

## Analytical Chemistry.

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**A New Desiccator.** By W. HEMPEL (*Zeit. ang. Chem.*, 1891, 200—201).—The author has some time ago explained that, as moist air is lighter than dry air, the practice of having the drying agent at the bottom of the desiccator is objectionable. By placing the drier (sulphuric acid, calcium chloride) as high as possible and above the substance, the drying proceeds rapidly and effectively, owing to circulation of the air. The author has now given a definite shape to his apparatus, which is composed of a glass cylinder fitted with a lid, in the shape of the well-known flycatcher, in which the sulphuric acid is placed. The top part of the lid is provided with an arrangement to connect the apparatus with an air-pump, if desired. To prove the superiority of this desiccator, the author took two watch-glasses of exactly equal size, filled each with 10 c.c. of water, and placed one in an ordinary desiccator and the other in the cylinder of the new apparatus. The result was, that whilst the first lot took nine days to evaporate, the second had dried up in three.

L. DE K.

**Improvement in Gasvolumetric Analysis.** By G. LUNGE (*Zeit. ang. Chem.*, 1891, 197—200).—The old-fashioned clamps used to support the author's gasvolumeter soon get worn out, or sometimes break down during the experiments, causing great annoyance to the operator. By the new arrangement this source of failure is eliminated. Figs. 1 and 2 represent the new compound screw and forked



FIG. 1.

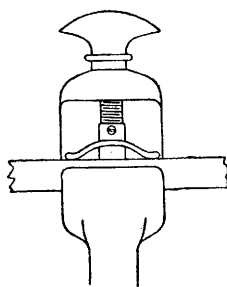


FIG. 2.

clamp; *a* and *b* being respectively a small and large cork-lined clamp, whilst *c* is a species of thumbscrew, shown on a larger scale in Fig. 2.

L. DE K.

**Simple and Rapid Method of Gravimetric Analysis.** By G. H. BAILEY and J. C. CAIN (*J. Soc. Chem. Ind.*, 10, 329—330).—A

100 c.c. flask is used, which is made in two parts, the neck being in a separate piece, and accurately ground into the body of the flask. It is desirable to make the neck almost capillary, for the purposes of accurate reading. The flask is first filled with the liquid and precipitate, after which the capillary neck is fitted in, and a little more liquid added, until a given mark on the stem is reached. The latter is graduated in millimetres. These serve, not only to adjust accurately 100 c.c. of liquid at 4°, but also (the capacity of the stem being, once for all, determined) enabled the authors to get rid of the troublesome cooling (or warming) process used in specific gravity determinations. For instance, if the liquid at 4° stood at division 6 for 100 c.c. liquid, and at 10° it stood at division 9, it is evident that when working at 10° the flask must be filled up to division 9, to represent 100 c.c. of liquid at the point of maximum density of water.

*Method of Working.*—First, the specific gravity of the substance precipitated is carefully determined by ascertaining (a) weight of flask and air, and then deducting from this the weight of 100 c.c. of air; (b) weight of flask and 100 c.c. of water at 4°; (c) weight of flask filled to 100 c.c. mark with water and the substance whose specific gravity was to be determined. The following values were obtained:—

Silver chloride precipitated hot, 5·298.

Silver chloride precipitated in the cold, 5·287.

Copper oxide precipitated hot, 6·514.

Ferric hydroxide precipitated hot, 2·583.

Barium sulphate precipitated hot, 4·651.

Barium sulphate precipitated in the cold, 4·612.

In order to make use of these numbers in the inverse manner for ascertaining the weight of a given precipitate, it is not necessary to replace the supernatant liquor by water, for, by using two similar flasks, 100 c.c. of the clear, supernatant liquor may be run off into one flask, leaving the precipitate and supernatant liquor occupying 100 c.c. in the other flask. Some of the supernatant liquid may also be transferred to a small wash-bottle for washing down any precipitate which remains attached to the sides of the funnel. The actual determination of the weight of the precipitate *W* now takes this form:—

$$W = \frac{(w' - w)V_s}{V_s - W},$$

where *w'* = weight of supernatant liquid and precipitate.

*w* = weight of known volume (say 100 c.c.) of liquid.

*s* = specific gravity of precipitate.

*V* = volume of the flask (say 100 c.c.). D. B.

**Use of Potassium Hydrogen Tartrate for Titrating Standard Acids and Alkalis.** By A. BOENTRÄGER (*Zeit. anal. Chem.*, **31**, 43—57).—After giving a summary of most of the methods in use, or suggested, for the standardisation of acid and alkaline solutions, and

pointing out the objections to each of them, the author proposes potassium hydrogen tartrate for the purpose. It is easily prepared and preserved in a pure and definite condition, is anhydrous and not hygroscopic; used directly, it serves as an acid for standardising alkalis, and if ignited it yields a definite quantity of potassium carbonate, by which an acid can be titrated. The author's material was prepared by heating white cream of tartar with dilute hydrochloric acid, cooling with stirring, washing the precipitate completely, recrystallising from water, and drying at  $100^{\circ}$ . It was regarded as pure when the alkali obtained by calcining a weighed quantity exactly neutralised an equal amount of the uncalcined substance. Sensitive violet litmus-paper was used as indicator.

M. J. S.

**Gasometric Alkalimetry and the Employment of Potassium Ferricyanide in Gasometry.** By J. QUINCKE (*Zeit. anal. Chem.*, **31**, 1—43).—The reaction  $K_3Fe_2Cy_{12} + 2KHO + H_2O_2 = 2K_4FeCy_6 + 2H_2O + O_2$  takes place with quantitative exactness, the amount of oxygen evolved being an accurate measure of the reagent present, so that it can be employed for the estimation of ferricyanides, alkaline hydroxides, or hydrogen peroxide. Assuming that the volume of gas evolved can be read to 0.05 c.c., the error of reading is about equal in magnitude to that involved in measuring a N/10 solution in a burette with float. The potassium hydroxide of the above equation can be replaced by sodium or barium hydroxide with equally accurate results; calcium hydroxide gives results of slightly less accuracy, but still amply sufficient for most purposes. Ammonia is inadmissible, since it decomposes far more of the peroxide than would correspond with an analogous equation. Neither can the alkali or alkaline earthy carbonates be estimated, since they only slowly and imperfectly decompose the peroxide. Weak solutions of the carbonates indeed act so slowly, whilst the reaction with the hydroxides is so rapid (being complete in from one to five minutes), that the presence of a little carbonate does not interfere with the estimation of a hydroxide. The reaction  $K_3Fe_2Cy_{12} + BaO_2 = BaK_4Fe_2Cy_{12} + O_2$  also takes place, but not very energetically. By dissolving the barium peroxide in dilute hydrochloric acid, and mixing this solution with excess of strongly alkaline ferricyanide, the reaction becomes rapid and exact.

There are many substances which are oxidised by alkaline solution of ferricyanide, as grape sugar, phosphorous acid, &c.; and, in cases where the products of the oxidation have no action on hydrogen peroxide, an estimation of the residual ferricyanide can be employed for their determination. Arsenious and antimonious trioxides in particular give highly satisfactory results. The potash solution should be strong, and used in large excess. The ferricyanide may be used in the form of standard solution, which, if kept in a bottle of yellow glass, retains its strength for some months. No warming is required. A precipitate of antimony trisulphide may be titrated by dissolving it in concentrated hydrochloric acid, boiling to expel hydrogen sulphide, and then treating the solution with the alkaline ferricyanide. Arsenic trisulphide presents difficulties. The author's

experiments were made with an improved form of Knop's azotometer, in which all parts of the apparatus could be plunged into the same mass of water. The hydrogen peroxide used was "commercial," containing 2.4 per cent. The numerous test analyses reported are highly satisfactory, and tables are given in the paper for facilitating the calculations.

M. J. S.

#### Estimation of Soluble Chlorides, Bromides, and Iodides.

By L. L. DE KONINCK and E. NIHOUL (*Zeit. ang. Chem.*, 1891, 294—298).—When a perfectly neutral solution of a chloride, bromide, or iodide (free from barium) is shaken with freshly precipitated silver chromate, the silver combines with the halogen, and the filtrate contains a chromate. The chromic acid may be readily estimated by adding sulphuric acid and potassium iodide, and titrating the liberated iodine with sodium thiosulphate. 3 mols. of this salt or 3 atoms of free iodine correspond with 1 atom of either combined chlorine, bromine, or iodine. Very small traces of chromate may also be estimated colorimetrically. The test analyses are satisfactory.

L. DE K.

#### A New Method for the Estimation of Organic Nitrogen.

By J. H. SMITH (*Chem. Zeit.*, 14, 1223—1229).—A preliminary communication as to an indirect process for the determination of organic nitrogen. It is based on the complete oxidation of the organic compound and the conversion of its nitrogen either into nitrogen or nitric acid or both, according to the substance oxidised.

The process is carried out by heating the substance with potassium permanganate, potassium bromide, and sulphuric acid. The amount of the bromide need not be large, as bromine is constantly being regenerated. After cooling, the excess of permanganate is titrated back in the usual manner, after the bromides have been first precipitated with argentic nitrate; and from the difference between the oxygen absorbed in this case, and that absorbed in a like oxidation in absence of bromide, the nitrogen is calculated.

L. DE K.

#### Estimation of Nitrogen in Nitrates.

By E. FRICKE (*Zeit. ang. Chem.*, 1891, 239—241).—A process which the author has successfully employed for some years is as follows:—20 grams of the sample is dissolved in a litre of water, and 50 c.c. is put into a 600 c.c. flask and diluted with an equal bulk of water. 20 grams of potassium hydroxide is now added, and after this has dissolved, 15 grams of zinc and iron dust, also 75 c.c. of alcohol. To prevent frothing, it is as well to add a few granules of animal charcoal. The flask is now closed and connected with a 200 c.c. Peligot's tube which contains 10 c.c. of normal sulphuric acid, and is partially immersed in cold water. After three or four hours, the spirit, which of course carries over the ammonia, is distilled off. The acid is then titrated back with N/4 soda. The process is really an excellent one, provided the potash is free from nitrates. Stützer's process, reduction by means of aluminium wire, cannot be recommended, as the pure metal nowadays, prepared by electrolysis, is far less active than the metal made

by the old process, which yields a product containing both sodium and silicon.

After mentioning Schmitt's process, based on the reduction of nitric acid by hydrogen in acetic acid solution, a process which would be an excellent one if it were not for the frothing and spurting, the author remarks: "This and similar processes must, however, make room for a method lately proposed by Ulsch, which is characterised by simplicity, economy, and celerity." 25 c.c. of a solution containing about 0.5 gram of the nitrate is put into a 600 c.c. flask, and reduced by means of 5 grams of reduced iron and 10 c.c. of dilute sulphuric acid (1—3). To prevent loss by spurting, the flask must be covered with a pear-shaped glass bulb. The liquid is now gradually heated to boiling, and, after six minutes, allowed to cool. After diluting with about 150 c.c. of water, 30 c.c. of aqueous soda is added, besides a few lumps of granulated zinc, and the ammonia is distilled off as usual. On trying this process on a sample of chemically pure sodium nitrate, the author found 16.44 instead of 16.47 per cent. of nitrogen. The process may also be employed for the estimation of nitrates in potable waters.

L. DE K.

**Estimation of Nitrogen in Pure and Mixed Nitrates.** By A. SÜLLWALD (*Chem. Zeit.*, **14**, 1673—1674, 1748).—The author has made several experiments to compare his own process (a modification of Jodlbauer's) with the one recommended by Förster.

The author's process is as follows:—0.5 gram of nitre, or 1 gram of a mixed nitrate, is put into a 150 c.c. flask and moistened with 0.5 c.c. of water. The object of this is to make the nitrate dissolve more easily in the acid, and it also prevents too violent an action. 25 c.c. of sulphuric acid containing 40 grams of phenol per litre is now put into the flask, and, after cooling, 2.5 grams of zinc-dust is added. After waiting for about 15 minutes, a few drops of mercury are added, and the mass gradually heated to boiling. All the nitrogen will thus be converted into ammonium sulphate. The ammonia is then estimated volumetrically as usual.

Förster's process is as follows:—0.5 gram of the nitre is dissolved in 15 c.c. of sulphuric acid containing 6 per cent. of salicylic acid. When the substance has dissolved, 5 grams of sodium thiosulphate is added, and when the action is over, 10 c.c. of pure sulphuric acid and a few drops of mercury are introduced. The conversion of the nitrogen into ammonia is generally accomplished in about  $1\frac{1}{2}$  hours.

The results of the experiments proved that the author's process is quite as accurate as Förster's method, although the latter is more simple and is performed in less time. In order to avoid loss, the author recommends treating the nitrate with the acid in the same flask, which is afterwards used for the ammonia distillation. When, however, Chili saltpetre has to be tested, the author prefers his own process to that of Förster, as this substance refuses to properly dissolve in the sulphosalicylic acid.

L. DE K.

**Estimation of Phosphorus in Iron and Steel.** By C. MALOT (*Zeit. anal. Chem.*, **31**, 78—79; from *Compt. rend. mens. de la Soc. de*

*l'ind. minér.*, Avril, 1887).—The phosphorus is precipitated as ammonium phosphomolybdate in the usual way; the precipitate is dissolved in ammonia and reprecipitated by magnesia mixture. The magnesia precipitate is dissolved in nitric acid of 1·2 sp. gr., a drop or two of cochineal tincture (prepared by treating cochineal with boiling water) is added, then ammonia just to violet coloration, which is again removed by one or two drops of nitric acid. There is then added 5 c.c. of a solution containing in the litre 100 grams of sodium acetate and 50 c.c. of acetic acid, the whole is heated to 100°, and titrated with a neutral uranium nitrate solution of known value. As soon as all the phosphoric acid is precipitated, the excess of uranium forms a green lake with the cochineal, so that the colour changes from red to greenish-blue. The uranium solution is of convenient strength if 1 c.c. precipitates 2 milligrams of phosphoric anhydride. Duplicate estimations do not show greater differences than 0·2 milligram.

M. J. S.

### Estimation of Phosphorus in Pig Iron, Steel, and Iron Ores.

By F. A. EMMERTON (*Zeit. anal. Chem.*, **31**, 71—75).—The phosphorus is converted into the yellow ammonium phosphomolybdate, and the amount of the latter ascertained by volumetric estimation of the molybdic acid. 5 grams of steel is dissolved in 75 c.c. of nitric acid (sp. gr. 1·2) in a covered basin, the solution boiled to dryness, and the residue heated for 30 minutes. The cooled residue is taken up by 40 c.c. of strong hydrochloric acid, and the solution evaporated to 15 c.c., taking care that none of the ferric chloride dries on the side of the basin. 40 c.c. of strong nitric acid is now added and again evaporated to 15 c.c., the basin being covered by a clock glass of smaller diameter than itself, placed with the concave side downwards, so that the condensed vapours flowing back prevent the formation of a crust at the edges. A highly concentrated, clear solution free from hydrochloric acid is thus obtained. This is washed into a 400 c.c. flask with about 60 c.c. of water, mixed gradually with a small excess of ammonia, and the precipitate just redissolved by strong nitric acid. The temperature is now brought to 85°, and 40 c.c. of molybdate solution added at once. The flask, stoppered and wrapped in a cloth, is shaken for five minutes, which completes the precipitation. The precipitate is now collected on a suction filter, washed with dilute nitric acid, and then rinsed with 30 c.c. of dilute ammonia through the pierced filter into a flask containing 10 grams of zinc; 80 c.c. of dilute sulphuric acid (1 : 4) is added, and the liquid boiled for 10 minutes, to complete the reduction of the molybdic acid, the colour of the liquid passing through pale-red to olive-green. It is then rapidly filtered and washed through a ribbed filter, and standard permanganate run in until the liquid is perfectly colourless. Since the reduction of molybdic acid by zinc produces a mixture of oxides corresponding with the composition  $\text{Mo}_{12}\text{O}_{19}$ , the equation for the titration is  $5\text{Mo}_{12}\text{O}_{19} + 34\text{KMnO}_4 = 60\text{MoO}_3 + 17\text{K}_2\text{O} + 34\text{MnO}$ , and since the phosphorus in the precipitate amounts to 1·794 per cent. of the molybdic acid present, a permanganate solution of which 1 c.c. oxidises 0·006141 gram of iron will correspond with 0·0001 gram of phosphorus per c.c.

Pig iron is dissolved in hydrochloric acid, and, after making up and filtering from insoluble matter, an aliquot part is treated as above. Iron ores are dissolved in hydrochloric acid, the solution evaporated to dryness, taken up with hydrochloric acid, concentrated, boiled with nitric acid, then made up, filtered, and an aliquot part of the filtrate employed.

M. J. S.

**Separation of Phosphoric and Arsenic Acids from Mercury, and of Nitric Acid, Chlorine, and Sodium from Mercury, Phosphoric, and Arsenic Acids.** By K. HAACK (*Zeit. anal. Chem.*, **31**, 79—81).—1. *Separation of Arsenic and Phosphoric Acids from Mercury.*—If a mercuric salt, the substance is dissolved with hydrochloric acid; if a mercurous salt, it is dissolved in nitric acid with the addition of hydrochloric acid and potassium chlorate, and warming until the odour of chlorine is removed. An excess of ammonia is added, and then, drop by drop, a rather strong solution of potassium cyanide until the precipitate is redissolved. Ammonia is now added until it amounts to one-fourth of the whole volume, and then an equal bulk of absolute alcohol. The arsenic or phosphoric acid can now be precipitated by magnesia mixture in the usual way, and the mercury from the filtrate by hydrogen sulphide.

2. *Nitric Acid from Mercury and Phosphoric and Arsenic Acids.*—The solid substance is boiled for a quarter of an hour with an excess of barium hydroxide. From the filtrate, the excess of barium is precipitated by carbonic anhydride and boiling. The remaining barium is precipitated as sulphate and calculated to nitrate. If alkali metals are present, part or the whole of the nitric acid will remain as alkali nitrate, and, should the alkali be in excess, this method is inapplicable. If otherwise, the alkali must also be determined and calculated as nitrate. For this purpose, the filtrate from the barium sulphate is evaporated, and the residue weighed as alkali sulphate. To separate sodium from mercury and arsenic acid, the substance may be ignited with excess of solid ammonium chloride in a porcelain crucible.

3. *Separation of Chlorine.*—The substance is boiled with baryta as above, and the chlorine estimated in the filtrate. The baryta precipitate is free from chlorine.

M. J. S.

**Direct Estimation of Arsenic in Minerals and Metals.** By J. CLARK (*J. Soc. Chem. Ind.*, **10**, 444—445).—In 1867, the author, in conjunction with Esilman, showed that ferric salts decomposed the sulphides of a large number of metals, separating sulphur, and forming a salt of the metal, which was accompanied by the reduction of the ferric salt employed to the corresponding ferrous salt. It occurred to the author to utilise this action for the estimation of arsenic when in the form of sulphide. For this purpose, known quantities of pure arsenic sulphide were distilled with a concentrated solution of ferric chloride in strong hydrochloric acid in an ordinary flask connected with a worm condenser, to the end of which a wide tube was attached which dipped into water. The arsenic was



then precipitated with hydrogen sulphide in the cold, and collected on a weighed filter, with the following results:—

- (1.) 5 grams  $\text{As}_2\text{S}_3$  gave  $5.02 = 100.40$  per cent.
- (2.) 5 grams  $\text{As}_2\text{S}_3$  gave  $4.95 = 99.00$  „

It is absolutely necessary, in estimating the arsenic in this way, that the hydrochloric acid should be concentrated, and it is always advisable to evaporate down the solution three times with a considerable quantity of strong hydrochloric acid. The ferric chloride decomposes the hydrogen sulphide which would be produced by dissolving the arsenic sulphide in hydrochloric acid, facilitates the solution, and enables the arsenic to be estimated directly.

The author has made numerous experiments with ferric chloride on mixed sulphides of arsenic, antimony, and tin with satisfactory results, and has found this to be the simplest and most trustworthy process for estimating arsenic in antimony ore. It is also applicable for the direct estimation of arsenic in iron and copper pyrites.

D. B.

**A New Apparatus for the Estimation of Combined and Free Carbonic Acid.** By G. LUNGE and L. MARCHLEWSKI (*Zeit. ang. Chem.*, 1891, 229—234).—In Fig. 1, which shows the apparatus in its proper proportions, but at about one-ninth of its actual size, A represents the generating flask. If used for the analysis of limestone, soda-ash, &c., it has a capacity of about 30 c.c.; but if required for the estimation of carbonic anhydride in water, or carbon in iron, it must have a larger size. The flask, which is shown on a larger scale in Fig. 2, has a side tube *a*, which is connected with a stop-cock *b* and a funnel *c*; it is closed by means of a ground, hollow stopper, which ends in a rather thin, capillary tube *d*, 35 cm. in length. This leads to the side tube of a Lunge's gasvolumeter. The bulb underneath the stop-cock *f* holds about 100 c.c., and is connected with a tube holding another 50 c.c., and calibrated into 0.1 c.c. The Friedrich's stop-cock *f* communicates with the capillary tubes *e* and *g*, which run at a right angle to one another. The tube *g* is connected by means of an india-rubber tube with another capillary tube which terminates in a doubly perforated stop-cock *h*. The latter is connected with the Orsat tube E, which is filled with aqueous potash or soda, and is conveniently supported by the same forked clamp (shown in the figure). All the india-rubber joints, except the one at *e*, are wired. The tube E is also fitted with a soda-lime tube *i*, which prevents the entrance of respiratory carbonic anhydride when the potash is blown up. C is the duct tube, which terminates in a sealed capillary *k*, and D the pressure tube, which should be 3.5 cm. wide; both are supported by the forked clamp *l*. To adjust the apparatus, the temperature and pressure must be, once for all, observed, the great advantage of the instrument over similar ones being that it serves as its own barometer. The gas is expelled from B by raising D; *f* is then closed and D lowered until the distance of its mercury level is over 760 mm. from *f*. D should be lowered sufficiently to cause the mercury to



sink below the bulb in B. D is then made fast, and one must wait a short time to see whether  $f$  fits tightly, and if the mercury level in B

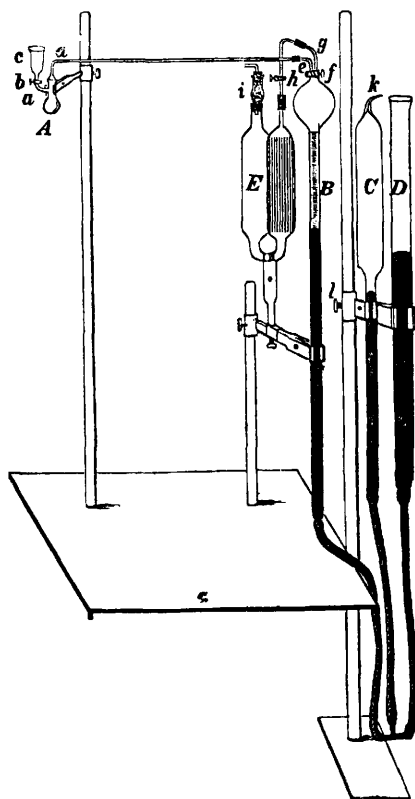


FIG. 1.

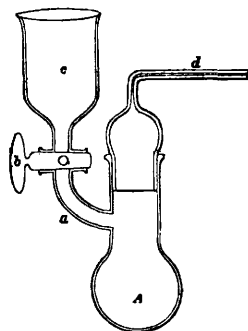


FIG. 2.

remains unchanged. The distance in millimetres between the two mercury levels in B and D is now ascertained by means of any suitable rule. 1 mm. is deducted on account of the tension of mercury vapour at temperatures under  $12^{\circ}$ , 2 mm. for  $13-19^{\circ}$ , and 3 mm. for  $20-25^{\circ}$ , and this corrected pressure is called  $b$ . The volume which 100 c.c. of dry air would occupy under these conditions is calculated by the formula

$$\frac{100.(273 + t)760}{273.(b - f)},$$

in which  $f$  is the tension of water vapour, which may be taken approximately as  $(t - 2)$  for temperatures between  $12^{\circ}$  and  $25^{\circ}$ , thus:—

$$\frac{100.(273 + t)760}{273(b - t + 2)}.$$

*f* is now opened, the mercury in C adjusted to the division corresponding with the calculated volume, and the column in B and D are brought to the same height. In order to saturate the air with water vapour, a minute drop of water is introduced by means of the capillary K into C. K is now sealed by means of a spirit flame, and, to prevent C getting warm, a screen of cardboard is used. If the mercury still stands at the same division, the apparatus is ready for use, and no corrections need be made for alterations in temperature and pressure.

Before commencing an analysis, it is necessary to conduct a blank experiment to see whether the apparatus is tight. 0.1 gram of fine aluminium wire is introduced into the flask A. B is filled with mercury by raising D, and is made to communicate with A by the stop-cock *f*, *b* being closed. D is then lowered, *h* opened, and D again raised to expel the air in B through E. When this has been repeated about three times, the apparatus is sufficiently exhausted. Hydrochloric acid, diluted with four times its bulk of recently boiled water, contained in the funnel *c*, is now allowed to run through *b* into A until this is one-third filled. On applying a gentle heat, a strong evolution of gas sets in, but care must be taken to have the mercury level in D lower than in B. After all the aluminium has dissolved, the stop-cock *b* is opened, and more acid is allowed to enter until it reaches the neck of the capillary *d*, and the stop-cock *f* is then closed. The three tubes are adjusted to the same level, the mercury in C standing at the 100 division, and allowed to remain so for 10 minutes to equalise their temperatures. In the meanwhile, the soda is forced by blowing through *i* as far as the stop-cock *h*, which is then closed. A is now detached and cleaned, so as to be ready for the actual analysis. After adjusting, if necessary, the levels of the tubes, the volume indicated by B is read off; *f* is now turned to make B communicate with *g*, and *h* is opened. D is then raised, the gas driven from B into E, and again lowered, which operation is repeated several times. Finally, the soda-ley is again forced up until it reaches *h*, which is then immediately closed, and the tubes are again brought to the same level, C standing at the 100 division. If the whole apparatus has been airtight, and the acid been free from carbonic anhydride, the mercury will occupy the same height before and after treatment with the soda.

After driving the gas completely out of B by raising D, the analysis of any carbonate may be performed. An accurately weighed quantity of the substance, together with about 0.8 gram of aluminium, is dissolved in A, as before. The hydrogen completely expels the last traces of carbonic anhydride from the fluid. All the operator has now got to do is to measure the gas both before and after the action of the soda-ley. The difference represents the volume of the carbonic anhydride, which multiplied by 1.966 gives its weight in milligrams.

L. DE K.

**Apparatus for the Estimation of the Carbonic Anhydride of the Atmosphere.** By O. SCHULZ (*Chem. Centr.*, 1891, ii, 726—727;

from *Münchener med. Wochenschr.*, **38**, 641—643).—The apparatus consists of a 500 c.c. cylinder which is graduated for each 20 c.c. At each end it is drawn out. The lower orifice is connected, by means of an india-rubber tube, with a movable reservoir. The upper orifice is closed by a three-way tap. One of the openings of the latter communicates with the air, whilst the other is connected with a stout glass test-tube. The latter is closed by an india-rubber stopper, through one of the holes of which passes a tube drawn out to a capillary at its lower end and reaching to the bottom of the test tube, whilst the other is in connection with the three-way cock of the cylinder. In the test-tube is placed 20 c.c. of a millinormal soda solution, with phenolphthaleïn as indicator. In using the apparatus, the movable reservoir is raised until the water flows from it into the measuring cylinder up to the zero mark, the three-way tap being in connection with the atmosphere. The tap is then reversed so as to come into connection with the test-tube, and the reservoir is now lowered and air allowed to bubble through the soda solution until it is just colourless. The volume of air passed through may then be read off on the cylinder and compared with the weight of carbonic anhydride which exactly neutralises the soda.

J. W. L.

#### Estimation of Calcium in Phosphates by Glaser's Process.

By J. H. VOGEL (*Zeit. ang. Chem.*, 1891, 357—359).—The author, after carrying out a large number of experiments, has come to the conclusion that the original process recommended by Glaser gives the calcium too low. Excellent results are, however, obtained by using the modified process recommended by Jones (compare this vol., p. 99). Nothing is gained by first drying and separating the calcium sulphate from the filter before calcining it, but too strong a heat should be avoided.

If the phosphate contains much magnesium, there is a danger of this substance coming down with the iron and aluminium phosphates. In this case, Stützer's plan (as explained in Jones's article) must be adopted.

L. DE K.

**Volumetric Estimation of Zinc.** By L. BLUM (*Zeit. anal. Chem.*, **31**, 60—61).—The author has pointed out (Abstr., 1890, 1191) that in Donath and Hattensauer's method for the titration of zinc, the presence of manganese leads to incorrect results. Moldenhauer prefers to omit the tartrate, and to titrate the zinc in a simple ammoniacal solution, in which the precipitated ferric hydroxide is suspended. If, before making alkaline with ammonia and boiling, a little bromine water has been added to the solution, the manganese will be all precipitated with the iron, and will no longer interfere with the titration of the zinc. Moldenhauer's ammoniacal reagent (5 c.c. for 50 c.c. of solution) consists of 50 grams of ammonium carbonate, 50 grams of ammonium chloride, and 100 c.c. of ammonia in the litre.

M. J. S.

**Separation of Copper and Cadmium.** By J. S. C. WELLS (*Chem. News*, **64**, 294).—Copper and cadmium may be separated, in the absence of ammonium salts, in the following manner:—The

neutral solution containing the two metals is rendered colourless by treating with sodium thiosulphate; the addition of sodium carbonate then precipitates the cadmium as carbonate, which is filtered off. The copper is obtained as sulphide by boiling the filtrate with hydrochloric acid.

D. A. L.

**Assay of Aluminium and its Alloys.** By F. REGELSBERGER (*Zeit. ang. Chem.*, 1891, 360—363).—Although an experienced dealer can readily distinguish a good sample of aluminium from an inferior one by its appearance and some physical tests, a chemical analysis is more satisfactory. The impurities almost exclusively present are iron and silicon; but it is as well to also make a direct estimation of the aluminium, so as to make sure there can be no other impurities to speak of. The author now recommends the following process:—*Silicon*.—From 2 to 4 grams of cut-up metal is dissolved, with the usual precautions, in 50 c.c. of water containing 12—24 grams of potassium hydroxide. A platinum basin is used, and when the bulk of the metal has dissolved, a gentle heat is applied. The solution is acidified with hydrochloric acid, evaporated to dryness to render silica insoluble, and the residue treated in the well-known manner. *Iron*.—3 grams of the metal is treated in a flask with 50 c.c. of 40 per cent. aqueous potash and the action assisted by a gentle heat. 200 c.c. of dilute sulphuric acid (1.16 sp. gr.) is now added, and, after boiling to dissolve the iron, the liquid is rapidly cooled and titrated with permanganate. *Aluminium*.—This estimation has already been described (this vol., p. 102).

As regards alloys, the author found that the treatment with aqueous potash completely dissolves out the aluminium from its alloy with 6 per cent. of copper or 20 per cent. of nickel. Even an alloy of aluminium and silver may be so separated, but in this case treatment with nitrohydrochloric acid is preferable.

L. DE K.

**Volumetric Estimation of Alumina.** By C. F. CROSS and E. J. BEVAN (*J. Soc. Chem. Ind.*, 10, 202).—Lunge (Abstr., 1891, 365) contends that the authors are in error in supposing that the change of colour takes place when the proportion  $5\text{SO}_3 : 2\text{Al}_2\text{O}_3$  is reached, the real ratio being  $3\text{SO}_3 : \text{Al}_2\text{O}_3$ . The authors have repeated their former experiments with precisely the same results.

D. B.

**Volumetric Estimation of Alumina.** By G. LUNGE (*J. Soc. Chem. Ind.*, 10, 314—317).—Referring to the preceding abstract, the author states that he has repeated his former experiments, and has confirmed his results and those of all previous observers, according to which the change in the colour of methyl-orange occurs when  $3\text{H}_2\text{SO}_4$  has been used for  $1\text{Al}_2\text{O}_3$ .

D. B.

**Commercial Assay of Alums.** By M. KRETZSCHMAR (*Chem. Zeit.*, 14, 1223).—Five grams of the sample is dissolved in 500 c.c. of water, 50 c.c. is taken, and the acidity estimated by means of N/2 potash with methyl-orange as indicator. The alumina is then approximately estimated by continuing the titration after addition of phenolphthalein. Another 50 c.c. is now taken and boiled with a few drops

of bromine. After cooling, sodium acetate is added, and then a known volume of sodium phosphate of known strength, in order to completely precipitate the alumina and any ferric oxide. As the washing of the precipitate is troublesome and slow, the author prefers to estimate the excess of phosphate volumetrically, in the cold liquid, by means of uranium nitrate solution. As the precipitate also contains ferric phosphate, it is necessary to estimate the iron in another 50 c.c. by reduction with zinc and titration with permanganate. From the amount of alumina obtained by the titration with uranium is then deducted the quantity of alumina corresponding with the ferric oxide.

The process is convenient and rapid. As the amount of alumina is already fairly accurately known by the titration with phenolphthaleïn, the operator knows almost exactly how much uranium will be required to precipitate the excess of phosphate. A single titration will therefore be sufficient. If the alum contains ammonia, the greater part of this must be expelled by heating with excess of alkali.

L. DE K.

**Glaser's Process for the Estimation of Iron and Aluminium in Phosphates.** By T. MEYER (*Chem. Zeit.*, **14**, 1730).—A joint committee of German manure makers and agricultural analysts has reported in favour of Glaser's spirit method, instead of the conventional ammonia and acetic acid process. The weak point in Glaser's process is, however, his complete ignoring of the magnesia. Although magnesium sulphate is practically insoluble in absolute alcohol, it is far from insoluble in a mixture of alcohol and dilute sulphuric acid. Manures containing much magnesia always yielded, in the author's hands, a mixture of aluminium, magnesium, and ferric phosphates. Without a serious modification, Glaser's method is therefore not yet fit to become the recognised trade process. (Compare, however, R. Jones, this vol., p. 99.)

L. DE K.

**Estimation of Manganese in its Ores and Alloys.** By J. PATTINSON and H. S. PATTINSON (*J. Soc. Chem. Ind.*, **10**, 333—338).—The authors had occasion to repeat to a certain extent the investigations of Wright and Luff, and of Pickering, on the products obtained by heating hydrated manganese dioxide in air. Their experiments were made with manganosomanganic oxide of known composition, prepared either from crystallised manganese sulphate, or from ferromanganese containing about 82 per cent. of manganese. It is shown that (1) manganosomanganic oxide is only formed from these substances by ignition for about an hour at almost a white heat, in a gas muffle furnace or other apparatus in which the oxide is protected from reducing agents; (2) an ordinary red heat, such as is obtained over a Bunsen burner, is not sufficient to reduce the oxide to the state of  $Mn_3O_4$ , and that an error of  $1\frac{1}{2}$  per cent. of manganese too much may be made in this way if the percentage of manganese is calculated on the assumption that the product obtained is manganosomanganic oxide; (3) there is a risk in all cases when the oxides are heated over a gas blowpipe of obtaining an oxide containing less

oxygen than  $Mn_3O_4$ , probably due to the action of reducing gas permeating the heated platinum or entering the crucible under the lid, and that an error of as much as  $3\frac{3}{4}$  per cent. of manganese too little may be made in this way; (4) all determinations of manganese made by weighing the product yielded by igniting manganous carbonate or hydrated manganese dioxide in air are untrustworthy, unless the available oxygen in the product is also determined and taken into consideration.

The authors conclude from their investigations and experience, that, just as in the case of the estimation of iron, the volumetric methods of estimating manganese are far superior to the gravimetric methods, both as regards accuracy and ease of execution. The volumetric method described by one of the authors in 1879 (*Trans.*, 1879, 365), with some slight modifications of detail, is still used by the authors, who have reason to believe that it is now largely adopted by chemists.

D. B.

**Separation of Metals in Alkaline Solution by Hydrogen Peroxide.** By P. JANNASCH and R. NIEDERHOFHEIM (*Ber.*, 24, 3945—3947).—The method recently described by Jannasch and Franzek (this vol., p. 240) for the quantitative separation of manganese, cobalt, and nickel by treating the alkaline solution of their double cyanides with hydrogen peroxide, may with equal advantage be employed for the separation of manganese and zinc. The solution of manganese and zinc in 50 c.c. of water is mixed with a 10 per cent. solution of potassium cyanide (10 c.c.) and a similar amount of 25 per cent. potash solution, the whole being stirred until the precipitate first formed is redissolved. The manganese is then precipitated by the addition of hydrogen peroxide (50—60 c.c.), the mixture warmed for 15—20 minutes at  $100^\circ$ , and filtered. The filtrate is saturated with hydrochloric acid, evaporated to dryness, warmed at  $110$ — $115^\circ$  in the air-bath for half an hour, dissolved in very dilute hydrochloric acid, and filtered from any silica present. The zinc is then precipitated by sodium carbonate and weighed as oxide.

The analytical data given in the paper show that the method is capable of yielding accurate results.

H. G. C.

**Action of Alkaline Sulphides on Metals of the Iron Group.** By L. L. DE KONINCK and M. LEDENT (*Zeit. ang. Chem.*, 1891, 202—203).—It is a well known fact that when salts of nickel are precipitated with ammonium sulphide in excess, the filtrate has a dark-brown colour, due to some dissolved nickel. According to Lecrenier, this is owing to the ammonium sulphide used for testing nearly always containing polysulphide. The authors thought it would be interesting to try the behaviour of sodium or potassium polysulphide, not only on salts of nickel, but also on the other metals of the same group. Nickel gives a brownish-black, but very clear filtrate, just the same as when using ammonium sulphide. Salts of cobalt give a dirty, violet-brown liquid. With iron, the colour is at first a nondescript dark brown, but gradually changes into a lively green, resembling chromic sulphate or potassium manganate. One

of the authors recommended this reaction some time ago as a delicate test for iron. Manganese salts give no peculiar colour, and, like those of zinc, seem insoluble in an excess of the reagent. The authors will continue the investigation. L. DE K.

**Estimation of Chromium in Steel.** By T. W. HOGG (*J. Soc. Chem. Ind.*, 10, 340—341).—It has been objected that in Galbraith's process (Abstr., 1877, 357) the whole of the chromium is not dissolved by the sulphuric acid. This objection has most probably arisen from insufficient boiling with the strength of acid originally given (1 to 6), and the author therefore recommends the use of a stronger acid (1 to 3), and to go on boiling the solution vigorously for a few minutes after all decomposition has apparently taken place. The solution should be filtered through an asbestos filter, as with the use of a filter paper a small quantity of chromic acid is reduced. With regard to the gravimetric method of estimating chromium in steel, Arnold's process (Abstr., 1881, 646) gives the best results.

The method recommended by the author is based on the fact that a solution of chrome steel in sulphuric acid is highly coloured, the intensity of the coloration being greater as the percentage of chromium increases. Ferrous sulphate itself has a green tinge, but the colour intensity of chromium sulphate is so very much greater that the presence of even 0.1 per cent. is sufficient to give a decided increase to the depth of colour of the solution, and the presence of 0.35 per cent. will almost double it. D. B.

**Gas-volumetric Analysis.** By A. BAUMANN (*Zeit. ang. Chem.*, 1891, 332—339).—Lunge has tried to show that Baumann's new methods of quantitative analyses (this vol., p. 103) do not show any special advantages, and give inaccurate results. The author attempts to prove that Lunge is prejudiced against his process; that, in fact, for the estimation of small quantities of chromic acid and sulphuric acid, no better methods are to be found in the analytical text-books. New gas-volumetric processes for the estimation of arsenious and antimonious acids, and also for the alkalis, will shortly be published. L. DE K.

**Gas-volumetric Analysis.** By G. LUNGE (*Zeit. ang. Chem.*, 1891, 339—340).—The author remarks that, notwithstanding Baumann's pretensions as to the accuracy of his process, he still admits that in the estimation of chromic acid an error may occur of 0.98 per cent., and that in the estimation of sulphuric acid an error of even 1.74 per cent. is possible. The author thinks that more accurate results are obtained by the use of his gas-volumeter than by the azotometer, and points to the fact that his own processes have been used successfully for many years by hundreds of analysts. L. DE K.

**New Methods in Quantitative Analysis.** By A. BAUMANN (*Zeit. ang. Chem.*, 1891, 203—210, 328—332; compare this vol., p. 103).—The reaction between chromic acid and hydrogen peroxide may be taken advantage of in the estimation of lead, bismuth, and barium. With chromic acid, these metals form insoluble salts, which,



during a quantitative analysis, are generally collected on a weighed filter, and dried at 100–120°. The estimation is, however, much more easily performed by the author's mode of procedure.

*Estimation of Lead.*—If the liquid should contain free nitric acid, it is first mixed with excess of sodium acetate, then heated to boiling. Potassium dichromate is then added in slight excess. The precipitate is collected and washed, the filter spread out on a glass plate, and the precipitate washed down with dilute sulphuric acid into the outer chamber of the evolution flask. After some time, 5 c.c. of 2 per cent. solution of hydrogen peroxide is added, and the process conducted as previously described. 1 c.c. of gas = 0.0049766 gram of lead oxide.

*Estimation of Bismuth.*—The solution, which must be free from excess of nitric acid, is poured into a warm solution of potassium dichromate. The precipitate is washed by decantation with hot water, and dissolved in a little weak hydrochloric acid. After transferring it to the evolution flask, and adding 10 c.c. of dilute sulphuric acid, the operation is conducted as in the previous case. 1 c.c. of gas = 0.005192 gram of bismuthic oxide.

*Estimation of Barium.*—The solution is precipitated with ammonium chromate in the presence of ammonium acetate. The precipitate is washed first with a weak solution of ammonium chromate and finally with very weak ammonia. It is then treated like the bismuth precipitate. 1 c.c. of gas = 0.00342 gram of barium oxide.

The estimations may, of course, also be performed by adding a known quantity of potassium or ammonium dichromate, making up to a definite bulk, and estimating the excess of chromic acid in an aliquot part of the filtrate.

The author has also studied the action of iodine and its oxygen acids on hydrogen peroxide, and obtained results which, if confirmed by other analysts, will revolutionise quantitative analysis. A freshly prepared solution of iodine in potassium hydroxide, which is really a solution of potassium hypoiodite, when brought into contact with hydrogen peroxide, evolves one molecule of oxygen for every molecule of iodine, the products being water and potassium iodide. This fact has been made use of by the author for the estimation of free iodine. 40–50 c.c. of a solution of iodine in potassium iodide is placed in the outer chamber of the flask, and 5 c.c. of 5 per cent. hydrogen peroxide is put into the glass cylinder with a solution of potassium hydroxide (1 : 2). After mixing and shaking, oxygen will be evolved, and after about five minutes' cooling, its volume may be read off. 1 c.c. of gas = 0.01133 gram of iodine. As an instance of the applicability of the method, it may be used to estimate with great rapidity mineral, and even organic, acids, by bringing them in contact with a mixture of potassium iodide and iodate, and estimating the liberated iodine in the manner described.

L. DE K.

**Estimation of Tungsten in Rich Alloys and in Steel.** By R. NAMIAS (*Chem. Centr.*, 1891, ii, 728–729; from *Stahl. u. Eisen*, 11, 757–760).—For the estimation of tungsten in rich alloys, the author prefers to oxidise with bromine in a concentrated sodium hydroxide

or carbonate solution, the finely divided metal being digested in a porcelain dish for 1—2 hours near the boiling point of the solution. The solution is evaporated to dryness several times with hydrochloric acid, the tungstic anhydride is then dissolved from the silica with ammonia, the silica removed by filtration, and the tungstic anhydride again precipitated with hydrochloric acid, and the whole evaporated to dryness. It is then collected, washed with ammonium nitrate solution, and ignited.

The author prefers this method to that of Schneider, by which the tungstic anhydride and silica are fused with potassium hydrogen sulphate. But the mixture of tungstic anhydride and silica may be ignited in a current of hydrogen, and the loss (oxygen) calculated into tungstic anhydride.

In the case of tungsten steel, the metal is digested with hydrochloric acid, in the absence of air, and the insoluble metal collected and washed with ammonium nitrate. A little tungsten passes into the filtrate, and this is recovered by bringing the latter to dryness with hydrochloric acid, sugar is added, and the mass ignited. It is then treated with hydrochloric acid, filtered, washed with hydrochloric acid, ignited, and added to the main portion.

J. W. L.

**Analysis of Tin Ores.** By J. S. C. WELLS (*Chem. News*, **64**, 294).—The author finds that the decomposition of cassiterite can be satisfactorily effected by treating it with dilute hydrochloric acid in the presence of zinc and platinum, heating, and shaking frequently; when the decomposition appears to be complete, the remaining zinc and reduced tin are dissolved in hydrochloric acid and filtered from insoluble matter. The residue is tested for tin, and, if necessary, is treated again. The tin is estimated in the solution by any ordinary method.

D. A. L.

**Quantitative Separation of Metals of the Hydrogen Sulphide Group by means of a Stream of Bromine Vapour.** By P. JANNASCH and P. ETZ (*Ber.*, **25**, 124—127; see also this vol., p. 385).—*Separation of Bismuth from Lead.*—A solution of the mixed chlorides is precipitated with hydrogen sulphide, and the precipitated sulphides dried at 100° and heated in a weighed porcelain boat in a stream of air containing bromine. The bismuth bromide completely volatilises, whilst the lead bromide remains behind in a state of purity. The details of the experiments have already been given in a former paper. The special precautions for this separation are, not to allow the precipitated sulphides to remain too long in contact with hydrogen sulphide, and to complete the drying of the sulphides by gently heating them in a stream of dry air before passing the bromine vapour over them. The results obtained were good.

E. C. R.

**Application of Electrolysis to Qualitative Analysis.** By C. A. KOHN (*J. Soc. Chem. Ind.*, **10**, 327—329).—*Antimony.*—The precipitated sulphide is dissolved in potassium sulphide, and the solution, after warming with a little hydrogen peroxide to decolorise any polysulphides that may be present, electrolysed with a current

giving 1.5—2 c.c. of electrolytic gas per minute, when the antimony is deposited as metal upon the negative electrode. 1 part of antimony in 1,500,000 parts of solution may be thus detected.

*Mercury.*—This metal is best separated from its nitric acid solution on a small, closely-wound spiral of platinum wire. The solution is electrolysed with a current of 4—5 c.c. The deposited metal is removed from the spiral by heating the latter gently in a test tube, when globules of mercury form on the upper portion of the tube.

*Lead.*—This metal is precipitated either as peroxide at the anode from a nitric acid solution or as metallic lead at the cathode from an ammonium oxalate solution. In both cases a current giving 2—3 c.c. of gas is sufficient to effect the deposition in one hour. 0.0001 gram of metal in 150 c.c. of solution can be easily detected. With both solutions, this amount gives a distinct discoloration to the platinum spiral, on which the deposition is best effected. As a confirmatory test, the deposited metal is dissolved in nitric acid, and the solution treated with hydrogen sulphide.

*Copper.*—0.00005 gram of copper can be very readily detected by electrolysing an acid solution in the usual way. A platinum spiral is employed as the cathode, and the presence of the metal confirmed by dissolving it in a small quantity of nitric acid, diluting with water, and adding potassium ferrocyanide.

To detect the above metals in cases of poisoning, it is necessary to destroy the organic matter associated therewith in the usual way by means of hydrochloric acid and potassium chlorate. Urine can, however, be tested directly for these poisons. Mercury or copper may be detected by acidifying the urine with 2—3 c.c. of nitric acid and electrolysing as described. 0.0001 gram of metal in 30 c.c. of urine can be thus detected. Lead does not separate well as peroxide from urine, but if ammonium oxalate be added, the reaction is quite as delicate as in aqueous solution, and 0.0001 gram of lead can be thus detected. Antimony is best detected by precipitation as sulphide and electrolysing with a current of 1.5—2 c.c. In all cases, it is advisable to continue the passage of the current for about twice the time required in the case of aqueous solutions.

*Silver.*—This metal is precipitated as chloride, dissolved in potassium cyanide, and the solution electrolysed with a current of 1—1.5 c.c. A platinum spiral forms the anode, from which the silver deposit may be dissolved by nitric acid, and tested for by hydrochloric acid or hydrogen sulphide. 0.0001 gram of silver in 150 c.c. can be detected, and one hour is sufficient for the deposition.

*Gold* is deposited under similar conditions to silver from cyanide solutions. The deposit, which is somewhat dark coloured, can be dissolved in *aqua regia* and tested by the purple of Cassius test. 0.0001 gram of metal in 150 c.c. of solution can be detected without difficulty.

D. B.

**Separation of Antimony from Arsenic.** By F. A. GOOCH and E. W. DANNER (*Amer. J. Sci.* [3], **42**, 308—312).—A very exact process for the separation of arsenic from antimony consists in distilling their hydrochloric acid solution with ferrous sulphate; but the

conditions are such that the antimony in the residue must be determined gravimetrically. The authors have so arranged the process that the estimation of the antimony may be made by a rapid volumetric method, and this they have accomplished by substituting for the iron salt, which interferes with the direct volumetric estimation, another reducing agent, namely, hydriodic acid. After a great number of experiments, the authors now recommend the following process:—

The solution, which must not contain more than 1 gram of the mixed oxides of antimony and arsenic, is mixed with a slight excess of potassium iodide, diluted with hydrochloric acid to 100 c.c., and then saturated with hydrogen chloride, which is also passed during the subsequent distillation. The apparatus consists of a 250 c.c. flask, provided with a hollow glass stopper tightly fitted in a ground joint, the stopper itself being sealed on a large glass tube bent suitably to connect the interior of the flask with a condenser, whilst through the hollow stopper, and sealed into it, passes a smaller glass tube reaching nearly to the bottom of the flask. By means of this arrangement, a current of gas entering the smaller tube passes nearly to the bottom of the flask, and then out through the hollow stopper into the condenser, without coming in contact with joints, rubber, or cork.

The distillation is continued until fully one-half of the liquid has passed over, when the residue is quickly cooled. Sulphurous acid is added, and, after a little while, the excess of this reagent is destroyed by cautiously adding solution of iodine. After the addition of 1 gram of tartaric acid for every gram of antimony supposed to be present, the free acid is nearly neutralised with sodium hydroxide, and 10 c.c. of a solution of sodium hydrogen carbonate is then added. The antimony is now estimated by titrating the solution with N/10 iodine (compare Gooch and Gruener, this vol., p. 242).

L. DE K.

**Estimation of Organic Matters in the Atmosphere.** By J. ARCHAROW (*Chem. Centr.*, 1891, ii, 887; from *Arch. Hygiène*, **13**, 229—246).—The author aspirates measured quantities of the air under investigation through a tube, 39 cm. long and 1 cm. wide, which contains solution of potassium permanganate acidified with sulphuric acid. The tube is closed at one end, and fitted, like a wash-bottle, with two tubes, one of which passes to the closed end, whilst the other merely passes through the stopper, and serves as an exit. The inlet-tube is furnished with a cap at its further end, and is perforated so that the bubbles of air are reduced in size as far as may be. Three such tubes are employed in connection with one another for such experiments, and the apparatus is kept warm (about 43°). The potassium permanganate solution has a strength such that 10 c.c. = 0.02 milligram of oxygen, and it is titrated with oxalic acid.

J. W. L.

**Apparatus for Determining the Flashing Point of Heavy Mineral Oils.** By J. GRAY (*J. Soc. Chem. Ind.*, **10**, 348).—The apparatus designed by the author is an adaptation of the Pensky-Marten slide and stirrer to the Abel cup.

D. B.

**Indirect Estimation of Alcohol.** By T. P. BLUNT (*Analyst*, 1891, 221—223).—When alcohol is estimated by the indirect or evaporation method, the spirit gravity is calculated, either by Tabarié's formula, which requires the division of the specific gravity of the sample by that of the extract, or by Mulder's formula, which is based on the assumption that the spirit gravity is obtained by deducting from the gravity of the sample the extract gravity minus 1000. The author has satisfied himself that in the case of sweet wines and liqueurs, the last formula is the most accurate. L. DE K.

**Estimation of Fusel Oil in Spirits.** By J. TRAUBE (*Chem. Zeit.*, 14, 1410).—The author, in reply to Stützer and Reitmair, still believes his process with the stalagmometer to be the most suitable for analysts who have but seldom occasion to perform a spirit analysis. If it is thought advisable to concentrate the fusel oil, the author operates as follows:—500—600 c.c. of aqueous potash, of 1.24 sp. gr., is put into a suitable vessel, and, after warming to 60°, 100 c.c. of the sample, which must contain 80 per cent. of alcohol, is gradually added. The top layer which forms is removed, and the bottom layer is twice extracted with 20 c.c. of (50 per cent.) pure spirit, in order to remove the last traces of fusel oil. The united spirituous fluids are then tested as usual. L. DE K.

**Decolorisation of Wines.** By A. BOENTRÄGER (*Zeit. ang. Chem.*, 1891, 340—343).—Vogel (Abstr., 1891, 1557) has found that most Portuguese red wines require an enormous quantity of basic lead acetate, sometimes three times their volume, in order to render them sufficiently colourless and clear for examination in the polarimeter. The author thinks, however, that as a rule a much smaller quantity will suffice if the sample is first exactly neutralised with potassium or sodium hydroxide, and then concentrated to half its bulk. After addition of the lead solution, the original volume must be restored, and the mixture filtered through a dry filter. Should the reaction be alkaline, this shows that too much lead has been added, and, although it may be remedied by adding a drop of strong acetic acid, it is perhaps better to carefully repeat the whole operation. If the wine is suspected to contain cane-sugar, another portion is first heated for 15 minutes with 10 per cent. of hydrochloric acid, cooled, neutralised, and treated as before. L. DE K.

**Alkalimetric Estimation of Phenol.** By R. BADER (*Zeit. anal. Chem.*, 31, 58—59).—This can be very accurately effected by the use, as indicator, of symmetrical trinitrobenzene (m. p. = 122°), which substance gives a blood-red coloration with aqueous alkalis. The solution of the indicator is prepared by shaking a pinch of the pure substance with 50 c.c. of absolute alcohol and filtering. It should only be feebly yellow; it requires to be protected from light, and must be freshly prepared from time to time. The phenol solution to be titrated should not contain less than 20 grams in the litre. Only two or three drops of the indicator should be added to 50 c.c. of the phenol solution, and then normal soda, drop by drop, with constant

shaking, until the reddish-yellow colour no longer disappears. 1 c.c. of normal soda = 0.094 gram of phenol. The test analyses leave nothing to be desired.

This method does not seem to be applicable to the cresols, or to catechol.

M. J. S.

**Estimation of Phenols in Human Urine.** By RUMPF (*Zeit. physiol. Chem.*, **16**, 220—242).—The gravimetric estimation of phenol as tribromophenol in the distillate from acidified urine gives most inaccurate results; Koppeschaar-Beckurt's (*Arch. Pharm.*, 1886, 561) method of titration with potassium iodide is trustworthy. Paracresol also cannot be estimated as the tribromo-derivative. The substance obtained by adding bromine to the distillate from urine contains a pigment, and two other substances; one of these, insoluble in sodium carbonate solution, is dibromoparacresol; the other, which is soluble, is tribromophenol; it is, however, present in very small quantities. Hence it is inferred that paracresol is the most abundant substance of the phenol group in the urine.

W. D. H.

**Estimation of Cholesterol.** By J. LEWKOWITSCH (*Ber.*, **25**, 65—66).—In consequence of the communication of Obermüller (this vol., p. 248), the author publishes the methods he has for some time adopted for the estimation of cholesterol. The first method depends on the quantitative formation of a diacetate of cholesterol when the latter is boiled with acetic anhydride; the precipitate formed is collected, and washed with warm water until no longer acid, its amount being determined by boiling with alcoholic potash of known strength, and titrating back with standard acid. In the second method, which depends on the formation of a di-iodo-additive product, the cholesterol is dissolved in chloroform (50 c.c.), mixed with v. Hübl's solution of iodine and mercuric chloride in alcohol (25 c.c.), and the excess of iodine determined by thiosulphate. Good results are obtained by both methods.

H. G. C.

**Estimation of Glycerol by Alkaline Permanganate.** By W. JOHNSTONE (*J. Soc. Chem. Ind.*, **10**, 203—204).—For the estimation of glycerol in butter fat, the author some time ago recommended the process of Benedikt and Zsigmondy, but although it gives accurate results in certain instances, he now finds it to be quite inapplicable for the estimation of glycerol in butter fat or in oils containing soluble fatty acids. He is, therefore, more inclined to support Wanklyn's statement that the theoretical quantity of glycerol cannot be obtained from butter fat, and that the large amount of soluble fatty acids calculated as butyric acid, recently found by the author, goes a long way to support his "iso-glyceride" theory (compare *Abstr.*, 1891, 849 and 868).

D. B.

**Analysis of Crude Glycerols.** By F. FILSINGER (*Chem. Zeit.*, **14**, 1729—1730).—Apart from mixed products, there are three different kinds of commercial glycerol:—1. Saponification glycerol obtained from the works where the fats are saponified with milk of



lime. This brand is characterised by a straw-yellow or brownish colour, has an agreeably sweet taste, and emits no smell when rubbed on the hands. The reaction is generally neutral, unless a trace of free lime should be present. The specific gravity should not be less than 1.24, and its boiling point, in Gerlach's apparatus, not below 138°. The ash should not exceed 0.5 per cent., and neither basic lead acetate nor hydrochloric acid should cause much turbidity.

2. Glycerol intended for distillation, the product of the treatment of fats with sulphuric acid with or without pressure.—This generally has a burning, astringent taste, which often completely obscures the sweet one, an unpleasant odour, and leaves several per cent. of ash consisting chiefly of salt and calcium sulphate. Basic lead acetate causes a voluminous precipitate, sometimes large enough to gelatinise the liquid. Hydrochloric acid often gives a copious fusible precipitate chiefly consisting of fatty matter. The boiling point is generally below 125°. Even the best brands of this glycerol are about 10 per cent. lower in price than the worst saponification glycerols.

3. Glycerol recovered from Soap Leys.—This is the most impure of all, and has to be put through many preliminary processes before it can be distilled. When sold for that purpose, it should not contain more than 10 per cent. of ash and 10 per cent. of water.

As regards the assay of these samples, a chemical one is scarcely needed for the purer kinds of glycerol. All that is wanted is to take the specific gravity with some delicate instrument, and the boiling point in Gerlach's apparatus. As regards the chemical analysis of crude samples, the author recommends the permanganate method, originally described by Benedikt and Zsigmondy, as being the only process which has stood the test of many years' experience.

The author communicates another method which certainly has the merit of being a very simple one, and which has been in use for some years in a Hamburg laboratory. To estimate the water, 20 grams of the sample is put into a flask, provided with a ground stopper, and exposed for 10 hours to a temperature of 100°. The loss represents the water. To estimate the fixed impurities, 5 grams of the sample is heated in a flat-bottomed platinum dish up to 180°, which will cause the glycerol to completely volatilise without perceptible charring. To complete the analysis, the ash may be taken.

L. DE K.

**Detection and Extraction of Trehalose.** By E. BOURQUELOT (*J. Pharm.* [5], 24, 524—526).—For the rapid extraction of trehalose from fungi, the plant is treated, as soon as possible after gathering, with boiling 90° alcohol. The solution is distilled, and the residue is concentrated to one-tenth of the weight of fungus employed. The residue, cooled and filtered, is treated with 3 or 4 vols. of 90° alcohol to precipitate certain salts and nitrogenous substances; after filtering and distilling, the residue is evaporated to a rather thick syrup. Now a glass plate is lightly rubbed about its centre with a crystal of trehalose, and a drop of the syrup is placed on the part touched. If the fungus contains trehalose, crystallisation occurs almost immediately, and under a slight magnifying power shows octahedra. A few of these crystals transferred to the syrup will immediately induce



crystallisation in the liquids. In the case where the drop on the glass gives no crystals, trehalose is not present in the extract. J. T.

**Estimation of Aldehydic and Ketonic Oxygen.** By H. STRACHE (*Monatsh.*, **12**, 524—532; compare Fischer, *Ber.*, **17**, 572).—Phenylhydrazine and its salts are rapidly decomposed when treated with hot Fehling's solution, the whole of the nitrogen being evolved in the free state; on the other hand, the hydrazones which phenylhydrazine forms with aldehydes and ketones give no free nitrogen when treated in a similar way. On a knowledge of these facts, the author has based a method of determining aldehydic and ketonic oxygen, which is as follows:—A known quantity (0.1—0.5 gram) of the substance under investigation is added to an accurately weighed excess of phenylhydrazine hydrochloride and some sodium acetate, the solution is heated at 100° for several minutes, made up to a known bulk, and an aliquot part withdrawn and heated with an excess of Fehling's solution in a specially constructed apparatus, whereby the nitrogen evolved, representing unchanged phenylhydrazine, may be readily measured, and from this the amount of hydrazine used in forming the hydrazone, and subsequently the amount of ketonic oxygen [ $\text{CO:N}_2\text{H}_3\text{Ph}$ ] present, can be calculated. Determinations with opianic acid, acetophenone, acetone, and several other compounds show that the method is on the whole trustworthy, the results, in most cases, coming out a little too high. The author suggests that the method may, perhaps, be usefully employed in estimating the amount of an aldehydic or ketonic compound present in certain mixtures, and is extending his experiments in that direction. G. T. M.

**Separation of Resin from Fatty Acids.** By J. A. WILSON (*Chem. News*, **64**, 204—205).—The author regards the following as the best method of detecting resin:—Boil 10 drops of the dry fat or fatty acids with 5 c.c. of acetic anhydride, cool, and add 2 drops of sulphuric acid (sp. gr. 1.84); 1 per cent. of resin is sufficient to produce the reddish-violet colour.

For the separation he strongly advocates the use of Twitchell's process (this vol., p. 389). D. A. L.

**Detection of Tartaric Acid in Citric Acid.** By L. CRISMER (*Bull. Soc. Chim.* [3], **6**, 23—24).—The test depends on the fact that the yellow coloration produced by citric acid with ammonium molybdate in presence of hydrogen peroxide is unaltered on warming, whereas, if tartaric acid be present, a fine blue colour results. 1 gram of citric acid is added to 1 c.c. of ammonium molybdate solution (10 per cent.) and 2—3 drops of hydrogen peroxide solution ( $\frac{1}{4}$ — $\frac{1}{5}$  per cent.  $\text{H}_2\text{O}_2$ ), and is heated at 100° for three minutes. The reaction will detect 1 milligram of tartaric acid in 1 gram of citric acid. T. G. N.

**Estimation of Uric Acid.** By E. W. GROVES (*J. Physiol.*, **12**, 485—489).—A comparison between various methods of estimating uric acid leads to the conclusion that Ludwig's is the only trust-

worthy method, and that Haycraft's, with which it was especially compared, gives a very considerable and variable error.

W. D. H.

**Revision of Constants employed in the Analysis of Fats and Oils.** By R. T. THOMSON and H. BALLANTYNE (*J. Soc. Chem. Ind.*, 10, 233—237).—In the table of "constants in oil analysis," which accompanies the original paper, will be found collected that portion of the authors' results which they regard as useful in oil analysis.

*Iodine Absorption.*—In a previous communication (*ibid.*, 9, 587), it was shown that the variation in iodine absorption for different olive oils was greater than usually stated. Since then, it has been found to be  $\frac{1}{2}$  per cent. higher still, so that the iodine value ranges from that of Gioja (79 per cent.) to that of Mogadore olive oil (86.9 per cent.). The lowest figure for rape oil now stands at 99.1, and the highest at 105.6 per cent.

*Potash Neutralising Power.*—The figures respecting olive and rape oils are in close accord with those obtained by Archbutt, and do not represent such a great variation between each individual oil as those given by other observers. The limits of five specimens of linseed oil examined by the authors vary from 19.00 to 19.28, whilst those of nine specimens tested by other observers and recorded by Allen range between 18.74 and 19.52.

*Unsaponifiable Matter.*—Olive, refined cotton-seed, unrefined arachis, and linseed oils contain about the same proportion of unsaponifiable matter, so that the determination of that constituent in a sample, say, of olive oil would not serve to show any adulteration with either of the other three oils. But the presence of a considerable proportion of rape oil would tend to reduce the percentage of unsaponifiable matter. In marine oils, it is noteworthy that seal oils contain only about one-third of that contained in whale, cod, and menhaden oils.

*Specific Temperature Reaction.*—This is merely a modification in recording the results of Maumene's reaction with strong sulphuric acid. It consists in mixing 50 grams of water with 10 c.c. of sulphuric acid, each at 20°, and registering the highest temperature reached. The amount of water is best measured from a pipette at 15.5°, and the sulphuric acid should be run in from a pipette which will deliver the 10 c.c. in one minute. During the addition, the mixture should be vigorously stirred with the thermometer, and the highest temperature reached rapidly read off, as it only remains constant for a few seconds. The oil being tested in precisely the same manner, it is only necessary to divide the rise in temperature obtained with water into that obtained by the oil under examination. The answer is the specific temperature reaction compared with water as 100. The oils must be carefully weighed, and the acid added to them exactly as with water, except that even more vigorous stirring is necessary during and after the addition of acid. In this way, the rise in temperature in all the experiments made by the authors was fairly steady up to the highest point, at which the temperature remained constant for 50 to 60 seconds. In the cases of linseed, cod,

seal, and menhaden oils, the tests had to be made with a mixture of 20 grams of these oils and 30 grams of olive oil of known specific temperature reaction. As a rule, an oil having a high iodine absorption has also a high specific temperature reaction, but the rise is not always directly as that in the former. The reason the specific temperature reaction cannot be depended on with the same assurance as the iodine value is that it shows, in some cases, large variations for the same class of oils.

*Valenta's Test.*—The authors conclude from the results of their experiments that this test is surrounded with too many conditions to be of any practical value in the general analysis of oils.

*Oleic Acid.*—Although the percentage of free acid cannot be looked on as a constant, the authors consider that it serves a purpose in indicating to some extent the condition of the oil, and shows how little, if at all, a high free acidity affects the results of analysis.

D. B.

**Detection of Rosin Oil in Fatty and Mineral Oils.** By A. GRITNER (*Zeit. ang. Chem.*, 1891, 265).—The original process proposed by Storch has only a limited application, as, when sulphuric acid is added to the solution of the oil in acetic anhydride, train oil gives a red colour, whilst with cholesterol, present in many fatty oils, a violet one is produced. With dark-coloured mineral oils, the process fails altogether. Morawsky has modified the process by using a weaker acid of 1.53 sp. gr. Holde also made use of this acid (without the acetic), but of late, he has increased the strength to 1.624 sp. gr., as with the weaker acid the violet-red colour takes a long time to develop. The author found that when mixing rape oil with 1 per cent. of rosin oil, the adulteration may be easily detected by Holde's original or modified process; but Morawsky's method was still more delicate, as it showed  $\frac{1}{2}$  per cent. Black rosin oil did not give such a characteristic reaction as was observed with oils of a lighter colour. Train oils, before being tested, must be shaken with alcohol, and the alcoholic solution tested for the rosin oil. The reaction is best observed by allowing sulphuric acid to run down the side of the test tube; if rosin oil be present, a red or violet ring will form at the point of contact. With dark-coloured oils, Holde's method is the best; but with light-coloured train oils, Morawsky's process is preferable. As the reaction is also caused by colophony and shellac, the absence of these substances must be ascertained, and should they be present, it is necessary to saponify the oil and to test the unsaponifiable portion. For dark mineral oils, it is advisable to use an acid of 1.53 sp. gr., as the use of a stronger acid often causes a dark-yellow coloration, which renders the reaction less characteristic. Samples of rosin oil examined by the author gave the reaction with this acid just as plainly as with the 1.624 sp. gr. acid.

Schädler remarks that train oil mixed with syrupy phosphoric acid (5—1) gives a red colour, which gradually turns very dark, and is even noticed in mixtures containing only 1 per cent. of the oil. The author never succeeded in obtaining this reaction, and only noticed a dirty-brown colour. The reaction depends on the nature of the rosin oil. The author occasionally succeeded in detecting

an admixture of 5 per cent., but often could not find it at all. The phosphoric acid process is therefore not to be recommended.

L. DE K.

**Estimation of Fat in Milk.** By E. GOTTLIEB (*Landw. Versuchs-Stat.*, 40, 1—27).—The method is a modification of Röse's method (Abstr., 1888, 1135), and is carried out as follows:—A weighed amount of milk (about 10 grams) is poured into a measuring cylinder (40 cm. high), exactly divided into 0.5 c.c. The milk is shaken, first with 10 per cent. aqueous ammonia (1 c.c.), and again, after the addition of alcohol (95° Tralles; 10 c.c.); ether (25 c.c.) is then added. The cylinder is closed with a good cork, which has been wetted, and shaken a few times; light petroleum (25 c.c.) is added, the whole shaken and then left for at least six hours. The volume of the upper layer—a solution of fat in ether and light petroleum—is read, and most of it transferred by means of a fine pipette to a weighed flask, when it is evaporated at a low temperature, the flask and residue dried for 1—2 hours at 100° and weighed. If free from foreign matters, the fat remains as a perfectly clear oil. The weight of fat is calculated to the volume of extract, and to the weight of milk used. The chief difference between the above method and Röse's is that the volumes of milk and alcohol are about the same; the result being that the lower layer has a higher sp. gr., and that the upper layer contains hardly any alcohol.

The method may be further simplified. The milk is measured in a 10 c.c. Geissler pipette, instead of being weighed, and treated as described above. The total volume will be 70.5 c.c.; the upper layer being 53 c.c., and the lower 17.5 c.c. If the pipette used to remove the extract is so adjusted in a cork that it will take out just 51.5 c.c., the weight of residue in milligrams multiplied by 10 will give the percentage of fat in the milk. 10 c.c. of milk weighs 10.33 grams, so that 51.5 c.c. of extract (out of 53 c.c.) corresponds with 10 grams of milk.

With regard to the reagents, normal (or even weaker) potash and soda solutions may be used instead of ammonia. The light petroleum is obtained from commercial petroleum after treatment with sulphuric acid; it boils at about 80°; sp. gr. = 0.671 at 15°. The addition of light petroleum causes the complete separation of the water and a nearly complete separation of the alcohol, as well as the small amounts of matter dissolved in them. This is of much importance, as it is difficult to completely dry fat which contains a little water. The fat is always more or less slightly coloured, according to the feeding of the cow, and when kept readily crystallises. The fat extracted in the usual way is white, and will not readily crystallise. The fat gives no ash when burnt, and is free from nitrogen, and the only foreign substance which could be detected was a small amount of a glyceride of the formula  $C_{18}H_{36}O_2$  or  $C_{20}H_{40}O_2$ , which melts at 75—80°. The milk fat was further examined, and the following results obtained: sp. gr. = 0.936 at 13°; the sp. gr. of fat extracted in the usual manner being 0.943. A determination of the saponification number by Koettstorfer's method showed that 1 gram of the fat required 233 milligrams of

potash, which is near the number obtained by Koettstorfer for pure butter fat. The examination for volatile acids, by Reichert and Wollny's method, showed that, using 5 grams of fat, 28.7 c.c. of decinormal baryta was required; fat obtained by the extraction method requiring 29.8 c.c. The fat showed a normal refractive power.

The results of many analyses made by the method, together with results obtained by Soxhlet's method, and by weighing, show that the method is trustworthy, the results agreeing among themselves even better than those obtained by the other two methods. The method is, moreover, very easily and quickly carried out; the author did 30 analyses in one day, a number which is not to be considered as a maximum.

One advantage in the method is that, owing to alcohol being an antiseptic, the milk, after the addition of alcohol, may be kept in an ordinary bottle for six months before the analyses is completed.

The cylinder employed for the analysis may also be used for estimating fat in cream, butter, and finely-powdered cheese.

N. H. M.

**The Lactocrite compared with other Methods for Estimating Fat in Milk.** By L. F. NILSON (*Bied. Centr.*, 20, 789; *Chem. Zeit.*, 1891, No. 37).—The estimation of fat by this process does not always give trustworthy results, especially when more or less of the cream has been removed. This is due to the action of the glacial acetic acid and sulphuric acid mixture on the fat. Good results are obtained by the employment of a mixture of ethylidene-lactic acid (90 c.c.) and hydrochloric acid (10 c.c.); the results are then very exact whether the milk is rich or poor in fat.

Kaolin burnt at a high temperature is preferable to pumice, &c., for the absorption of liquids which have to be extracted, and it can always be used again after re-ignition. Adams' method gives concordant and trustworthy results, provided the paper is thoroughly extracted with ether before it is used. The areometric method gives too high results when employed with milk containing under 2.5 per cent. of fat; with milk richer in fat, the results are not uniform, being sometimes too high and sometimes too low. The method is not so good as the gravimetric method (when kaolin or filter-paper are used) or the lactocrite method (compare Abstr., 1890, 1346).

N. H. M.

**Milk Analysis.** By J. SZILASI (*Chem. Zeit.*, 14, 1202—1203; see this vol., p. 517).

**Soap Analysis.** By J. PINETTE (*Chem. Zeit.*, 14, 1442).—The author proposes a simple process for the analysis of soap, which, however, involves the use of Röse's milk fat burette.

The analysis is carried out as follows:—2 grams of the sample is dissolved in boiling neutral alcohol, and any insoluble matter is filtered off and tested if required. The filtrate is mixed with a drop of phenolphthalein solution, and if this should cause a reddening, the free alkali is estimated by means of N/10 sulphuric

acid. The liquid is now diluted with water to about 80 c.c. and put into the burette. After cooling, 10 c.c. of normal sulphuric acid is added, and then a mixture of equal parts of ether and light petroleum up to nearly the top division. After thorough shaking, the whole is allowed to remain quietly for some time, and the volume of the two layers is recorded. For the estimation of the fatty acids, 25 c.c. of the ethereal layer is pipetted off and evaporated in a tared basin. If desired, the residue may be dissolved in alcohol and titrated with N/10 alkali. To estimate the combined alkali, 25 c.c. of the aqueous layer is taken, and the excess of acid is titrated with N/10 soda. If the soap is supposed to contain both potash and soda, the alkalis may be weighed as mixed sulphates, and their relative proportion calculated from the sulphuric acid. Allowance must, of course, be made for the amount of soda introduced during the titration. The author claims the following advantages for his process:—The troublesome washing and collecting of the fatty acids is avoided, and there is plenty left of both ethereal and aqueous layers in case of an accident.

L. DE K.

**Assay of Bees' Wax for Vegetable Wax.** By H. RÖTTGER (*Chem. Zeit.*, 14, 1442—1443, 1473—1474).—The author criticises the various processes from time to time proposed. *Rabineaud's Method.*—1 part of wax is shaken with 50 parts of ether until the mass is thoroughly disintegrated. The insoluble portion is collected on a weighed filter, washed with ether, air-dried, and weighed. If the sample is pure, 50 per cent. remains undissolved; but, suppose it were all vegetable wax, only 5 per cent. of insoluble matter would be found. In regard to this process, Dullo remarks that it is not strictly the case that Japan or vegetable wax is always soluble in ether. There certainly are commercial vegetable waxes which are soluble in ether, but six different samples have been met with which behaved almost like pure bees' wax.

The author repeated the experiment, and tested four samples of Japan wax. Only one gave an unweighable trace of insoluble matter, but the other three completely dissolved in ether. As, however, other wax adulterants, such as stearic acid, paraffin, rosin, tallow, are also soluble in cold ether, the ether test cannot be depended on without further confirmation.

According to Dullo, the best way to test for Japan wax is as follows:—10 grams of the sample is boiled with 120 c.c. of water and 1 gram of soda and potash for one minute. If the comparatively easily saponifiable Japan wax is present, a soap is formed, which will more or less solidify on cooling. Pure wax is not affected. The author, however, finds this process to be absolutely useless, as pure wax also gives an emulsion.

According to Hager, Japan wax is sure to be present when the sample, which must be completely soluble in chloroform, has a sp. gr. above 0.980. According to Mène, the adulteration with Japan wax can be detected only by determining the sp. gr., not by taking the fusing or solidifying point.

According to another process by Hager, the fraud is easily detected



by boiling 0.5 gram of borax with 8 c.c. of distilled water and 0.4 gram of the sample. The milky liquid gradually separates into a clear, watery liquid with the wax floating on the top, but when Japan wax is present, it gelatinises more or less. The author tested this process with pure wax, and noticed a clear, watery layer, an emulsion, and a solid mass on the top. In testing for foreign admixtures such as Japan wax, tallow, stearic acid, and rosin, the process is generally very uncertain, as the separation in layers does not always take place as readily as one may think. Stearic acid or rosin may, however, be readily recognised. If the amount of the former is as little as 5 per cent., no solid cake is obtained, but, instead, a pasty emulsion, which, after a few days, separates slightly. In the case of rosin, the liquid also gets pasty, but does not separate at all. The author cannot, however, recommend the process for the detection of tallow or Japan wax, as it will only lead to serious mistakes.

A process lately recommended by the German Pharmacists Association is as follows:—If 1 gram of wax is boiled with 10 c.c. of water and 5 c.c. of sodium carbonate, the wax, after cooling, ought to float on a clear liquid; but if adulterated with Japan wax, stearic acid, or rosin, an emulsion is formed, which, even after remaining for a day, shows no inclination to separate. The author finds that when wax is adulterated with the said substances, or with tallow, a solid cake is obtained, besides an emulsion and a clear liquid. The emulsion is largest when rosin is present, and least in presence of tallow. In conclusion, the author states that by this process, 2 per cent. of Japan wax, stearic acid, or rosin may be detected by the abnormally large emulsion. Tallow does not show well under 5 per cent.

L. DE K.

**Salicylsulphonic Acid as a Test for Albumoses and Peptones.** By J. A. MACWILLIAM (*Brit. Med. J.*, 1, 1892, 115; compare Abstr., 1891, 872).—The precipitate produced by adding salicylsulphonic acid to heat-coagulable proteids (albumin, globulin, myosin, &c.), or to derived albumins (acid-albumin and alkali-albumin) does not dissolve on heating, but becomes coagulated. If it is added to a solution of a primary albumose (proto-albumose or hetero-albumose), the precipitate dissolves on heating, and reappears on cooling. If it is added to a solution of deuterio-albumose, this reaction does not take place unless a considerable amount of neutral salt, such as twice its bulk of saturated ammonium sulphate solution, is added also.

True peptone gives no precipitate, unless the solution is fully saturated with ammonium sulphate; the occurrence of this precipitate is stated to be a more delicate test for peptone than the biuret reaction. The precipitate readily dissolves on the addition of a little water or glycerol; it also clears up on heating, and reappears on cooling.

W. D. H.