in a dilute solution used in technical practice, for example, in deliming hides. [See, further, *J. Soc. Chem. Ind.*, 343A.], F. C. T.

An Optical Method for the Estimation of Malic and Tartaric Acids in the same Solutions. J. J. Willaman (*J. Amer. Chem. Soc.*, 1918, **40**, 693—704).—The method depends on the facts that uranyl acetate enhances the rotation of *l*-malic and *d*-tartaric acids, whilst ammonium heptamolybdate reverses the direction of the rotation in the case of *l*-malic acid, giving strongly positive solutions in each case. A chart is made connecting the rotations of solutions, containing up to 1% of the acids, activated by uranyl acetate on the one hand (the curves slope down from left to right) with those of solutions activated by ammonium heptamolybdate on the other (the curves slope up from left to right). The point of intersection of a given pair of curves will give, therefore, the number of grams of tartaric acid on the abscissæ and the proportion of malic acid on the ordinates.

The details of the method are based on Yoder's work on malic acid (A., 1911, ii, 1141) and further developments by Gore and others in America, which have been embodied in an official process (*J. Assoc. Off. Agric. Chemists*, 1916). An amount of the sample is taken which, judged by titration, will contain at least about 0·1 gram of either acid and not more than 0·6 gram of tartaric acid or 0·8 gram of malic acid. This is neutralised with *N*-ammonia solution, mixed with 2 vols. of 95% alcohol, filtered from pectins, and the filtrate slowly mixed with an excess of a 10% solution of barium chloride in 50% alcohol, and then made up to fourteen times the original volume with 95% alcohol. The precipitate is collected by centrifuging, boiled with water, mixed with 10 c.c. of 20% ammonium sulphate solution, the mixture is concentrated to about 80 c.c., cooled, mixed with 6 c.c. of glacial acetic acid, and diluted to 100 c.c. Two 25 c.c. portions of the clear solution, after centrifuging, are taken, mixed with 10 c.c. of an 8% solution of pure uranyl acetate and 10 c.c. of 10%

ammonium heptamolybdate respectively, left for three hours in the dark, and then polarised at about 20° in a 2-dcm. tube. If the molybdate solution becomes green through reduction, a drop of bromine water may be added. J. C. W.

Estimation of Fatty Acids in Butter Fat. E. B. Holland and J. P. Buckley, jun. (*J. Agric. Research*, 1918, **12**, 719—732).—Direct esterification of butter fact (with absolute alcohol containing hydrogen chloride or concentrated sulphuric acid). and subsequent fractional distillation of the resulting esters, affords a trustworthy method for the estimation of certain of the fatty acids. The following quantities of fatty acids were found in butter fat: hexoic acid, 1·36%; octoic acid, 0·975%; decoic acid, 1·831%; lauric acid, 6·895%; myristic acid, 22·618%. Butyric acid (3·153%) and palmitic acid (19·229%) were estimated by difference, stearic acid (11·384%) by crystallisation, and oleic acid (27·374%) from the iodine number of the insoluble fatty acids. [See, further, *J. Soc. Chem. Ind.*, 846A.] W. P. S.

Test for Acetone in Urine. M. Wagenaar (*Pharm. Weekblad*, 1918, **55**, 57—60).—The presence of 0·5 mg. of acetone in 10 c.c. of urine can be detected by mixing the sample with a solution of acetic acid, tartaric acid, and sodium nitroprusside, and covering the liquid with a concentrated solution of ammonia. A coloration like that of permanganate solution is developed at the junction of the liquids. A. J. W.

Detection of Acetone in Urine. P. Bohrisch (*Pharm. Zeit.*, 1918, **63**, 173—174. Compare this vol., ii, 179).—The author finds that Legal's test is more sensitive than Lange's ring test, and mentions that Arends and Urban had shown in 1911 that it was not necessary for the sodium nitroprusside solution used in the tests to be freshly prepared. W. P. S.

Detection of Arbutin. Hans Salomon (*Ber. Deut. pharm. Ges.*, 1918, **28**, 138—139).—The tests commonly applied for arbutin in urine, for example, after the ingestion of bearberry leaf tea, are not specific to this substance. D. F. T.

Direct Estimation of Urea and Ammonia in Placenta Tissue. Frederick S. Hammett (*J. Biol. Chem.*, 1918, **33**, 381—385. Compare Sumner, A., 1916, ii, 655).—The methods of estimation recommended by the author are essentially those devised by Sumner (*loc. cit.*), the chief modification being the addition of potassium carbonate in a solid form to liberate the ammonia prior to aeration. H. W. B.

Estimation of Uric Acid in Urine and Blood. D. G. Cohen Tervaert (*Arch. Néerland. physiol.*, 1918, **2**, 337—345).—In the case of urine, the uric acid is precipitated by ammonium chloride as ammonium urate under conditions described by the author in detail. The precipitate is washed with ammonium chloride by

centrifugalisation, then dissolved in lithium carbonate solution, and the uric acid is estimated colorimetrically by Folin's phosphotungstate reagent in a solution made alkaline by sodium carbonate. In the case of blood, the proteins are separated by coagulation of the solution acidified by acetic acid, the filtrate is concentrated to a small bulk, and the uric acid is precipitated as urate and the amount estimated in a manner similar to that described for the estimation of uric acid in urine. S. B. S.

Estimation of Uric Acid in the Blood by Titration with Permanganate. J. Lucien Morris (*Proc. Amer. Soc. Biol. Chem.*, 1917, xxi; *J. Biol. Chem.*, 1918, **33**. Compare A., 1917, ii, 279).—The uric acid from 20 c.c. of blood is isolated as zinc urate. It is then dissolved in hydrochloric acid and disodium hydrogen phosphate added until all the zinc is reprecipitated. A saturated solution of sodium hydrogen carbonate (25 c.c.), 10% potassium iodide (5 c.c.), and 0·5% starch solution (1 c.c.) are added, and 0·002*N*-permanganate run in from a burette until the blue colour of iodide of starch appears. In the slightly alkaline solution, the oxidation of the potassium iodide, and consequent production of the blue iodide of starch, does not occur until all the uric acid has been oxidised. The results are accurate to within 5%. H. W. B.

Homatropine and the Vitali Test. H. Droop Richmond (*Analyst*, 1918, **43**, 167—168).—Although the Vitali test serves to distinguish homatropine or its hydrobromide from atropine, hyoscyamine, or hyoscine, it is untrustworthy when applied to homatropine sulphate, since the sulphuric acid in this salt causes the production of a violet coloration. In testing the sulphate, the alkaloid should be isolated and the reaction applied to it instead of to the original salt. W. P. S.

Microchemical Tests for Choline. N. Schoorl (*Pharm. Weekblad*, 1918, **55**, 363—369).—A description of the microchemical characteristics of double salts of choline hydrochloride with platinum chloride, gold chloride, mercuric iodide, bismuth iodide, and of the picrate and picrolonate. A. J. W.

Estimation of Creatinine and of Creatine in the Blood. Isidor Greenwald and Grace McGuire (*J. Biol. Chem.*, 1918, **34**, 103—118).—The new method consists in removing the blood-proteins by heat coagulation in dilute acetic acid solution, and then shaking with kaolin, which almost completely removes the creatinine, leaving the creatine unaffected. After filtration and concentration, the creatine is hydrolysed by hydrochloric acid and estimated by Folin's colorimetric method. H. W. B.

Detection and Estimation of Quinine in Blood and Urine. W. Ramsden and I. J. Lipkin (*Ann. Trop. Med. Parasitol*, 1918, **11**, 443—464).—The thalleioquinine reaction is rendered more delicate

(1:40,000 with certainty) by adding to 10 c.c. of the quinine solution, feebly acidified with hydrochloric acid and shaken in a test-tube, one-tenth saturated bromine water drop by drop until the pale yellow colour is no longer instantly discharged (white background). At intervals of five seconds, lots of about 2 c.c. are poured into test-tubes containing one drop of concentrated ammonia. Finally, all ammonia solutions are mixed, and the green pigment is extracted with chloroform. The Herapath test may, with Christensen's reagent and a polarising microscope, be employed for the recognition of 1/500 mg. of quinine. Mayer's reaction (ordinarily 1:500,000) may be rendered twenty times as delicate by extracting the alkaloid with ether free from all traces of aldehyde or acetone, dissolving in saturated ammonium sulphate solution, and adding 1/100 volume of the reagent. Potassium triiodide (limit, 1:1,500,000) is less suitable, on account of the colour; phosphotungstic acid and bismuth potassium iodide are much less delicate.

Blood is boiled with ammonium sulphate, urine is precipitated with lead acetate and ammonium sulphate in the presence of acetic acid; in either case, after addition of ammonia to the filtrate, the quinine is extracted with ether free from ketones, the ether is evaporated, and the residue is dissolved in saturated ammonium sulphate solution (at least 10 c.c. for each mg. of quinine). The turbidity due to Mayer's reagent is compared nephelometrically with that in saturated ammonium sulphate solutions containing known amounts of quinine (gauged test-tubes in box with slit for illumination, dark-room, best dilution of quinine 200—300 c.c. per mg.). Thus 0·02—0·03 mg. of quinine in 5 c.c. of blood may be estimated with an error of less than 5%. Larger quantities of quinine (100 mg.) may be precipitated as periodide, from which the quinine is recovered with sodium hydrogen bisulphite and ether, so that it may be weighed or titrated. Gordin's volumetric method (A., 1900, ii, 114, 777; 1907, ii, 487; 1902, ii, 186) is found to be accurate. G. B.

Detection of Proteins by Bleaching Powder and Hydrochloric Acid. Adolf Jolles (*Deut. med. Woch.*, **43**, 1620—1621; from *Chem. Zentr.*, 1918, i, 303—304).—The test mentioned in the title is not sufficiently sensitive, and can be replaced by the following "three-tube test." The specific reagent contains 10 grams of mercuric chloride, 20 grams of citric acid, and 20 grams of sodium chloride in 500 c.c. of water. To three tubes are added 5 c.c. of filtered urine, to the first tube 1 c.c. of 30% acetic acid + 5 c.c. of the reagent; to the second, 1 c.c. of acetic acid + water, and to the third, water only. All tubes are made to contain the same volume of liquid. By comparing differences in the turbidities after remaining for ten minutes, it is possible to ascertain whether traces of proteins are present. S. B. S.