APRIL, 1916. Vol. XLI., No. 481.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

AN ordinary meeting of the Society was held on Wednesday evening, March 1, in the Chemical Society's Rooms, Burlington House. The President, Mr. G. Embrey, F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

A certificate of proposal for election to membership in favour of Mr. F. T. Alpe was read for the second time; and a certificate in favour of Mr. Maurice S. Salamon, B.Sc. (Lond.), 79, Mark Lane, London, E.C., analytical and consulting chemist, was read for the first time.

Messrs. N. T. Foley and T. J. Hitchcock were elected members of the Society.

The following papers were read: "The Manufacture of English Chemical Filter-Paper," by E. J. Bevan, F.I.C., and W. Bacon, B.Sc., F.I.C.; "Pink Colour on the Surface of Margarine," by A. W. Knapp, B.Sc., F.I.C.; "A Rapid Method for the Estimation of Fat in Powders," by S. B. Phillips; and "A New Colour Reaction for Aloes," by C. E. Stacy.

Specimens were shown of laboratory porcelain ware manufactured by the Worcester Royal Porcelain Co., Ltd., Worcester.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Estimation of Caffeine in Coffee. G. Fendler and W. Stüber. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1915, **30**, 274-277; through *Chem. Zentralbl.*, 1915, ii, 1315.)—In addition to other methods which have been examined (*cf.* ANALYST, 1914, **39**, 394), the authors have investigated a method proposed by Burmann for the estimation of caffeine in coffee (ANALYST, 1910, **35**, 202) and find that this method does not yield accurate results. W. P. S.

Estimation of Free Alkali in Soap. F. H. Newington. (*J. Soc. Chem. Ind.*, 1916, **35**, 95-96.)—The usual method of dissolving the soap in alcohol, filtering and titrating the free alkali is unsatisfactory in many respects; the author therefore prefers a method based on the salting out of the soap from its aqueous solution by means of sodium sulphate and titrating the free caustic alkali with standard acid, using silver nitrate as a spot indicator. Ten grms. of soap from the interior of the sample are weighed out into a wide-necked flask; 50 c.c. of freshly-boiled hot distilled water are added, the flask is lightly corked, and the whole heated until the soap is dissolved. Fifty c.c. of a hot saturated solution of sodium sulphate are added, the liquids thoroughly mixed by shaking, the contents of the flask transferred to a narrow-mouthed separating-funnel, and the flask is rinsed out into the separator with a further 5 c.c. of sodium sulphate solution. The funnel, lightly corked, is placed upright in a hot-water oven until the soap separates out on the surface. The bottom layer, which contains the free alkali, is run off and titrated with $\frac{N}{10}$ sulphuric acid, using a 5 per cent. solution of silver nitrate as a spot indicator on a porcelain plate. As long as free hydroxide is present a brown stain of silver oxide is produced; carbonates, silicates, etc., do not interfere. If an appreciable amount of free hydroxide be found, the soap residue may be washed in the funnel with a further quantity of 50 c.c. of sodium sulphate solution and a second titration performed. The sodium sulphate and silver nitrate must be tested for neutrality. The results are accurate and the method is sufficiently delicate to permit of the estimation of 0·01 per cent. of caustic alkali when added to a neutral soap. J. F. B.

Some Japanese Essential Oils. S. Uchida. (*Bull. Forest Exper. Stat.*, Tokyo, 1915.)—*Hinoki Oil.*—Both leaves and wood of the hinoki tree (*Chamæcyparis obtusa*), a valuable timber grown extensively in Japan, contain an essential oil. The oil, obtained by dry distillation of the wood, is brown in colour, has an empyreumatic odour, and the yield is about 2·4 per cent. of the weight of the wood. When rectified by steam distillation and subsequent treatment with dilute sodium carbonate solution the oil has a sp. gr. at 15·5° C. of 0·8821, and $[\alpha]_{D\,15^\circ C.} -50{\cdot}61^\circ$.

The fraction of the oil distilling below 170° C. amounts to about 60 per cent. of the total, and consists mainly of α-pinene; it forms a good substitute for turpentine oil.

Sansho Oil.—This essential oil is obtained by steam distillation of the berries of a wild shrub (*Xanthoxylum piperitum*) growing in Japan, the yield being about 5·7 per cent. The oil begins to boil, under ordinary pressure, at 110° C., the boiling-point of the last fraction being 239° C.; the fraction distilling between 176° and 186° C. constitutes 80 per cent. of the oil. The sp. gr. at 15·5° C. is 0·8504, $[\alpha]_{D\ 20^\circ C.} + 46{\cdot}5^\circ$, $[n]_{D\ 20^\circ C.}$ 1·46, acid value 3·3, ester value 19·28, ester value of the non-aldehyde constituent of the oil 23·23. The composition of the oil is approximately as follows: Free acid (chiefly palmitic acid) 2·0, aldehyde 15, ester (as acetic ester of $C_{10}H_{17}OH$) 5·7, free alcohol ($C_{10}H_{17}OH$) 1·1, and terpene (chiefly dipentene) 77 per cent. The oil has an agreeable odour, and can be used in the preparation of perfumes, confectionery, etc.

Sugi Oil.—The sugi (*Cryptomeria japonica*) is a coniferous tree indigenous to Japan; the green leaves of the tree yield 0·7 per cent. of volatile oil boiling between 155° and 350° C. The oil has a sp. gr. at 15·5° C. of 0·9217, $[\alpha]_{D\ 20^\circ C.} + 19{\cdot}29$, $[n]_{D\ 20^\circ C.}$ 1·4895, acid value 1·0, ester value 6·56, and ester value after acetylation 14·35; the relative proportions of the constituents are approximately as follows: Free acid (as acetic acid), 0·1; free alcohol (as $C_{10}H_{17}OH$), 3·14; ester (as caprylic ester of $C_{10}H_{17}OH$), 3·28; terpene (chiefly α-pinene), about 34; sesquiterpenes, about 30; a sesquiterpene alcohol ($C_{15}H_{25}OH$), 12; diterpene (α-cryptomerene), about 18 per cent.
W. P. S.

Estimation of Essential Oils in Vermouth. X. Rocques. (*Ann. Chim. anal.*, 1916, **21**, 35-38.)—French vermouth, which is made by macerating the plants with a white wine softened by the addition of liquorice wine and brought to an alcoholic strength of 18 per cent., is shown by the author's experiments not to contain any appreciable proportion of essential oils. The volumetric method of estimating essential oils by determining the iodine value is not applicable to spiced or aromatic wines, since these invariably contain a certain proportion of sulphurous acid.
C. A. M.

Analysis of Waxes, with Special Reference to Beeswax and Wool-Wax. F. W. Richardson and G. A. Bracewell. (*J. Soc. Chem. Ind.*, 1916, **35**, 160-163.) —*Beeswax:* Unsaponifiable matter was separated by evaporating 5 grms. of the wax with 50 c.c. of $\frac{N}{2}$ alcoholic potassium hydroxide solution nearly to dryness, and extracting the mass with successive large volumes of hot petroleum spirit. The unsaponifiable matter thus extracted from three English waxes and one foreign wax gave the following values: Iodine value (Hübl), 5·42 to 10·04; butyro-refractometer reading at 75° C., 20·9 to 22·00; and refractive index at 75° C., 1·4383 to 1·4392. The presence of ceresin (11·6) and paraffin wax (9·6) would be indicated by the butyro-refractometer readings. The saponifiable matter from the waxes gave the following results: M.-pt., 50·0° to 58·4° C.; butyro-refractometer reading at 50° C., 30·3 to 32·5; refractive index at 50° C., 1·4454 to 1·4470; calculated butyro-refractometer reading at 75° C., 14·8 to 17·0; and iodine value (Hübl), 7·7 to 14·06. The presence of tallow would be indicated by these tests, but the most reliable test for glycerides is to estimate the glycerol, and after making allowance for any other reducing substance, to multiply the result by 10. Before estimating the glycerol it is necessary to extract the sample repeatedly with boiling water until the concentrated filtrate no longer reduces Fehling's solution. Five grms. of the purified mass are boiled with 50 c.c. of $\frac{N}{2}$ alcoholic potassium hydroxide solution, and the mixture heated on the water-bath with successive additions of water to expel all alcohol. Finally, the pasty mass is neutralised with acetic acid, treated with a slight excess of basic lead acetate, and filtered, and the precipitate washed with cold water. The filtrate is freed from lead by means of sulphuric acid and concentrated, and the glycerol estimated by the bichromate method. Rosin and stearic acid, when present, may be estimated by treating the wax with hot carbon tetrachloride, neutralising the solution with alkali, and extracting glycerides, myricyl-alcohol, etc., by means of boiling petroleum spirit. The fatty acids are liberated from the aqueous soap solution, and their refractive index and iodine value determined. In the presence of rosin both values will be high. After separation of free cerotic acid (practically insoluble in 50 per cent. alcohol) and resinic and stearic acids as described, saponification of the residual portion will yield the fatty acids from glycerides, such as Japan wax and tallow. In Buisine's method of measuring the hydrogen evolved on heating the wax with potash lime, it was proved that oleic acid did not yield hydrogen at 250° C., but that the glycerol liberated by the alkali from any glyceride in the wax gave off a large volume of hydrogen mixed with methane. The presence of stearin in a wax would therefore vitiate the accuracy of the results given by this method. With regard to the acetyl value, three genuine samples of beeswax yielded unsaponifiable matter with acetyl values of 91·14, 109·7 and 94·52 respectively, and the variations would thus be too great to allow of the detection of less than 10 per cent. of adulterant. *Wool-wax :* The unsaponifiable matter is best extracted by saponifying the wax with strong alcoholic potassium hydroxide solution, removing the alcohol, treating the residue with a mixture of boiling alcohol and water (1 : 1), and shaking the hot liquid with petroleum spirit in a separating funnel. About seven extractions are required to separate all the unsaponifiable matter. Five samples of pure and commercial wool-fat yielded about 42 per cent. of unsaponifiable matter, with an iodine value 40·38 to 50·49; refractive index at 50° C., 1·4895 to 1·4950; and acetyl saponification value, 129·2 to 142·8. The saponifiable matter contained on the average 2 per cent. of free fatty acids, and 56 per cent. of neutral esters. C. A. M.

Zingiberol: A New Sesquiterpene Alcohol occurring in Essential Oil of Ginger. B. T. Brooks. (*J. Amer. Chem. Soc.*, 1916, **38**, 430-432.)—From 150 grms. of ginger oil, freed from terpenes and sesquiterpenes, there was obtained about 42 grms. of oil distilling between 130° and 160° C. at 14 mm. pressure. By repeated fractionation, this crude fraction yielded 24 grms. of oil distilling between 154° and 157° C. at 14·5 mm. Combustion gave 81·2 C, 11·6 H. Calculated for $C_{15}H_{26}O$, 81·08 C, 11·71 H, and for $C_{15}H_{24}O$, 81·81 C, 10·90 H. The substance is neither a ketone nor an aldehyde, but appears to be a tertiary alcohol. It is the only constituent of ginger oil possessing the peculiarly characteristic fragrance of ginger. Sodium in dry ether readily forms a sodium compound with it and, on gently heating with potassium hydrogen sulphate, water is split off, yielding zingiberene or *iso*-zingiberene, $C_{15}H_{24}$. On treating the alcohol with hydrogen chloride or bromide in glacial acetic acid solution and allowing to stand for two days, there are deposited crystals of isozingiberene dihydrochloride or dihydrobromide. G. C. J.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Agglutination Test as a Means of Studying the Presence of *Bacterium Abortus* in Milk. L. H. Cooledge. (*J. Agric. Research*, 1916, **5**, 871-875.)—The following procedure was adopted: Antigen was prepared for the test by growing a culture of *B. abortus* on ordinary agar for forty-eight hours. The growth was then washed off with a solution containing 0·9 per cent. of sodium chloride and 0·5 per cent. of phenol, the suspension filtered through paper and standardised, so that the turbidity compared with tube 1·5 of McFarland's nephelometer. Four c.c. of this suspension were then placed in each of a number of small test-tubes, which then received the addition of 0·1 c.c., 0·05 c.c., 0·025 c.c., 0·01 c.c., and 0·005 c.c. of milk respectively. In this way dilutions from 1 : 40 to 1 : 800 were obtained. The turbidity due to the whole milk added did not interfere with the reading of the reaction. A pure culture of *B. abortus* introduced into a cow's udder caused the appearance of agglutinins in the milk. In every case in which *B. abortus* was found to be present in a milk by animal inoculation tests the agglutinins for this organism were also found, but the bacterium was not found in every case in which the presence of agglutinins was demonstrated. If *B. abortus* is, in the future, found to be pathogenic for human beings, the above test may be of value in safeguarding milk supplies.
W. P. S.

Quantitative Estimations of Glyoxalase in Blood. H. W. Dudley. (*Biochem. J.*, 1915, **9**, 253-259.)—The enzyme glyoxalase converts α-ketonic aldehydes into α-hydroxyacids according to the general equation:

$$R.CO.CHO + H_2O = R.CHOH.COOH$$

(Dakin and Dudley, *J. Biol. Chem.*, 1913, **14**, 155, 423; **15**, 463; 1914, **16**, 505.) This enzyme rapidly converts methylglyoxal into lactic acid, and is, therefore, most probably of importance in the intermediary metabolism of carbohydrates. On account of the difficulty of the quantitative extraction of lactic acid from liquids containing proteins and of its determination, methylglyoxal is not employed as the substrate, phenylglyoxal being used instead. The ease with which the optically active mandelic acid thus produced can be extracted from digestion mixtures containing protein and its high optical activity renders quick estimations of the activity of glyoxalase preparations possible. The method consists in allowing 5 c.c. of defibrinated blood to act upon 10 c.c. of a 1·2 per cent. solution of phenylglyoxal-hydrate at 37° C. in presence of chalk for twenty to twenty-four hours. Phenylglyoxal displays distinctly antiseptic properties, thus rendering the addition of toluene unnecessary. After completion of the reaction 25 grms. of ammonium sulphate are added to the mixture, which is then heated in a boiling-water bath for three or four minutes, thereby precipitating the protein. After cooling 12 c.c. of 50 per cent. phosphoric acid are added, and the liquid allowed to stand, with frequent shakings, for half an hour. The protein precipitate is filtered off and washed with 40 per cent. ammonium sulphate solution. The clear filtrate is extracted with small quantities of ether and the mandelic acid separated as a crystalline residue by distilling off the ether. From the optical activity of this residue the amount of *l*-mandelic acid formed can be estimated, this being a measure of the glyoxalase content of the blood. In descending order of glyoxalase activity the species so far examined arrange themselves thus: dog, ox, rabbit, sheep, pig. The glyoxalase of the blood is remarkably constant in different animals of the same species under normal conditions. Attention is drawn to the observation that animals displaying high glyoxalase values are those having high sugar tolerance, while those with low glyoxalase activities have less well-developed powers of assimilating sugar. H. F. E. H.

Improved Hæmin Test for Blood. W. Beam and G. A. Freak. (*Biochem. J.*, 1915, **9**, 161-170.)—The following test is very sensitive, even with blood-stains twelve years old, and with blood heated at 110° C. or mixed with earth or iron-rust. A small quantity of the suspected material is placed in a tube a few mm. in diameter and about 35 mm. in length, a few drops of acetic acid containing from 0·01 to 0·1 per cent. of sodium chloride are added, and a very fine cotton thread is inserted in the tube, so that its upper end is near the top of the tube and the lower end reaches to the bottom of the liquid; the thread should be in contact with the side of the tube, to which it readily adheres when moistened with a drop of the liquid. The tube is then placed in a vertical position, and hæmin crystals usually appear near the top of the thread within about one hour; they gradually increase in size, and, after some hours, can be seen with a good hand lens. There is no advantage in substituting other salts for the sodium chloride, and even the latter is not necessary when dealing with unwashed blood-stains. W. P. S.

Studies of the Formation and Translocation of Carbohydrates in Plants. I. The Carbohydrates of the Mangold Leaf. W. A. Davis, A. J. Daish, and G. C. Sawyer. (*J. Agric. Sci.*, 1916, **7**, 255-326.)—In the present paper the methods described previously for the estimation of carbohydrates were employed (ANALYST, 1913, **38**, 504, 591; 1914, **39**, 312, 555). The results are calculated on the total vacuum-dried material, cane-sugar being determined from the increase of cupric reducing power after inversion with citric acid, since the results obtained by the change of polarisation on inversion with invertase were always high, especially when working on leaves or mid-ribs (see following abstract).

For full details of the very numerous analyses recorded the original paper must be consulted, the general results being summarised as follows: Starch is entirely absent from the leaf after the very earliest stages of growth. As soon as the root begins to develop so that the sugar formed in the leaf can be translocated to it, starch disappears almost entirely from the leaf. Maltose is absent from leaf, mid-ribs, and stalks at all stages of growth and at all times of night and day. During the early stages of growth of the mangold, when leaf formation is the principal function, cane-sugar is present in the leaf tissue in excess of the hexoses. Later in the season, when sugar is being stored in the root, the reverse is true, hexoses largely predominating in the leaf. In the mid-ribs and stalks the hexoses always predominate greatly over the cane-sugar, and vary widely in amount during the day and night, and throughout the season, whilst the cane-sugar remains practically constant. In passing from leaves to mid-ribs, from mid-ribs to the tops of stalks, and from the tops of stalks to the bottoms, the ratio of hexoses to cane-sugar steadily and rapidly increases. As the season advances the predominance of the hexoses in leaf, mid-ribs, and stalks becomes more and more marked. The facts brought forward can apparently be best explained by Brown and Morris's view that cane-sugar is the primary sugar formed in the mesophyll of the leaf under the influence of the chlorophyll; it is transformed into hexoses for the purpose of translocation. This transformation occurs in the veins, mid-ribs, and stalks, the proportion of hexoses increasing more and more as the root is approached. The sugar enters the root as hexose, and is therein reconverted into cane-sugar; once in this form it cannot leave the root until it is put under contribution for the second season's growth. As regards the mechanism by which cane-sugar is synthesised from the hexoses, it is improbable that this change is effected by invertase by a process of reversible zymo-hydrolysis, the entire absence of invertase from the root being against this view. Pentoses form only a small proportion of the total sugars in the tissues; they are probably formed from the hexoses, and appear to be precursors of the pentosans. H. F. E. H.

Studies of the Formation and Translocation of Carbohydrates in Plants. II. The Dextrose-Lævulose Ratio in the Mangold. W. A. Davis. (*J. Agric. Sci.*, 1916, **7**, 327-351.)—The main purpose of the present paper is to show that, whilst it is possible to take the actual polarimetric readings very accurately (the probable error did not exceed 0·005°), the values are falsified, in the case of most plant material, by the presence of optically active substances other than sugars, which are not completely precipitated by the basic lead acetate (or other clarifying substance) used to purify the solutions. Typical substances of this kind are amino-acids and amides, such as glutamic acid and glutamine, aspartic acid and asparagine. The first three of these have a pronounced positive rotation, which is greatly enhanced by acids, whilst asparagine is lævo-rotatory in aqueous solution and dextro-rotatory in acid solution. The effect of these and other impurities on the results obtained for the dextrose-lævulose ratio in plant material has not hitherto been taken into account.

These impurities occur in the leaves, but are much more abundant in the mid-ribs and stalks. In the leaves the dextrose and lævulose appear to be present in approximately equal amount, as would be expected if they were formed from cane-sugar by inversion. When the ratio $\frac{D}{L}$ departs from unity it is probably owing to the presence of a dextro-rotatory impurity (glutamine ?), which increases the amount of dextrose apparently present; but at certain times of the day a lævo-rotatory impurity seems to predominate, so that the ratio $\frac{D}{L}$ becomes less than unity. In the mid-ribs and stalks, especially at the bottoms of the latter, the dextrose always appears to be in very large excess as compared with the lævulose; this is probably due to the proportion of the dextro-rotatory impurity being relatively greater in these parts than in the leaf, as is shown by the divergences between the polarisation and reduction values of saccharose being far greater. The apparent fluctuations in the ratio of dextrose to lævulose are probably due to fluctuations in the optically active impurities rather than to variations in the sugars themselves. The fluctuations of the apparent dextrose and apparent lævulose take place more or less regularly during the twenty-four hours; this points to a regular variation in the optically active impurities. In the leaves the values of cane-sugar obtained by the double polarisation method are almost always higher than the reduction values; in the stalks, however, they are sometimes very high and sometimes very low. This is probably due to the presence of at least two different optically active substances at different times of the day. Until more reliable results can be obtained for the true dextrose and lævulose by methods which are independent of the polarimetric data, it seems justifiable, from the results brought forward, to assume that the dextrose and lævulose exist in the leaves and stalks as invert sugar, and travel in nearly, if not exactly, equal proportions to the root, where retransformation into cane-sugar occurs. It is impossible in the present state of our knowledge to draw any conclusions from the proportion of apparent dextrose or lævulose in plant tissues as to whether either of these sugars is better adapted than the other to tissue formation or to respiration. All such conclusions hitherto drawn are valueless, because the analytical methods at present existing do not give true values for these sugars. H. F. E. H.

Studies of the Formation and Translocation of Carbohydrates in Plants. III. The Carbohydrates of the Leaf and Leaf-stalks of the Potato. W. A. Davis and G. C. Sawyer. (*J. Agric. Sci.*, 1916, **7**, 352-384.)—In addition to cane-sugar, invert sugar, pentoses, and pentosans, "soluble starch," or dextrin, and true starch were estimated in the leaf and leaf-stalks. It was found that at certain times of the day the dried potato leaf obtained after the complete extraction of sugars and other substances soluble in 80 per cent. alcohol, still contained large quantities of a substance possessing a high dextro-rotation. This material proved to be soluble in water, and on treatment with taka-diastase yielded a mixture of maltose and dextrose. The true starch in the leaf was estimated by treating with taka-diastase, the leaf material remaining after extraction with water, as described in a previous paper (ANALYST, 1914, **39**, 312). Contrary to the finding of Brown and Morris (*J. Chem. Soc.*, 1893, **63**, 604), maltose was invariably absent from the potato leaf and also from the leaves of other plants, which form much starch in the leaf. The degradation of starch in the leaves is probably affected by a mixture of enzymes, similar to those of *Aspergillus oryzæ* (taka-diastase); maltose is always in relative excess, so that the starch is degraded completely to dextrose. The series of changes is therefore:

$$\text{Starch} \longrightarrow \text{dextrins} \longrightarrow \text{maltose} \longrightarrow \text{dextrose}.$$

In the potato leaf when the tubers are beginning to develop the principal sugar present is cane-sugar; its amount increases from sunrise up to 2 p.m., following approximately the curve of temperature. It then falls during the rest of the day and night. The rise and fall are both linear. The hexoses are present in the leaf in very small amounts—generally less than 1 per cent. of the total dry weight of the leaf. The starch is apparently formed from the hexoses. Directly the amount of cane-sugar has reached its maximum the hexoses begin to increase in the leaf, owing apparently to hydrolysis of the cane-sugar to invert sugar. At the same time, "soluble starch" (or dextrin) is first detected in the leaf, and its amount increases regularly up to 6 p.m. At 6 p.m., two hours before sunset, the true starch in the leaf reaches a maximum value, far greater than any previous value during the day. The starch and "soluble starch" subsequently fall rapidly until between midnight and 2 a.m. the amount left is exceedingly small (0·2 per cent.). The starch is apparently converted directly into hexose (dextrose), the amount of which increases in the leaf. In the stalks reducing sugars predominate greatly over cane-sugar, in spite of the fact that in the leaf the latter is in excess. As in the mangold, it is probable that cane-sugar is the first sugar formed in the leaf, and that it is hydrolysed by invertase in the veins, mid-ribs, and stalks, for the purpose of translocation. As in the mangold, the true proportions of dextrose and lævulose cannot be determined in the leaves and stalks owing to the presence of soluble optically active bodies, which vitiate the polarimetric data. It is shown that the presence of these impurities also falsifies the results obtained for cane-sugar by the double polarisation method. The fluctuations of the "apparent dextrose" and "apparent lævulose" in the leaf really indicate variations in other optically active bodies, rather than variations in the hexoses, which are perhaps present mainly as invert sugar. H. F. E. H.

Reaction and Calcium Content of Milk as Factors in the Coagulation Process. T. H. Milroy. (*Biochem. J.*, 1915, **9**, 215-228.)—The two main factors which govern the coagulant action of rennin on caseinogen are the $[H^{\bullet}]$ and the calcium content of the medium. The method employed for the determination of the hydrogen ion concentration $[H^{\bullet}]$ was the electrometric one as described by Sörensen and others (see also Milroy, *Quart. J. Exp. Physiology*, 1914, **8**, 141). It was found that during the course of rennin action there is no change in the $[H^{\bullet}]$ of milk, either in the earlier stage or in the actual separation of the clot. The addition of an alkali oxalate to milk lowers, while that of calcium chloride raises the $[H^{\bullet}]$. Fresh milk which has been subjected to a temperature slightly below boiling-point for one hour shows a rise in $[H^{\bullet}]$ and a fall in the calcium content. Such milk is only very slowly acted upon by rennin. The coagulability of heated milk may be raised either by the addition of calcium chloride or by raising the $[H^{\bullet}]$. The former does not act simply by raising the $[H^{\bullet}]$, nor the latter from its effect upon the soluble calcium content. The acid precipitation zone of caseinogen lies on the acid side of the rennin zone of action, but the latter gradually approaches the former as the calcium content of the mixture is lowered, so that in all probability the latter is an extension of the former towards the neutral point. Calcium chloride, apart from its effect upon the $[H^{\bullet}]$, increases the activity of the rennin ferment from the beginning of the digestion process. H. F. E. H.

Volumetric Estimation of Total Sulphur and Sulphates in Small Quantities of Urine. J. C. Drummond. (*Biochem. J.*, 1915, **9**, 492-507.)—The benzidine method is found to be quite satisfactory for the estimation of inorganic and total sulphates in urine, as well as for the estimation of total sulphur in biological material after oxidation of the latter. For the estimation of inorganic sulphates, 5 c.c. of the urine are acidified with 0·5 c.c. of dilute (1 : 3) hydrochloric acid, and mixed with 20 c.c. of Raschig's benzidine reagent. After five minutes the precipitate is collected on a small pulp-filter, and the precipitating vessel and precipitate are washed twice with 2 to 3 c.c. of a saturated solution of benzidine sulphate in distilled water, taking care not to suck the precipitate dry. The precipitate, pulp, and porcelain disc, and the washings of the funnel are then transferred to the precipitation vessel. The suspension of benzidine sulphate is then titrated at boiling-point with $\frac{N}{50}$ potassium hydroxide, using phenolphthaleïn or neutral red as indicator. If the precipitate be sucked dry, substantial flakes of benzidine sulphate form, which cannot be directly titrated. They can, however, be dissolved in an excess of hot alkali hydroxide, the excess of which can then be titrated with standard acid.

Total sulphates are estimated by hydrolysing the ethereal sulphates by boiling 5 c.c. of urine with 0·5 c.c. of dilute (1 : 3) hydrochloric acid for thirty minutes, cooling the solution, adding 20 c.c. of the benzidine reagent, and proceeding as above described.

For the estimation of total sulphur, 2 c.c. of urine are evaporated to dryness with 0·5 c.c. of Benedict's reagent (*J. Biol. Chem.*, 1909, **6**, 363), and the residue is ignited over a spirit-lamp, cooled, and warmed with 1 c.c. of dilute hydrochloric acid. When solution is complete, the solution is evaporated to dryness, the residue taken up in 1 c.c. of water, 10 c.c. of benzidine reagent added, and the analysis completed as described.

Comparison of results obtained by the above method with results obtained by the barium sulphate method shows that the benzidine method is as accurate a method as could be desired for the above purposes. G. C. J.

Cause and Significance of an Abnormal Reaction obtained in Testing Urine for Sugar with Fehling's Solution. W. Cramer. (*Biochem. J.*, 1915, **9**, 71-77.)—A sample of urine, containing about 1·5 per cent. of dextrose, yielded a scarlet transparent solution when heated to boiling and then mixed with an equal volume of hot diluted Fehling's solution. After some time the mixture darkened and deposited a black precipitate of extremely finely divided metallic copper. This reduction of cupric salts to metallic copper can be brought about by concentrated aqueous solutions of reducing sugars, provided that the latter are present in excess of the amount necessary to reduce all the cupric salt. It takes place even more readily when the reducing sugar is present in a urine of normal concentration as regards the usual urinary constituents, and is facilitated by the fact that some of the constituents (creatinine) of normal urine are capable of holding cuprous oxide in solution. The reaction, when it takes place, indicates marked glycosuria unaccompanied by polyuria, and not typical diabetes mellitus. W. P. S.

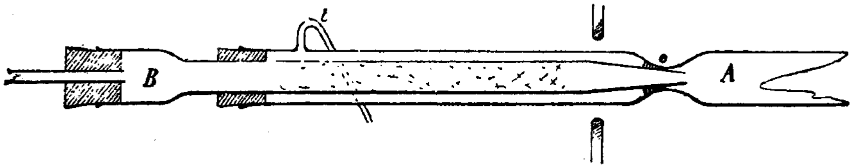
New Test for Reducing Sugars in Urine. W. Cramer. (*Biochem. J.*, 1915, **9**, 156-160.)—The test depends on the reduction of mercuric oxide in slightly alkaline solution to metallic mercury. The degree of alkalinity is important, as the test becomes more sensitive but less specific the greater the alkalinity. The reagent is prepared by dissolving 0·4 grm. of mercuric oxide and 6 grms. of potassium iodide in 100 c.c. of water, and adjusting the alkalinity of the solution by the addition of $\frac{N}{10}$ alkali or acid solution, until 10 c.c. of the reagent require exactly 2·5 c.c. of $\frac{N}{10}$ acid for neutralisation, using phenolphthaleïn as the indicator. To apply the test, 3 c.c. of the reagent are heated in a test-tube to boiling, 0·3 c.c. of the urine is added, the mixture again boiled, and, after the lapse of thirty seconds, acidified with acetic acid. The latter dissolves precipitated phosphates which obscure the reaction. Normal urine containing the usual quantity (0·1 to 0·2 per cent.) of dextrose yields a very slight turbidity; when the sugar content increases to 0·5 per cent. a distinct turbidity is produced. If the alkalinity of the reagent is increased beyond the degree stated above, the normal quantity of sugar in urine then interferes by producing a turbidity; very strongly alkaline mercuric oxide solutions are also reduced by such substances as creatinine, and even by glycerol. W. P. S.

ORGANIC ANALYSIS.

Colorimetric Estimation of Acetylene. E. K. Weaver. (*J. Amer. Chem. Soc.*, 1916, **38**, 352-361.)—The gas containing the acetylene is first freed from hydrogen sulphide and from large amounts of oxygen and carbon dioxide, if present, and then passed through one or more fine orifices (0·2 mm. in diameter) into an absorbing solution, prepared as follows: About a quarter of a grm. of gelatine is dissolved in 500 c.c. water, the solution diluted with 500 c.c. alcohol, and 1·25 grm. hydroxylamine hydrochloride added. Of this stock solution, 20 c.c. are mixed with 10 c.c. of concentrated ammonium hydroxide solution, and one or two cgrms. of cuprous chloride are added. This serves to absorb the acetylene, the gelatine acting as a protective colloid with the result that a clear colloidal sol of copper acetylide of an intense red colour is obtained. After the absorption, the solution is diluted to 100 c.c. and compared in a colorimeter with a standard which has been chosen. The standard used by the author contained 0·21 mgrm. Chromanilbraun R, 0·04 mgrm. Carmoisine B, and 2·5 grms. gum arabic in 100 c.c. If 10 c.c. of this solution be used as standard, the amount of acetylene in 100 c.c. of colloidal sol may be calculated from the equation $x = 0{\cdot}143\ y + 0{\cdot}03$, where x = number of mgrms. of acetylene and y = the reciprocal of the number of c.c. of colloidal solution required to match the standard. A more convenient but less accurate standard is a fixed depth of a solution of azolitmin. Amounts of acetylene as small as 0·03 mgrm. may be detected and amounts up to 2 mgrms. may be estimated within 0·05 mgrm. G. C. J.

Modification of Mohler's Reaction for Benzoic Acid. J. Grossfeld. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1915, **30**, 271-273; through *Chem. Zentralbl.*, 1915, ii., 1313-1314.)—Whilst the method described by Von der Heide and Jacob for the detection of benzoic acid (ANALYST, 1910, **35**, 166) is trustworthy, the reduction of the dinitrobenzoic acid with ammonium sulphide, as recommended by Mohler (*cf.* Halphen, ANALYST, 1908, **33**, 420), is not satisfactory. The following modification of the test is therefore proposed, the reduction being brought about by means of hydroxylamine: The dry alkali benzoate residue is heated on a water-bath for twenty minutes with a mixture of 0·1 grm. of potassium nitrate and 1 c.c. of concentrated sulphuric acid, then cooled, diluted with 2 c.c. of water, again cooled, and treated with 10 c.c. of 15 per cent. ammonia and 2 c.c. of 2 per cent. hydroxylamine hydrochloride solution. A red coloration develops more or less rapidly according to the quantity of benzoic acid present; if the mixture is heated, the reaction is accelerated, and the coloration reaches its maximum intensity when the solution is again cooled. W. P. S.

Simultaneous Estimation of Carbon, Hydrogen, and Mercury in Organo-Mercuric Compounds. Grignard and A. Abelmann. (*Bull. Soc. Chim.*, 1916, **19**, 25-27.)—In the combustion analysis of organic compounds containing mercury no satisfactory method was previously available for the complete condensation of the mercury vapours separately from the water. The authors have devised a form of combustion-tube, in which gold is employed for absorbing the mercury, while the water vapours pass on to the absorption vessels. The combustion-tube, *A* (see Fig.), of the usual open type, is constricted (as at *e*) a short distance before the point at which it emerges from the furnace. The tube is continued for about 20 cm. beyond the constriction, and a narrower tube, *B*, is inserted from the open end, being held in position by a cork. The tube *B* is drawn out to a point, which penetrates the constriction, *e*, and is packed therein by a conical sleeve of calcined asbestos. This sleeve is composed of strips of thin asbestos paper about 15 mm. wide, moistened and moulded to the correct form on the point of the tube *B*, then dried and ignited in a crucible. The mercury vapours passing forward during the combustion enter the tube *B* through the constricted orifice, and are absorbed therein by gold leaf, or preferably by a helix of gold wire 0·2 mm. thick, 12-15 cm. long, and a few mm. in diameter. If the asbestos joint were perfectly gas-tight, there would be no difficulty in collecting the whole of the mercury in the tube *B*, but in practice there is a slight tendency for the mercury and water vapours to leak through into the space between the two tubes. This trouble is avoided by fusing a small tubulure, *t*, into the further end of the tube *A*, and introducing through this tubulure a current of dry purified oxygen at a pressure slightly higher than that existing in the combustion-tube. The slight inward leakage of oxygen through the joint is sufficient to ensure that the whole of the products of combustion pass down the tube *B*. The heat of the furnace is so manipulated that no condensation can occur in the packing,



while the projecting portion of the tube is surrounded with metal gauze and gently heated so that the water vapour passes on to the absorption apparatus. Certain organo-mercury compounds are difficult to burn, and must be mixed with powdered copper oxide; the presence of halogens necessitates the use of lead chromate in the ordinary manner. The mercury tube is cooled in the desiccator and weighed after the combustion; the mercury is then expelled by connecting the tube with a water aspirator and heating at 400° C. J. F. B.

Differentiation and Valuation of Erythrosin and Bengal Rose. A. Leys. (*Ann. Chim. anal.*, 1916, **21**, 25-32.)—Erythrosin, the sodium salt of tetraiodofluorescein, usually contains only a trace of chlorine attached to the resorcinol nucleus, and shows little or no fluorescence, whereas Bengal rose (the sodium salt of tetraiododichlorfluorescein) in which the chlorine is largely present in the phthalic nucleus, shows pronounced fluorescence. In estimating the value of these dyestuffs it is therefore necessary to determine the amount of substitution, and their relative proportions of chlorine and iodine. *Moisture.*—A weighed quantity is dried at 100° C. *Mineral Matter.*—From 2 to 3 grms. are extracted in a Soxhlet apparatus with 200 to 250 c.c. of absolute alcohol, until the extract is nearly colourless. The alcoholic solution is evaporated to obtain the pure colouring matter, the residue is extracted with boiling water, the extract made up to definite volume, an aliquot portion evaporated, and the saline residue ignited and weighed. *Estimation of the Amount of Substitution.*—From 2 to 3 grms. of the pure dyestuff separated by the extraction with alcohol are fused with 5 to 10 grms. of potassium hydroxide and 10 to 15 grms. of sodium carbonate, the melt dissolved in boiling water, and the solution made up to 200 c.c. The total halogens in 25 c.c. of the solution are then estimated by titration with $\frac{N}{10}$ silver nitrate, and the number of c.c. required by 100 grms. of the pure dyestuff compared with those required by erythrosin and Bengal rose. The following table gives the values shown by compounds found in commercial products:

Formula.	Molecular Weight.	$\frac{N}{10}$ Silver Nitrate required per 100 Grms.	Chlorine.	Iodine.	$\frac{Cl}{I} \times 127$.
		C.C.	Per Cent.	Per Cent.	
$C_{20}H_6O_5I_4Na_2$, Erythrosin	880	4·54	—	57·7	—
$C_{20}H_6O_5I_3CNa_2$, Chloriodo-erythrosin	788	5·07	4·50	48·35	11·8
$C_{20}H_7O_5I_2Na_2$, Incomplete erythrosin	754	3·97	—	—	—
$C_{20}H_7O_2I_2ClNa_2$, Incomplete chloriodo-erythrosin ...	662	4·53	5·36	38·36	17·7
$C_{20}H_8O_5Cl_2Na_2$, Sodium dichlorfluorescein	445	4·49	15·95	—	—
$C_{20}H_7O_5Cl_2INa_2$, Sodium dichlormonoiodofluorescein	571	5·25	12·43	22·24	71·0
$C_{20}H_6O_5Cl_2I_2Na_2$, Sodium dichlordi-iodofluorescein ...	697	5·73	10·18	36·44	35·5
$C_{20}H_5O_5Cl_2I_3Na_2$, Sodium dichlortri-iodofluorescein ...	823	6·07	8·62	46·29	17·7
$C_{20}H_6O_5Cl_4Na_2$, Sodium tetrachlorfluorescein	514	7·78	27·62	—	—
$C_{20}H_5O_5Cl_4INa_2$, Sodium tetrachlormonoiodofluorescein	640	7·81	22·18	19·84	142·0
$C_{20}H_4O_5Cl_4I_2Na_2$, Sodium tetrachlordi-iodofluorescein	766	7·83	18·53	33·15	71·0
$C_{20}H_3O_5Cl_4I_3Na_2$, Sodium tetrachlortri-iodofluorescein	892	7·84	15·91	42·71	47·3
$C_{20}H_2O_5Cl_4I_4Na_2$, Tetra-Bengal rose	1,018	7·85	13·94	49·90	35·5

If the number of c.c. of silver nitrate approximate 4·54 or less, the product belongs to the erythrosin class, and should show only a negligible fluorescence in aqueous solution. But if the number exceeds 5·07 the dyestuff belongs to the Bengal rose class, and should show more or less pronounced fluorescence. *Estimation of Iodine.*—Fifty c.c. of the solution of the fused saline mass are acidified with dilute sulphuric acid, and shaken with carbon bisulphide and a little potassium nitrite. The bisulphide extract of the iodine is decanted, washed with water, treated with a few c.c. of sodium bicarbonate solution, and titrated with $\frac{N}{10}$ sodium thiosulphate solution. The nature of the dyestuff may then be ascertained by comparing the ratio of iodine to chlorine with the values in the table. C. A. M.

Estimation of Methyl Alcohol in Ethyl Alcohol. G. Reif. (*Arbeit. Kaiserl. Gesundheitsamte,* 1915, 50-56; through *Chem. Zentralbl.,* 1915, ii., 1056-1057.)—The method depends on the fact that methyl iodide reacts with methyl sulphide in the cold, with the formation of trimethylsulphine iodide according to the equation: $CH_3I + (CH_3)_2S = (CH_3)_3SI$, whilst the corresponding ethyl compound is not formed at ordinary temperature. If, however, the proportion of methyl iodide in the mixture of methyl and ethyl iodides is very small, the ethyl compound is produced, but its formation may be prevented by the addition of ether. The trimethylsulphine iodide behaves as a salt of hydriodic acid, and can be titrated with silver nitrate solution. Wirthle's method (ANALYST, 1912, **37**, 367) is used for the preparation of the mixed alkyl iodides, and the specific gravity of the mixture will indicate approximately the amount of methyl iodide present. Two c.c. of the iodide mixture are placed in a 20 c.c. flask, and, if the quantity of methyl iodide in the mixture exceeds 5 per cent., 2 c.c. of methyl sulphide are added; if the proportion of methyl iodide is less, only 1·5 c.c. of methyl iodide is added, together with 0·5 c.c. of dry ether. The flask is closed, kept in a desiccator for twenty-four hours, the crystals of trimethylsulphine iodide are then rinsed on to a dry filter with dry ether and washed with the latter. The crystals are next dissolved in water and titrated with standard silver nitrate solution. One molecule of silver nitrate is equivalent to 1 molecule of methyl alcohol.

In the estimation of methyl alcohol in brandy, a preliminary test is made as described by Wirthle (*loc. cit.*) to ascertain whether this alcohol is actually present. One hundred c.c. of the sample are then treated with 10 c.c. of $\frac{N}{1}$ sodium hydroxide and distilled in an apparatus fitted with a Le Bel still-head; the distillate should measure about 10 c.c. more than the quantity of alcohol in the sample. The total amount of alcohol in the distillate is now estimated, and from the result is calculated the number of c.c. of distillate corresponding with 10 c.c. of alcohol. This quantity of the distillate is used for the preparation of the mixed iodides. The boiling-point observed during the distillation of the sample will give an indication of the proportion of methyl alcohol present, and consequently of the quantities of iodine and phosphorus required, but an excess of 1 grm. of iodine per 10 c.c. of the alcohols is without influence. When large quantities of methyl alcohol are present, observation of the boiling-point of the sample will enable a distillate containing approximately 10 per cent. of methyl alcohol to be obtained. The mixed iodides are then treated with methyl sulphide and the estimation completed as described. W. P. S.

Methods of Analysing Vegetable Parchments. R. W. Sindall and W. Bacon. (*Paper Makers' Monthly J.*; through *Chem. News*, 1916, **113**, 67-68.)—The usual method of disintegrating paper for microscopic examination is not applicable to parchment papers in which the fibres are firmly cemented together by colloidal cellulose. Methods which depend on the use of chemicals for overcoming this adhesion, *e.g.*, with cuprammonium solution or sulphuric acid, are unsatisfactory, because such chemicals, while dissolving the cementing substance, may attack the fibres and introduce appreciable errors in their subsequent estimation. The authors therefore prefer a mechanical method of disintegration such as is conveniently afforded by a small vertical coffee-mill. About 0·5 grm. of the sample is crumpled up with hot water, torn into small pieces, and passed several times through the mill with a little water until the fibres are sufficiently separated. In preparing the fibres for microscopic examination, the specimen should be mounted in zinc chloride-iodine reagent somewhat more dilute than is suitable for ordinary paper analysis, since the strong reagent gives such a dense blue coloration with the parchmentised fibres as to make them practically opaque. The correct degree of dilution of the staining reagent suitable for the differentiation and estimation of mixed rag and wood cellulose fibres in vegetable parchments, is best found by trial. In highly parchmentised specimens the wood cellulose fibres may lose some of the characteristics by which they are recognised in the microscopic examination of ordinary papers. J. F. B.

Reducing Action of Phenylhydrazine on Certain Metallic Oxides. E. Puxeddu. (*Gazz. Chim. Ital.*, 1916, **46**, 71-76.)—Yellow oxide of mercury when added to an ethereal solution of phenylhydrazine is instantly reduced to finely divided metallic mercury. Red mercuric oxide is also reduced to the metallic form, with the simultaneous formation of a small amount of a crystalline compound—probably diphenyl mercury. A similar reduction of mercurous oxide is effected. Of the oxides of lead, only the peroxide PbO_2 reacts with phenylhydrazine at the ordinary temperature, while the oxide Pb_3O_4 reacts on heating. In both cases the suboxide Pb_2O is produced. C. A. M.

Hopkins-Cole Reaction for Protein. H. G. D. Breidahl. (*Biochem. J.* 1915, **9**, 36-37.)—Hopkins and Cole investigated (*Proc. Roy. Soc.*, 1901, **68**, 21) the Adamkiewicz protein reaction, and found that the test failed when pure acetic acid was used. They showed that the presence of glyoxylic acid in the acetic acid was a necessary factor, and on this account they modified the test by dispensing with the use of acetic acid, employing instead a glyoxylic acid reagent, prepared by reducing oxalic acid solution with sodium amalgam. The author now points out that the sulphuric acid has also an influence on the reaction, and that it is necessary, in the case of old sulphuric acid, to subject the same to a preliminary treatment with granulated zinc; the concentrated acid should be well mixed with the metal and left for a few days. The test is carried out by mixing the protein solution with the glyoxylic acid reagent, and then pouring the concentrated sulphuric acid down the side of the tube; a brilliant violet coloration is produced at the junction of the two liquids.

W. P. S.

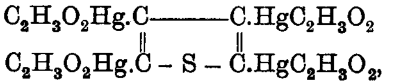
Action of Normal and Basic Lead Acetate on the Sugars, with Remarks on Rübner's Test for Dextrose and Lactose. H. Rogerson. (*Biochem. J.*, 1915, **9**, 245-252.)—Rübner's test (*Zeitsch. anal. Chem.*, 1885, **24**, 447) is carried out by adding some lead acetate to a dilute solution of dextrose, and then adding ammonia drop by drop until a permanent precipitate is formed. On standing in the cold the liquid becomes coloured gradually, first yellow, and then rose or flesh red; on heating the coloration takes place more quickly. The author finds that dextrose, lævulose, and galactose gave a flesh colour and a precipitate; mannose a yellow colour, but no precipitate; lactose a yellow colour and precipitate; and maltose a yellow to buff colour and precipitate. These tests clearly indicate the futility of adopting this reaction of Rübner as specific for dextrose and lactose. Many experiments are described in which normal and basic lead acetate in the presence of different alkalis are allowed to react at varying temperatures with all the ordinary sugars and polyhydric alcohols, and it is concluded that their action is somewhat complex, and that it is impossible to correlate the effects observed with any particular sugar in order to identify it. There is little doubt that the precipitates obtained in many cases are complex derivatives of the sugar and the acetates of lead.

H. F. E. H.

Use of Nickel Hydroxide in Tannin Estimation. P. Singh and T. P. Ghose. (*J. Soc. Chem. Ind.*, 1916, **35**, 159-160.)—Nickel hydroxide powder is recommended as a substitute for hide-powder for the absorption of tannin. In the discussion Procter pointed out that as an empirical method it offered no advantages to the tanner over the well-established hide-powder method, while nickel hydroxide appeared to be inferior to hide-powder as a reagent for distinguishing between gallic acid and tannin. He regarded most of the tannin compounds as colloidal precipitates rather than as true salts.

C. A. M.

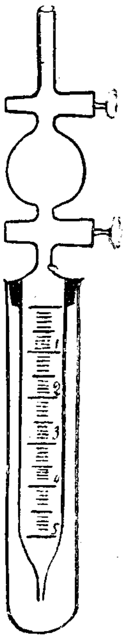
Estimation of Thiophen in Benzene. O. Paglini and B. Silbermann. (*Gazz. Chim. Ital.*, 1915, **45**, 385-390.)—The method depends on the formation of thiophen mercuri-acetate,



which is insoluble in all ordinary solvents, and may be separated from glacial acetic acid, in which, when hot, it dissolves in the proportion of 1 : 5. A little more than the calculated quantity of mercuric oxide is dissolved in twice its weight of hot glacial acetic acid, and the solution cooled for the separation of the mercuric acetate. The sample of benzene under examination is then added, and the mixture boiled on the water-bath for about fifteen minutes. The precipitate of thiophen mercuri-acetate is separated from the cold solution, washed several times with ether, dried at 100° C., and weighed. The method gives accurate results in the estimation of quantities of thiophen ranging from 1 to 0·5 per cent. C. A. M.

INORGANIC ANALYSIS.

Analysis of Mixed and Spent Acids. L. Wuyts. (*J. Soc. Chem. Ind.*, 1916, **35**, 149-151.)—The proportion of nitrous and nitric acids in the mixed acids used for nitrating toluene may be rapidly estimated by a modification of Schloesing's method. The apparatus required consists of a pear-shaped Kjeldahl flask with a capacity of about 200 c.c., provided with a 40 c.c. tap-funnel and a delivery tube. The stem of the tap-funnel is of capillary tubing, and is bent twice at right angles, with the object of preventing its being immediately above the Bunsen burner, whilst the delivery tube has a tap, and is bent so as to deliver the gas beneath a eudiometer, graduated in 0·1 c.c. In making an estimation 40 c.c. of ferrous chloride solution (450 grms. per litre with a few drops of HCl), and 30 c.c. of hydrochloric acid (1 vol. of concentrated acid with 2 vols. of water, boiled to expel air), are placed in the Kjeldahl flask, and the stopper with funnel and delivery tube inserted. In the case of the mixed acids for preparing mononitro-toluene, about 15 c.c. of water are first introduced into the funnel, and sufficient run into the flask to expel the air from the tube. The contents of the flask are boiled until all air is removed, and a eudiometer tube filled with water is placed over the end of the delivery tube. To prevent water being drawn back into the flask through back pressure, the end of the delivery tube is formed into a T-piece, one end of which passes up into the eudiometer while the other dips into mercury below the water in the trough. About 0·7 c.c. of the acid is introduced into the tap-funnel by means of the bulb pipette shown in the diagram, and, together with the water already there, is allowed to fall drop by drop into the boiling ferrous chloride solution in the flask. A further 10 c.c. of water is subsequently added to rinse the funnel, and the rinsing is twice repeated after the evolution of gas has nearly stopped. Meanwhile the amount of acid used in the estimation is weighed. After all the gas has been collected, the eudiometer is transferred to a cylinder containing water saturated with nitric oxide, while a fresh estimation may be made without interrupting the boiling. In the case of the mixed acids for preparing trinitro-toluene, about 0·9 c.c. is used, while with "spent acid T.N.T." a weight corresponding to about 4 c.c. is taken, and the liquid is diluted with 25 c.c. of water. For "spent acid M.N.T." a weight corresponding to about 10 c.c. is required, and 25 c.c. of water should be added. Blank estimations with 20 c.c. of a standard solution of nitrate (16·5 grms. of dry sodium nitrate or 20 grms. of dry potassium nitrate per litre) are made before and after a series of estimations, and the results are compared with the volumes of the gas yielded by the mixed acids. If V_o represent the volume of gas from the standard nitrate solution, and V_o^1 the



volume from the sample of acid of weight W, the percentage of nitric acid corresponds to $24{\cdot}4629 V_o^1 \div V_o W$. For preserving the tubes of gas until convenient to read, the best results were obtained by the use of water saturated with nitric oxide. This was prepared by leaving two or more burettes containing the gas in a gas cylinder filled with water, until the volume showed no further sign of reduction when reduced to standard temperature and pressure. C. A. M.

Analysis of a Mixture of Alkali Sulphides, Thiosulphates, and Dithionates. J. A. Muller. (*Bull. Soc. Chim.*, 1916, **19**, 8-9.)—In a mixture of alkali salts of this nature the pre-existing sulphates are estimated by precipitation as barium sulphate, in highly dilute solution, slightly acidulated with acetic acid and maintained in an atmosphere of carbon dioxide. In another portion of the solution the sulphides and thiosulphates are estimated together by iodimetric titration in highly dilute solution slightly acidified by acetic acid. Another portion is placed in a small flask, acidified with acetic acid, and the flask connected with a water suction-pump. The contents of the flask are vigorously shaken for about ten minutes under the vacuum of the pump, until all the sulphuretted hydrogen is expelled and the thiosulphates are then titrated iodimetrically. The difference between the two titrations gives the sulphides. In these titrations the presence of compounds of the thionic series does not interfere, provided the titration be carried out rapidly, as the action of iodine on these compounds in the cold is practically negligible. The thionates are estimated in another portion of the liquid, after acidulating as before and removing the sulphuretted hydrogen *in vacuo*. The solution is filtered, then made distinctly alkaline with potassium hydroxide, and evaporated to dryness in a silver capsule, with the addition of a little nitre. The dried residue is carefully roasted and the total sulphates are estimated as barium sulphate, from which the equivalent of the thiosulphates and pre-existing sulphates is subtracted. The result may be expressed in terms of dithionate, since in presence of excess of alkali sulphides the tri- and tetra-thionates are decomposed. If the original solution contained disulphides and polysulphides, sulphur would be precipitated on acidification with acetic acid. This sulphur, at first colloidal, coagulates when the last traces of sulphuretted hydrogen have been expelled. It is then easily collected on a small filter which, after washing, is heated in a silver capsule with potassium hydroxide and nitrate, and the sulphur estimated as barium sulphate. J. F. B.

Estimation of Bromine and Iodine in the Presence of Chlorides. L. W. Winkler. (*Zeitsch. angew. Chem.*, 1915, **28**, 477-480, 494-496.)—The permanganate method is recommended for the estimation of bromine in bromides. In the case of sodium or potassium bromide containing but traces of chloride, about 0·1 grm. of the salt dissolved in 100 c.c. of water is acidified with 25 c.c. of 50 per cent. sulphuric acid, the mixture is boiled, and permanganate solution is run in until a permanent pink coloration is obtained. The permanganate solution should be standardised against pure potassium bromide. When the bromide contains its own weight of chloride, the titration is carried out in a distillation flask, the jet of the burette being fitted into the neck of the flask. After each addition of permanganate

2 c.c. of the liquid are distilled, and this is continued until the distillate is colourless —*i.e.*, until bromine no longer distils over. If iodide is present, iodine distils before the bromine, and can be collected separately; nitrates and carbonates may be present in the solution, but reducing substances and ammonia must be removed previously. In the case of sea-water, the bromine is concentrated by acidifying 100 c.c. of the water with hydrochloric acid, adding sulphuric acid, and distilling with the addition of permanganate, the distillate being collected in dilute sulphurous acid. Traces of iodine do not interfere, but if a large quantity is present, the greater part should be removed by first distilling the water with the addition of sulphuric acid and ferric chloride solution. The distillate is then placed in a flask, sulphuric acid is added, and, when all the air has been expelled by boiling, permanganate solution is run in under the conditions described above. The liberated bromine is collected in hydrogen peroxide solution, and the hydrobromic acid thus formed is estimated either by titration with lime-water, by titration with silver nitrate solution after neutralisation with lime-water, or gravimetrically as silver bromide. The following method is recommended for the estimation of bromine in mineral waters, and is similar to the above, except that the distillate containing the bromine is collected in 10 c.c. of $\frac{N}{1}$ sodium hydroxide solution. A few c.c. of carbon tetrachloride are then added to the distillate, the mixture is acidified with 2 or 3 drops of concentrated hydrochloric acid, and shaken. After fifteen minutes $\frac{N}{100}$ arsenious acid solution is added in small quantities at a time, and the mixture shaken until the carbon tetrachloride layer is colourless. Iodine, if present, is also titrated; in this case the carbon tetrachloride changes in colour from brownish-yellow to colourless and then rose-red, at which point the titration is ended.

For the estimation of iodine in the presence of bromides and chlorides modifications of the nitrite method are described. The sample is acidified, and shaken with sodium nitrite solution in the presence of carbon tetrachloride; after several of these treatments, the separated carbon tetrachloride solutions are treated with a small quantity of urea to decompose traces of nitrite, and then titrated with thiosulphate solution. If bromides are present, the bromine may be estimated in the same portion of the sample; the solution remaining after the carbon tetrachloride has been separated is acidified with sulphuric acid, boiled until all nitrous acid and dissolved carbon tetrachloride have been expelled, and then titrated with permanganate solution as described. W. P. S.

Estimation of Carbon Dioxide in Air by Haldane's Apparatus. R. C. Frederick. (*J. Soc. Chem. Ind.*, 1916, **35**, 96-99.)—For the estimation of small quantities of carbon dioxide in air, Haldane's apparatus is the most convenient and, in skilled hands, it gives sufficiently accurate results. The carbon dioxide of the air sample is absorbed by caustic potash, and the consequent diminution in volume, measured on a graduated scale, gives a direct reading of the quantity of carbon dioxide in 10,000 parts of air. The author employs a potash solution of 10 per cent. strength coloured with methyl orange. The apparatus is fully described and illustrated, and complete details are given for its correct manipulation. A more definite reading may be obtained by placing a sheet of black material behind the tubes on which the adjustment marks are situated. Instead of agitating the water-jacket by blowing air through the water by mouth, a rubber bulb is preferably supplied for the purpose. Thin pipe cleaners which can pass right through the stopcocks serve admirably for dislodging dirt from the glass tubes and air burette, which must be perfectly clean. The simplest operation is when the air to be analysed can be taken directly into the instrument. For analyses at a distance small sample bottles of special pattern are required. The method of collecting the samples and discharging the contents of the bottles into the Haldane apparatus, with the assistance of a mercury bath, is described. Certain modifications have been introduced in the details of the Haldane apparatus, notably a device for extending the range of the instrument designed to register up to 100 parts of carbon dioxide per 10,000, so that it can be used to record up to 500 parts by the temporary attachment of a special scale. Another adjunct described by the author relates to a counterbalancing device to reduce the physical effort required for raising and lowering the mercury reservoir of the apparatus when several analyses have to be performed. Stops are also provided which render it impossible for the mercury to run over into the potash vessel, or *vice versa*. J. F. B.

Analysis of Copper-Aluminium-Zinc Alloys. H. Graefe. (*Chem. Zeit.*, 1916, **40**, 102.)—Alloys consisting of copper, 25 to 27 per cent., aluminium, 14 to 18 per cent., and zinc, 54 to 57 per cent., together with small quantities of silicon, cadmium, tin, lead, and iron, may be analysed as follows: A weighed quantity of about 0·5 grm. of the turnings is dissolved in 15 c.c. of nitric acid (sp. gr. 1·4), and any insoluble metastannic acid is separated by filtration. The filtrate is diluted to 150 c.c., heated to 60° to 70° C., and submitted to electrolysis; using a current of 0·2 to 0·4 ampère and 2 to 3 volts, the copper and lead are deposited within forty-five minutes. Solid potassium hydroxide is now added to the solution until the acidity of the latter is nearly neutralised, and the aluminium, iron, and zinc are precipitated together by the addition of sodium carbonate. The precipitate is collected, washed with hot water, dissolved in the minimum quantity of dilute sulphuric acid (1 : 4), and the solution treated with an excess of potassium hydroxide. The solution, containing a small quantity of undissolved ferric hydroxide, is diluted to 150 c.c. and electrolysed; a copper-coated cathode is employed, and this is rotated at 600 to 800 revolutions per minute, the current being increased from 1 to 5 ampères in steps of 1 ampère every ten minutes. The zinc is thus deposited in forty minutes, and is washed without interrupting the current, then dried with alcohol, and weighed. The iron and aluminium remaining in the electrolyte are then estimated in the usual way. W. P. S.

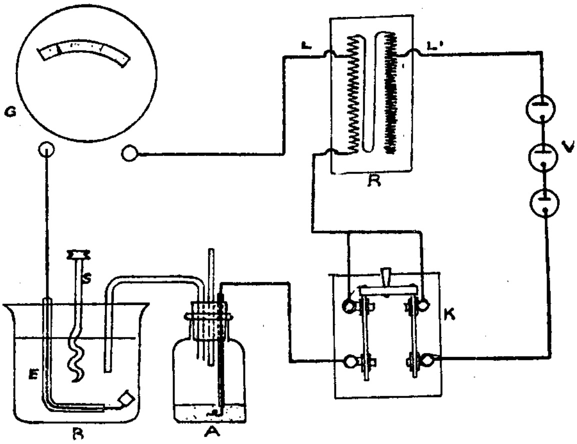
Differential Iodimetry. Estimation of Periodates, Iodates, Bromates, and Chlorates in Presence of Each Other. O. L. Barnebey. (*J. Amer. Chem. Soc.*, 1916, **38**, 330-341.)—Periodates, iodates, bromates, and chlorates can be estimated iodimetrically in presence of each other, and of perchlorate, by regulation of the concentration of reagents, especially the acidity, the temperature, and time of reaction. Periodate reacts completely with iodide in saturated boric acid solution containing sufficient borax to diminish the acidity to a slight extent, forming iodate and free iodine; under these conditions the other salts are unaffected. Iodate as well as periodate is acted on by $\frac{N}{10}$ iodide in $\frac{N}{4}$ acetic acid solution, and the free iodine can be titrated; bromate, if present, is not unaffected under these conditions, but a simple correction makes the method substantially exact even in presence of bromate. In $\frac{N}{2}$ hydrochloric acid containing $\frac{N}{10}$ iodide, bromate as well as periodate and iodate is completely decomposed, and the free iodine can be titrated. Finally 6 N hydrochloric acid acting in presence of $\frac{N}{10}$ to ^{N} iodide decomposes chlorate completely, as well as periodate, iodate, and bromate, whilst perchlorate is unaffected. After rendering the solution alkaline and then acidifying, the iodine can be titrated with thiosulphate.

Of a mixture of the above salts, 2 grms. are dissolved and the solution is diluted to 1,000 c.c. Periodate is estimated in one 100 c.c. portion by adding 2 grms. borax, an excess of boric acid, and 20 c.c. of $\frac{N}{1}$ iodide, allowing to stand three minutes, and then titrating the liberated iodine with $\frac{}{10}$ thiosulphate. In another 50 c.c. portion, periodate and iodate are estimated by cooling to 3° or 4° C. (to limit the tendency of bromate to react), adding 20 c.c. water, 10 c.c. $\frac{N}{1}$ iodide, and 20 c.c. $\frac{N}{4}$ acetic acid, also cooled, and commencing to titrate the liberated iodine with thiosulphate after one or two minutes. When the titration is complete, which should not be less than three minutes after adding the iodide and acid, the mixture is allowed to stand for a further equal interval of time, after which any iodine which appears is titrated with thiosulphate. This is a fairly exact measure of the error due to decomposition of bromate, and is deducted from the consumption of thiosulphate in the estimation proper. Periodate, iodate, and bromate are estimated in another 50 c.c. portion by adding 40 c.c. water, 10 c.c. $\frac{N}{1}$ iodide, and 5 c.c. 6 N hydrochloric acid and titration of the liberated iodine with thiosulphate. Finally, periodate, iodate, bromate, and chlorate are estimated in a 50 c.c. portion which is placed in 500 c.c. stoppered flask. The air in the flask is replaced by carbon dioxide, 10 to 20 c.c. $\frac{N}{1}$ iodide and 50 c.c. concentrated hydrochloric acid are added, and the mixture is allowed to stand twenty minutes. The flask is then placed in running water, 50 c.c. of sodium hydroxide solution at least equivalent to the acid used (520 grms. NaOH per 1,000 c.c.) are added all at once and the mixture cooled to room temperature. Hydrochloric acid (1 : 1) is then added slowly until iodine is liberated in quantity, followed by 3 to 5 c.c. more of the acid. The free iodine is then titrated with thiosulphate. G. C. J.

Detection of Nitrates in the Presence of Organic Matter. A. Tingle. (*J. Soc. Chem. Ind.*, 1916, **35**, 77-78.)—The reagent consists of 3 grms. of salicylic acid in 100 c.c. of concentrated sulphuric acid. A control test should show that the reagent, after being warmed, contains no unsulphonated salicylic acid; in the absence of nitrates an ethereal extract of the reagent should give no red coloration with ferric chloride solution. The solution to be tested—*e.g.*, 2 c.c. of a saturated solution of cane-sugar, containing 0·1 per cent. of potassium nitrate—is treated with an equal volume of the reagent, and warmed until a charred mass is formed. The char is extracted with 10 c.c. of boiling water, and the solution, after cooling, shaken with an equal volume of ether. One half of the ethereal extract is shaken with a little aqueous ammonia, which produces an orange colour with the nitrosalicylic acid; the other half is shaken with 2 c.c. of a 1 per cent. aqueous solution of ferric chloride, which in presence of nitrosalicylic acid gradually forms a red coloration in the aqueous layer. J. F. B.

Electrometric Titration of Vanadium. G. L. Kelley and J. B. Conant.

(*J. Amer. Chem. Soc.*, 1916, **38**, 341-351.)—Vanadates may be titrated with ferrous sulphate, using a change in the electromotive force of a suitable cell as the indicator. It is not necessary to measure the potential involved, and an ordinary suspension galvanometer serves, with a resistance box containing two sliding contacts. The resistance box used by the authors consists of two coils and sliding contacts with resistances of 195 and 5 ohms, and is wired so that one resistance serves to vary the E.M.F. of the dry cells used as a source of current, and the other serves as the potentiometer box. *A* represents a calomel electrode, the dotted area representing the mercury, and the bottle being filled with a solution of potassium nitrate, and fitted with a plunger, slight pressure on which causes the ejection of a portion of the liquid through a capillary tube. *B* is the beaker in which the titration is made, and contains the platinum electrode, *E*, and the stirrer, *S*. The galvanometer is shown at *G*, and the resistance box at *R*, with its two sliding contacts, *L* and *L*¹. The batteries—ordinary dry cells—are shown at *V*, and *K* is a double knife switch, which throws in both the galvanometer and main circuits at one operation. The solution containing the vanadium as vanadate is placed in the beaker, and 25 c.c. of dilute (1 : 1) sulphuric acid are added, with water enough to bring the volume up to 200 c.c. The connections are then made, and resistances so adjusted that the needle comes to rest on the scale. Standard ferrous sulphate solution is then run in from the burette. At the first addition of ferrous sulphate the needle begins to move off the scale, showing a drop in potential. The sliders on the resistance box are then changed in such a manner as to keep the needle on the scale during the addition of further quantities of the reducing agent. This movement of the needle is slow, and continues until half to three-quarters of the equivalent quantity of ferrous sulphate has been added. During the addition of the last quarter the needle remains practically at rest until the end-point is reached, when the addition of a few tenths of a c.c. in excess causes a sudden movement. To obtain exact adjustment, dilute standard dichromate solution is added in small amount until the needle is returned to the position which it occupied before ferrous sulphate was added in excess. The addition of 2 or 3



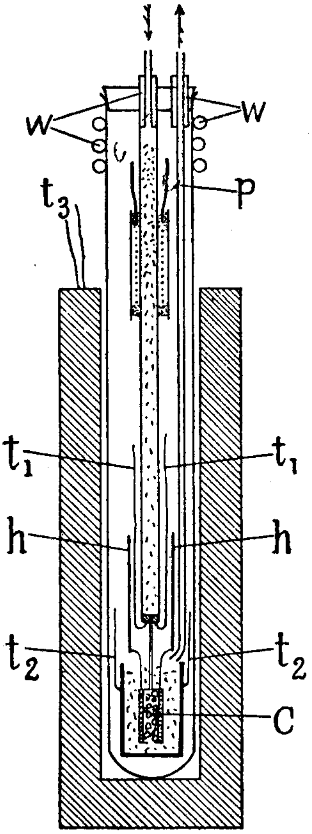
drops of ferrous sulphate solution is now usually enough to cause a sudden movement over several divisions. This is followed by the addition of exactly the quantity of dichromate necessary to return the needle to the original stationary position. The end-point of the reaction is thus indicated by a sharp throw of the needle, which is easily distinguished from the slow movement which occurs when the first portions of ferrous sulphate are added. Sharpness of end-point is promoted by low temperature (preferably 10° C.), high acid concentration, small rather than large amounts of vanadium, and low concentration of chromic and ferric salts.

The mean of the results obtained by this method is substantially identical with the mean of results obtained by reduction with sulphur dioxide, hydrogen peroxide, and oxalic acid, whilst duplicate estimations by the new method agree more closely than do duplicates obtained by the other methods. G. C. J.

Rapid Method for Converting Scrap Platinum into Chloroplatinic Acid. J., B. Tingle and A. Tingle. (*J. Soc. Chem. Ind.*, 1916, **35**, 77.)—The solution of scrap platinum in *aqua regia* is a tedious process, necessitating the use of a disproportionate amount of acid. The authors employ a far more rapid process by fusing the platinum with fifteen times its weight of zinc under a layer of borax or other flux. The mass is fused for about ten minutes, and the temperature then raised until the zinc begins to boil and its vapour to burn above the layer of flux. Small quantities may be thus treated in a porcelain crucible over a Bunsen burner. The metallic mass is cooled and separated, then dropped into dilute commercial hydrochloric acid. The zinc dissolves rapidly, leaving a black residue which is washed by decantation. The residue, still containing a little zinc, dissolves rapidly in *aqua regia;* the solution is evaporated to dryness and then redissolved in warm, very dilute hydrochloric acid. The solution may then be treated with pure zinc sheet or rod, and the precipitate of platinum washed with dilute hydrochloric acid until the washings are free from zinc. Alternatively, the platinum may be precipitated with sulphuretted hydrogen, and the sulphide ignited on the filter. The metallic platinum is re-dissolved in *aqua regia*, the solution evaporated almost to dryness, the residue dissolved in hydrochloric acid, and the solution again evaporated. The residue is dissolved in water, the solution of chlorplatinic acid filtered and diluted to the desired strength. J. F. B.

Estimation of Free Sulphur in Antimony Sulphide Pigments. A. Hutin. (*Ann. Chim. anal.*, 1916, **21**, 32-33.)—Carbon bisulphide is unsuitable for the extraction of sulphur from antimony sulphides, since when heated it tends to transform antimonic sulphide into a sulphide, Sb_2S_4. Acetone is a more suitable solvent, notwithstanding the fact that the solubility of sulphur therein is very small (0·25 per cent. at 56° C.). For the complete separation of free sulphur from 2 grms. of an antimony sulphide from six to eight hours' extraction beneath a reflux condenser are required. The extract must be evaporated at a low temperature, preferably under diminished pressure. C. A. M.

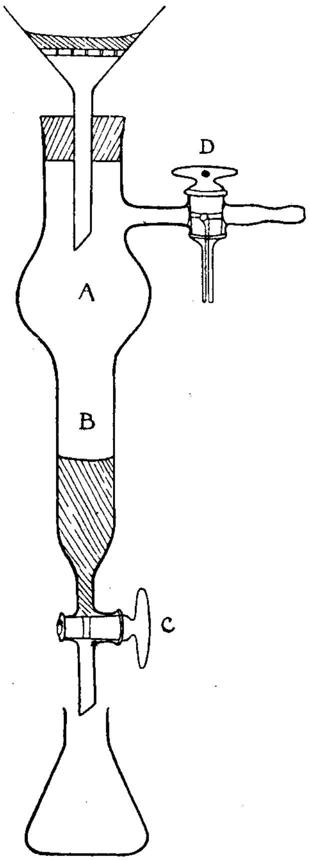
APPARATUS, ETC.



Calorimetric Measurements at High Temperatures. S. Tamaru. (*J. Soc. Chem. Ind.*, 1916, **35**, 81-88.)—Calorimetry at high temperatures is far more difficult than at ordinary temperatures, because of the difficulty of getting an effective thermostat at such temperatures—*e.g.*, at red heat—also because of the difficulty of measuring the temperature accurately, and because of the higher heat conductivity of materials under such conditions. The paper contains an account of a study of the heat of formation of synthetic ammonia at temperatures between 500° and 1,000° C. The principles employed are capable of large extension to thermochemical problems, and are also applicable to the determination of specific heats at high temperatures. A constant flow calorimeter is employed (see Fig.). Pure synthetic ammonia gas enters the top of the pre-heater, which consists of a silver tube containing silver filings, and is provided with a heating coil outside. The gas passes through the filings and leaves the pre-heater at a temperature which is measured by the thermocouple, t_1, attached to the bottom of the pre-heater, and enters the decomposer through a thin platinum capillary. The decomposer consists of a silver crucible containing silver filings and a small silver tube. The latter—the decomposer proper—contains a catalyst, *C*, and is provided outside with a heating coil consisting of a long thin platinum wire embedded in kaolin and asbestos, and connected with the wires, *h*. The ammonia is split up into nitrogen and hydrogen, and cools the decomposer through absorption of heat in the reaction. The fall of temperature is compensated by the supply of heat electrically through the heating coil. The products of decomposition pass out through silver filings at a temperature recorded by the thermocouple, t_2, finally leaving the apparatus by the porcelain tube, *p*. The whole apparatus is enclosed in a porcelain tube, and this, again, is inserted in a thick block of copper heated electrically to a temperature measured by the couple, t_3. *W* represents a water cooling coil, and the object of the silver filings is to facilitate the recording of the temperature changes of the gas. In the complete arrangement, ammonia gas from a cylinder enters an automatic pressure regulator, passes then through a drying tube containing solid caustic potash, next through a differential manometer into the calorimeter, which is kept in a thermostat with flowing water, so that the measurement is not affected by variations of room temperature. The decomposed gas passes through an absorption apparatus, containing sulphuric acid, to remove undecomposed ammonia, then through a second differential manometer, a rotameter, and finally through a gas meter. A constant stream of ammonia is supplied, and the supply of electricity to the copper thermostat, the pre-heater, and the decomposer is regulated so that all three are at the same temperature. The amount of energy supplied to the decomposer to compensate for the absorption of heat through decomposition gives the heat of formation at that temperature for the amount of ammonia decomposed. The correction necessary, owing to the inequality of temperature between the decomposer and its surroundings, may be determined by the passage of an inert gas. The precautions required for obtaining accurate results, the sources of error and the minute details of construction of the various parts of the apparatus, are fully discussed.

J. F. B.

Apparatus for Filtration under Reduced Pressure. J. C. Irvine. (*Biochem. J.*, 1915, **9**, 321-322.)—The illustration is almost self-explanatory. *D* is a three-way stopcock by means of which the apparatus may be put in communication with the pump during filtration, or with the atmosphere when it is desired to draw off the filtrate through *C*. An advantage of the apparatus is that it can be enclosed in a hot air-bath, thus facilitating the filtration of viscous or saturated solutions. The most generally useful size is 12 inches long over all, with a bulb 3 inches in diameter. This serves for the collection of 150 c.c. of filtrate, but apparatus of twice this capacity and smaller ones down to 20 c.c. capacity have been made. G. C. J.



Simple Hydrogen Electrode. H. P. Barendrecht. (*Biochem. J.*, 1915, **9**, 66-70.)—A simple arrangement for the estimation of the true reaction of liquids is described. It is in some respects an improvement on Walpole's electrometric titrating apparatus for open bench work, special provision being made for allowing instantaneous and accurate estimations even in liquids containing carbon dioxide and oxygen. The electrode consists of a bent piece of glass tubing with a platinum wire sealed in its wall; the upper end of the tube is connected with a perforated brass syringe. The point of the platinum wire, which is blacked, is near the lower end of the tube, which is narrowed. Hydrogen is first admitted through the tap of the syringe, while the piston is pushed home; the tap is then closed, and, by drawing the piston in and out several times, equilibrium between the hydrogen and the dissolved carbon dioxide is attained. Oxygen is removed by drawing the liquid several times up and down over the blacked wire, and the liquid is then forced down the tube until its surface is near the platinum point. The other half of the galvanic cell consists of a bottle filled with saturated potassium chloride solution, and closed with a rubber stopper through which pass three tubes. One of these is closed at the bottom, but has two openings in its side and constitutes the mercury-calomel-saturated-potassium-chloride electrode proper; the second tube is a capillary provided with a tap and connected with a capillary filled with cotton-wool, this capillary extending into the liquid under examination; the third tube is open to the air, and serves to prevent air pressure in the top of the bottle from causing inconvenience. W. P. S.

Viscosity of Oils in the Redwood and Ostwald Viscometers. C. A. Savill and A. W. Cox. (*J. Soc. Chem. Ind.*, 1916, **35**, 151-153.)—The Ostwald viscometer was calibrated by determining the time taken by concentrated sulphuric acid to flow through the capillary tube at 25° C. The specific gravity of the acid at 25° C. and its percentage strength, as shown by titration, were also estimated, and from these results the viscosity of the acid was found by reference to the graph (Dunstan, *J. Chem. Soc.*, 1914, **93**, 30). By substituting these figures in the formula $K = \frac{\eta}{td}$, the value of the constant K, depending on the size of the capillary tube of the particular viscometer, was found to be 0·00108. Absolute viscosity was calculated by means of the formula $\eta = Kdt$, where η represents the absolute viscosity, K the constant for the viscometer, t the time of flow in seconds, and d the specific gravity at the temperature of the determination. Parallel determinations were made by means of the two viscometers with eleven distinct types of oils, and the results plotted on graphs. It was found that the relationship between absolute viscosity and the efflux velocity in Redwood's viscometer could be expressed by a straight-line graph. For example, in the case of rape oil, the following results were obtained:

Temperature ° C.	Absolute Viscosity η.	Seconds' Flow in Redwood's Viscometer.	
		Observed.	Read from Graph.
50	0·304	149	149
70	0·173	86	87

C. A. M.

Electrically-heated Bomb Furnace. D. F. Calhane and H. A. Lavene. (*Met. and Chem. Eng.*, 1916, **14**, 140-143; through *J. Soc. Chem. Ind.*, 1916, **35**, 330.)—The furnace contains eight small iron pipes arranged around a central pipe of 1¼-inch diameter, and two heating elements, an inner one around the central pipe and an outer one enclosing the set of smaller pipes, which serve as receptacles for the sealed glass tubes to be heated. Each heating element consists of nichrome wire of the ribbon type, wound on an iron pipe and insulated from the pipe by alundum cement. The wire on the inner element is covered with a thin layer of alundum cement and enclosed in a jacket of thin sheet iron; that on the outer element is also covered with alundum cement and is enclosed in two jackets, an inner removable one of alundum cement composition and an outer one of magnesium insulation, as used for steam pipes. The furnace is fitted at each end with a door of a new type, the door proper consisting of an iron disc fastened by screws to cross-strips on the hinged door-frame, and held in position against the end of the furnace by springs on the shanks of the screws. The door-frame is fastened by means of a catch, and in the event of an explosion the door is forced backwards against the action of the springs until the pressure is relieved, and then returns to its original position. Hoods are fitted over the doors to prevent danger from fragments of glass which may be projected from the furnace.

REPORT.

Committee for the Investigation of Atmospheric Pollution. First Report: April, 1914, to March, 1915. (*Lancet Suppl.*, 1916, February 26, pp. i. to xl.)—The Committee consists of C. J. P. Cave, J. G. Clark, J. B. Cohen, H. A. Des Voeux, W. L. Hawkesley, J. B. C. Kershaw, R. Lessing, J. S. Owens, E. J. Russell, Sir N. Shaw, E. D. Simon, W. Smith, and F. J. W. Whipple. It was appointed "to draw up details of a standard apparatus for the measurement of soot and dust and standard methods for its use." For the purpose of this investigation, the question of variations of the normal gaseous constituents of the atmosphere is ignored and the interpretation of the term "pollution," adopted by the Committee, relates to such matter, solid, liquid, or gaseous, as reaches the surface of the earth or falls upon the buildings, etc., either by its own gravity or with the assistance of falling rain. As a preliminary step an analysis was made of a specimen of the grosser solid pollution, in the form of suspended particles, intercepted by the ventilator filters of a large institution in London. The analysis showed: Sodium chloride, trace; calcium carbonate, 2·17 per cent.; ferric oxide, 2·44; calcium sulphate, 5·09; alumina, 8·34; magnesium carbonate, 0·33; sand, 37·99; carbon, 35·48; ammonium sulphate, 5·77; tar (extracted by carbon disulphide), 1·49; and fibrous matter, 0·95 per cent. It is evident that the two main sources of pollution are earthy matters from the surface of the land and the products of imperfect combustion of coal. In connection with the latter, analyses were made of samples of soot deposited in chimneys in use with ordinary domestic hearths. These samples showed a large proportion of matter soluble in ether (tar), about 6 per cent. of combined sulphuric anhydride, and 4 per cent. of combined ammonia. It is noted that, whereas a considerable proportion of the sulphur of coal is discharged in the form of sulphuretted hydrogen and the oxides of sulphur in gaseous form, and whereas the ammonia may also rank as gaseous pollution, yet these constituents are found united in the form of ammonium sulphate, which as a solid and in solution comes prominently within the scope of this investigation. Among the constituents of smoke, the vapours of tarry hydrocarbons play an important part in condensing on the solid particles of carbon and imparting to these an extraordinary power of adhesion and resistance to the cleansing action of rain.

Methods of Work.—The objects to be attained in the course of the inquiry were formulated as follows: (*a*) To measure the amount of matter deposited from the air on a given area at any place during a fixed period. (*b*) To measure the amount of impurity held in suspension in the air at any time and place. (*c*) To find the nature and composition of the deposited or suspended matter. On consideration, it was recognised, however, that no single method could afford a simultaneous solution of problems (*a*) and (*b*), and since the method of measuring the amount of deposited impurity was capable of easy development, it was decided to put this into operation first, leaving the measurement of suspended impurity to a later stage of the inquiry. Nevertheless, a certain amount of preliminary investigation has been carried out on the latter problem, and some nine possible methods of procedure have been more or less thoroughly examined. The method of filtration through cotton- or asbestos-wool presents difficulties in connection with the accuracy of weighing the deposit on the filter and its quantitative detachment for analysis. Aitken's dust-counting method is interesting, but hardly suitable, as it affords no measure of the size and weight of the various particles nor of their composition. Optical methods, based on measurements of the opacity of a column of air or of a glass plate coated with a sticky substance, on which a jet of air is caused to impinge for a short time, might be capable of development, and a method of this order has been under investigation by J. S. Owens with some measure of success. The apparatus used consists of two bottles serving as aspirators; water, allowed to syphon from one to the other, draws a measured volume of air through a disc of filter-paper of standard area, the discoloration of which is compared with a scale of shades. An observation can be taken in about ten minutes, and the instrument has been approved by the Committee for the measurement of suspended impurity.

The major portion of the present report is concerned with the measurement of the deposited impurities, including those precipitated by the rainfall. A standard gauge has been devised and distributed in many of the large industrial centres of the kingdom, where systematic observations and records have been carried out during the period under review. The standard gauge consists of a galvanised iron stand supporting a circular enamelled iron gauge vessel of 4 square feet collecting area. Projecting upwards and outwards above the edge of the vessel is a wire screen, open at the top, to prevent birds from settling on the edge of the vessel and contaminating the contents. The vessel has a conical bottom communicating by a glass tube and rubber connexion with a group of three or more bottles connected together, capable of holding one month's rainfall. The rain and solid matters falling on the gauge area are collected in the bottles and removed once a month for analysis. The gauges are to be placed, if possible, on the ground level in open spaces free from abnormal dust. Before removing the bottles containing the water and deposit, the gauge vessel is to be washed down with some of the collected water, using a brush and squeegee of standard pattern (supplied with the instrument) to remove any adherent matter. Analyses are to be made (generally by the local Public Analyst) according to a uniform scheme briefly outlined below. The bottles are to be allowed to remain at rest in the laboratory for several days until the contents are completely settled; the approximate volume of contents should be noted at the time of receipt.

Undissolved Matter.—The whole of the amount collected should be used for the estimation. The filtration is best done in the following manner: A syphon-tube is inserted in the bottle to within about 1 inch from the bottom and the bottle placed at a convenient height for control of the discharge. The suspended matter is collected in a Gooch crucible supported in a graduated bottle, at least as large as the station bottle, and connected with a suction-pump. The filter-bed is prepared from a tared filter-paper, 12·5 cm. in diameter, which is heated with about 200 c.c. of the sample water, specially syphoned off for the purpose. The paper is shaken violently with the water in a flask until reduced to a pulp, a portion of which is collected in the Gooch crucible to form a filter, and the rest reserved. Filtration of the sample is then proceeded with, the clear liquid being syphoned first. Should the filter become clogged, it should be sucked dry and removed together with the collected deposit, and a fresh bed prepared. Towards the end, the contents of the bottle are shaken up and poured on the filter, the bottle being rinsed out with some of the filtrate. Any of the paper pulp left over or previously removed is finally placed on the top of the matter collected in the crucible. If there be more than one station bottle, it is best to filter the clear liquid of all before collecting the sediment of any. The contents of the Gooch crucible are washed, dried at 105° C., and weighed.

Tarry Matter.—The crucible is placed in an extraction apparatus and extracted with carbon disulphide; the difference found after drying represents tarry matter. *Ash.*—The residue is carefully ignited, loss of particles through the perforations being avoided.

Dissolved Matter.—250 c.c. of the filtrate are evaporated to dryness for estimation of total solids, and the residue is gently ignited. One litre is concentrated, acidified, and treated with barium chloride; *sulphates* are expressed as SO_3. *Chlorides* are estimated as Cl by titration of 500 c.c. with $\frac{N}{10}$ silver nitrate. *Ammonia* is estimated by slightly acidifying 1 litre with sulphuric acid, concentrating to 100 c.c. and distilling from alkali; alternatively 10 to 25 c.c. of the water may be treated with 1 c.c. of Nessler solution. *Lime* is estimated in 1 litre, after adding ammonium oxalate and ammonia and concentrating to 100 c.c. *Alkalinity* or *acidity*, expressed as H_2SO_4 or NH_3, is estimated in 500 c.c. by titration with $\frac{N}{100}$ solutions in presence of methyl orange. All results are expressed as per cent. of total solids, and calculated as metric tons per square kilometre.

Classification aud Discussion of Results.—Full results for the six months, from October, 1914, to March, 1915, have been returned from twenty-five of the thirty-nine recording stations, and are reproduced in the form of tables, illustrated by maps, in the report. The figures to hand have enabled the Committee to classify them according to group limits for each constituent to facilitate comparison and record variations due to season, weather, type of locality, etc. For each constituent a suitable (low) figure has been selected as the unit, all values below which are grouped as Class A. Class B then includes from 1 to 3 units, Class C from 3 to 5 units, and Class D above 5 units. Thus, in terms of metric tons per square kilometre per month, the units are as follow: For total deposited solids, 5 tons; for insoluble matter—tar, 0·05; carbonaceous matter other than tar, 1; ash, 2; for soluble matter—loss on ignition, 0·75; ash, 1·5; total solids, 5; sulphates, 1; chlorine, 0·3; ammonia, 0·05. The units have been selected so as to include a few stations in Class A and a few in Class D, leaving the majority of the stations to be included in one or other of the intermediate classes. Certain exceptional conditions may influence the results from particular stations—*e.g.*, the prevalence of tarred roads may increase the tar, and proximity to the sea the chlorine, etc. The dividing line between Class B and Class C represents, in a manner, the average atmosphere of the winter months, a period during which pollution by domestic fires is at a maximum.

The effect of copious rainfall appears to be to increase the precipitation of solid matters in the immediate neighbourhood of the polluting areas, although its effect over the whole country would show an improvement due to the "washing" of the atmosphere and diminution of mineral dust. J. F. B.

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INSTITUTE OF CHEMISTRY.

THIRTY-EIGHTH ANNUAL GENERAL MEETING, MARCH 1, 1916.

SIR JAMES DOBBIE, the President, referred briefly to the work of the Institute during the war, and the importance of the services of professional chemists to the nation, particularly in the production of munitions and other material of war. The Institute had fulfilled a useful function which he knew was fully appreciated by the authorities. Both in the interests of the profession and of the industries of the country, the Institute had encouraged by every means possible the production of laboratory requirements of all kinds hitherto obtained almost entirely from Germany and Austria.

In co-operation with the Society of Public Analysts, steps were taken to ensure supplies of satisfactory chemical reagents, and a number of British firms immediately undertook the manufacture according to standards prescribed by a Joint Committee of the two Societies.

In connection with the problem of ensuring supplies of laboratory glassware and other forms of glass, the work of the Glass Research Committee of the Institute had been remarkably successful. At the end of six months' work, formulæ had been produced for practically all the various kinds of glass required in chemical operations, in addition to glasses for miners' lamps, pharmaceutical ampoules, and X-ray tubes. A number of manufacturers had taken up these industries, and were now able to supply immediate requirements, so that there was good reason to expect that within a short time they would have completely mastered the technique involved in the production of such articles.

Sir James Dobbie appealed to the chemists to support British manufacturers who had come to their aid, for the country should be independent of foreign sources of supply of a material so indispensable as glass, and, whatever might be their views on the larger question of trade policy, there could be but one opinion as to what the policy should be where those industries usually spoken of as "key industries" were concerned.

The chief credit for this achievement was due to Prof. Herbert Jackson of King's College, London, assisted by Mr. Thomas R. Merton.

The work of the Committee had received the recognition of the Advisory Council on Scientific and Industrial Research, from whom grants had been received for the furtherance of investigations with a view to the determination of formulæ for other glasses required for scientific purposes, including certain forms of optical glass.

Referring to the necessity of taking adequate measures for equipping ourselves for the economical struggle which must ensue when peace is restored, he said that the discussions which had taken place on the subject revealed a wide divergence of views, both as to the cause of the unsatisfactory position in which we found ourselves and the steps required to remedy it. In chemical industries, however, it was generally agreed that the relations between chemical science and chemical manufactures should be more intimate in the future than they had been in the past. That condition could only be fulfilled if the country possessed an ample supply of highly trained chemists. He agreed with Dr. Beilby in the belief that the phenomenal development of chemical industry in Germany had resulted much more from the large command of chemists and engineers of sound professional training than from the possession of an even larger supply of research chemists of mediocre ability. That opinion should not be taken as giving the impression that the value of research was to be under-rated. So far as the supply of chemists of sound professional training was concerned, he thought we could face the future with some confidence, and he instanced the remarkably increased facilities for training chemists which had been afforded in this country within his memory. It had to be admitted, however, that the great public schools were, for the most part, unsympathetic towards the study of science and, even when they were excellently equipped for the purpose, the results were meagre and unsatisfactory.

Referring to the older Universities, he thought it must be allowed that Cambridge had lately achieved an extraordinary measure of success in adapting its teaching to the needs of modern times, while the fact that Oxford was rousing herself to meet her responsibilities was shown by the terms of a Memorandum issued by the Natural Sciences Board in support of a reform in the regulations for the Honours Degree in chemistry, whereby research would become a compulsory part of the curriculum. He advocated a system of general education on broad lines throughout, including both classics and science, up to the proper age for specialisation.

Should they fail to answer the expectation of the country the inevitable result will be that schools established on more modern lines will gradually replace the old public schools as the training ground of the leaders of the nation.

Sir James Dobbie intimated that the Council of the Institute were about to give further consideration to the problem of promoting a more complete organisation of professional chemistry in the interests of the industries of the country.

Referring to the position of the Institute in its relation to scientific and more especially chemical education, he expressed the view that the list of institutions recognised by the Council for the training of candidates for the Associateship should be extended. Colleges which could train candidates satisfactorily for the degree of B.Sc. ought to be encouraged by every means to prepare candidates for professional qualifications. Further, he hoped that the Institute would, without lowering the standard of its requirements, act as an attesting rather than an examining body in the case of graduates who had secured honours in chemistry, following on a course of at least four years' systematic training.

Chemistry was a comparatively young profession which was gradually establishing itself in the knowledge, and, he hoped, the good opinion of the community. It would be successful in this in proportion as it attracted men of strong character and individuality, efficient and capable of holding their own as professional men. As it gained in strength, its services would become more widely recognised and would meet with the same appreciation as that accorded to the older learned professions. The fact that the title "chemist" had long been identified in this country, alone of all European countries, with the craft of pharmacy was responsible for much of the confusion existing in the public mind, but the public were learning at present so much about the work of the chemist that he did not despair of seeing the day when it would be common knowledge that while in law all pharmacists were chemists, all chemists were not pharmacists.

The Officers and Council for the ensuing year were elected as follows:

President: Sir James J. Dobbie, LL.D., D.Sc., F.R.S. *Vice-Presidents:* E. J. Bevan, M. O. Forster, D.Sc., F.R.S.; A. Harden, D.Sc., F.R.S.; O. Hehner, H. Jackson, A. Smithells, B.Sc., F.R.S. *Hon. Treasurer:* A. G. Salamon, A.R.S.M. *Members of Council:* H. Ballantyne, R. F. Blake, R. Bodmer, W. T. Burgess, H. C. H. Candy, B.A., B.Sc.; C. F. Cross, B.Sc.; A. Findlay, M.A., D.Sc.; G. J. Fowler, D.Sc.; G. G. Henderson, M.A., D.Sc., LL.D.; W. R. E. Hodgkinson, Ph.D.; G. T. Holloway, A.R.C.S.; P. H. Kirkaldy, A. H. Knight, A. Lauder, D.Sc.; H. R. Le Sueur, D.Sc.; B. McNeill, A.R.S.M.; G. T. Morgan, D.Sc., F.R.S.; D. Northall-Laurie, F. R. O'Shaughnessy, A.R.C.S.; P. A. E. Richards, W. H. Roberts, M.Sc.; R. Robertson, M.A., D.Sc.; G. Stubbs, F. N. Sutton, W. L. Sutton, T. Tickle, B.Sc.; W. H. Willcox, M.D., B.Sc.

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REVIEWS.

PRACTICAL ORGANIC AND BIO-CHEMISTRY. By R. H. A. PLIMMER. London: Longmans, Green and Co. 1915. Price 12s. 6d. net.

A moderate sized textbook of practical bio-chemistry was undoubtedly wanted, and Dr. Plimmer's pre-eminent qualifications for supplying that want will be admitted on all hands. This book is based upon an earlier work of the author which, under the title "Practical Physiological Chemistry," was originally compiled as a handbook for students of biological chemistry at University College, London. The scope of this earlier work has been very greatly extended, and much new matter has been added, including the description of a number of experimental methods necessary for advanced study.

It may be said at once that the general arrangement is good, and that the author has exercised a wise discrimination in regard to the subjects selected for inclusion. If any fault is to be found at all with the general plan, it is that Dr. Plimmer has perhaps attempted to compress too much matter within a volume of some 600 pages. The majority of the substances with which the bio-chemist is concerned are of a very complex character, and it is doubtful whether medical students, for whom the book is mainly intended, and students of biology generally, will be able to understand and to derive much advantage from the very numerous complicated constitutional formulæ with which the work abounds.

The chapter headed "Complex Aromatic Compounds," and dealing with plant pigments, simply bristles with such formulæ, and constitutes an extreme example; but the tendency to excessive compression is noticeable in a good many other sections of the work.

The writer's descriptions of practical laboratory methods are so clear, and his experience in that direction so wide and so exceptional, that one feels inclined to regret that he has not devoted more space to the laboratory handbook side of the work, and left the advanced student to extract from textbooks of descriptive organic chemistry such information as he might require in regard to the chemical constitution of many of the more complex substances in question.

The work as a whole is so good and so useful that the writer may perhaps be permitted to call attention to a few defects—not in any carping spirit, but solely in the hope that they may be remedied in future editions.

In dealing with oxalic acid, reference should surely have been made to the production of oxalates from formates—a method which is coming widely into use, since it has been found possible to effect the economical synthetic production of formic acid on a commercial scale.

On p. 171 there is an important slip, the author stating that Jaffé's picric acid reaction is characteristic of creatinine. It is scarcely necessary to point out that it has long been known that a great many reducing substances produce the same coloration, and that the reaction, so far from being characteristic of creatinine, is in fact a very general one. Some reference might possibly have been made to the work done on this subject by the writer of this review.

On p. 302 nucleic acid is said to be "very insoluble in water," and immediately afterwards it is stated that its aqueous solutions are not precipitated by acetic acid. Doubtless by "aqueous solutions" the author means solutions in dilute alkalies, but even then the statement is not correct, since such solutions, if they contain but little alkaline acetate and are of sufficient strength, afford precipitates of nucleic acid on the addition of acetic acid, although the precipitation is not as ready or as complete as when mineral acids are used.

The chapter on the terpenes must be pronounced decidedly disappointing. The author commences with the following statement: "The volatile or essential oils, excluding the esters, such as oil of wintergreen, oil of aniseed, mustard oil, etc., which are contained in the flowers and stems of plants, especially the various kinds of conifer and citrus, are grouped together under the term Terpenes. They are the constituents of turpentine, india-rubber, and various resins. The various kinds of camphor are terpenes containing oxygen in their molecule." He also refers to the "solid terpenes such as camphor," and speaks of santalol as a sesquiterpene.

The above definition is clearly incorrect, the word "terpene" in its widest sense being restricted by all chemists to hydrocarbons having the empirical formula $(C_5H_8)_n$, these being again divided, as the author correctly states, into hemiterpenes, terpenes proper, sesquiterpenes, and polyterpenes.

This chapter affords evidence of excessive compression, and is, in fact, little more than a collection of structural formulæ. It would surely have been better, in a book intended mainly for medical students, to have devoted more space to a description of the more important terpenes and their oxygen derivatives, particularly to those which are of practical importance. As an example of the excessive condensation, it may be pointed out that pinene—in many respects the most important of all the terpenes—is dismissed in two or three lines.

In speaking of the strength of acids, it would have been better if the author had given a numerical statement in every case, and had devoted a paragraph to explaining the meaning of the numbers so given. In this way the student would be able to appreciate the great differences shown by organic acids in this respect, and to form some idea of the influence on this property of certain substituent elements or groups of elements, as in the case of the chloracetic acids mentioned on p. 100.

On p. 67 it is stated that in the determination of alcohol by distillation the specific gravity of the alcoholic distillate "is taken with a hydrometer." It may be pointed out that such a method would not be sufficiently accurate for most purposes.

Among matters of minor importance, I would suggest that the expression "under reduced pressure" should be substituted for the expression "in vacuo" as applied to operations involving distillation and evaporation; and also that it would be well if the terms "ligroin" and "gasolin," both of which appear in this work, were replaced by the expression "light petroleum," with an added statement as to the range of boiling temperatures when necessary. It may also be pointed out that the expression "·1 N" for $\frac{N}{10}$ is unusual, and that it is more liable to be misprinted through the accidental omission of the decimal point.

The reviewer has noticed very few typographical errors, the paper is good, the printing is clear, and there is a really useful index. The illustrations are very good, and special commendation must be given to those representing various crystal forms, some of which are reproduced from Funke's "Atlas of Physiological Chemistry."

A good knowledge of bio-chemistry and of bio-chemical methods is becoming increasingly important to a considerable section of the analytical profession, and to all analysts who are interested in the examination of foodstuffs and similar products this book may be warmly recommended. The practitioner will find within its covers a clear account of a good many analytical methods which have stood the test of experience, and of which it is not always easy to find a description in the usual books of reference.

A. CHASTON CHAPMAN.

SURFACE TENSION AND SURFACE ENERGY, AND THEIR INFLUENCE ON CHEMICAL PHENOMENA. By R. S. WILLOWS and E. HATSCHEK. Pp. viii + 80. Fifteen diagrams. London: J. and A. Churchill, 1915. Price 2s. 6d.

This excellent little book is based on a course of lectures given at the John Cass Institute, and deals with the fundamental laws of surface tension and surface energy. It must be confessed that to many students of chemistry and biology who are anxious to gain an insight into this important branch of physical chemistry the mathematical treatment to be found in the larger works acts as a deterrent, and for this reason in the present little volume the mathematical development has been omitted.

The subject is treated in five chapters, of which the first two deal with surface tension and surface energy. In the third chapter the relation between surface tension and osmotic pressure is discussed, and reference made to the theories of Traube, Gibbs, and Van der Waals. In the fourth chapter we have a short account of adsorption, while the last chapter deals with the effect of electric charge on surface tension.

The presentation of the subject is exceedingly clear, and cannot but find favour with those students who wish for a concise account and discussion of the phenomena without the necessity for their following the many steps in the mathematical arguments. The book is well printed, and is singularly free from typographical errors.

E. C. C. BALY.