

## Analytical Chemistry.

---

**Asbestos Filters.** By OTTO LOHSE (*Ber.*, 1899, 32, 2142—2146).—The author describes a modified asbestos filter tube constructed on the same principle as a Gooch filter (*Chem. News*, 1878, 37, 181). It consists of a glass tube, 12 cm. long and 15 mm. in diameter, closed at one end and slightly widened at the open end; the closed end is perforated with a number of minute holes, so arranged that the tube can be heated without risk of breaking. The advantage the tube appears to have over the ordinary Gooch filter is that the precipitate can be somewhat more conveniently heated in a reducing atmosphere.

Convenient forms of aluminium holder, desiccator, and tongs for removing the asbestos from the tube, are also described. J. J. S.

**Weighing Corrosive or Fuming Liquids.** By RUDOLF SCHWARTZ (*Chem. Zeit.*, 1899, 23, 451).—The apparatus is essentially a pipette, ending in two capillary tubes, and weighing about 2—3 grams. One end is placed in the liquid, and suction is applied at the other end with a pump until the bulb is three parts filled; the top tube is then sealed. By removing the pipette and turning it upside down, it becomes possible to seal the other end, and the pipette is then reweighed. The pipette may be readily broken under water to obtain the required solution. L. DE K.

**Apparatus for the Extraction of Liquids with Ether.** By FRITZ BAUM (*Chem. Zeit.*, 1899, 23, 249—250).—Ether is boiled in a flask, and the vapour being condensed, the ether (or similar non-miscible solvent) flows down the condenser tube to the bottom of a cylindrical vessel containing the liquid (or solid matter) to be extracted. While ascending through the liquid, it dissolves any fatty matter, and when a certain quantity has collected on the top it is syphoned back into the boiling flask. An illustration is given.

When the liquid has been thoroughly extracted, it is removed with a pipette, thus leaving plenty of room for the collection of the recovered ether. L. DE K.

**Estimation of some Inorganic and Organic Acids by means of the Volume of Hydrogen evolved on Treatment with a Metal.** By KARL ULSCH (*Chem. Zeit.*, 1899, 23, 624—625).—Platinised iron (iron powder treated with solution of platinum chloride) is quantitatively acted on by fairly concentrated solutions of sulphuric, hydrochloric, and phosphoric acids, and the amount of these acids may be readily ascertained from the volume of hydrogen evolved. Phosphoric acid causes some trouble as the platinised iron gets gradually coated with ferrous phosphate.

The process may also be extended to the estimation of oxalic, acetic, and lactic acids; the author has not, as yet, applied it successfully to tartaric and citric acid (compare this vol., i, 868). L. DE K.

**Iodometric Acidimetry.** By FRANZ FESSEL (*Zeit. anal. Chem.*, 1899, 38, 449—453; from *Inaug. Dissert. Tech. Inst. Univ. Würzburg*, 1898).—The author has submitted to a careful study the conditions under which the reaction  $\text{KIO}_3 + 5\text{KI} + 6\text{HCl} = 6\text{KCl} + 3\text{H}_2\text{O} + 6\text{I}$  can be employed for the estimation of a free acid. It is important that both the iodate and the iodide should be in excess, otherwise too little thiosulphate will be required. A deficiency of the former will be indicated by a liberation of iodine on adding iodate to the colourless liquid at the end of the titration. A deficiency of the latter is shown by the deposition of solid iodine.

Boiled water must be used, since carbonic anhydride liberates iodine from the mixture of iodide and iodate. The iodide solution (10 per cent.) should be preserved from air and light, but even if coloured by free iodine it may be used after decolorising with thiosulphate. Excessive dilution lowers the results, but as this seems to be due to a retardation of the reaction, it is recommended that where great dilution is unavoidable there should be a delay of half an hour before titrating the liberated iodine. The method cannot be employed for organic acids.

J. KJELDAHL points out that, in applying the iodometric method to the estimation of ammonia, accurate results are only obtained by working strictly in the same manner in all cases. Sulphuric acid either alone or after absorption of ammonia does not immediately liberate the theoretical amount of iodine from a mixture of iodide and iodate, but the deficiency in the quantity of thiosulphate required will be the same in the two cases if the volumes of the solutions are equal. The thiosulphate should be standardised by titrating with it the acid to be employed both before and after the absorption of ammonia from a known weight of an ammonium salt. M. J. S.

**Delicate Reaction of Hydrogen Peroxide.** By EDGAR S. BARRALET (*Chem. News*, 1899, 79, 136).—Ferrous ferrocyanide, prepared from a solution of a ferrous salt and potassium ferrocyanide, is quickly turned deep blue by hydrogen peroxide, even when the amount of the latter is as little as 1 part in 165,000 parts of water.

D. A. L.

**Estimation of Hydrochloric Acid in the Stomach Contents.** By GIUSEPPE SIRINGO (*Gazzetta*, 1899, 29, i, 476—479).—The author finds that the use of Günzberg's reagent—an alcoholic solution of phloroglucinol and vanillin (*Abstr.*, 1888, 617)—does not afford an infallible test for the presence of hydrochloric acid in the stomach contents, and suggests instead the employment of salts of nitrohydroxylamine which even in very dilute solutions are decomposed by hydrochloric and the other more energetic acids, nitric oxide being immediately evolved in almost theoretical quantity; the weak organic acids, as also acid phosphates, are without effect. 5 c.c. of the stomach contents are placed in a graduated tube which is filled with mercury and inverted in a mercury trough; a small piece of the nitrohydroxylamine salt, for instance, the sodium salt, is then introduced into the tube and after a few minutes the volume of the evolved gas is read off and corrected in the usual way. T. H. P.

**Estimation of Chlorine, Bromine, and Iodine.** By J. BOUGAULT (*J. Pharm.*, 1899, [vi], 10, 18—20).—Instead of converting the mixed silver haloids into silver chloride, they are reduced by pure metallic zinc and dilute sulphuric acid (1 of acid to 10 of water), and the precipitated metallic silver weighed. The reduction takes place easily and the results obtained are accurate. H. R. LE S.

**Detection of Chlorates, Bromates, and Iodates in the Presence of Each Other.** By DIOSCORIDE VITALI (*Chem. Centr.*, 1899, i, 1083—1084; from *Boll. Chim. Farm.*, 38, 201—209).—When manganous sulphate is treated with sulphuric acid and potassium bromate, a violet-red solution is obtained; this coloration is not due to permanganic acid, but to a manganic salt. Chlorates or iodates do not give this reaction. On boiling, the manganese is completely precipitated as hydrated dioxide.

Salts of hydroxylamine (but not the free base) act on iodates in the cold with liberation of iodine; bromates are only reduced on heating, and chlorates are not affected at all; salts of phenylhydrazine act similarly. Phosphorous and hypophosphorous acids do not act on

bromates or chlorates, but in contact with iodates, iodine is first liberated, and then redissolved as hydriodic acid. The author recommends the following process for the separation of the three compounds. By cautious addition of silver nitrate, the iodates and bromates are alone precipitated; the silver compounds are then decomposed with hydrogen sulphide, and after filtering from the silver sulphide, the excess of the former is expelled by gentle warming, and the filtrate neutralised with soda and concentrated. The iodine is liberated by means of sulphuric acid and potassium nitrite, and removed by shaking with chloroform; the liquid is then tested for bromine with chlorine water.

L. DE K.

[Detection and Estimation of Small Amounts of Iodine in Cuprite and Malachite.] By WILHELM AUTENRIETH (*Chem. Zeit.*, 1899, 23, 626—627).—The finely divided mineral is fused with 3—4 times its weight of pure sodium hydroxide (from metal) and a small quantity of water in a nickel crucible; the mass is then extracted with water, the filtered solution transferred to a glass cylinder and mixed with 10—20 c.c. of chloroform, a few drops of a 1 per cent. solution of pure sodium nitrite solution, and then acidified, while well cooled, with 20 per cent. sulphuric acid; after vigorous shaking, the colour of the chloroform solution is compared with that produced by the addition of a known quantity of potassium iodide solution (0.0001 gram iodine in 1 c.c.) to 20 c.c. chloroform, several c.c. of saturated sodium sulphate solution, dilute sulphuric acid, and a little sodium nitrite solution. The results are correct to within 0.1 milligram of iodine. If siliceous ores are present in the mineral, it is necessary to filter after fusing and rendering acid with sulphuric acid. The fusion must not be carried out at too high a temperature, as in such circumstances the sodium iodide volatilises.

J. J. S.

Detection of Fluorine in Wine. By GIULIO PARIS (*Chem. Zeit.*, 1899, 23, 685—686).—The ash obtained from 50 c.c. of wine is mixed with a little precipitated silicic acid, introduced into a platinum crucible, and 0.5—1 c.c. of sulphuric acid added. The crucible is then covered with an inverted lid containing cold water, the under side being also moistened with a drop of water. The mixture is now heated over a very small flame for 5 minutes, and when cold the drop of liquid adhering to the lid is put on to a slide covered with a thin layer of Canada balsam, and a few particles of sodium chloride are added. After about an hour, the slide is examined under the microscope for the very characteristic crystals of sodium silicofluoride.

L. DE K.

Estimation of Sulphur in Iron, Iron Pyrites, Slags, Coal, Coke, Asphalt, Rubber, and Gas Purification Material. By OTTO HERTING (*Chem. Zeit.*, 1899, 23, 768—769).—Mainly a review of recent improved methods for the estimation of sulphur in these materials.

The use of stannous chloride for reducing ferric sulphate before precipitating with barium chloride is suggested.

L. DE K.

**Estimation of Sulphuric Acid in the Presence of Iron.** By GEORG LUNGE (*Zeit. anorg. Chem.*, 1899, 21, 194—200).—An answer to Küster and Thiel (this vol., ii, 247).—A comparison of the results obtained in the estimation of sulphur in iron pyrites by the author's and Küster and Thiel's methods shows that there is no practical difference in the results obtained, being 49·93 per cent. and 49·86 per cent. respectively, and the time employed on each method is about equal. In the estimation of sulphur in pyrites residues, the results obtained by the different methods are as follows: Watson-Lunge, 1·96 per cent.; Meineke's, by fusion with sodium carbonate and potassium chlorate, 2·00 per cent.; Lunge's ammonia method, 1·94 per cent.; Küster and Thiel's, 1·94 per cent.; Meineke's, by reduction with zinc (this vol., ii, 518), 1·92 per cent. E. C. R.

**Valuation of Persulphates.** By G. H. MONDOLFO (*Chem. Zeit.*, 1899, 23, 699).—2 or 3 grams of the sample are dissolved in 100 c.c. of cold water, and 10 c.c. then heated at 60—80° in a stoppered bottle with half a gram of potassium iodide for 10 minutes. The liberated iodine is titrated with *N*/10 sodium thiosulphate, 1 c.c. of which represents 0·0114 gram of ammonium, or 0·0135 gram of potassium persulphate. L. DE K.

**Gas Washing Apparatus.** By OTTO FOERSTER (*Chem. Zeit.*, 1899, 23, 196—197).—The apparatus is based on the principle of the dephlegmator used in fractional distillation. The steam evolved when boiling a solution of an ammonium salt (such as a reduced nitrate) with excess of alkali, partly condenses, and when a certain quantity of liquid has accumulated, it is syphoned back into the boiling flask. By this means, the liquid cannot become over concentrated whilst the ammonia is still fully expelled. Two drawings are given. L. DE K.

**The Diphenylamine Test for Nitric Acid in Natural Waters.** By RAFFAELE CIMMINO (*Zeit. anal. Chem.*, 1899, 38, 429—431).—The sensitiveness of this test depends greatly on the mode of performing it. That recommended by Emmerich and Trillich has hitherto been the most sensitive, being capable of detecting nitric acid at a dilution of 1:100,000, but by the addition of a little hydrochloric acid the sensitiveness is increased tenfold. The reagent employed therefore is a solution of diphenylamine and sulphuric acid in 5 per cent. hydrochloric acid; three or four drops of this reagent are added to 1 c.c. of the liquid to be tested, then 2 c.c. of concentrated sulphuric acid, and the whole shaken. In presence of nitric acid, the mixture acquires a blue colour. No other constituent of natural water gives a similar reaction. M. J. S.

**Estimation of Ammonia and Nitric and Nitrous Acids in Natural Waters.** By LUDWIG W. WINKLER (*Chem. Zeit.*, 1899, 23, 454—455; 541).—Preferred ammonia may be estimated without previous distillation by removing the calcium and magnesium salts by means of sodium hydroxide and carbonate; the clarified solution may then be at once Nesslerised. If too little standard ammonia has been added to the comparison liquid, it does not do to add a little more, but a fresh solution must be prepared. To obviate this, and also to save the

removal of the calcium and magnesium salts, the author recommends the addition of a soluble tartrate (Rochelle salt) both to the water and to the comparison liquid.\*

The ordinary Nessler solution is not well adapted to this process, but a delicate solution may be prepared by dissolving 10 grams of mercuric iodide and 5 grams of potassium iodide in a little water, and then adding a solution of 20 grams of sodium hydroxide, and finally diluting to 100 c.c.

For the estimation of nitric acid, a slight modification of Lunge and Lwoff's process is recommended (Abstr., 1894, ii, 398). Nitrous acid is estimated by the amount of iodine liberated by the addition of hydrochloric acid and potassium iodide; the liquid is then titrated with standard solution of sodium thiosulphate, using starch-water as indicator. To get trustworthy results, the air should be excluded, which is best effected by operating in a sufficiently small flask with narrow neck, and displacing the air by carbonic anhydride generated by dropping into the acid liquid a sufficiency of solid potassium hydrogen carbonate.

L. DE K.

**A Source of Error in the Estimation of Nitric Nitrogen by Ulsch's Method.** By L. BRANDT (*Chem. Zeit.*, 1899, 23, 22).—Ulsch (Abstr., 1891, ii, 617) reduces nitrates to ammonia by the action of sulphuric acid and reduced iron. The author shows the necessity of always making a blank experiment to ascertain whether the reagents are free from nitrogen. In one case, it was found that the iron used in the experiment contained a considerable amount of nitrogen in some form capable of conversion into ammonia when the metal was dissolved in dilute sulphuric acid.

L. DE K.

**Gasometric Estimation of Nitric Oxide.** By GEORG VON KNORRE and KURT ARNDT (*Ber.*, 1899, 32, 2136—2141).—As the usual methods for the estimation of nitric oxide, namely, (1) absorption by solution of a ferrous salt; (2) titration with potassium permanganate; (3) conversion into nitric peroxide by the aid of oxygen, are considered unsatisfactory, the authors have elaborated another method which consists in passing the gas, together with an excess of hydrogen, through a Drehschmidt's platinum capillary tube heated to bright redness and measuring the contraction. According to the equation  $2\text{NO} (2 \text{ vols.}) + 2\text{H}_2 (2 \text{ vols.}) = \text{N}_2 (1 \text{ vol.}) + 2\text{H}_2\text{O}$ , the amount of nitric oxide is equal to  $2/3$  of the contraction. Very good results have been obtained by this method, but it is absolutely necessary that the mixed gases should pass very slowly through the capillary, as otherwise a certain amount of ammonia is formed. The method also admits of the estimation of nitrous oxide in the presence of nitric oxide. From the equation  $\text{N}_2\text{O} (1 \text{ vol.}) + \text{H}_2 (1 \text{ vol.}) = \text{N}_2 (1 \text{ vol.}) + \text{H}_2\text{O}$  and the one given above, it follows that if  $x = \text{vol. of nitric oxide}$  and  $y = \text{vol. of nitrous oxide}$ , then  $x + y = V$  (volume of mixture),  $1.5x + y = C$  (contraction on leading through the capillary, and  $x = 2(C - V)$ .

\* NOTE BY ABTRACTOR.—Romijn (*Ber. Deut. Pharm. Ges.*, 1898, 8, 9) has already advocated the use of Rochelle salt.

If nitrogen is present in addition to nitrous and nitric oxides, then the amount of hydrogen used up must be determined, and the volumes of the three constituents can be calculated. J. J. S.

**Detection and Estimation of Free Phosphorus in Oils and Fats.** By ÉMILE LOUISE (*Compt. rend.*, 1899, 129, 394—395).—When a solution of silver nitrate is added to a solution of phosphorus in oil, the silver solution is reduced, and a black precipitate is produced as long as any free phosphorus is present. By previously mixing the oil with 20 times its volume of acetone, the production of any turbidity is prevented when the aqueous silver nitrate solution is added. A 10 per cent. and a 1 per cent. solution of silver nitrate are prepared, and these titrated by taking 5 grams of oil, containing a known quantity of phosphorus, and diluting to 100 c.c. with acetone. To 10 c.c. of this solution, the 10 per cent. silver nitrate solution is added drop by drop until the filtered liquid is no longer darkened in colour on the addition of another drop of silver nitrate. Suppose two drops of the silver nitrate solution were insufficient, but that after the addition of three drops the filtered liquid was not darkened on the addition of another drop, then evidently the correct amount of silver nitrate solution was between two and three drops. To determine the exact amount, another 10 c.c. of the solution are taken, two drops of the 10 per cent. solution added, the liquid filtered, and then the 1 per cent. solution added drop by drop until the filtered liquid is not darkened in colour by the further addition of silver nitrate. In this way, the strengths of the silver solutions in terms of phosphorus are ascertained. The determination of an unknown amount of phosphorus in an oil is carried out in exactly the same way. To obtain drops of exactly the same size, Duclaux's apparatus was used. H. R. LE S.

**Limits of Error in the Estimation of Phosphoric Acid Soluble in Citric Acid.** By MAX MAERCKER (*Landw. Versuchs-Stat.*, 1899, 52, 119—136).—A Report to the Verband der Versuchs-Stationen on the results of two series of determinations of phosphoric acid soluble in citric acid in samples of basic slag, the first comprising 468 analyses of 116 samples, the second 504 analyses of 252 examples.

In the second series of determinations, 92.1 per cent. showed a difference of, at most, 0.5 per cent. between the highest and the lowest result, or an error of  $\pm 0.25$  per cent. N. H. J. M.

**Estimation of Total Phosphoric Acid in Basic Slags.** By C. ASCHMAN (*Chem. Zeit.*, 1899, 23, 435—436).—The following solutions are required: nitrosulphuric acid, made by taking 420 grams of nitric acid of sp. gr. 1.2, 50 grams of strong sulphuric acid, and diluting with water to 10 litres. Solution of citric acid, made by dissolving 500 grams of the acid to 1 litre. Wagner's magnesia mixture, made by dissolving 110 grams of crystallised magnesium chloride and 140 grams of ammonium chloride in 700 c.c. of ammonia (of 8 per cent. strength) and 1300 c.c. of water. Molybdate solution, made by dissolving 150 grams of ammonium molybdate and 400 grams of ammonium nitrate to 1 litre, and pouring the solution into a litre of nitric acid of sp. gr. 1.19.



5 grams of the sample are put into a half-litre shaking bottle, which is then filled up to the mark with nitrosulphuric acid. After rotating for half an hour in a Wagner apparatus, the liquid is left overnight. 50 c.c. of the filtrate are then mixed with 10 c.c. of citric acid solution and neutralised with ammonia; 50 c.c. more water are added, and the whole left to cool for 20 minutes. To the liquid, which must be perfectly clear, are now added 20 c.c. of magnesia mixture; after a few hours standing with frequent stirring, the magnesium phosphate precipitate is collected as usual.

If the neutralised liquid deposits silicic acid, another portion of the acid solution must be precipitated with molybdate in the usual manner.

L. DE K.

**Basic Slag.** By ARMAND DEZSÖ HERZFELDER (*Landw. Versuchs-Stat.*, 1899, 52, 291—314. Compare Abstr., 1897, ii, 344).—The assimilable phosphoric acid in basic slag is in the form of tricalcium phosphate. The slag contains also calcium phosphates of higher basicity, iron phosphates, iron silicophosphates, calcium silicophosphates, calcium oxide and carbonate, silicon chiefly in combination with calcium, iron oxide, sulphide, and carbonate, &c.

The difficulties in the way of determining the tricalcium phosphate by extraction with citric acid are the presence of lime, which neutralises some of the acid, and the dissolution of a portion of the silicate, most of which is precipitated with the molybdate, redissolves in ammonia, and is again precipitated by the magnesia mixture. In this way, 0.01 and 0.02 gram of  $\text{SiO}_2$  will cause an error of 0.6 and 1.27 per cent. in the phosphoric acid. This source of error and the error (in the other direction) caused by the presence of lime, can be avoided by altering the process, but the presence of iron in the slag involves another difficulty, since iron compounds diminish the solubility of tricalcium phosphate in citric acid.

It is concluded that the error of the Wagner method is as much as 2—3 per cent., and varies according to the nature of the slag. With slags which do not contain much total phosphoric acid and silica, relatively little soluble phosphoric acid, but much lime and iron, the results will be too low; whilst with slags which contain much phosphoric acid (both total and soluble) and silica, but only a little lime and iron, the results for soluble phosphoric acid may be even higher than the total phosphoric acid.

N. H. J. M.

**Estimation of Boric Anhydride in Boracite.** By RUDOLF SCHWARTZ (*Chem. Zeit.*, 1899, 497).—Gilbert's process of carefully estimating the other constituents of the mineral and thus obtaining the boric anhydride by difference is difficult of application. Höning and Spitz's volumetric process yields very good results. In order to prepare the solution for titration, 1—2 grams of the finely powdered sample are digested for several hours with 100 c.c. of water, and 5—10 c.c. of hydrochloric acid; if a reflux apparatus is employed, half-an-hour's heating on the water-bath will be sufficient. The filtrate is carefully neutralised with  $N/5$  soda, using methyl-orange as indicator. The solution may also be prepared by attacking the mineral suspended in water with carbonic anhydride for about half-an-hour;



after remaining for a few hours in a closed flask, the liquid is filtered, evaporated to about 20 c.c., and carefully neutralised with dilute hydrochloric acid, using methyl-orange as indicator; the carbonic anhydride is then expelled by boiling for a short time. The liquid obtained in either way is then titrated with *N*/5 soda in the presence of glycerol, using phenolphthalein as indicator (*Abstr.*, 1897, ii, 159).

L. DE K.

**Estimation of Carbon in Iron Products.** By ADOLPHE CARNOT and E. GOUTAL (*Chem. Centr.*, 1899, i, 903; from *Anal. Chim. Appl.*, 4, 73—76).—Blair separates carbon from iron by treating the sample with a solution of potassium cuprous chloride containing 15 c.c. of hydrochloric acid in 200 c.c. The authors having tested the method, state that the temperature should not exceed 70°, otherwise there will be a loss of carbon. They, however, prefer their own process of treating the sample at 95° with the slightly acidified copper solution for about half-an-hour in a current of carbonic anhydride. Ferromanganese with more than 75 per cent. of manganese should be treated in the cold. The drying of the carbonaceous residue is often a source of decided loss.

L. DE K.

**A Rapid Eggertz Process.** By JOSEF SPÜLLER (*Chem. Zeit.*, 1899, 23, 476—478).—Some practical hints as to the quickest way of applying the well-known Eggertz test for estimating carbon in iron. The details do not admit of condensation.

L. DE K.

**Two Simple Methods for estimating Carbonic Anhydride in Quick Lime, Limestone, Animal Charcoal, Marls, &c., and Soils.** By V. SCHENKE (*Chem. Zeit.*, 1899, 23, 612—614).—The first method and apparatus present no novel features.

A new process is proposed for the estimation of carbonic anhydride combined with lime and magnesia in soils. The soil is ignited, finally over the blowpipe and the ash weighed; it is then moistened with ammonium carbonate and again dried over a low rose-burner; the increase in weight represents the carbonic anhydride in combination with the lime and magnesia.

L. DE K.

**Estimation of Potash.** By E. WIGHTMAN BELL (*Chem. News*, 1899, 79, 135—136).—In the case of manures, 5 or 10 grams are boiled with about 150 c.c. of water, with or without addition of hydrochloric acid; ammonia is then added to slight alkalinity, and is followed by barium carbonate to the extent of about double the weight of the manure taken, the boiling being continued for half-an-hour. The mixture is filtered, the precipitate is washed, and the combined filtrate and washings made up to 500 c.c., of which 50 to 100 c.c. are evaporated to dryness, along with a small quantity of ammonium oxalate, gently ignited, treated with hot water, and filtered. The filtrate is evaporated with hydrochloric acid and platinic chloride, and the estimation completed in the usual way. With soils, either the solution in hydrochloric acid is boiled, treated with ammonia, &c., as above, or the soil is moistened with strong sulphuric acid, gently ignited, boiled with water, treated with barium carbonate, boiled again, filtered, and an aliquot portion of the filtrate evaporated with

hydrochloric acid and platinic chloride, &c. The sulphuric acid treatment serves also for the estimation of potash in vegetable substances.

D. A. L.

**Estimation of Alkalis in Natural Waters.** By E. BOHLIG (*Zeit. anal. Chem.*, 1899, 38, 431—432).—Half a litre of the water is rapidly boiled down to 50 c.c. in a large porcelain basin, then strongly acidified with sulphuric acid, and evaporated without ebullition until fumes of sulphuric acid are given off. The residue is then washed into a 200 c.c. flask with 150 c.c. of water, some barium carbonate added, and carbonic anhydride passed through the mixture as long as any is absorbed; the insoluble matter is removed by filtration and washing, the filtrate evaporated to dryness, and the dry residue digested with 50 c.c. of a mixture of equal parts of alcohol and water. The alkali carbonates dissolve in this menstruum, whilst all other carbonates remain undissolved. After filtration, the total alkalis present can at once be estimated by titration with *N*/10 hydrochloric acid. The potassium is then precipitated as platinochloride, and the sodium calculated from the difference.

M. J. S.

**Estimation of the Alkalis in Urine.** By RICHARD PRIBRAM and GEORG GREGOR (*Zeit. anal. Chem.*, 1899, 38, 401—411).—With the object of establishing a method which, without loss of accuracy, should be sufficiently rapid and convenient for clinical purposes, the authors have examined the methods of Neubauer, Lehmann, Bunge, Salkowski-Munk and Heintz. Neubauer's method is far from satisfactory, Lehmann's process (*Abstr.*, 1885, 609) is theoretically free from sources of error, and as it gives higher values for the potassium found than any of the other processes, it is presumed to be the most accurate. It may be improved by substituting concentrated sulphuric acid for the ammonium sulphate added before incineration of the urine residue; a white ash is then much more rapidly obtained, and there is less danger of loss of alkali. Another improvement introduced by Kretzschmar consists in evaporating to dryness, and drying at 110° after the addition of barium chloride, ammonia, and ammonium carbonate. On treating the residue with hot water containing a little ammonia, the alkali chlorides can be washed out with great facility. Both this method and Bunge's are, however, tedious. That of Salkowski-Munk is equally so, and of lower accuracy, whilst Heintz's process is confined to the estimation of the potassium, and gives seriously low results. The authors therefore propose a new process which gives results identical with those of Lehmann, but is more rapid and requires less caution on the part of the operator. 50 c.c. of urine are mixed with 10 c.c. of dilute (1:10) sulphuric acid and a sufficient quantity of a 10 per cent. solution of barium permanganate (10—20 c.c.), and the mixture is boiled; after boiling for 10—15 minutes, a red coloration must still remain; this is removed by a few drops of oxalic acid. Barium chloride is then added in small excess followed by ammonia and ammonium carbonate; the precipitate is removed by filtration, and the filtrate evaporated in a platinum basin; the alkali chlorides thus

obtained are usually quite free from carbonaceous matter. Should magnesium be present, as may occur in all processes in which the alkalis are weighed as chlorides, it may be eliminated by keeping the chlorides at a dull red heat in a covered platinum crucible for an hour or longer, by which means magnesium chloride is converted into oxide. It is then desirable to estimate the total chlorine by silver, and the potassium by platinum, and calculate the sodium indirectly.

M. J. S.

**Separation of Zinc from Nickel.** By E. DÖHLER (*Chem. Zeit.*, 1899, 23, 399—400).—Zinc may be separated from nickel by precipitation with hydrogen sulphide in the presence of free acetic acid when mineral acids are absent. Good results may be obtained by using a very large excess of acetic acid, but even then the precipitated zinc sulphide may be of a dark colour and contain an admixture of nickel sulphide. It may be freed from nickel by dissolving it in dilute hydrochloric acid, which leaves most of the nickel sulphide undissolved, and after neutralising the filtrate with ammonia and adding sodium acetate and free acetic acid, again precipitating by means of hydrogen sulphide, pure white zinc sulphide being then obtained. Instead of using sodium acetate and acetic acid, the neutralised solution may be advantageously mixed with sodium formate and formic acid; the precipitate produced by hydrogen sulphide is then free from nickel, and needs no purification.

L. DE K.

**Electrolytic Estimations and Separations.** By LILY G. KOLLOCK (*J. Amer. Chem. Soc.*, 1899, 21, 911—928).—Some of the older methods for electrolytic determinations and separations in potassium cyanide solutions have been repeated. The exact conditions as to current density, ampère and voltage and other factors have been worked out for the estimation of cadmium, silver, and mercury, and for the separation of silver, mercury, or gold from platinum, copper, zinc, nickel, or cobalt; of silver or mercury from cadmium or iron, and of gold from palladium.

Mercury may be deposited from alkali sulphide solution under the following conditions: 25 c.c. of sodium sulphide are added to a solution of mercuric chloride containing 0.1403 gram of mercury; this solution, diluted to 125 c.c., is electrolysed at 70° by a current of  $N.D._{100} = 0.11$  ampère, and voltage = 2.5, and, at the end of 5 hours, the mercury is completely deposited. Copper can be deposited from nitric acid solution by using a current of  $N.D._{100} = 0.09$  and voltage = 1.9 in 5 hours at 65°; mercury can also be deposited in a similar manner.

Cadmium is deposited from sulphuric acid solution in 5 hours at 65° when a current of  $N.D._{100} = 0.078$  and voltage = 2.61 is employed. Mercury may also be estimated in a somewhat similar manner.

The conditions necessary for the accurate determination of bismuth in citric acid, ammonium citrate, or potassium citrate solution have also been determined. In the case of ammonium citrate, the solution is made ammoniacal and is electrolysed at 65°, first with a current of 0.02 ampère and 1.8 volts, the current being finally increased to

0.09 ampère and 2.3 volts. A certain amount of bismuth peroxide is always deposited on the anode and must be weighed. A current of much the same density was employed in both citric acid and potassium acetate solutions, and in the latter case the solution was made strongly alkaline by the addition of potassium hydroxide.

Iron may be deposited in the course of 7 hours from a solution containing sodium citrate and free citric acid when a current of 0.58 ampère and 5.7 volts is employed, or in 4.5 hours at 50° with a current of 0.4—1 ampère and 7—11 volts. J. J. S.

**Precipitation of Cadmium by Hydrogen Sulphide.** By ROBERT MELDRUM (*Chem. News*, 1899, 79, 170—171).—The author attributes failure to detect small quantities of cadmium, by means of hydrogen sulphide in the way usually employed, to the too liberal use of hydrochloric acid, which dissolves the sulphide. He has further investigated the precipitation by means of hydrogen sulphide of neutral solutions of cadmium sulphate varying in strength from 0.5 to 0.00125 per cent. Solutions from 0.5 to 0.25, in the cold give a curdy precipitate which settles quickly and a dense, opaque, yellow solution; when heated to boiling, the precipitate becomes orange-yellow, settles rapidly, and turns paler on cooling, but does not regain its former colour; the solution is clear with a slight yellow tinge. Agitation while cooling, and when cold, makes the precipitate settle less rapidly and renders the solution opaque. Solutions containing from 0.2 to 0.02, yield less precipitate on boiling, give little or no precipitate in an hour in the cold, and the solution remains opalescent. Solutions of greater dilution (except the 0.00125, which shows no colour in a thickness of 2 inches) yield yellow solutions, but no immediate precipitate. The filtrates from the solutions of greater strength than 0.0175 per cent. are almost free from colour in a thickness of 2 inches, but from the solutions of still greater dilution the filtrates are always coloured. By repeated filtrations, this colour may be removed from the 0.0175 to the 0.015 per cent. solutions but not from the weaker filtrates; these, however, deposit a small precipitate in 24 hours, but still retain a yellow colour.

A colorimetric method of estimating cadmium is suggested for solutions not exceeding in strength 0.02 per cent. of the sulphate.

D. A. L.

**Solubility of Lead Sulphate in Aqueous Ammonium Acetate.** By J. C. LONG (*Amer. Chem. J.*, 1899, 22, 217—218).—Lead sulphate is most soluble in a solution of ammonium acetate containing 33 grams per 100 c.c. To dissolve lead sulphate in analysis, it is best to add, for each gram, 10 c.c. of the ammonium acetate solution, heat to boiling, and filter promptly; to recover the lead sulphate, 0.5 gram of sulphuric acid should be added, this facilitating the evaporation and expulsion of the ammonium acetate. W. A. D.

**Separation of Copper and Zinc.** By W. DEDERICHS (*Chem. Centr.*, 1899, i, 950; from *Pharm. Zeit.*, 44, 198).—Zinc is estimated in brass by dissolving in the smallest possible amount of nitric acid,

diluting with water containing 2—3 per cent. of hydrochloric acid, and removing the copper by means of hydrogen sulphide; the filtrate is then boiled and the zinc precipitated with sodium carbonate.

To estimate copper, the nitric acid solution of the brass is digested with sodium hydrogen sulphite, and an excess of a standard solution of ammonium thiocyanate is added to precipitate the copper; the excess of the reagent is then titrated with silver nitrate in an aliquot part of the filtrate.

Traces of lead are estimated in the usual way as sulphate.

L. DE K.

**Separation of Copper from Cadmium as Oxalate.** By G. BORNEMANN (*Chem. Zeit.*, 1899, 23, 565—566).—The solution of copper and cadmium, which must not be too concentrated, is mixed with a moderate quantity of nitric acid, heated to boiling, and then mixed with a slight excess of oxalic acid previously dissolved in boiling water; the liquid is then placed in a water-bath until it has become quite clear. The precipitated copper oxalate is first washed by decantation with boiling water containing a little nitric and oxalic acids, and then collected on a suitable filter and washed with boiling water containing at first a few drops of oxalic acid; in this way, the precipitate is obtained free from cadmium. The oxalate is finally converted into sulphide by ignition with sulphur in a current of hydrogen.

L. DE K.

**Modification of Eschka's Mercury Assay.** By K. E. CHISM (*Chem. Centr.*, 1899, i, 996; from *Trans. Amer. Inst. Min. Eng.*, Oct., 1898).—Eschka's method of mercury assay by collecting the mercury vapour on a weighed golden lid, gives accurate results, but is somewhat expensive, as the gold soon wears out. The author now substitutes a lid made of silver of about 0.02 mm. thickness; this may be used three times, and costs but little.

0.5—1 gram of the ore is mixed in a porcelain crucible with 5 grams of clean iron filings, another gram of the iron being sprinkled on the top. The crucible is placed on a perforated plate and the lid is put on, and cooled by placing on it a silver or copper basin filled with cold water. The bottom of the crucible is now heated for 15 minutes with a spirit lamp, and when cold the lid is taken off and re-weighed. On applying heat, the mercury volatilises, and the lid may be used again.

L. DE K.

**Volumetric Estimation of Manganese in Manganates by means of an Alkaline Solution of Arsenious Acid.** By C. REICHARD (*Chem. Zeit.*, 1899, 23, 801—802).—The process is based on the fact that on adding an alkaline solution of arsenious acid of known strength to a solution of a manganate, the latter is reduced to manganous hydroxide. The unoxidised arsenious acid is then estimated by standard iodine; or the manganous hydroxide may be at once redissolved by adding sulphuric acid and the arsenious acid then estimated in acid solution with standard permanganate.

Five molecules of arsenious acid used for reduction are equivalent

to four atoms of metallic manganese. The test experiments are satisfactory. L. DE K.

**The Presence of Carbon in Electrolytically Deposited Iron.** By SAMUEL AVERY and BENTON DALES (*Ber.*, 1899, 32, 2233—2238. Compare this vol., ii, 251, and Verwer and Groll, *ibid.*, 387)—The authors maintain the accuracy of their former conclusion that the ordinary methods for the electrolytic estimation of iron do not yield satisfactory results, because a small amount of iron remains in the solution, and the deposited metal contains carbon.

In the presence of oxalic acid, the reaction with potassium thiocyanate is not delicate, and cannot therefore be depended on for indicating the completion of the deposition. Oxalic acid is converted by electrolytic reduction into glycollic acid, and the presence of this in the solution causes the deposited metal to contain carbon. A. H.

**Potassium Ferrocyanide as a Source of Iron in the Volumetric Analysis of Iron.** By K. SCHRÖDER (*Chem. Zeit.*, 1899, 23, 533—534, 540—541, 557—559).—The author states that potassium ferrocyanide may be obtained almost absolutely pure and be advantageously utilised as a source of pure iron when standardising solutions for the technical estimation of iron.

Four or five grams of the pure salt are heated in a Kjeldahl flask with 20—25 c.c. of strong sulphuric acid; heat is applied, moderately at first, and then gradually increased until boiling sets in, which is then kept up for half-an-hour. When cold, 25 c.c. of water and 10—15 c.c. of hydrochloric acid of sp. gr. 1.19 are added and the mixture warmed until completely dissolved. This solution may now be reduced with zinc, or by means of stannous chloride followed by mercuric chloride, and will then be fit for standardising purposes. L. DE K.

**Estimation of Iron in Organic Matter.** By FRANZ RÖHMANN and FRANZ STEINITZ (*Zeit. anal. Chem.*, 1899, 38, 433—435).—Instead of destroying the organic matter by incineration, it is far preferable to employ Neumann's method of heating with concentrated sulphuric acid and ammonium nitrate. The operation is performed in a Jena glass flask over the naked flame, the ammonium nitrate (1 gram for each c.c. of acid) being added in several portions, and the heating continued until a clear, pale yellow liquid is obtained. This is now made alkaline with ammonia and the iron precipitated as sulphide by adding a few drops of colourless ammonium sulphide with the usual precautions. The iron sulphide is collected on an ash-free filter, from which it is re-dissolved by sulphuric acid, the solution being run back into the precipitation flask, where it dissolves any iron sulphide adhering to the sides. Here it is concentrated by boiling and transferred to a platinum basin, in which, meanwhile, the filter has been incinerated and its ash fused with potassium hydrogen sulphate. By introducing a rod of zinc for about half-an-hour, the ferric salt is completely reduced, and is then titrated with permanganate.

In dealing with faeces, the large amount of calcium sulphate formed is an inconvenience. It is best not to attempt to remove it from the



original acid solution, but to precipitate the iron sulphide in the turbid liquid. Urine (300—400 c.c.) should be mixed with 25—30 c.c. of fuming nitric acid and boiled down to a small volume before adding sulphuric acid (20—30 c.c.) and ammonium nitrate. M. J. S.

**Iron Separations with Alkali Salts. I.** By HARRY BREARLEY (*Chem. News*, 1899, 79, 193—194).—It is pointed out that for the separation of iron from manganese, zinc, nickel, cobalt, and sometimes copper, alkali acetates, phosphates, sulphates, chromates, tungstates, and molybdates are amongst the available reagents. *Separation with acetates* has already been investigated (Abstr., 1896, ii, 676; 1897, ii, 388; 1898, ii, 96 and 143); it is, however, now shown that it is best not to add the acetate to the cold solution, and then to heat to boiling, but that the acetate should be added to the hot solution. Furthermore, it is shown that with a constant quantity of nickel or other metal and varying quantities of iron, the exactness does not necessarily increase with the decrease in the proportion of iron; but with fixed proportion of iron and a varying proportion of nickel, probably the separation is better for small than for large amounts of nickel.

D. A. L.

**Detection of Cobalt in the Presence of Ferric Compounds.** By HENDRIK WEFERS BETINK (*Chem. Centr.*, 1899, i, 904; from *Ned. Tydschr. Pharm.*, 11, 64—66).—When a solution of cobalt is mixed with potassium thiocyanate and a mixture of ether and alcohol poured on to the surface, a blue coloration is observed in the upper layer. In the presence of ferric salts, however, the red colour given by ferric thiocyanate interferes with the test, but in this case the iron compound may be removed by first extracting the mixture with pure ether, which dissolves the ferric thiocyanate but not the cobalt compound; the ether-alcohol mixture is then added to the residual liquid.

The author has improved the method by operating as follows: Potassium thiocyanate is added and the red coloration is destroyed by adding powdered sodium thiosulphate; the liquid is then covered with a layer of ether-alcohol. 0.001 gram of cobalt nitrate may thus be detected in the presence of 1 gram of ferric chloride.

L. DE K.

**Reduction of Chromic Acid by Acetic Acid and its Effect in Anthracene Testing.** By HENRY BASSETT (*Chem. News*, 1899, 79, 157).—When the mixture of acetic acid with chromic acid used in anthracene testing is stored, the latter suffers some reduction, a reaction that is still more marked when undiluted acetic acid is boiled with chromic acid; but it also takes place with aqueous solutions of acetic acid when boiled, not, however, when the volumes of the acetic acid and water are equal. The reduction is more marked with acid crystallising at 13.5° than with the purer acid crystallising at 17°.

In anthracene testing, the less pure acetic acid and mixed solutions of acetic and chromic acids that have been kept too long (more than 15 days) will give results which are too high.

D. A. L.

**Testing Oil of Turpentine for Mineral Oils.** By C. SCHREIBER and F. ZETZSCHE (*Chem. Zeit.*, 1899, 23, 686—687).—The following



method is suggested as a good preliminary test: 1 c.c. of the sample of turpentine is diluted to 50 c.c. with alcohol of 90—95 per cent. 20 c.c. of this solution are put into a 75 c.c. glass cylinder and 20 c.c. of a solution containing exactly 15 grams of potassium bromate and 50 grams of potassium bromide in a litre are added, followed by 20 c.c. of dilute sulphuric acid (1:3). The stopper is inserted and the mixture well shaken for half a minute. If the liquid should not be quite decolorised, the sample is likely to contain mineral oil and should be submitted to further analysis, but otherwise it may pass as commercially pure.

L. DE K.

**Estimation of Dry Substance in Glycerol.** By G. BENZ (*Zeit. anal. Chem.*, 1899, 38, 436—438).—It is commonly accepted that glycerol cannot be estimated by drying at 100°, because the glycerol begins to volatilise as soon as all the water has evaporated. The author shows, however, that this occurs only in shallow, uncovered vessels. By employing a wide-necked flask covered with a loose glass cap resting on the neck, glycerol can be dried to a constant weight either at 100° or 110°, and that this takes place without loss is shown by dissolving glycerol, so dried, in water and re-evaporating the solution in the same flask, when exactly the same weight of dry substance will be obtained.

M. J. S.

**Estimation of Glycerol in Fermented Liquids.** By J. B. VINCENT LABORDE (*Chem. Centr.*, 1899, i, 905—906, 1086; from *Ann. chim. anal. appl.*, 4, 76—80; 110—114. Compare *Abstr.*, 1896, ii, 77).—The process is based on the fact, noticed by the author, that at 150—200°, glycerol is quantitatively decomposed by sulphuric acid with liberation of the whole of the carbon; 1 part of carbon represents 2.56 parts of glycerol. A quantity of liquid containing from 0.1—1.0 gram of glycerol is mixed with 10 drops of sulphuric acid and boiled down in a 250 c.c. flask with small, flat bottom until about 2 c.c. are left. 6 c.c. of sulphuric acid are then added and the flask closed with a rubber cork through which passes a glass tube drawn out to a fine point. The mixture is now heated on a sand-bath to 150°, and when the action takes place the temperature rises to 200°. After a few minutes, 5 c.c. of dilute hydrochloric acid (1:1) are added, and the whole is heated on the sand-bath until white fumes begin to form; the carbon is now thoroughly washed by decantation with boiling water, and then transferred to a weighed platinum dish; a little ammonia is added, and the whole dried first at 110° and then over a small rose-burner until no more ammoniacal odour is noticed. When cold, the carbon is weighed and allowance made for any ash.

To estimate glycerol in saccharine liquids, any sugar must first be removed. If the amount does not exceed 0.5 gram per litre, 50 c.c. are mixed with 100 grams of small shot, evaporated down to 2—3 c.c. and gradually mixed with 1—2 grams of calcium hydroxide; 75 c.c. of ether-alcohol mixture (2:1) are then added to dissolve the glycerol; the liquid is filtered, and the residue is washed with more of the ether mixture until the filtrate reaches 200 c.c. After adding 10 drops of sulphuric acid, the ether-alcohol is distilled off and the residue treated as already described.

If the amount of sugar amounts to 5—20 grams per litre, the sufficiently concentrated solution is mixed with 2 grams of calcium hydroxide which has been moistened with alcohol; if the mass does not readily set, a little more dry lime should be added; the mass must then be extracted as before with ether-alcohol mixture. If the liquid contains more than 20 grams of sugar per litre, a volume of it which will contain about 5—6 grams of sugar is mixed with 2 grams of calcium hydroxide suspended in 10—20 c.c. of proof spirit and then boiled with 100—200 c.c. of 80 per cent. alcohol. When cold, an aliquot portion of the filtrate is acidified with tartaric acid, and the alcohol having been expelled, the residue is treated as before with the ether-alcohol mixture.

The test analyses are very satisfactory.

L. DE K.

**Behaviour of Iron Salts with Pyrogallol.** By A. HIRSCH (*Chem. Centr.*, 1899, i, 927; from *Pharm. Zeit.*, 44, 205).—When 2 drops of a solution of ferric chloride containing 10 grams of liq. ferri. sesquichlor., in 90 of water are added to a few c.c. of a 1 per cent. solution of pyrogallol, an amethyst-blue coloration is first formed, which rapidly changes to reddish-brown, but if 1 c.c. of a 0.1 per cent. solution of sodium hydroxide or 0.5 c.c. of a 0.5 per cent. solution of sodium carbonate be then added, the solution becomes deep blue. The brown coloration is restored, however, by adding very small quantities of mineral acids. Zinc oxide or barium carbonate acts like the alkalis, hence the brown coloration must be due to the mineral acid contained in the ferric salt. Organic iron salts give blue colorations, which become brown only on addition of mineral acids. A solution of pyrogallol, rendered reddish-brown by the addition of a very dilute solution of ferric chloride, may be used to test for faint alkalinity, as, for instance, in the case of dilute solutions of alkaloids, which give the blue coloration. When pure ferrous salts are mixed with solutions of pyrogallol, a white turbidity is formed, and if a very dilute solution of sodium hydroxide is then added, the solution becomes blue, probably owing to partial oxidation of the ferrous salt.

E. W. W.

**Estimation of Mannose in Mixtures of Sugars.** By ÉMILE BOURQUELOT and HENRI HÉRISSEY (*Compt. rend.*, 1899, 129, 339—341).—The solution of mannose is mixed with a solution of equal volumes of phenylhydrazine and glacial acetic acid, allowed to remain for 8 hours at the ordinary temperature, and the precipitate washed successively with ice-cold water, alcohol, and ether, and dried in a vacuum. The results are not affected by the presence of galactose, arabinose, maltose, or dextrin, and are accurate if the solution contains 3 to 6 per cent. of mannose, and the operations are conducted at as low a temperature as possible. A correction should be made by adding 40 milligrams to the weight of the precipitated hydrazone for every 100 c.c. of solution.

C. H. B.

**Influence of Sucrose on the Estimation of Pentosans by the Phloroglucinol Method, with special reference to Products from Sugar Refineries.** By KARL ANDRLIK (*Chem. Centr.*, 1899, i, 905; from *Zeit. Zuck. Ind. Böhm.*, 23, 314—323).—When pentoses

are estimated by the phloroglucinol method, the result, calculated as arabinose, may be from 0.60—0.89 per cent. too high if cane sugar is present.

Substances yielding furfuraldehyde are found in the diffusion syrup to the extent of only 0.2 part for every 100 parts of cane sugar; the concentrated juice contains 0.13, and the first crop of crystals 0.03 part; the greater portion of these passes into the "scum" or the molasses.

L. DE K.

**Cobalt Derivatives of Sucrose and of Dextrose.** By W. HERZOG (*Chem. Zeit.*, 1899, 23, 627—628).—Papasogli (Abstr., 1898, 194) states that an aqueous solution of cane-sugar, when treated with a few drops of a solution of a cobalt salt and then with an excess of sodium hydroxide, gives an amethyst-violet coloration which is moderately stable, whereas an aqueous solution of dextrose, when treated in the same manner, yields a blue colour which is remarkably evanescent, changing quickly to a dirty green, and he recommends this reaction as a test for sucrose in the presence of dextrose. The author finds that the colours produced in the two cases are very similar, that with dextrose, however, is not so stable—it immediately turns blue and then a dirty green; with saccharose, too, the final colour is dirty green. The method is therefore of no use in testing for sucrose in the presence of dextrose. With regard to the nature of the products, it is thought probable that they are compounds of the cobalt salts with alkali sucates or dextrosates, the colour depending to a certain extent on the actual cobalt salt employed.

J. J. S.

**Analyses of Honey.** By C. HOITSEMA (*Zeit. anal. Chem.*, 1899, 38, 439—441).—In 10 samples of honey of known origin, the following estimations have been made: (1) specific gravity at 15° of a filtered solution of 1 part of honey in 2 parts of water; (2) pollen and wax, the constituents insoluble in warm water; (3) moisture estimated by drying without heat in a vacuum desiccator; (4) polariscopic rotation (after König, *Unters. landw. gew. wicht. Stoffe*, 1898, 474; (5) ash; (6) reducing and invertible sugars. The sp. gr. ranged from 1.102 to 1.14, water from 8.3 to 17.8 per cent.; polarisation from -3.1 to -9.1, being in all cases negative, ash, 0.12 to 0.34 per cent., pollen and wax, 0.02 to 0.46 per cent.; reducing sugar, 71.2 to 74.4; and sucrose, 0.2 to 2.6 per cent., whilst a sample of unknown origin showed 6.4 per cent. of the latter.

M. J. S.

**Estimation of Sugar in Fodder containing Molasses.** By OTTO FOERSTER (*Chem. Zeit.*, 1899, 23, 196).—The analysis is generally made by mixing a weighed quantity of the sample with water up to a definite volume, and afterwards making a correction for the volume of the undissolved matter; but this process can hardly lay claim to accuracy. The author recommends weighing out a multiple of 13.024 grams of the fodder and then extracting this in a Sickel's apparatus with absolute alcohol; after 12 hours, it may be taken for granted that all the sugar has been extracted. After distilling off the spirit, the residue is dissolved in water and diluted to a volume representing 75 c.c. for each 13.024 grams of the sample, and the sugar is then estimated either chemically or polarimetrically.

L. DE K.

[**Estimation of Caramel.**] By N. FRADISS (*Chem. Centr.*, 1899, i, 1160—1161; from *Bull. de l'Assoc.*, 16, 280).—The amount of caramel contained in sugar products is determined by treating the dry substance with a small quantity of methylic alcohol of 95°, evaporating on the water-bath, drying the gummy residue in a vacuum or in a current of dry air, and again digesting it with methylic alcohol for 2 hours. The caramel is precipitated from the solution by amylic alcohol, the precipitate redissolved, reprecipitated once or twice more, and finally dried at 90° and weighed. The caramel may also be determined by titrating with Violette's or Fehling's solution, but if dextrose is present it is necessary to titrate the original solution before and after treating with an excess of lead acetate. Then if  $A$  = c.c. of copper solution required,  $a$  = c.c. of original solution required for 5 c.c. of copper solution, the volume of the copper solution corresponding with the caramel is  $5(1 - a/A)$ . Allowance must also be made for the amount of lead acetate solution (1/10 vol.) used. The dextrose may also be destroyed by boiling with calcium carbonate and then precipitating with carbonic anhydride. Traces of caramel are estimated by treating 200 c.c. of the solution with excess of ammoniacal lead acetate. The precipitate, after washing with water free from carbonic anhydride, is suspended in water and decomposed with hydrogen sulphide. The filtrate is again treated with lead acetate, and the solution, from which the hydrogen sulphide has been expelled by evaporating to 10 c.c., is titrated with copper solution. Determinations of the amount of caramel in various sugar products are quoted.

E. W. W.

**Estimation of Glycogen.** By EDUARD PFLÜGER and JOSEPH NERKING (*Pflüger's Archiv*, 1899, 76, 531—542. By EDUARD PFLÜGER, *ibid.*, 543—551. Compare this vol., ii, 529).—These two papers contain more criticisms of Külz's method, and show that it can be improved by certain modifications. A new method, which is simpler and gives practically the same results as the improved Külz method, depends on the fact that in an alkaline solution of glycogen, or extract of flesh or liver, alcohol will completely precipitate the glycogen, and if the solution contains potassium iodide, the proteid matter remains in solution.

W. D. H.

**Detection of "Saccharin" in Foods.** By ALFRED HASTERLIK (*Chem. Zeit.*, 1899, 23, 267—268).—The author, having tried a large number of sweetened beverages for "saccharin" by Börnstein's resorcinol test, finds that this process is absolutely worthless, as the green fluorescence is often obtained when the liquids are quite free from "saccharin," as proved by other tests. The green colour is often caused by succinic acid, traces of which are always present in fermented liquids.

L. DE K.

**Detection of Formaldehyde in Milk.** By ALEXANDER LEYS (*J. Pharm.*, 1899, [vi], 10, 108—114).—Gayon's solution (*Abstr.*, 1888, 326), as used by the author for the detection of small quantities of formaldehyde in milk, is prepared by adding 10 c.c. of pure hydrochloric acid to a mixture of 10 c.c. of sodium hydrogen sulphite solu-

tion (30° B.) and 1000 c.c. of a 0.1 per cent. aqueous solution of magenta. It produces a well marked coloration with the distillate from milk containing one part of formaldehyde in a million.

H. R. LE S.

**Estimation of Formaldehyde by the Aid of Hydrogen Peroxide.** By OSKAR BLANK and HERMANN FINKENBEINER (*Ber.*, 1899, 32, 2141. Compare this vol., ii, 188).—A reply to Harden's criticism (*Proc.*, 1899, 15, 158). The authors' contention that their method is extremely accurate has been proved by numerous determinations.

J. J. S.

**Iodine Number of Fatty Acids.** By ALEXANDER ZEGA and R. MAJSTROVIĆ (*Chem. Zeit.*, 1899, 23, 597).—From experiments with fatty acids from maize oil, olive oil, sesame oil, and cotton seed oil, the conclusion is drawn that the iodine number largely decreases when the fatty acids are kept too long in the air-bath. In the case of maize oil, it was found that the dried acids, when kept at the ordinary temperature for some days, gave a gradually decreasing iodine number, falling in 8 days from 118.6 to 102.3 per cent.

L. DE K.

**Modification of Kaemer's Method for the Analysis of Cream of Tartar.** By G. LOMBARD (*Chem. Centr.*, 1899, i, 1086; from *Staz. sperim. agrar. ital.*, 32, 123—126).—In Kaemer's method, a weighed quantity of the sample is dissolved in the calculated amount of normal soda, the solution diluted to 200 c.c., and 20 c.c. are acidified with acetic acid and evaporated to dryness; the residue is washed with alcohol until the washings are no longer acid, and the undissolved potassium hydrogen tartrate then estimated by means of standard alkali.

The author has successfully modified this test. 20 c.c. of the solution are mixed with 1.5 c.c. of acetic acid and then with 100 c.c. of a mixture of equal parts of commercial absolute alcohol and ether. After the lapse of 4 hours, the potassium hydrogen tartrate has separated completely, and may be freed from acid by washing with ether-alcohol mixture; it is then titrated as before.

L. DE K.

**Calcium Citrate and its Analysis.** By ARTURO SOLDAINI and E. BERTÈ (*Gazzetta*, 1899, 29, i, 489—497).—The authors assign the considerable differences found in the results of analyses by different workers of commercial calcium citrate to two causes. Firstly, the amount of water retained by the sample varies with the method of storage, with the time of exposure to the air, &c. Secondly, when estimated as calcium citrate, the solubility of the latter in the washing water is not properly allowed for. Error is also sometimes caused by excessive drying of the citrate which then becomes to a slight extent insoluble in acetic acid. The authors find that crystallised calcium citrate loses  $2\text{H}_2\text{O}$  at 100—130°, and the remaining  $2\text{H}_2\text{O}$  at 175—185°. The following method of analysis is recommended. 1 gram of the finely powdered substance is heated in an Erlenmeyer flask with 10 c.c. of water, about 20 drops of hydrochloric acid of sp. gr. 1.18 added, and when solution has taken place, the liquid is cooled, made distinctly alkaline to phenolphthalein with  $N/2$  sodium hydroxide, and then about 3 c.c. of acetic acid added. The liquid is filtered and the residue washed until it is no longer acid to litmus paper.



The filtrate is evaporated to dryness on the water-bath, the dry mass being taken up with 15–20 c.c. of boiling water and the solution filtered on a hot water funnel through an ashless filter of 9 cm. diameter. The residue is then washed with 55 c.c. of water in such a way as to have the last drops of the washings almost or quite colourless. The precipitate is dried, burnt, and calcined strongly for 10 minutes.

T. H. P.

**Estimation of Non-volatile Organic Acids in Tobacco.** By RICHARD KISSLING (*Chem. Zeit.*, 1899, 23, 2–4. Compare Abstr., 1898, ii, 659).—I. *Separation and estimation of oxalic, citric, and malic acids by the baryta method.*—The barium salts of these acids may be obtained practically anhydrous by drying them at 70° under 100 mm. pressure; they then yield almost the theoretical amount of barium carbonate. Calcium oxalate also becomes quite anhydrous when thus treated.

The mixed acids are titrated with standard baryta, and 20 per cent. by volume of alcohol is then slowly added with constant stirring. Barium citrate and oxalate are almost completely precipitated, whilst the malate practically remains in solution, the two errors fairly compensating each other. The malate is then recovered from the filtrate by adding alcohol until its amount reaches 70 per cent. by volume, and leaving the mixture for 24 hours. Both precipitates are collected and weighed. The oxalic acid is estimated in a fresh portion of the solution of the acids by precipitation with calcium chloride in the presence of ammonium acetate.

II. *Estimation of oxalic, citric, and malic acids in tobacco.*—The process (*loc. cit.*) has been slightly modified. The first extraction of the tobacco with ether is now continued for 40–50 hours, and then again for 20 hours, this being repeated two or three times. After making up to 200 c.c., it is better to use 50 c.c. in place of 25 c.c. After shaking out the ether with 90 c.c. of water, the acids are separated as already described.

L. DE K.

**Influence of the Quality of the Ether on the Estimation of Fat in Food-stuffs.** By THEODOR METHNER (*Chem. Zeit.*, 1899, 23, 37–38).—When foods contain substances readily soluble in water or alcohol, it is as well to extract the fat by means of ether which is free from either water or alcohol. Absolutely anhydrous ether may be obtained by allowing ether to stand over metallic sodium, but the author states that a very good ether may be prepared by placing the commercial article of sp. gr. 0.720 for three weeks over quicklime and then distilling. This will extract about 0.1 per cent. more than the ether prepared by means of sodium.

L. DE K.

**Estimation of Fat in Animal Organs.** By WILHELM KNÖPFEL-MACHER (*Chem. Centr.*, 1899, i, 860–861; from *Österr. Chem. Zeit.*, 2, 122–124. Compare Dormeyer, Abstr., 1895, ii, 540; Frank, 1898, ii, 174; Voit, *ibid.*, 175).—Fat occurs in organs in two forms, one readily extractable with ether, whilst the other seems to be mechanically combined with proteid matters and cannot be extracted by the Soxhlet apparatus in 1000 hours. According to the

author, the best plan is to digest the matter with pepsin; this dissolves the proteids and so renders all the fat soluble in ether.

L. DE K.

**Preservation of Hübl's Reagent.** By RANDOLPH BOLLING (*Amer. Chem. J.*, 1899, 22, 213—214).—The author's experiments show that the alteration of Hübl's reagent as regards the free iodine present is very rapid and is only slightly delayed by using absolute alcohol in preparing it or by excluding air and light. W. A. D.

**Estimation of Fat in Milk.** By RICHARD WINDISCH (*Landw. Versuchs-Stat.*, 1899, 52, 209—211).—A number of samples of milk were analysed by the methods of Liebermann and Székely (*Abstr.*, 1893, ii, 308) and Gerber. The results, which are given, indicate that both methods are satisfactory. N. H. J. M.

**Aërometric Estimation of Fat in Milk.** By H. TIMPE (*Chem. Zeit.*, 1899, 23, 436—437, 455—457. Compare *Abstr.*, 1895, ii, 95).—When milk is mixed with aqueous potash, a small but fairly definite quantity of the fat is saponified and becomes insoluble in ether; this constitutes a slight error in Soxhlet's aërometric process. Another slight source of error is the fact that the usual figures representing the mutual solubility of ether and water are not absolutely correct. Finally, the following modified formulæ are proposed:

(1)  $Z = A - 0.075 W$ ; (2)  $F = 1.4566 Z \frac{S - 0.7222}{(0.9520 - S) \frac{v}{100}}$ , where  $A$  represents the weight of the ether saturated with water,  $W$  the weight of the water,  $Z$  the weight of the aqueous ether left after shaking,  $S$  the sp. gr. of the ethereal fat solution,  $v$ , the original volume of the milk,  $s$ , its sp. gr., and  $F$ , the weight of the fat it contains.

L. DE K.

**Estimation of Fat in Milk.** By A. A. BONNEMA (*Chem. Zeit.*, 1899, 23, 541—542).—10 c.c. of milk are put into a 100 c.c. cylindrical glass and mixed with 1.5 c.c. of aqueous caustic potash (20 grams per 100 c.c.). After shaking for a moment, 25 c.c. of ether are added and the whole thoroughly shaken for 5 minutes, the glass being held in the warm hand. The glass is placed in cold water, the stopper removed for a moment, 2 grams of tragacanth are added, and the whole is again thoroughly shaken. The tragacanth absorbs all the water and forms a clot, whilst the ether becomes quite clear; 10 c.c. of the ethereal solution are now pipetted off and evaporated in a weighed dish. The results agree with those obtainable by the "Gerber process."

L. DE K.

**Varying Composition of Butter.** By J. J. L. VAN RIJN (*Chem. Zeit.*, 1899, 23, 453—454).—The results obtained by the author again confirm the fact that the Reichert-Meißl number for genuine butter varies from 17—32, although the majority of butters give figures varying from 23—26. The variation is due to the condition of the cows and to the quality of the food.

L. DE K.



**Adulteration of Butter.** By THEODOR PFEIFFER (*Chem. Zeit.*, 1899, 23, 39—40).—A sample of absolutely pure butter gave, on analysis, a Reichert-Meissl number of only 19·4, and a Koetstorfer number as high as 219·4. It would therefore have been liable to be reported as adulterated with a considerable amount of margarine. It was made from the milk of three cows fed on the usual mixed diet.

L. DE K.

**Butter Analysis.** By ALEXANDER ZEGA (*Chem. Zeit.*, 1899, 23, 312).—Butter is melted and filtered, and some of the fat is put into a test-tube and heated for 2 minutes in the boiling water-bath. One c.c. is drawn off with a small pipette previously heated and put into a glass-stoppered cylinder containing 20 c.c. of a mixture of 6 parts of ether, 4 parts of alcohol, and 1 part of glacial acetic acid. The cylinder is placed in water at 15—18°, when, if the sample is pure, the liquid remains clear or only deposits an inappreciable amount of fatty matter. With butter containing 10 per cent. or more of margarine, however, a more or less abundant deposit is obtained which may be examined microscopically. Drawings are given showing the considerable differences in appearance between the deposits from butter and from margarine. The process is also serviceable for the detection of tallow in lard; drawings of deposits from lard and tallow being also given.

L. DE K.

**Butter Analysis.** By W. LAWRENCE GADD (*Chem. News*, 1899, 80, 113).—In the application of Reichert's test, the use of alcoholic potash that has been kept may lead to erroneous results which may be obviated by the use of solid potash in the following manner: about 2·5 grams of butter fat just at its melting point is treated in an 8 oz. flask with 20 c.c. of methylated spirit freshly distilled from caustic potash; about 1 gram of solid caustic potash is added, and the flask plunged into hot water. The saponification being complete, the alcohol is evaporated with the aid of a current of filtered air, the mass dissolved in hot water, acidified with sulphuric acid, and subjected to distillation, &c.

D. A. L.

**Halphen's Test for Cotton Seed Oil.** By PAUL SOLTSIEN (*Chem. Centr.*, 1899, i, 1049; from *Zeit. öffent. Chem.*, 5, 106—107).—Halphen's test for cotton seed oil (*Abstr.*, 1898, ii, 358) works well even without addition of amylic alcohol and sodium chloride. 10 grams of the sample of fat are mixed with 2 grams of a 1 per cent. solution of sulphur in carbon bisulphide and introduced into a test-tube which is closed with a cork through which passes a tube blown out to a bulb; the mixture is then heated for 15 minutes on a water-bath, and the colour compared with that of a known mixture of fat and cotton seed oil treated in like manner. The colour, which is very permanent, strongly resembles that of a solution of potassium dichromate.

The test is very useful, because age or rancidity of the fats do not impair it in the least, and it shows the presence of cotton seed oil, even if this has been previously heated to 200°. Lard, tallow, fats from cheese, sesamé oil, olive oil, rape oil, linseed oil, poppy oil, arachis oil, hazelnut oil, and cod-liver oil are not affected.

L. DE K.

**Presence of Chlorinated Organic Compounds and Absence of Sulphur Compounds in Cotton Seed Oil.** By P. N. RAIKOW (*Chem. Zeit.*, 1899, 23, 769—770 and 802).—Although cotton seed oil does not contain sulphur, it gives the phloroglucinol-vanillin test (this vol., ii, 52); this is due to the presence of chlorinated compounds, which, on burning, yield hydrogen chloride. By means of the tubular lamp previously described, the oil was burnt in a beaker placed on its side and the inner surface of the beaker was kept moistened with potassium hydroxide solution. Dupont's method (*Abstr.*, 1896, i, 409) has also been employed, but no sulphur could be detected. The chlorinated compound is not extracted by water, but is somewhat volatile in superheated steam.

Olive oil and walnut oil, although they give the phloroglucinol reaction, are free from both sulphur and chlorine (compare Fox and Riddick, *Chem. News*, 1895, 71, 296; also Dupont and Charabot, *Bull. Soc. Chim.*, 1896, [iii], 15, 341). The presence of chloro-derivatives in cotton seed oil may be made use of in determining whether this oil has been employed in adulterating other oils. J. J. S.

**"Vegetale" (Cotton-Stearin): Isolation of Cholesterol and Phytosterol from Fats.** By F. WIRTHLE (*Chem. Zeit.*, 1899, 23, 250).—"Vegetale" consists of crude cotton-stearin, and occurs as a yellow, semi-solid mass which, after being melted, does not readily solidify. Its iodine number is 89.24, and its saponification number 196.1. It gives the Bechi test in a very marked degree, and shows a refraction of  $63.3^\circ$  at  $25^\circ$ . By using Böhmer's process for the isolation of cholesterol and phytosterol, the latter may be readily detected, and mixed crystals of both these substances may be obtained from lard adulterated with only 10 per cent. of "vegetale." Böhmer's process gives satisfactory results even with very rancid fats, and it is only when the samples have been kept for many years under unfavourable conditions that no crystals can be obtained. Kreis's process for the isolation of cholesterol and phytosterol (this vol., ii, 343) requires 50 grams of fat, whilst Böhmer's test may be made with 25 grams only, so it possesses no decided advantages. L. DE K.

**Isolation of Cholesterol and Phytosterol from Fats.** By OTTO FOERSTER (*Chem. Zeit.*, 1899, 23, 188).—The author objects to the process recommended by Kreis and Wolf (this vol., ii, 343), as in the case of a sample of oil of mustard it only gave 0.03 gram of phytosterol, whilst in another experiment, conducted by the author's process (this vol., ii, 121), 0.6 gram was obtained. L. DE K.

**New Reactions of Sesamé Oil.** By FERDINAND BREINL (*Chem. Zeit.*, 1899, 23, 647).—The author states that, in place of testing for sesamé oil with hydrochloric acid and a 2 per cent. alcoholic solution of furfuraldehyde, a 3 per cent. alcoholic solution of hydroxybenzaldehyde, vanillin, or piperonal may, with advantage, be substituted. L. DE K.

**Bishop's Test for Sesamé Oil.** By HANS KREIS (*Chem. Zeit.*, 1899, 23, 802—803).—Stale sesamé oil, when shaken with an equal

volume of hydrochloric acid of sp. gr. 1.19, turns green, and if sugar is also added, a blue colour is developed.

A remarkable result was obtained by the author: a very rancid olive oil gave no colour with hydrochloric acid, but on adding some fresh sesamé oil, which also gave no test with hydrochloric acid, the green colour was at once apparent. Further results are promised.

L. DE K.

**Analysis of Human Urine.** By WILLIAM CAMERER and FRIEDRICH SÖLDNER (*Zeit. Biol.*, 1899, 38, 227—290).—The principal methods of urine analysis are criticised and experimentally compared. The most important outcome appears to be that, in the estimation of urea by the Mörner-Sjöquist method, part of the nitrogen of hippuric acid, allantoin, and creatinine is reckoned as urea nitrogen. The methods of estimating uric acid, alloxuric bases, ammonia, and other substances are also examined.

W. D. H.

[Estimation of Allylthiocarbimide in] Seeds of some Varieties of Brassica and Sinapis. By WILHELM KINZEL (*Landw. Versuchs-Stat.*, 1899, 52, 169—193).—The method employed was that of Dirck as modified by Schlicht (*Abstr.*, 1892, 1035), except that the substance was steam-distilled, as proposed by Förster (*Landw. Versuchs-Stat.*, 1898, 50, 419). Powdered rape cake (15 grams) and white mustard (4 grams) are mixed with water (150 c.c.) and tartaric acid (0.25 gram) in a 500 c.c. Erlenmeyer flask. The heating of the steam flask is so arranged that steam begins to pass over in about half-an-hour; the Erlenmeyer flask is now heated with a small flame until, in  $1\frac{1}{2}$  hours, 200 c.c. has distilled over. The vapours pass through a 0.6 cm. tube, 70 cm. long, into three receivers (an Erlenmeyer flask, a Will-Varrentrapp apparatus, and a second Erlenmeyer flask) containing 100, 18, and 10 c.c. respectively of alkaline permanganate solution. As the distillation proceeds, the heating of both flasks must be increased to avoid violent rising of the liquid in the Will-Varrentrapp apparatus. The safety tube of the steam flask is only opened in the case of the permanganate flowing backwards to the flask containing the substance. The liquid in the three receivers is mixed, heated for an hour on a water-bath, and after cooling to 70°, treated with alcohol (25 c.c.) and diluted to 501 c.c. Every 3.333 grams of precipitate ( $\text{KH}_3\text{Mn}_4\text{O}_{10}$ ) corresponds with 1.012 c.c. of solution at 17.5°. After filtration and addition of a little acid and iodine, the sulphuric acid is determined in the manner described by Schlicht (*loc. cit.*), the result being corrected by adding 0.00319 to the amount of barium sulphate from 400 c.c.

The results obtained with a number of samples of different varieties of brassica from India are given.

N. H. J. M.

**Estimation of Urea.** By SERGEI SALASKIN and J. ZALESKI (*Zeit. physiol. Chem.*, 1899, 28, 73—87).—The estimation of urea in urine by the Mörner-Sjöquist method gives results which are too high. This is because part of the hippuric acid nitrogen is reckoned as urea nitrogen; and the more hippuric acid the urine contains the greater the error. The method suggested is to heat the residue after the ether

and alcohol are evaporated off, in a glass tube, and to estimate the urea from the ammonia thus given off.

W. D. H.

**Volumetric Estimation of Aniline and Bromine.** By GEORGES DENIGÈS (*J. Pharm.*, 1899, [vi], 10, 63—66).—A claim for priority with respect to the method proposed by François (this vol., ii, 713) for the estimation of aniline.

H. R. LE S.

**Methods of Estimating Alkaloids.** By OTTO LINDE (*Arch. Pharm.*, 1899, 237, 392—408. Compare this vol., ii, 534).—Keller's method (compare Abstr., 1897, ii, 84) is open to the following objections. (1.) The assumption is made that the solvent either extracts all the alkaloid, or, at least, contains it in equal concentration throughout, whether within or without the drug; it is found, however, that the residual part, including the much-swollen drug, contains proportionally more alkaloid. (2.) The amount of solution removed is assumed to contain 100/120 of the alkaloid; as a matter of fact, the total amount of the solution may have suffered an increase owing to the solution in it of fat, and even of soap. (3.) The repeated shaking and the large amount of solvent used are objectionable; shaking once with a smaller quantity suffices if the volume of the aqueous solution is kept relatively small; it may also be saturated with some salt. (4.) It is better to measure the quantities of solvent used than to weigh them; pipettes graduated for use with water may be employed.

Ekroos' method (this vol., ii, 74) cannot be used with drugs from which ammonia or an aliphatic amine is liberated by treatment with soda; neither can it be used with drugs which contain oil, as in these cases a soap is formed which dissolves in the chloroform-ether, and uses up some of the sulphuric acid.

Katz's method (Abstr., 1898, ii, 547) is equally unavailable for drugs from which soda liberates ammonia or an amine.

It is better not to estimate an alkaloid only by titrating its solution, but first to weigh it after evaporation of the solution, in which process volatile alkalis are removed, and then to titrate the residue; a serious discrepancy between the two results would indicate adulteration.

C. F. B.

**Hydrastine Pentiodide Hydriodide.** Volumetric Estimation of Hydrastine and Berberine in the Root of *Hydrastis Canadensis*. By HARRY MANN GORDIN and ALBERT B. PRESCOTT (*Arch. Pharm.*, 1899, 237, 439—446).—When a dilute solution of hydrastine is added to a large excess of a solution of iodine in aqueous potassium iodide, a dark brown compound,  $C_{21}H_{21}O_6N, HI, I_5$ , is precipitated; this melts readily under water; it loses 5I when treated with sulphurous acid or sodium thiosulphate.

For the estimation of the alkaloids in *Hydrastis canadensis*, 10 grams of the powdered root are stirred into a paste with a mixture of alcohol, concentrated ammonia, and ether (1 : 1 : 6 parts by volume), and allowed to remain in a well-closed vessel for several hours. The mixture is then dried, at first in a good draught and then over sulphuric acid under diminished pressure; the residue is transferred to a Soxhlet apparatus, being rinsed out with powdered barium nitrate, and the hydrastine is

extracted completely with absolute ether; the ether is evaporated from the extract, and the residue dissolved in acidified water, and the solution diluted to 100 c.c. In a graduated 100 c.c. flask, 20—30 c.c. of a standard iodine solution (of about 1 per cent. strength) are placed, 20 c.c. of the filtered hydrastine solution run in, and the mixture diluted to the mark and shaken until the pentiodide has all separated; the mixture is then filtered, and the excess of iodine determined by titrating 50 c.c. of the filtrate with standard sodium thiosulphate solution. Every 1 part of iodine used corresponds with 0.60403 part of hydrastine. Or the alkaloid may be estimated gravimetrically by shaking 20 c.c. of the filtered hydrastine solution with benzene and ammonia, removing the alkaloid from the benzene solution by shaking with acidified water, and then from the acid solution with ammonia and ether, the ethereal solution is finally evaporated in a dark place at the ordinary temperature, and the residue of hydrastine weighed.

The residue in the Soxhlet apparatus contains the berberine, which is not soluble in absolute ether; it is dried by passing a current of dry air through the apparatus, and is then extracted with alcohol. The alcohol is removed from the extract by heating it with 200 c.c. of water on the water-bath; the residual liquid is acidified with acetic acid, cooled, and filtered into a conical flask; in this it is shaken for 10—15 minutes with 6—8 c.c. of acetone and enough 10 per cent. caustic soda solution to render the reaction alkaline, and set aside for 2—3 hours. The precipitated acetone compound is washed, and warmed in the same flask with 200—300 c.c. of very dilute sulphuric acid until it has all dissolved, the solution is poured into a long-necked Kjeldahl flask and boiled for  $1\frac{1}{2}$ —2 hours; when cold, it is added to 100 c.c. of *N*/20 potassium iodide solution contained in a graduated 1000 c.c. flask, diluted to the mark, shaken, and left overnight. Then 500 c.c. are filtered from the precipitate of berberine hydriodide into another 1000 c.c. flask, treated with 50 c.c. *N*/20 silver nitrate and nitric acid, diluted to the mark, and filtered; the excess of silver is determined by titrating 500 c.c. of the filtrate with *N*/40 ammonium thiocyanate. The number of c.c. of the iodide solution used, multiplied by 0.167125, gives the percentage of berberine in the root.

C. F. B.

**Vitali's Test for Veratrine.** By IWAN L. KONDAKOFF (*Chem. Zeit.*, 1899, 23, 4).—The author confirms Kunz-Krause's statement that when veratrine is evaporated with fuming nitric acid and the residue treated with alcoholic potash, the mixture turns blood-red and evolves a strong odour of coniine. The odorous substance is really coniine, and not  $\beta$ -picoline.

L. DE K.

**Melzer's Picrotoxin Reaction.** By HANS KREIS (*Chem. Zeit.*, 1899, 23, 21—22).—The author warns toxicologists that cholesterol and phytosterol also give reactions with benzaldehyde and sulphuric acid very similar to that described as characteristic for picrotoxin by Melzer (*Abstr.*, 1898, ii, 650).

L. DE K.

**Estimation of Proteids in Blood Serum.** By GUSTAVE PATEIN (*J. Pharm.*, 1899, [vi], 10, 244—249).—*Alkaliseralbumin* is the name given by the author to the albumin precipitated by acidifying diluted

human blood serum with 10 per cent. acetic acid. For its estimation, 100 c.c. of the serum are diluted with 900 c.c. of water, and the liquid is rendered faintly acid with 10 per cent. acetic acid. The mixture is left for 24 hours, and the precipitate washed by decantation with water slightly acidified with acetic acid. It is then dissolved in 60 c.c. of water previously acidified with acetic acid, 1 to 2 grams of sodium sulphate or chloride are added, and the solution boiled. From this stage, the process is the same as for the estimation of albumin.

*Estimation of total albumin.*—10 c.c. of the serum are diluted to 100 c.c. with water, the liquid is rendered very faintly acid with acetic acid, and 2 grams of sodium sulphate are added. The mixture is boiled, and the precipitate, collected on a tared filter, washed with boiling water, alcohol, and ether, and dried and weighed.

*Estimation of serin and serum-globulin.*—The serum previously diluted with 9 times its volume of water, is rendered faintly acid with acetic acid and the mixture left for 24 hours. It is then heated to boiling, and the precipitate collected, dried, and weighed.

*Estimation of serin.*—The serum is diluted with 9 times its volume of water, and 100 c.c. of this solution are saturated with magnesium sulphate. The liquid is filtered, and an aliquot part of the filtrate, faintly acidified with acetic acid, is boiled, and the precipitated serin dried and weighed. The serum-globulin is estimated by difference. 1000 grams of blood serum were found to contain : total proteids, 75.4 ; alkaliseralbumin, 2.45 ; serin and other proteids, 45.03 ; and globulin, 27.92.

H. R. LE S.

**Estimation of Albumin in Urine.** By GEORGES DENIGÈS (*J. Pharm.*, 1899, [vi], 10, 97—102).—The author's method for the estimation of casein in milk (*Abstr.*, 1897, ii, 532) is applicable to the estimation of albumin in urine. For urine containing not more than 1.1 grams of albumin per litre, the method is as follows: 20 c.c. of mercuric potassium iodide and 2 c.c. of glacial acetic acid are added to 150 c.c. of the urine, and the whole diluted to 200 c.c. The mixture is filtered, and 25 c.c. of an ammoniacal solution of potassium cyanide (equivalent to  $N/20$  silver nitrate) are added to 125 c.c. of the filtrate, the whole left for 2 or 3 minutes, and again filtered. To 120 c.c. of the last filtrate,  $N/10$  silver nitrate is added until a faint, but permanent, opalescence is produced. The number of tenths of a c.c. of silver nitrate less 48 (a constant) gives the number of decigrams of albumin in a litre of urine.

If the urine contains more than 1.1 grams of albumin in a litre, then the number of c.c. of urine used must be such that it shall not contain more than 0.15—0.16 gram of albumin, and shall be a simple ratio of 150. If the urine contains less than 0.2 gram of albumin in a litre, the amount is determined by comparing the opalescence produced by the addition of 2 c.c. of a 5 per cent. solution of sodium metaphosphate and 4 drops of sulphuric acid to 10 c.c. of the urine and heating in boiling water for 5 minutes, with that produced, under the same conditions, with urine solutions containing a known weight of albumin.

H. R. LE S.