

THE ANALYST.

OBITUARY.

CLAYTON BEADLE.

THROUGH the death of Clayton Beadle on August 16, in his fiftieth year, the Society loses a member who attained considerable prominence in the chemistry of cellulose and its technical applications. Beadle started work at a relatively early age in the paper mills of his cousin, Edmund Joynson, at St. Mary Cray, and there acquired a thorough knowledge of the art of paper-making, which stood him in very good stead in later years when brought face to face with analytical and technical problems connected with the manufacture of paper. After a few years at St. Mary Cray he entered into partnership for a period with Cross and Bevan, during which the discovery of viscose was made. Later he left to develop a viscose product known as viscoïd in experimental works at Erith. In 1903 he returned to London and commenced to practise in conjunction with the writer as analytical and consulting chemists, the partnership continuing till the date of Beadle's death.

Mr. Beadle was author or joint-author of a number of papers published in this and in other scientific and technical journals. He was also the author of a series of small technical works entitled "Chapters on Paper-making." He took part in the investigation of fibres for the Indian and Colonial Exhibition, and earned distinction as an authority on the Chemistry of paper-making. His work received recognition, and he was awarded medals by the Royal Society of Arts, Société d'Encouragement pour l'Industries Nationale (Paris), and the Franklin Institute, Philadelphia, the last being for work undertaken in conjunction with his colleagues, Messrs. Cross and Bevan.

H. P. STEVENS.

JOHN JOSEPH EASTICK.

JOHN JOSEPH EASTICK, whose death on September 7, 1917, was somewhat sudden, although he had been ailing for a considerable time, was for many years a member of the Society. He was born on February 6, 1855, at Great Yarmouth, his father being manager of the Southtown gas-works there, and afterwards at Peel, Isle of Man. When the family came to Lancashire, he took up the study of science, and gained an exhibition at Owens College. He subsequently obtained a Royal Exhibition at the Royal School of Mines, and threw himself with ardour into the various science subjects taught there, finally obtaining the Associateship of the school. His bent was always towards the technical utilisation of scientific results, and he found

considerable scope for his abilities as first chemist at the sugar refinery of Messrs. Abram Lyle and Sons, where he initiated their special methods of making Brewers' Saccharum, Invert Sugars, and Golden Syrup. From 1890 to 1894 he built and managed the Australasian Sugar Refinery, Melbourne, and acted as Honorary Adviser to the Victorian Government on beet cultivation and sugar manufacture. Leaving Melbourne for Bundaberg, Queensland, he spent a year on improvements and the rearrangement of Messrs. Cran and Tooth's juice mills. During the next ten years he converted the Millaquin and Yengarie juice mills into juice and raw-sugar refineries (carbonatation process), was general manager for the Queensland National Bank of their refineries, three raw-sugar mills, several juice mills and plantations. He was also director and chairman of the Bundaberg Distillery for several years. In 1906 he inspected tropical and subtropical agriculture in the East, but owing to failing health he returned to England, and has since then been consulting sugar expert and chemical engineer, in addition to which, at the death of his father-in-law, Mr. B. E. R. Newlands, he took over the analytical and consulting practice of Newlands Bros. at No. 2, St. Dunstan's Hill, E.C. He leaves a widow, four daughters and three sons, the latter of whom are carrying on the traditions of their father in applied chemistry.

Tribute will always be paid to his memory by his many pupils, co-workers and employees, and all who were acquainted with him for his never-failing kindly advice and aid.

L. J. DE WHALLEY.

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

TREATMENT OF CORKS USED IN SOXHLET AND OTHER EXTRACTION APPARATUS.

By T. J. WARD.

THE difficulties which arise from the porosity of cork and the solubility of certain constituents of cork when used in extraction apparatus may be overcome in the following manner: Gelatin is soaked in cold water for five or six hours, the unabsorbed water poured off, and the gelatin warmed gently until it is melted, when one quarter of the volume of glycerol and two volumes of water are added.

The corks, which have previously been softened and bored, are completely immersed in the glycerol-jelly solution contained in a covered beaker, and heated on a boiling water-bath for two hours. They are then removed, the whole of the surface wiped with a cloth, the corks dried in a warm place for a few hours, and then used in a Soxhlet extraction apparatus for one hour.

Corks treated in this manner may be safely exposed to the vapour of ether,

petroleum ether, benzene, carbon disulphide, and chloroform, but must not be used with solvents in the vapour of which water and glycerol are readily soluble.

The corks should be stored at the ordinary temperature away from any source of heat, as a warm, dry atmosphere causes them to shrink and harden.

Four corks, two of which were very porous, treated in the manner described, were used to connect the flask, Soxhlet extractor, and condenser, in a series of ether extractions. The jelly was then completely removed with hot water and the corks dried at the ordinary temperature, after which a further series of extractions was made. The extraction in all cases lasted for one hour.

The following results were obtained:

		<i>Mgrams. of Ether-soluble Matter extracted.</i>	
<i>Extraction.</i>	<i>Corks A and B (very porous).</i>	<i>Corks C and D.</i>	
After treatment with glycerol jelly	1	5	1
	2	0	0
	3	0	0
	4	0	0
After removal of glycerol jelly	5	13	7
	6	10	3
	7	7	9
	8	7	6
	9	8	5
	10	6	3

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

New Unit for the Estimation of Food Values. H. Suchting. (*J. für Landwirtschaft*, 1916, **64**, No. 3, pp. 163-170; through *Int. Rev. Sci. and Pract. of Agric.*, 1916, **7**, 1797.)—Kellner's "starch value" is a unit sometimes preferred to the "calorie" or energy unit, for the practical reason that it is much larger and therefore simplifies calculations. The author questions the inferiority of the calorie, and shows how it may be modified for use. Kellner stated his starch values to a tenth of a kgrm., and with such a degree of accuracy the equivalent calorie value certainly runs into large and unwieldy numbers. The author proposes that a unit of 1,000 calories be adopted in the place of starch values (and decimals of starch value), and points out that even this unit is too small, as 1,000 calories are equivalent to 0.25 kgrm. of starch. He considers this new unit to be more practical, simpler, and more exact than starch values. From the physiological point of view, too, it is more satisfactory to compare the animal body to an engine supplied with

fuel, and to obtain the food value in terms of the fuel, which can then be easily converted into force, than to obtain the food value in terms of one of the substances in the food.

The value of digestible food substances would thus be expressed in heat units or large calories, for which the name of "Kellner values" is proposed, and 1 kgrm. of starch, protein, and fat, would be equivalent to 4, 4 and 9 Kellner values respectively. For example, the Kellner value of 100 kgrms. of wheat (95 per cent. digestible), containing 9 per cent. of protein, 1 per cent. of fat, and 64 per cent. of nitrogen-free extract, would be:

9 kgrms. protein	=	9	×	4	=	36	
1 kgrm. fat	=	1	×	9	=	9	
64 kgrms. N-free extract	=	64	×	4	=	256	
Tota.											301

$$\text{or } \frac{301 \times 95}{100} = 286 \text{ Kellner values.}$$

Analysis of Milk. E. Ackermann. (*Ann. Chim. anal.*, 1917, 22, 152-158.)—To distinguish between abnormal and watered milk, it is suggested that the quantity of lactose present be estimated, and that this quantity be subtracted from the percentage of non-fatty solids of the milk. In the case of normal milk free from added water the result of this subtraction is usually above 4; when the milk is abnormal owing to ill health of the cow, the quantity of lactose will be less than usual, but the subtraction result will be higher than 4. With watered milk all the results will be low. The lactose content of milk may be calculated from the refraction of the serum, and tables are given for this purpose.

W. P. S.

Examination of Milk. G. Wilhelm. (*Zeitsch. Unters. Nahr. Genussm.*, 1916, 32, 573-576; through *Int. Rev. Sci. and Pract. of Agric.*, 1917, 8, 648-649.)—The refractometer value of the serum of cow's milk (prepared by the calcium chloride method) does not fall below 38 in the case of genuine milk, and there is a certain relation between the refraction and sp. gr. of milk. If the sp. gr. expressed in degrees of the lactodensimeter (L) be subtracted from the refractometer value (R), a practically constant number is obtained, usually varying between 9 and 10. If the milk contains added water, this number will be lower; for instance, if $R - L = 5$, the milk contains 20 per cent. of added water.

To some extent the quantity of whey yielded by a milk is an indication of presence of added water. If the volume of whey from 100 c.c. of milk exceeds 68 c.c., the milk has been watered. The author has also observed that the acidity of watered milk is lower than that of genuine fresh milk. In the case of genuine milk the acidity usually lies between 17° and 22° (Thörner's scale; number of c.c. of $\frac{N}{10}$ sodium hydroxide solution required to neutralise 100 c.c. of milk when a mixture of 10 c.c. of milk and 20 c.c. of water is titrated); in watered milk it may fall to 15° or even 13°

W. P. S.

Estimation of Phenol in Commercial Cresylic Acid. J. J. Fox and M. F. Barker. (*J. Soc. Chem. Ind.*, 1917, **36**, 842-845.)—A method has been worked out to estimate the amount of residual phenol left in cresylic acid after the bulk of the crystallisable phenol has been taken out. The method depends on the fact that the addition of a suitable proportion of *o*-cresol furnishes a mixture from which the whole of the phenol can be recovered in the first fractions of the distillate. The proportion of *o*-cresol to be added is ascertained by a preliminary distillation of the tar acids. The volume of the cresylic acid distilling between 195° and 205° C is noted, and an equal volume of *o*-cresol is added to the original tar acids. Good commercial *o*-cresol is, as a rule, sufficiently pure for use, but it is advisable to assay it by distilling off other matters in a current of steam from a sodium hydroxid solution, and fractionally distilling the acidified residue. The examination of the sample of cresylic acid is carried out as follows: 100 c.c. of cresylic acid are shaken with 200 c.c. of sodium hydroxide (sp. gr. 1.2), and the liquid is thoroughly extracted with ether, making three extractions with 20 to 30 c.c. of ether each time. The alkaline solution is acidified during cooling, the tar acids are separated, and the clear aqueous layer extracted with ether, the extracts being added to the bulk of the tar acids. The tar acids are washed with sodium sulphate solution, dried with calcium chloride, and the ether is removed by distillation. The fractionation of the tar acids is then effected in a suitable column. The first distillation is carried up to 210° C. This fraction is again distilled, the first fraction up to 195° C., and the second from 195° to 196.5° C., being collected separately and weighed. Provided the first fraction amounts to about 50 per cent. of the volume of the total, the phenol can be estimated at once by the freezing-point method; but if it is much less, sufficient *o*-cresol is added to the product of the first distillation to bring the fraction boiling up to 195° C. to at least 50 per cent. The fraction up to 195° C., whether obtained direct or after the addition of *o*-cresol is weighed, dried with calcium chloride, and used for the determination of freezing-point after the addition of pure phenol sufficient to yield a mixture containing at least 80 per cent. of phenol. About 15 grms. of this mixture are required for the determination. This liquid is placed in a test-tube 6 inches by $\frac{3}{4}$ inch, and this is surrounded by another test-tube 6 inches by $1\frac{1}{4}$ inches, and placed in a bath of water at about 20° C. The solidifying-point is taken while the liquid is stirred regularly with a stout copper wire; at the solidifying-point the thermometer rises to a maximum, and remains stationary for some time. Reference is then made to the curve of solidifying-points of known mixtures of phenol and cresols for the amount of phenol in the mixture. Data for these curves, obtained with mixtures of pure phenol and commercial distillates free from phenols, are as follows: 100 per cent. phenol, 40.1° C.; 95 per cent., 37.4°; 90 per cent., 34.5°; 85 per cent., 31.7°; 80 per cent., 28.9°. Having found the percentage of phenol in the enriched portion of the distillate, the estimation of phenol in the original sample is a matter of calculation. The fraction distilled between 195° and 196.5° C. should be tested for the presence of phenol; the sp. gr. at 15.5° C. should be less than 1.0516, and treatment of 0.1 to 0.2 gm. with bromine-water in presence of dilute hydrochloric acid should not show a voluminous flocculent precipitate consisting of microscopic needles.

J. F. B.

Estimation of Phenol in Crude Carboic Acid and Tar Oils. J. M. Weiss and C. R. Downs. (*J. Ind. and Eng. Chem.*, 1917, 9, 569-580.)—The authors have worked out a standard method for the estimation of phenol in crude carboic acid and tar oils, which, however, requires great care and attention to details. If the oil is at all dirty, it must be distilled in a copper tar-still, taking the total distillate up to the coking-point. The tar acids are then extracted by shaking a quantity of the oil containing 300 to 350 grms. of acids with 300 c.c. of 20 per cent. caustic soda solution. The solution of sodium carbolate should separate completely from the oil after standing fifteen to thirty minutes; if the oil contains solid matter, it is maintained all the time at a temperature sufficient to keep the solids in solution. The separated oil is washed successively with three more portions of 200 c.c. each of the caustic soda solution, and the washings are added to the original carbolate extract. The carbolate is then extracted in a separating funnel with five successive portions of 100 c.c. of pure benzene. The benzene is added to the carbolate, shaken, and allowed to separate; the carbolate is drawn off into another separating funnel, and the benzene into a third one. Finally, after running the washed carbolate into a beaker, the two funnels in which it has been treated are rinsed out with 25 c.c. each of water. The combined benzene extract is washed twice with 50 c.c. of water, which is added to the carbolate. The latter is boiled gently to drive off the benzol; no smell of creosote should be perceptible. The purified carbolate is acidified in a separating funnel with sulphuric acid of 25 per cent. strength. During acidification the mass must be kept below 40° C.; the test for acidity is made with litmus-paper. More than a very slight excess of acid (5 to 10 c.c.) must not be added. The liquor is allowed to settle until practically clear (three to four hours); the aqueous portion is drawn off, and the acid layer is transferred to a weighed round-bottomed flask. The sodium sulphate solution is returned to the separating funnel and extracted three times with 100 c.c. portions of benzene, the procedure being the same as before; the benzene is washed twice with 25 c.c. of 20 per cent. caustic soda. The latter is acidified, and the separated acids added to the main portion. The weight of wet crude acids is recorded. The crude acids are then subjected to fractional distillation in an apparatus the details of which are minutely prescribed. The distillate is collected at the rate of one drop per second in a 100 c.c. separating funnel until the temperature has reached 170° C. Distillation is interrupted, and sufficient salt added to the distillate to cause the separation of the acids; the aqueous layer is removed and the acids returned to the distillation flask; the weight of the dry acids is then determined. Distillation is then resumed, the first few drops, which contain water, being collected in a separating funnel. When no more water comes over, the distillation is carried on to 190° C. The acid separated from the water is added to this first fraction, and a second fraction is collected from 190° to 202° C. in a 250 c.c. round-bottomed flask. This is substituted for the distillation flask, and the second fraction is redistilled up to 197° C., the distillate being collected together with the first fraction; the weight of the combined fraction up to 197° C. is recorded. This fraction is tested for sp. gr. and solidifying-point. The solidifying-point is determined under prescribed conditions in a test-tube 1 inch inside diameter and 5 to 6 inches long, in which the liquid stands 2 inches high. The

tube is placed in a bath 4.5° to 5.5° C. below the solidifying-point, and the melted sample allowed to cool with constant stirring. When crystals begin to separate, a rise in temperature is noted, which reaches a maximum point at which it remains constant; duplicate tests should show identical values. The sp. gr. is taken at 25°/25° C. or at 45°/45° C. according to the solidifying-point, with a Westphal balance. The results for solidifying-point and sp. gr. are compared with curves plotted with solidifying-points as ordinates and sp. gr. as abscissæ made with known mixtures of phenol and cresols. These curves have been constructed by the authors, and contain "indeterminate areas." If the results fall outside the determinate areas, it is necessary to add to the sample a known quantity of pure phenol sufficient to bring them within the required limits. The percentage of phenol in the oil is then calculated from the data obtained.

J. F. B.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Studies on the Culture Media employed in the Bacteriological Examination of Water. IV. Neutral Red Lactose Peptone Media. E. M. Chamot and C. M. Sherwood. (*J. Amer. Chem. Soc.*, 1917, **39**, 1755-1766.)—Neutral red—dimethyldiaminotoluphenazine—discovered by Witt in 1879, was not applied to bacterial diagnosis until 1898, when it was stated by Rothberger that its presence in bouillon agar afforded a useful means of distinguishing between *B. coli* and *B. typhosus*. Stokes (*J. Infect. Dis.*, 1904, **1**, 341) employed a sugar medium containing neutral red in a Smith fermentation tube, thus enabling the reduction of the neutral red, the quantity of gas produced, and the ratio of carbon dioxide to hydrogen, to be noted; he also recognised the selective value of lactose for the diagnosis of *B. coli*. The present paper is an attempt to ascertain the nature of the changes produced by the reducing bacteria, and the best diagnostic concentration of the components in the Stokes medium. It was found that a neutral red medium composed of from 3 to 4 per cent. peptone, 0.8 per cent. potassium chloride or sulphate, 0.6 per cent. lactose, and 0.008 per cent. neutral red, with a reaction of +1 per cent., affords a very sensitive and accurate medium for the speedy detection of fæcal pollution by bacteria. The addition of meat broth increases the sensitiveness of the medium, but is not essential. The Stokes neutral red medium is a convenient and reliable one for the detection of fæcal contamination in water, and is more sensitive than lactose bile. The yellow fluorescent compound formed by the action of the bacteria is most probably dimethyldiaminomethylhydrophenazine, a simple reduction product of neutral red. Ammonia does not enter into the formation of the reduction product. (*Cf. also ANALYST*, 1915, **40**, 446, 467.)

H. F. E. H.

Substitute for Litmus for Use in Milk Cultures. W. M. Clark and H. A. Lubs. (*J. Agric. Res.*, 1917, **10**, 105-111.)—Since litmus is liable to undergo reduction, or even destruction, during sterilisation in the presence of milk, the use of dibromo-*o*-cresol-sulphonphthaleïn is recommended in estimating the degree of acid or alkaline fermentation in milk cultures. This substance is not affected by sterilisation, may be used in dilute solution, and its cost is not excessive.

W. P. S.

ORGANIC ANALYSIS.

Identification of Acids—III. E. Lyons and E. E. Reid. (*J. Amer. Chem. Soc.*, 1917, **39**, 1727-1750.)—The authors continue the preparation of crystalline esters by the methods already described (*ANALYST*, 1917, **89**, 215); and, of sixty-four carboxylic acids studied, fifty gave crystalline *p*-nitrobenzyl esters (two of which did not melt at 300° C.), and fourteen gave oils. Sulphonic acids could not be induced to form esters. More trouble was encountered with the aliphatic acids, since the esters of many monobasic acids are oils. It was found that 0.5, or even 0.25, of the reagent is quite sufficient for a determination. A beginning has been made on the identification of acids in mixtures; thus, benzoic acid is readily identified in the presence of acetic, tartaric, citric, salicylic, and *p*-toluene sulphonic acids. Modified details are given for the preparation of *p*-nitrobenzyl bromide (*ANALYST*, 1917, **42**, 89), which crystallises in fine nearly white needles and melts at 99° C. Since *p*-nitrobenzyl chloride can be readily obtained in large amount, its transformation into the bromide was studied, the best yield being obtained by boiling the chloride in 95 per cent. alcohol with an excess of sodium bromide. A good yield is thus obtained, but it is contaminated with unchanged chloride, so that by the time it is purified up to m.-pt. 99° C. only about 25 per cent. is obtained.

H. F. E. H.

Action of Sulphur Monochloride on Bituminous and Tarry Substances and Hydrocarbon Oils. J. V. Meigs. (*J. Ind. and Eng. Chem.*, 1917, **9**, 655-658.)

—It is shown that sulphur chloride acts on bituminous matter, tarry substances, and hydrocarbon oils, giving hydrochloric acid gas as one product of the reaction. Sulphur monochloride is proposed as a reagent for testing the comparative stabilities of transparent lubricating oils. The sulphur monochloride (1 c.c.) is added to 5 c.c. of the oil in a test-tube, and the mixture is allowed to stand in the cold for fifteen minutes, and the results are then compared with those obtained on an oil of known refinement. Highly refined gas-engine oils, for example, show but a slight wine-coloured tinge, whereas lower-grade oils change to a deep wine or even black colour.

Sulphur monochloride, in carbon disulphide, is also proposed as a reagent for investigating the nature of bitumens and hydrocarbon oils, a known weight of the dried substance being dissolved in carbon disulphide, treated with excess of the reagent under standard conditions, and the resulting hydrochloric acid separated from sulphur chloride by bubbling through carbon disulphide, absorbed by water and titrated with standard alkali. The number of c.c. of alkali required multiplied by 100 and by the normality of the solution, and divided by the weight of the sample, is termed by the author the "hydrogen number" of the sample. G. C. J.

Influence of Calcite Inclusions on the Estimation of Organic Carbon in Soils. E. C. Shorey and W. H. Fry. (*J. Ind. and Eng. Chem.*, 1917, **9**, 588-589.)

—Many soils derived from limestones are peculiar in containing calcite and iron oxide enclosed in quartz. It is evident that so long as the quartz particles are intact the included calcite is protected from the action of acids, and consequently is not

estimated in the determination of the carbonates of the soil. These calcite inclusions may be detected microscopically by mounting the grains in an oil of approximately the same refractive index as quartz. Two methods are available for the estimation of organic carbon in soils: the cupric oxide combustion method, which is extremely tedious, but is regarded as an absolute method; and the moist combustion method with sulphuric and chromic acids, which is simple, but is less direct. Both these methods give the total carbon in the soil, and the difference between this value and that of the carbonates separately estimated is calculated as organic carbon. In this way a source of error is introduced into the dry cupric oxide combustion method, since the carbonate included in the quartz has escaped the separate estimation, but appears in the total carbon owing to the rupture of the quartz particles at the high temperature employed. By the moist combustion method, on the other hand, the inclusions of calcite are not attacked, and this method consequently may yield more accurate results for organic carbon than the absolute combustion method. It should be noted that, in order to obtain the whole of the carbon dioxide of the calcite inclusions by the cupric oxide method, the heating must be prolonged far beyond the time usually allowed for organic combustion. Much depends on the fineness of the sample, but the quartz grains containing the calcite are very small, since a sample passed through an 80-mesh sieve may show less than 0.01 per cent. of carbon dioxide on treatment with acid, and yet yield 0.12 per cent. by cupric oxide combustion in excess of the quantity properly attributable to the organic matter. J. F. B.

Testing of Motor Gasoline. E. W. Dean. (*Techn. Paper 166*, Bureau of Mines, U.S.A. Pp. 1-27.)—The commercial types of gasoline may be classified into—(1) "Straight" refinery gasoline; (2) blended casing-head gasoline; and (3) cracked and blended gasoline. Speaking generally, No. 1 is produced by distilling the crude oil in a fire-still until the sp. gr. of the distillate reaches a definite point. The so-called "crude naphtha" or benzine is refined by acid, and distilled with steam. "Straight" refinery gasolines usually contain a low proportion of unsaturated and aromatic hydrocarbons, and show few pronounced irregularities when distilled. The second type of products consists of gasoline compressed from natural gas, and blended with sufficient naphtha to give a cheap and safe mixture. In chemical characteristics it resembles "straight" refinery products with the same range of distillation. The third type resembles "straight" refinery products in most physical and chemical properties, but differs therefrom in composition in containing varying proportions of unsaturated and aromatic hydrocarbons. As practical specifications for motor gasoline, the following suggestions are made: *Colour*: The sample should appear "water-white" when examined in a 4-ounce sample bottle. *Acidity*: On shaking 10 c.c. of the gasoline with 5 c.c. of water, the aqueous extract should not give a pink colour to blue litmus-paper. *Volatility*: When distilled by the method described below, the gasoline should answer to the following requirements: (a) The temperature shown on the thermometer after the distillation of 20 per cent. should not be below 70° C. (158° F.), nor above a limit fixed in accordance with the conditions of use. (b) The temperature after the distillation of 90 per cent. should not exceed another limit similarly chosen. (c) The

temperature after the distillation of 50 per cent. should not be higher than a mark halfway between the 20 per cent. and 90 per cent. limit. (d) The dry-point should not exceed the actual 90 per cent. reading by more than 55° C. (99° F.). If either the 20 per cent. or the 90 per cent. temperature exceed the required limit by not more than 10° C. (18° F.), the gasoline may be considered acceptable, provided that the sum of the two temperatures for the 20 and 90 per cent. marks does not exceed the sum for the adopted limits. *Distillation Method and Apparatus:* The method is essentially that recommended by Subcommittee XI. of Committee D of the American Society of Testing Materials. The distillation flask is the standard Engler 100 c.c. flask, with the following dimensions: Diam. of bulb, 6.5 cm.; diam. of neck, 1.6 cm.; length of neck, 15.0 cm.; length of vapour tube, 10.0 cm.; diam. of vapour tube 0.6 cm. It should be supported on a ring of asbestos with a circular hole $1\frac{1}{2}$ inches in diameter in the middle. The condenser consists of a thin-walled tube of brass or copper, 22 inches long and $\frac{1}{2}$ inch internal diameter. It is set at an angle of 75° from the perpendicular, and is surrounded by a water-jacket of the trough type. The lower end of the condenser is cut off at an acute angle, and is curved down for a length of 3 inches. The condenser jacket should be 15 inches in length. The thermometer should have a diameter, between 5.5 and 7 mm. The bulb should be of Jena normal or Corning normal glass, and should be 10 to 15 mm. long, and have a smaller diameter than that of the stem. The total length of the thermometer should be about 380 mm., and the length of the graduated portion between 210 and 250 mm. The 35° C. mark should be not less than 100 nor more than 120 mm. from the top of the bulb. *Method of Distillation:* The flask should be charged with 100 c.c. of gasoline, and heat gradually applied, so that the distillation proceeds at the rate of not less than 4 or more than 5 c.c. per minute. Readings are taken after the distillation of each 10 c.c., and the "dry-point" or highest reading at the end of the distillation is also recorded. The distillation loss is determined by adding the percentage of cooled residue in the flask to that of the total distillates. If the loss exceeds 3 per cent., and the initial rate of distillation has not been too rapid, the gasoline contains very volatile constituents, such as are present in casing-head gasoline. The condenser trough should be filled with a mixture of crushed ice and water, and the temperature of the cooling water should not be allowed to exceed 8° C.

C. A. M.

Composition of Menhaden Oil Fatty Acids. E. Twitchell. (*J. Ind. and Eng. Chem.*, 1917, 9, 581-584.)—The method described by the author (*ANALYST*, 1914, 39, 448), for determining the composition of a mixture of fatty acids by the lowering of the melting-point produced by the addition of a known percentage of the mixture to a pure acid of known melting-point, has been applied to the analysis of the fatty acids obtained from menhaden oil. In most cases the lowering of the melting-point of the solvent acid is proportional to the quantity of foreign acid up to about 20 per cent.; but when the solvent acid has an unusually low melting-point as compared with the added acid—as, for instance, in the case of behenic acid dissolved in myristic acid—proportionality is not insured beyond 10 per cent. of the added acid of higher melting-point. When the added acids are unknown, but are derived

from common fats not differing greatly in molecular weight, an average value of 4° C. may be assumed for the depression of the melting-point caused by adding 20 parts of one acid to 80 of another; but when the acids are to some extent known, it is better to use values which have been directly determined. This method in the ordinary way is limited to fatty acids solid at the ordinary temperature, but it can be extended by applying it to the solid acids produced from liquid acids either by the hydrogenation process or by fusion with caustic potash. The results recorded in the previous paper (*loc. cit.*) for some of the fatty acids from menhaden oil have been extended by the preparation of pure specimens of myristic and arachidic acids, and the distillation fractions of hydrogenated menhaden oil have been examined. Myristic acid (9.2 per cent.) was found in the same proportions in the fatty acids both from the original and the hydrogenated oils; it is therefore not produced by the hydrogenation. On the other hand, arachidic acid (22.2 per cent.) was found only in the acids from the hydrogenated oil, and must have been produced by the addition of hydrogen to an unsaturated acid with 20 atoms of carbon. The extended results of the examination of the fatty acids from menhaden oil now read: Palmitic acid, 22.7 per cent.; myristic acid, 9.2; stearic acid, 1.8; unsaturated acids with 16 carbon atoms, none; unsaturated acids with 18 carbon atoms, 24.9; unsaturated acids with 20 carbon atoms, 22.2; unsaturated acids with 22 carbon atoms, 20.2. By fusion of the oil with caustic potash only a small and doubtful quantity of arachidic acid was found, hence it is concluded that the unsaturated acid with 22 carbon atoms, which, like erucic acid, is converted into behenic by hydrogenation, is nevertheless not erucic acid, since it is not converted into arachidic acid by fusion, but yields apparently stearic or palmitic acid. J. F. B.

Estimation of Succinic Acid. E. C. Grey. (*Bull. Soc. Chim.*, 1917, 21, 136-141).—The mixture of acid products of a fermentation is distilled in a current of steam, and the residual solution of non-volatile acids is extracted with ether for several hours. The acid separated from the ethereal extract is dissolved in about 100 c.c. of hot water, and the solution gradually neutralised with calcium carbonate, and finally with lime-water, and then heated for at least an hour to complete the conversion of lactone into calcium lactate. The liquid is then filtered, and made up to 200 c.c., and 50 c.c. taken for the estimation of the calcium corresponding to the succinic, lactic, and other acids present, the calcium being precipitated as oxalate, and the oxalic acid estimated by titration with permanganate solution. Another portion of the same solution of calcium salts is concentrated slowly to 13 c.c., and this residue is brought to 110 c.c. by means of 96 per cent. alcohol, the final alcoholic strength being 85 per cent. The flask is thoroughly shaken, and left for several hours until the precipitate becomes crystalline. The liquid is then filtered, and the calcium estimated in 100 c.c. of the filtrate. The amount multiplied by $11/10$ gives the quantity of calcium corresponding to the acids the calcium salts of which are soluble, and the difference between the result, and that previously obtained, gives the amount of calcium corresponding to the succinic acid. The experimental error should not exceed 1 per cent. of the weight of succinic acid present. If the amount of acid is very small, it is not necessary to make a

separate estimation of the total calcium and of the calcium salts soluble in alcohol, but, after filtration of the 100 c.c. of alcoholic solution, the residual 10 c.c. and the calcium succinate precipitate may be dissolved in water and the calcium estimated. The difference between the result and the amount of calcium corresponding to the 10 c.c. of alcoholic solution gives the amount of calcium in the precipitate.

C. A. M.

Chemistry of Wood. I. Methods and Results of Analysis of Some American Species. A. W. Schorger. (*J. Ind. and Eng. Chem.*, 1917, 9, 556-561.)—The author has made a revision and selection of analytical methods to be used for the chemical examination of wood. Since most of these methods are empirical, it is obvious that the conditions of concentration and time of treatment must be sharply defined, and that the physical preparation of the sample is of primary importance. Starting from cross-sectional discs about 2 inches thick cut about 20 feet from the ground, this being the most uniform part of the tree, two diagonally opposite sectors are split out. The material is employed in the form of sawdust and shavings not more than 0.005 inch in thickness; with some very soft woods a satisfactory sample may be obtained by scraping with glass. Shavings are ground in a mill, having a shredding effect, to fragments 3 to 5 mm. long; powder which passes through a 40-mesh screen is rejected. The remaining portions of the sectors are cut down with a circular saw with a guide adjusted to reduce the material entirely to sawdust. A portion of the sawdust is reserved for the estimation of moisture and volatile oil, while the remainder is air-dried and ground so as to pass through a 40-mesh sieve. The moisture in the finely ground sample is estimated by drying in the oven for six hours at 105° C., and the oven-dried weight serves as the basis of calculation of the other results. Twenty-five grms. of the coarse sawdust are distilled with 75 c.c. of xylene saturated with water, and the percentage of water in the distillate is read off directly. Ten grms. of the same sample are placed in a flask in an oil-bath heated to 110° C., and the volatile oil is driven off by the passage of a current of steam. The wood residue is dried in the flask and weighed. The loss of weight, less the moisture determined by the xylene method, represents the volatile oil. All the other estimations except the cellulose are performed on the fine sifted sawdust. Waxes, fats, and resins, are determined by extraction for four to six hours with ether and weighing the dried ethereal extract. Alkali-soluble matter is determined on 2 grms. of material digested in the boiling water-bath with 100 c.c. of 1 per cent. sodium hydroxide for one hour, collecting, acidifying, and weighing the residue. Hot-water-soluble matter is estimated in a similar manner by boiling for three hours with 100 c.c. of water under a reflux condenser. Cold-water-soluble matter is extracted by infusion for forty-eight hours with 300 c.c. of water at the room temperature. The method for the estimation of furfural and methylfurfural is described; it follows the usual lines. The mixed phloroglucides in the Gooch crucible are extracted by placing the crucible in a beaker with 20 c.c. of 95 per cent. alcohol, and digesting at 60° C. for ten minutes. The extraction is repeated four or five times until the alcohol is practically colourless. For cellulose, 2 grms. of fine shavings are extracted with a mixture of equal parts of alcohol and benzene for

three to four hours, washed with hot water on the pump, and subjected to chlorination in a beaker; the chlorinated mass is treated with sulphurous acid, washed and digested with 100 c.c. of 2 per cent. sodium sulphite solution for thirty minutes in the boiling water-bath. The product is collected, washed in a crucible, and subjected to further treatments with chlorine for fifteen to thirty minutes each, until the colour reaction with sulphite has practically disappeared. The cellulose is finally bleached with 20 c.c. of 0.1 per cent. permanganate and dried at 105° C. The methoxyl value is determined by the usual method. Acid hydrolysis: Two grms. of fine sawdust are boiled under a reflux condenser with 100 c.c. of 2.5 per cent. sulphuric acid for three hours; the mixture is made up to 250 c.c. with recently boiled water, allowed to stand for several hours, and filtered; 200 c.c. of the filtrate are distilled under a pressure of 40 to 50 mm. in an oil-bath at 85° C. In the case of hard woods 100 c.c. are taken. The distillation is continued after the liquid has been concentrated to about 20 c.c., by adding water through a dropping funnel drop by drop. The volatile acids (mainly acetic acid) are estimated in the distillate.

RESULTS OF ANALYSIS OF SEVEN SPECIES OF AMERICAN WOODS.

Calculated on Oven-dried Material.

Species.	Ash.	Ether Extract.	Loss of Weight in—			Acetic Acid by Hydrolysis.	Methoxyl Value	Pentosan.	Methyl Pentosan.	Cellulose.	Ash-free Cellulose.	Pentosan in Cellulose.	Methyl Pentosan in Cellulose.	Volatile Oil.
			Cold Water.	Hot Water.	1% NaOH.									
Longleaf pine (<i>Pinus palustris</i>) ..	0.37	6.32	6.20	7.15	22.36	0.76	5.05	7.46	3.60	58.48	58.16	7.71	1.16	1.30
Douglas fir (<i>Pseudotsuga taxifolia</i>) ..	0.38	1.02	3.54	6.50	16.11	1.04	4.95	6.02	4.41	61.47	61.31	5.34	1.20	—
Western larch (<i>Larix occidentalis</i>) ..	0.23	0.81	10.61	12.59	22.14	0.71	5.03	10.80	2.81	57.80	57.51	8.94	1.19	—
White spruce (<i>Picea canadensis</i>) ..	0.31	1.36	1.12	2.14	11.57	1.59	5.30	10.39	3.55	61.85	61.55	9.63	0.72	—
Basswood (<i>Tilia americana</i>)	0.86	1.96	2.12	4.07	23.76	5.79	6.00	19.93	3.73	61.24	61.05	24.28	1.54	—
Yellow birch (<i>Betula lutea</i>)	0.52	0.60	2.67	3.97	19.85	4.30	6.07	24.63	2.69	61.31	61.18	28.30	1.16	—
Sugar maple (<i>Acer saccharum</i>) ..	0.44	0.25	2.65	4.36	17.64	4.46	7.25	21.71	2.39	60.78	60.50	24.48	0.96	—

J. F. B.

Chemistry of Wood. II. Discussion of Methods and Results. A. W. Schorger. (*J. Ind. and Eng. Chem.*, 1917, 9, 561-566.)—The methods adopted in the preceding paper are discussed, and the reasons for their selection are set forth. Drying *in vacuo* over sulphuric acid shows no advantages, and is rather less efficient than drying at 105° to 107° C. in an electric oven. When heated in the oven, the dry weight of the wood remains substantially constant for eighteen hours, and only decreases slowly when heated further over very prolonged periods. The volatile oil cannot be completely expelled by heating in the oven, and distillation with steam is necessary for its accurate estimation. The action of alkali on the wood is not specific, and the substances extracted include representatives of all the principal groups. The same remark applies to the action of boiling water; no final and sharp separation of extractive from non-extractive matters is attained, and it is necessary to limit the time of treatment and specify the conditions of boiling on purely empirical lines. For the estimation of pentosans and methylpentosans, 2 grms. of wood are taken in the case of conifers and 1 grm. in the case of hard woods; the method of extracting the methylfurfural phloroglucide described is more accurate than extraction in a Soxhlet apparatus, but there is reason to suppose that, in any case, the figures for methylfurfural are somewhat too high. It appears to be legitimate to regard wood cellulose as a complex of which pentosans, and possibly methoxy groups (*e.g.*, methylglucosides), form an integral part; thus, "cellulose" is defined by the author as the residue remaining after alternate treatment with chlorine and sodium sulphite up to the point where the colour reaction or the Maûle reaction ceases. The preparation of the sample for chlorination plays an important part in the results; reduction of the wood to powder must be avoided. The skilful use of a cabinet-maker's scraper on wood containing 25 to 30 per cent. of moisture gives, perhaps, the most satisfactory material. An apparatus for chlorinating material in four beakers simultaneously is described; the duration of the successive treatments with the gas is more a matter of judgment than of prescription; one hour for the first chlorination is in many cases too long. Previous extraction of the wood with 1 per cent. sodium hydroxide in the boiling water-bath gives lower results for cellulose than extraction with alcohol-benzene; the author has adopted the latter procedure. Caustic alkali should not be used with the sodium sulphite, as the alkalinity of the latter is sufficient for complete extraction if the material has been thoroughly washed. There is an advantage in using a rapid stream of chlorine in the first treatment, but in subsequent chlorinations the speed should not exceed forty bubbles per minute. Results by different chemists following the same procedure may be considered satisfactory if they agree within 1 per cent. The cellulose content of the various species of wood is remarkably uniform, especially when calculated on the weight of the wood freed from matters soluble in hot water and ether. The method for the estimation of volatile acids formed on hydrolysis has been specially worked out by the author. Though somewhat tedious, it is necessary that distillation be carried out at a low temperature, to avoid the decomposition of the carbohydrates in the solution.

J. F. B.

INORGANIC ANALYSIS.

Analysis of Some Aluminium Alloys. J. H. Stansbie. (*J. Soc. Chem. Ind.*, 1917, 36, 802-803.)—Certain rapid methods suitable for factory use have been selected for the analysis of alloys of aluminium; the metals to be estimated comprise—zinc, copper, manganese, nickel, magnesium, tin, lead, and iron, the last two being present as impurities, as a rule; silicon is always present as an impurity. The sample is most conveniently prepared for analysis in the form of moderately fine sawdust. For the estimation of silicon, copper, and manganese, 1 grm. of the metal is treated with a mixture of 5 c.c. of nitric acid and 20 c.c. of dilute sulphuric acid (1: 4) with heating; when the metal is nearly dissolved, 1 to 2 c.c. of hydrochloric acid is added and evaporated to the fuming-point. The mixture is taken up with water and heated till all soluble matter is dissolved and the solution is filtered into the beaker used for electrolysis. The filter-paper is ignited in a platinum crucible, and the ash is weighed; 1 to 2 c.c. of hydrofluoric acid is added and evaporated to dryness. The residue is moistened with water and sulphuric acid, heated, and ignited; the difference in weight is taken as silica and calculated as silicon. The copper is deposited electrolytically on a platinum cathode after adding 2 c.c. of nitric acid to the solution. The electrolysed solution is treated with 1 to 2 grms. of sodium bismuthate and shaken well for three or four minutes. It is set aside to settle, and filtered through an asbestos filter. The filter is washed with a 2 per cent. solution of nitric acid, a measured volume of standard ferrous ammonium sulphate is added, and the excess titrated back with permanganate; the manganese can then be calculated. Another portion of the alloy, 2 grms. in weight, is digested with 60 c.c. of 10 per cent. sodium hydroxide until the reaction ceases. The solution which contains the aluminium and zinc is filtered, and the zinc is deposited electrolytically on a rotating copper-covered cathode by a current of 0.6 to 0.8 ampère passed for forty-five minutes. The deposit is free from aluminium, and the zinc is weighed after washing and drying. An aliquot portion of the electrolysed solution may be used for estimating the aluminium. The black residue on the filter may contain copper, iron, lead, nickel, manganese, and magnesium; it is treated with nitric acid (5 c.c. of strong acid and 7 c.c. of water) in small quantities at a time, washing with water each time. The solution is collected in an electrolytic beaker, 3 c.c. of sulphuric acid are added, and the copper is deposited on the cathode. If lead is present, it will show on the anode; in that case the peroxide is allowed to dissolve off the anode, and the solution evaporated to the fuming-point; a little water is added, and the lead sulphate allowed to settle. The lead is determined in the usual way. If more than 1 per cent. of iron be present, the pulp filter will remain dark-coloured, and should be treated with hydrochloric acid and washed. A few c.c. of hydrochloric acid are added to the solution, and then ammonia in slight excess, together with 10 c.c. of ammonium acetate if manganese is present. The liquid is boiled for two minutes, and the precipitate of iron oxide and alumina is filtered off. The filtrate may contain manganese and magnesium. The former is removed by hydrogen sulphide and the filtrate boiled to expel the excess of the gas, or the manganese may be separated by bromine and ammonia in the

usual way. The magnesium is precipitated as phosphate. The precipitate containing the iron and aluminium is dissolved in hydrochloric acid, the solution neutralised with sodium carbonate, and one or two drops of acid are added to clear. This solution is heated to boiling, and poured slowly into a boiling solution of 5 grms. of sodium hydroxide. The iron oxide is filtered off, washed, dissolved in hydrochloric acid, and reprecipitated with ammonia. An alloy containing tin can be treated directly with nitric acid, and the tin separated as oxide. A method for the estimation of nickel after the deposition of the copper by electrolysis and addition of tartaric acid to hold up the aluminium is described, by precipitation with dimethylglyoxime.

J. F. B.

Rapid Method for Estimation of Water-Soluble Arsenic in Lead Arsenate.

H. A. Scholz and P. J. Waldstein. (*J. Ind. and Eng. Chem.*, 1917, 9, 682-683.)—0.5 gm. of the dry sample or 1 gm. of paste is weighed into a 250 c.c. measuring flask. Recently boiled distilled water (200 c.c.) is added, and boiled vigorously for about four minutes. After ten minutes' standing, the mixture is cooled, made up to the mark, and filtered. To 200 c.c. of the filtrate, potassium iodide and 7 c.c. sulphuric acid are added, and the mixture is boiled down to 50 c.c. It is then diluted, made alkaline to methyl orange by addition of sodium hydroxide, re-acidified with dilute sulphuric acid, excess of bicarbonate is added, and the arsenious acid titrated with $\frac{N}{10}$ iodine solution. There is no provision for the removal of lead, the authors never having found soluble lead in commercial arsenates, and the attempt to remove lead as sulphate carrying a risk of loss of arsenic by volatilisation. The results obtained either agree with those given by the A.O.A.C. method or are slightly higher. In the latter cases they are believed to be more exact, the A.O.A.C. methods using cold water for extraction, and certain arsenites which may be present being only soluble with difficulty in cold water.

G. C. J.

Fuller's Earth, and its Valuation for the Oil Industry. T. D. Richert.

(*J. Ind. and Eng. Chem.*, 1917, 9, 599-600.)—The bleaching and purifying properties of fuller's earth when brought into contact with refined oils depends on the formation of colloidal aggregates with the traces of soap and colouring matter. The main points to be determined in the valuation of earths are the relative bleaching power and the quantity of oil absorbed by the earth in the process. In testing these earths, a standard refined cottonseed oil is treated with quantities of the earths ranging from 1 to 8 per cent. The "degrees" of yellow and red colour left in the treated oil are determined by the tintometer, and curves are plotted with the "degrees" of red as ordinates and percentages of earth as abscissæ. The curves show the percentage of earth required to produce the maximum bleaching effect in each case, the maximum bleaching effect obtainable with reasonable quantities of the various earths, and the respective quantities of the various earths which will bleach the oil to a standard colour. The loss of oil due to absorption by the earth is determined by shaking 300 grms. of oil with 10 grms. of each of the earths for a given time at a given temperature, filtering on a Buchner funnel with a filter pump, and maintaining the suction for fifteen minutes after the oil is apparently drained out. The residue

of oil and earth is weighed, and from the amount of oil retained a presumptive figure is obtained, based on the known behaviour of a sample of standard earth in the factory bleaching process. The valuation of the oil is then calculated from the quantity of earth required to bleach to a standard colour, the presumptive loss of oil in the process, and the price of the earth and the oil. The formula employed for calculating the bleaching cost per 100 pounds of oil is

$$V = \frac{x(100P + AO)}{100 + \frac{xA}{100}} \text{ cents,}$$

where x is the percentage of earth required, A is the presumptive loss of oil, P and O the price in dollars per 100 pounds of earth and oil respectively. J. F. B.

Absorption of Atmospheric Gases by Water. J. H. Coste. (*J. Soc. Chem. Ind.*, 1917, 36, 846-853.)—The published data with regard to the atmospheric gases, chiefly oxygen and nitrogen, dissolved in water are collated and discussed. Comparisons between distilled water and sea-water show that the effect of saline solutes is to decrease the solubility of the gases. The solubilities of carbon dioxide and argon have also been the subject of investigation. It is probable that the principal cause of discrepancies is the difficulty of obtaining satisfactory equilibrium between the water and the air or other gas above it. If the bubbling method of saturation be adopted, there is the fact that the water is saturated at a pressure rather above atmospheric pressure owing to the column of water through which the bubbles have to pass. Shaking appears to be a good method of saturation, but temperature variations may occur between the time of shaking and determination of gaseous content. The gaseous exchanges, as the point of saturation is reached from either side, are very slow. The conditions of equilibrium between water and the constituents of the air above it are dependent on the actual solubility in water of the gases, the temperature of the water, and the atmospheric pressure. The rate of approach to a state of equilibrium in either direction (*i.e.*, from supersaturation or partial saturation) follows the normal course of a heterogeneous reaction—*i.e.*, it is proportional to the divergence from saturation. Natural water in free contact with air, provided it contains no substances which react chemically with the gases, contains those gases in the same proportions and amounts as pure water, except in so far as the amounts, but not the proportions, are affected by other matters in solution—*e.g.*, salts. The gases dissolved in water can be extracted either (*a*) by boiling the water and evolving the gases with the steam; (*b*) by pumping out and boiling under reduced pressure; or (*c*) by driving out with a current of some other gas. There are also several chemical methods for the estimation of oxygen and carbon dioxide. Winkler's apparatus is perhaps, on the whole, the most practical of any for general use for the determination of oxygen and nitrogen in water. The gases are driven off by means of carbon dioxide generated in the water by the action of hydrochloric acid upon calcite; the gas is collected over a 20 per cent. solution of sodium hydroxide, and subsequently analysed over water. The apparatus can easily be used on board ship for sea-waters. Argon is determined as "nitrogen" unless special means are

adopted for its separate estimation. Of the chemical methods for the estimation of oxygen, Winkler's manganous iodimetric process is the best. Carbon dioxide can be titrated in the water by acidimetric processes or driven out by hydrogen. If a natural water is found to contain very much less oxygen than the saturation value for its temperature, there is a presumption that it contains some substance which is consuming oxygen; but, as a similar result might be caused by a recent fall in temperature or other cause of deficient saturation, the question can in some cases only be definitely settled by a determination of the dissolved "nitrogen." If the volume of nitrogen is approximately double that of the oxygen, the chemical condition of the water is normal.

J. F. B.

Inadequacy of the Basic Ferric Acetate Test for Acetates. L. J. Curtman and B. R. Harris. (*J. Amer. Chem. Soc.*, 1917, **39**, 1315-1317.)—This test is not very sensitive; whilst 20 mgrms. of acetic acid (as sodium acetate) can be detected in 50 c.c. of solution, using 5 mgrms. of iron (as ferric chloride), 15 mgrms. of acetic acid do not give a reaction. The sensitiveness of the test is decreased by dilution and by increasing amounts of iron. Further, the amount of acetate present is not even approximately indicated by the reaction.

W. P. S.

Testing Galvanised Iron. J. C. Witt. (*Philippine J. of Science*, 1916, **11**, 147-165.)—The following method is recommended as the best means of estimating the zinc in galvanised sheet. A 15 cm. square or larger piece is immersed in dilute (1:1) hydrochloric acid, at a temperature of about 45° C., until perceptible effervescence ceases, which should require only two or three minutes. It is then removed and washed, and, if any zinc still remains, it is again placed in the acid, at most for another one or two minutes, until this has dissolved. It is removed, washed in water, then in alcohol, placed in an oven for a few minutes, cooled in a desiccator, and weighed. The strength of the acid should not be less than is recommended or the action is slow; nor much greater, or more iron will be dissolved. Similarly, the temperature must not exceed 60° C., nor should it be much less than 45° C. Under these conditions the amount of iron dissolved from hot galvanised sheets is seldom far from 4 per cent. of the weight of zinc removed, and is almost wholly derived from the layer of iron-zinc alloy.

G. C. J.

Stripping and Analysis of Galvanised Iron. G. W. Heise and A. Clemente. (*Philippine J. of Science*, 1916, **11**, 135-146.)—Various methods of analysing galvanised iron by stripping have been studied and developed. The separation of zinc from iron with phosphoric acid, sodium bisulphate, hydrochloric acid, or sulphuric acid, is sufficiently rapid and complete to serve as a quantitative analytical method. Acetic acid gives accurate results, but the action is slow. The separation can also be effected by metallic salts, notably salts of lead, cadmium, copper, cobalt, chromium, and aluminium. Stripping and analysis may be accomplished with the aid of the electric current, by making galvanised iron the cathode in sulphuric acid, or by making it the anode in concentrated caustic alkali solutions or in oxidising solutions like sodium nitrate. The iron removed from galvanised plate by these methods is

largely that contained in the zinc-iron alloy present in the coating, and is not due to corrosion of the iron base. The amount of iron so dissolved can be kept very small, and when great accuracy is required can be readily estimated and allowed for.

G. C. J.

Estimation of Manganese. Travers. (*Comptes rend.*, 1917, 165, 187-189.)—

The persulphate, silver nitrate method is said to give low results when the liquid is boiled for some minutes, as is advised by many authors who have recommended the Proctor Smith method, with or without minor modifications. The author obtains excellent results as follows:

The steel (0.2 grm.) is dissolved in 20 c.c. of dilute (1:3) nitric acid. Cold water (30 c.c.) is added to reduce the temperature to 40 or 50° C., and then 5 c.c. $\frac{N}{10}$ silver nitrate and 1.5 c.c. of a saturated solution of ammonium persulphate are added. The contents of the flask are shaken, and allowed to stand for three minutes after the appearance of the permanganate colour. They are then poured into 100 c.c. of cold water, and the mixture titrated with arsenious acid containing 0.65 grm. per litre (1 c.c. = 0.2 mgrm. manganese). The method is empirical, the end point not corresponding to reduction to MnO, Mn₂O₃, or MnO₂, but to a mixture of MnO and MnO₂, the latter beginning to appear as a precipitate soon after the titration is finished. Chromium up to 5 per cent. does not interfere, but beyond that percentage the colour of the chromate makes it difficult to determine the end point.

G. C. J.

Recovery of Molybdenum Residues. V. Lenher and M. P. Schultz. (*J. Ind.*

and Eng. Chem., 1917, 9, 684-685.)—The method consists in the precipitation of molybdenum sulphide from a solution slightly acid with nitric acid by means of hydrogen sulphide, or the formation of a sulphomolybdate solution by adding hydrogen sulphide to the alkaline solution of the "yellow precipitate," and subsequently adding hydrochloric acid to precipitate the molybdenum sulphide. This, when ignited, yields molybdic acid, which is in condition to be used again. In applying the method to waste liquors, which are usually strongly acid, the solutions should be largely diluted, or the acid so far neutralised that the acidity corresponds to from 0.1 to 0.4 per cent. acid. The solution is warmed, and hydrogen sulphide passed through it. The precipitate is washed repeatedly with water, which is siphoned off, and is finally filtered off, dried, and roasted. When much iron is present in the liquors, the water used for washing the sulphide should be acidified with hydrochloric acid.

The "yellow precipitate" is dealt with by dissolving in sufficient sodium hydroxide to make a solution just slightly alkaline to phenolphthaleïn. If ferric hydroxide is present, it is filtered off. The filtrate is then diluted and saturated with hydrogen sulphide, after which dilute hydrochloric acid is added and the molybdenum sulphide washed with hot water, acidified with hydrochloric acid, dried, and roasted.

G. C. J.

Nitrate Estimations in the Presence of Chlorides. W. F. Gericke. (*J. Ind.*

and Eng. Chem., 1917, 9, 585-586.)—The presence of chlorides interferes with the

estimation of nitric acid by the phenoldisulphonic acid method through loss of nitric acid at the point where the acid comes in contact with the dry salt containing the nitrates and chlorides. Heat is produced with the liberation of chlorine and nitric acid. This may be avoided by the addition of sulphuric acid before evaporation, and then completing the reaction with phenoldisulphonic acid without carrying the solution to dryness. It is necessary that the quantity of nitrate present be neither too large nor too small for the quantity of acid reagent employed for the test. With nitrates present to the extent of 1 to 5 parts per million, at least 50 c.c. of the sample should be used; 1.5 c.c. of concentrated sulphuric acid is added with constant stirring and then 2 c.c. of the phenoldisulphonic acid reagent. This reagent is prepared with fuming sulphuric acid according to Chamot's method to insure the absence of the monosulphonic acid. The mixture of the sample and acid reagent is evaporated in a capsule at first at the temperature of the boiling water-bath, and later at a temperature not above 70° C. Evaporation should continue until the original solution is concentrated to 6-14 c.c., depending on the amount of nitrate present; a small quantity of nitrate requires a greater concentration of the original solution. The proper point is determined by the colour, which resembles that of phenoldisulphonic acid slightly tinged with yellow; at the same point acid due to the chlorides will be detected in the vapours. This final evaporation must be conducted at a relatively low temperature. The solution should not be materially coloured or turbid. The liquid is diluted with about 50 c.c. of water, and neutralised without being allowed to get too hot; it is then compared with a standard in the colorimeter. In cases where the proportion of nitrate is very low and the chloride high, evaporation is preferably conducted under reduced pressure. In this way the loss of nitrate caused by chlorides in the usual method is completely avoided. J. F. B.

Note on the Phenolsulphonic Acid Method for Nitrates in Waters containing much Magnesium Salts. M. S. Nichols. (*J. Ind. and Eng. Chem.*, 1917, 9, 186-187.)—In the colorimetric estimation of nitrate with phenolsulphonic acid, the presence of magnesium salts may produce a turbidity on neutralisation of the nitrated acid solution. The presence of iron will also change the colour of the liquid. These difficulties may be surmounted by filtering the liquid after neutralising and before estimating the colour, but the filtration and washing occupy time, and there is a danger of leaving a trace of the colour in the filter-paper. In absence of iron the precipitation of magnesium hydroxide may be prevented, and filtration avoided by adding ammonium chloride after the neutralisation and before the final dilution. The quantity required depends on the alkali employed for neutralising. When neutralising with ammonium hydroxide, 5 c.c. of a saturated solution of ammonium chloride will be sufficient to hold up the magnesium present in 20 to 50 c.c. of most waters. In the case of sodium or potassium hydroxide a larger excess of ammonium salt is required, which must be increased in proportion to the amount of alkali added in excess. J. F. B.

Estimation of Ozone (in Air). David. (*Ann. Chim. anal.*, 1917, 22, 145-146.)—The method proposed depends on the oxidation of ammonium ferrous

sulphate by ozone. A measured volume of the air is shaken in a flask with 5 c.c. of the reagent (3.920 grms. of ammonium ferrous sulphate and 20 c.c. of concentrated sulphuric acid per litre), and the excess of the ferrous salt is then titrated with permanganate solution (0.316 grm. of potassium permanganate per litre). The ferrous sulphate and permanganate solutions are equivalent to each other, and 5 c.c. of either corresponds with 0.4 mgrm. of oxygen. The reaction between the ozone and the ammonium ferrous sulphate is immediate, but the latter is not oxidised by air; 20 litres of air free from ozone may be passed slowly bubbled through 5 c.c. of the reagent without any oxidation taking place. W. P. S.

Estimation of Strontium and Lithium in Water. S. D. Averitt. (*J. Ind. and Eng. Chem.*, 1917, 9, 584-585.)—With careful work, indirect methods of analysis are capable of giving satisfactory results while avoiding some extremely tedious and complicated separations; the chief essential is that the precipitates be pure. The following methods have been employed in water analysis for the estimation of strontium in presence of calcium, and lithium and other alkali metals. *Calcium and Strontium*: The weighed oxides (CaO and SrO) are dissolved in hydrochloric acid, and precipitated again as oxalates; these are washed, dissolved in sulphuric acid, and titrated with standard permanganate. The oxalates are washed with 0.5 per cent. solution of ammonium oxalate. Before titration they are washed with 15 c.c. of water delivered from a 5 c.c. pipette fast enough to cover the precipitate, and the filter is drained between each washing. This treatment is sufficient for 0.03 grm. of mixed oxides, which is approximately the amount which should be taken in the portion to be titrated. *Lithium, Sodium, and Potassium*: The mixed chlorides are dissolved in water to a convenient volume. An aliquot portion is taken for the estimation of potassium, from which the equivalent of chlorine is calculated. Another portion is titrated with silver nitrate for total chlorine..

J. F. B.

Detinning and Analysis of Tin Plate. G. W. Heise and A. Clemente. (*Philippine J. of Science*, 1916, 11, 191-199.)—A number of methods of stripping tin plate without attack on the iron base have been studied. Of these, detinning by means of an electric current, making the plate the anode in a bath of sodium nitrate, and stripping by immersion in a solution of a lead salt made alkaline with sodium hydroxide, are recommended for rapid and accurate analytical work. G. C. J.

Volumetric Estimation of Tin and Phosphorus in Phosphor-Tin. R. E. Lee, W. H. Fegely, and F. H. Reichel. (*J. Ind. and Eng. Chem.*, 1917, 9, 663-668.)—Sawings of the alloy are preferred to drillings, any iron derived from the saw being removed by magnets. The sample (0.5 grm.) is introduced into a flask, fitted with a cork carrying a tap-funnel and two bent tubes, one of the latter connecting to a train of three wash-bottles, the other serving for the introduction of carbon dioxide or coal gas. Each wash-bottle is charged with 90 c.c. of approximately $\frac{N}{10}$ permanganate and 10 c.c. of nitric acid. The air in the apparatus is displaced by carbon dioxide or coal gas, and 30 c.c. of hydrochloric acid introduced into the

first flask by means of the tap-funnel. The tap of the funnel is closed, and the acid is heated slowly (five minutes) to boiling, and maintained boiling until the alloy is decomposed. Gas is forced through the system for three minutes to carry any phosphine into the wash-bottles. The flask containing the stannous chloride is disconnected, about 2 grms. ferric chloride added, and the resulting ferrous iron titrated with dichromate.

The contents of the absorption flasks are united, boiled for a minute, excess permanganate being destroyed by addition of nitrite, and the solution boiled again for a minute to expel nitrous acid. The solution is cooled to 50° C. and made slightly alkaline with ammonia, a precipitate of manganese phosphate indicating when this point is reached. This precipitate is just redissolved by means of nitric acid, of which 10 c.c. excess is then added. The solution is heated to 85° C., and 50 c.c. of filtered ammonium molybdate solution added. The contents of the flask are vigorously agitated for five minutes, and the yellow precipitate filtered off and washed with an acid solution of ammonium sulphate until the washings no longer give a brown coloration with ammonium sulphide. Any precipitate adhering to the flask is dissolved in 20 c.c. of dilute (1:2) ammonia, which is then poured on to the precipitate in the funnel. This is then washed with water until about 60 c.c. have been collected, 10 c.c. sulphuric acid are added, and the solution is passed twice through a Jones reductor, which is then washed with 200 c.c. water. The solution is finally titrated with $\frac{N}{10}$ permanganate, 1 c.c. of which equals 0.000883 gm. phosphorus.

G. C. J.

Separation of Zinc from Cadmium, and Estimation of Cadmium Iodometrically. E. J. Ericson. (*J. Ind. and Eng. Chem.*, 1917, 9, 671.)—19.2 grms. of spelter are taken for analysis, and, lead having been removed as sulphate and iron as hydroxide, the filtrate is evaporated until a white precipitate appears. Dilute (1:3) sulphuric acid (50 c.c.) is added, and the solution concentrated to 80-100 c.c. and allowed to stand overnight. By next morning the bulk of the zinc will have crystallised as sulphate. The supernatant liquid is filtered off, and the zinc sulphate washed three times with cold water. The filtrate, diluted to 200 c.c., is saturated with hydrogen sulphide, and the cadmium sulphide filtered off. Cadmium is then estimated in any convenient manner, but the following is recommended for technical purposes:

The precipitate is transferred to a flask with 125 c.c. water, a measured and more than sufficient quantity of $\frac{N}{10}$ iodine solution is added, and then about 40 c.c. of dilute hydrochloric acid. The mixture is shaken, and then titrated with $\frac{N}{10}$ thiosulphate, using starch towards the end as indicator. 1 c.c. $\frac{N}{10}$ iodine = 0.00562 gm. cadmium.

G. C. J.

Electrometric Titration of Zinc. F. R. v. Bichowsky. (*J. Ind. and Eng. Chem.*, 1917, 9, 668-671.)—The ore is dissolved in the least possible quantity of hydrochloric acid, a pinch of chlorate is added, then 100 c.c. of dilute ammonia, the solution made up to 250 c.c., filtered, and 100 c.c. of the filtrate treated as follows: Hydrochloric acid is added until the mixture contains about 10 per cent., in which

zinc ferrocyanide is but slightly soluble, whilst the ferrocyanides of lead, manganese, iron, and copper, are readily soluble, and that of cadmium fairly soluble. The solution is saturated with sulphur dioxide to reduce oxidising agents which act on ferrocyanide, and the excess of sulphur dioxide is expelled by boiling and passing a stream of air through. At this concentration of acid ordinary indicators fail, but the zinc can be easily titrated with ferrocyanide, using an electrometric end-point. All that is required is a galvanometer, a slide wire resistance, a calomel electrode, a piece of platinum wire for the platinum electrode, a couple of dry cells and some copper wire for connections, although a voltmeter is a convenience. The two electrodes are dipped in the solution to be titrated, which should be hot, and the slider adjusted until the pointer of the galvanometer indicates zero. As the titration proceeds, the pointer swings slowly until the end-point is reached, when one drop in excess causes a large deflection in the opposite direction. Or, if a voltmeter is available, the slider can be set so that the voltmeter reads 0.24 volt, and titration continued until the galvanometer reads zero. If large quantities of cadmium are present, this should be separated by some standard method, such as Breyer's (*8th Int. Cong. Appl. Chem.*, 1911, 25, 7), but the electrometric end-point should be used.

G. C. J.

APPARATUS, ETC.

New Alloys to replace Platinum. F. A. Fahrenwald. (*J. Ind. and Eng. Chem.*, 1917, 9, 590-597.)—The principal uses of platinum are for chemical, electrical, and dental work, and for jewellery. In framing a specification for a substitute, the following points must be satisfied as far as possible: It must withstand a temperature of 1,300° to 1,400° C.; it must not be affected by mineral acids or alkalis, either fused or in solution, and must not be oxidised at any temperature up to and including its melting-point. It must be malleable and ductile, and sufficiently strong to withstand stresses tending to change its form while in use. Theoretical considerations as well as practical researches indicate that suitable alloys are to be sought for only among the metals iridium, rhodium, palladium, silver, and gold. The two first are eliminated on account of their rarity, and silver does not possess sufficient resistance for chemical purposes. A series of alloys consisting mainly of gold and palladium, in some cases with a small proportion of rhodium, has been introduced under the name of "rhotanium." It is necessary to observe every precaution in the preparation of these alloys, because of the affinity of palladium for many gases and solids ordinarily encountered in the process of manufacture. The most careful heat treatment is necessary to insure freedom from segregation. The slightest lack of homogeneity is fatal to uniformity of results in practically every application, especially for chemical purposes. As regards chemical resistance, an alloy may be chosen from the "rhotanium" series which will give service equal to, or better than, that of platinum, except when exposed to the action of hot concentrated nitric acid or when used as anodes for electrolytic work. "Rhotanium" is superior to platinum in its resistance to hot concentrated sulphuric acid and fused sodium hydroxide. Losses by volatilisation at temperatures below 1,300° C. are less than for commercial platinum. Its mechanical properties, ductility and welding properties, are highly satis-

factory, and, owing to the higher strength and lower sp. gr., articles of "rhotanium" weigh only half as much as similar articles of platinum. For electrical purposes "rhotanium" is a satisfactory substitute for platinum except in certain cases—*e.g.*, high-duty magneto contacts, where it is necessary to use a high percentage of iridium with the platinum. For dental work satisfactory results have been obtained, and in jewellery work "rhotanium" is superior to platinum. Rhotanium alloys contain 60 to 90 per cent. of gold, and the supply is limited only by the quantity of palladium obtainable. Exhaustive tests have shown that most of the uses for which platinum has hitherto been regarded as indispensable can be filled by one of these alloys, some of which, moreover, are scarcely distinguishable from platinum. J. F. B.

Fallacies in Colorimetry. W. M. Dehn. (*J. Amer. Chem. Soc.*, 1917, **39**, 1392-1398.)—In respect to different optical distributions, the matched solutions may have four geometrical conditions as regards volume, depth, and cross-section, and the greatest error occurs when the volumes, depths, and cross-sections of the two solutions are all unequal. Other errors are introduced by disturbances of equilibria resulting from ionisation and hydrolysis, and by disturbances due to chromoisomerisation. In the Folin method of estimating creatinine, possible errors may be introduced by mechanical and optical faults of the colorimeter, varied light, dilution, temperature, time of standing, concentration of the sodium hydroxide and picric acid, presence of interfering substances, and the action of light (*cf.* Chapman, *ANALYST*, 1909, **33**, 475). W. P. S.

Comparison of the Efficiency of Some Common Desiccants. M. V. Dover and J. W. Marden. (*J. Amer. Chem. Soc.*, 1917, **39**, 1609-1614.)—Baxter and Warren (*J. Amer. Chem. Soc.*, 1911, **33**, 340) and Baxter and Starkweather (*ibid.*, 1916, **38**, 2038) have recorded the comparative efficiencies as desiccants of calcium chloride, sodium hydroxide, and potassium hydroxide; and the methods of these authors are applied in the present work to aluminium oxide, magnesium oxide, and copper sulphate (*cf.* *ANALYST*, 1915, **40**, 298). In view of the fact that aluminium hydroxide does not lose all its water below about 700° C., it was necessary to determine whether the drying efficiency of this material was affected by the temperature to which it had been heated; but it was found that between the temperatures 220° and 500° C. no marked difference was observable. For the sake of comparison the values obtained in the present work are tabulated, together with those found by Baxter (*loc. cit.*), whose values are marked "B."

The figures given represent the weights in mgrms. of residual moisture left in 1 litre of air at 25° C. after it has passed through a U-tube 15 mm. wide and containing 30 cm. of desiccant, the air being driven at a rate of 1 to 3 litres per hour: Copper sulphate, 1.4; zinc bromide, 1.1 (B); zinc chloride, 0.8 (B); calcium chloride, 0.36 (B); sulphuric acid (95.1 per cent.), 0.3; calcium bromide, 0.2 (B); calcium oxide, 0.2; sodium hydroxide, 0.16 (B); magnesium oxide, 0.008; alumina ($\text{Al}_2\text{O}_3 \cdot x\text{H}_2\text{O}$), 0.003; potassium hydroxide, 0.002; phosphorus pentoxide (resublimed), practically none.

Potassium hydroxide has the advantage over alumina in that it is readily

prepared by fusing the commercial material; on the other hand, the alumina is much more easily handled.

H. F. E. H.

Granular Calcium Chloride as a Drying Agent. A. T. McPherson. (*J. Amer. Chem. Soc.*, 1917, **39**, 1317-1319.)—Anhydrous, granular calcium chloride (prepared by heating the material at 260° C. in a current of dry air) rapidly removes every trace of moisture from a comparatively large volume of gas. The granular calcium chloride usually employed contains a small quantity of surface moisture, and its efficiency is less than that of the anhydrous salt, but greater than that of the fused chloride. The surface moisture should be driven off by heat before the granular chloride is used. The desiccating action of the chloride is one of adsorption rather than of hydration.

W. P. S.

Chemical Laboratory Porcelain. H. Watkin. (*J. Soc. Chem. Ind.*, 1917 **36**, 749-753.)—Porcelain for laboratory purposes must be expected to stand changes of temperature rapidly produced over as great a range as possible. The glaze must resist the action of the chemical reagents which may reasonably be employed with it. The colour should be white or cream, but this is not an essential condition. The glaze should resist fusion at temperatures well above that at which the articles are generally used. The weight of the material is not a matter of prime importance, but should preferably be kept as low as is consistent with strength. The composition of the material is of little importance provided the other conditions are satisfied. Translucency is not essential, and is no guarantee of satisfactory quality for the purpose in view. In testing porcelain, the German authorities rightly attach the greatest importance to the quality of resisting sudden and large changes of temperature. Tests of this order comprise heating repeatedly for two days over a Bunsen burner; heating to a high temperature and suddenly placing on a cold metal plate; immersing in water for six days and then heating to redness; heating to redness, turning off gas, and subjecting to a blast of cold air; heating to 950° C. in a muffle furnace for ten minutes, taking out quickly and placing on a cold stone; the same experiment at 1,150° C. Four tests are described for application to English porcelain crucibles (1) The crucible is filled with wax and heated to 130° C., then allowed to cool to 125° C. to insure that the outer layers of the porcelain do not exceed this temperature. (2) The crucible is plunged into cold water, and the same experiment is repeated at 175°, 200°, 225°, and 250° C. (3) The crucible is placed in a muffle heated to about 900° C., removed, and allowed to cool in the air. (4) It is plunged into a furnace at dull red heat, about 550° C., and allowed to cool in the air. The action of water on the porcelain is determined in the manner usually employed for glass ware: carefully prepared distilled water is heated in the vessels at 80° C. for three hours. The action of the water is estimated by iodeosin solution; the volume of iodeosin required to restore the colour measures the amount of acid or alkali dissolved from the porcelain. A second test is made as before, but the action is continued for six hours at 100° C. The action of alkali is tested by heating with $\frac{N}{4}$ sodium hydroxide at 80° C. for three hours, and determining the loss of weight; a similar test is made with potassium hydroxide for five hours at 100° C. The resistance to sulphuric acid is

tested by evaporating 5 c.c. of $\frac{N}{1}$ sulphuric acid in the crucible, and estimating the loss of weight. The action of nitric acid is tested for five hours at 100° C.

J. F. B.

Vacuum-Jacketed Pycnometer for Liquids. F. Hall. (*J. Amer. Chem. Soc.*, 1917, **39**, 1319-1320.)—The pycnometer consists of a cylindrical bulb provided with two capillary tubes at the top; one of these tubes extends down the centre to the bottom of the bulb, and the other is provided with a mark to which the instrument is filled. The cylindrical bulb is surrounded by a vacuum jacket. Before filling the pycnometer, the liquid must be brought to the desired temperature.

W. P. S.

Determination of the Viscosity of Starch for Mill Purposes. G. M. MacNider. (*J. Ind. and Eng. Chem.*, 1917, **9**, 597-599.)—In testing starch for cotton-mill work, it is desirable to determine the viscosity of the solutions after they have been boiled with steam for an hour or more under similar conditions to the boiling of size in the mill. The author describes a small size boiling pan of $1\frac{3}{4}$ gallons capacity, provided with double stirrers of the propeller type and a perforated steam-coil at the bottom of the pan connected with a steam-supply which passes through a draining trap. The quantity of starch taken per gallon of water for a test varies according to the grade of the starch. With thick-boiling starches 0.5 pound is used, the quantity being increased to 1 pound for medium and 2 or 2.5 pounds for thin modified starches. One gallon of water is measured into the pan, the agitators are started, and the weighed quantity of starch is added. After mixing for ten to fifteen minutes to make a uniform cream, steam is turned on, and the size is boiled for one hour after it comes to the boil. When the boiling is finished, a sample of the solution is quickly poured into the cup of a Scott viscosimeter, and the viscosity determined as described in the original method (*ANALYST*, 1912, **37**, 368). The advantage of this method over the laboratory method lies in the fact that the various grades and concentrations of starch may be compared exactly under the same conditions as in practical use and the effect of the addition of chemicals to the mixing investigated. J. F. B.