THE ANALYST.

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

THE following letter was addressed by the Council of the Society to the Food Controller in reference to the recently issued Flour Order:

THE CONTROLLER,
  MINISTRY OF FOOD,   LONDON,
    GROSVENOR HOUSE, W. 1.   *July* 31*st*, 1917.

SIR,

The Council of our Society have instructed us to draw your attention to the fact that Public Analysts are being asked by their Authorities to make analyses of flour and bread under the Regulations recently issued by your Department.

In this connection our Council desire to point out that there is no scientific method in existence whereby it can be ascertained whether any sample of flour or bread is of the composition prescribed by these Regulations.

Under these circumstances, we should be glad to be informed whether your Department proposes to take any action in the matter, such as a communication to this effect to the Local Authorities.

We are, Sir,
Yours faithfully,
P. A. ELLIS RICHARDS,
E. RICHARDS BOLTON,
Hon. Secretaries.

THE CAKE AND PASTRY ORDER, 1917.

The following method has been proposed by the Government Laboratory for the determination of the amount of sugar present in baked articles examined under the Cake and Pastry Order. This process was considered and approved by a conference of members of the Government Laboratory, of the Council of the Society of Public Analysts and other Analytical Chemists, and representatives of biscuit manufacturers.

METHOD FOR THE DETERMINATION OF THE AMOUNT OF SUGAR IN BAKED ARTICLES.

I.—PREPARATION OF SAMPLE.

A. Biscuits and Similar Articles in a Fairly Dry Condition.—The sample, or a thoroughly representative portion of it, is rapidly ground in a mortar or, by passing through a mincing-machine, thoroughly mixed, and used for analysis.

B. Bread and Cakes in a Moist Condition or with an Outside Crust.—A slice, not less than half an inch in thickness, is cut through the loaf, weighed, and dried at a moderately low temperature to a moisture content of about 10 per cent., when it can easily be ground. It is weighed again, ground and mixed, and the powder used for analysis.

C. Buns and Cakes in a Moist Condition.—Several buns or cakes are taken, weighed, broken coarsely, and dried at a low temperature to a moisture content of about 10 per cent. The weight is again taken, and the pieces ground and mixed, and the powder used for analysis.

D. Articles containing Fruit (Raisins, Currants, Dates) in which Sugar naturally occurs.—A considerable portion of the cake or bun is weighed, broken rapidly on a sheet of paper, and the fruit removed and weighed. The drying of the cake from which the fruit has been removed is continued as under B; and the fruit is reserved for the determination of the sugar.

II.—ANALYSIS.

I. Moisture.—A portion, about 5 grms., is weighed out from the prepared sample, and dried at 100° C. until the weight is constant. The loss in weight is corrected for the total loss in those cases in which preliminary drying previous to grinding has been carried out, and in those cases where fruit has been removed to the cake containing fruit.

II. Sugars.—Ten grms. of the prepared sample are ground up with water in a mortar, and transferred to a 250 c.c. flask, using in all about 200 c.c. of *cold* water. The flask is shaken at intervals during thirty minutes. The solution in the case of some articles—for example, biscuits—does not usually need a clearing agent. If it is necessary to use a clearing agent, basic lead acetate followed by sodium sulphate to remove excess of lead, or alumina cream, or copper sulphate solution, may be employed. The liquid in the flask is then made up to 250 c.c., filtered, and the sugars determined in the filtrate.

Fifty c.c. of the filtrate are measured into a 100 c.c. flask and inverted as follows: 5 c.c. of hydrochloric acid of 38·8 per cent. are added, and the flask placed in a water-bath maintained at 70° C. The solution should reach a temperature of 67° to 69° in two and a half to three minutes. It is maintained at 69° C. for seven to seven and a half minutes, the total period of heating being ten minutes. It is then cooled at once, neutralised, bulked to 100 c.c., and filtered.

The reducing sugar in the filtrate is determined either by gravimetric or volumetric process, the total copper-reducing power being calculated as cane sugar.

The quantity of " cane sugar " obtained is to be corrected to the original

moisture of the sample and, if fruit has been removed, to the cake containing fruit. From the total quantity thus found, the sugar derived from added fruit in the case of fruit-cakes is to be deducted.

This is estimated by determining the amount of sugar in the fruit removed from the cake, and the loss in sugar it has sustained, on the basis of the following average amount of sugar in natural dried fruits, calculated on *water-free* sample:

	Total Reducing Sugars as Sucrose.
Raisins	80
Currants	80
Figs	70
Dates (without stones)	70

A deduction of 3 per cent. (3 units) is made from the total amount obtained for sugars naturally present in flour, or derived from flour in course of baking.

To provide for variations in sampling, in methods of analysis, and in the amount of sugar in the different materials employed, an allowance of 2 per cent. (2 units) is made.

GOVERNMENT LABORATORY,
May 23rd, 1917.

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A COMBINED REICHERT-POLENSKE AND MODIFIED SHREWSBURY-KNAPP PROCESS.

By G. D. ELSDON, B.Sc., F.I.C.

(Read at the Meeting, June 6, 1917.)

IN a recent communication the author and another (Elsdon and Bagshawe, ANALYST, 1917, 72) described a modified method of carrying out the Shrewsbury and Knapp process for the estimation of coconut oil in mixtures. In the ensuing discussion certain criticisms were offered by C. Revis, to which it will be convenient to reply here, as they will apply equally to the present paper.

In order to obtain satisfactory results, the main requirement is that the alcohol has a sp. gr. of exactly 0·9200 at 60° F., with an error of not more than one in the fourth decimal place. Using a sp. gr. bottle of 100 c.c. capacity, the adjustment is, of course, quite easy, the difficulty being in the correct adjustment of the temperature. The author uses a thermometer graduated from 55° to 65° F., in tenths of a degree (made by R. Bailey, Bennett's Hill, Birmingham), which has been verified by comparison with a standard National Physical Laboratory thermometer. With this thermometer (which cost about seven shillings and sixpence) it has been found quite easy to adjust the sp. gr. of the alcohol with an error of less than one in the fourth decimal place, in a comparatively short time. Time is saved by preparing a large quantity, about seven litres, at one operation. The complete drying of the acids is another important point.

Where untreated coconut oil is known to occur in a margarine, unmixed with other oils of the same family, everyone will agree that the Polenske process is as

useful as any that is likely to be devised, but in any such mixtures with which the analyst has to deal the composition is usually unknown, and the presence of palm-kernel oil (which is now used in increasingly large quantities in the manufacture of margarine) would render the Polenske figure comparatively useless. It is in such cases that the Shrewsbury-Knapp process is extremely useful, as it will not only give the true total percentage of the various members of the coconut-oil group present, but, as it is hoped to show in another paper, may even give the proportion of coconut oil to palm-kernel oil.

In the communication already referred to (*vide supra*) the authors expressed the hope that it would be possible to combine the Shrewsbury-Knapp process with the Reichert-Polenske-Kirschner process. As a result of further experiment carried out in this connection, it has been found possible to do this.

Although the results obtained are not quite so good as those given in the former paper (*loc. cit.*), yet the method has the great advantage of speed. During the boiling in connection with the Reichert-Polenske process the fatty acids are completely coagulated, so that it is not necessary to use filter-paper for their separation, a fine sieve being all that is required. The drying of the cake therefore takes much less time, and it is possible to do the combined Reichert-Polenske-Shrewsbury-Knapp process in from two hours to two hours and a half. The process is carried out in the following manner:

The flask containing the residual fatty acids after the distillation of 110 c.c. in the Reichert-Polenske process is removed from the condenser, and the contents cooled in water until the acids have become a solid cake. The cake is broken and the liquid strained through a fine wire sieve, the flask and fatty acids being washed with 50 c.c. of cold water. The fatty acids are allowed to drain on the sieve until practically free from water, when they are returned to the flask, the last portions being removed with a thin iron spatula; no difficulty has been experienced in removing the last traces in this way. The flask and contents are then dried in the oven, air being blown through the flask at intervals. One hundred c.c. of alcohol (sp. gr. 0·9200 at 60° F. from industrial methylated spirit) are then added, and the process continued from this point exactly as is given in the previous paper (*loc. cit.*).

Figures have been obtained in this way for the mixtures which were used in the previous paper and with which they may be compared; they are given in the following table:

MIXTURES OF COCONUT OIL WITH BUTTER AND WITH MARGARINE.

(*Sp. gr. of Alcohol 0·9200 at 60° F.*)

Percentage of Coconut Oil	0	10	20	30	40	50	60	70	80	90	100
With Butter..	12·7	14·5	16·5	20·6	24·0	30·4	—	44·4	58·3	76·5	89·8
With Oleo-Margarine	8·9	10·6	13·7	16·9	20·8	25·4	31·8	41·8	53·4	73·2	89·8

These figures are given also in the form of curves, which, it will be seen, are similar in shape to those given in the previous paper, being somewhat steeper, however, for the smaller percentage of coconut oil. From these curves the percentage of coconut oil (or other oil of similar nature) in a given mixture may be read off.
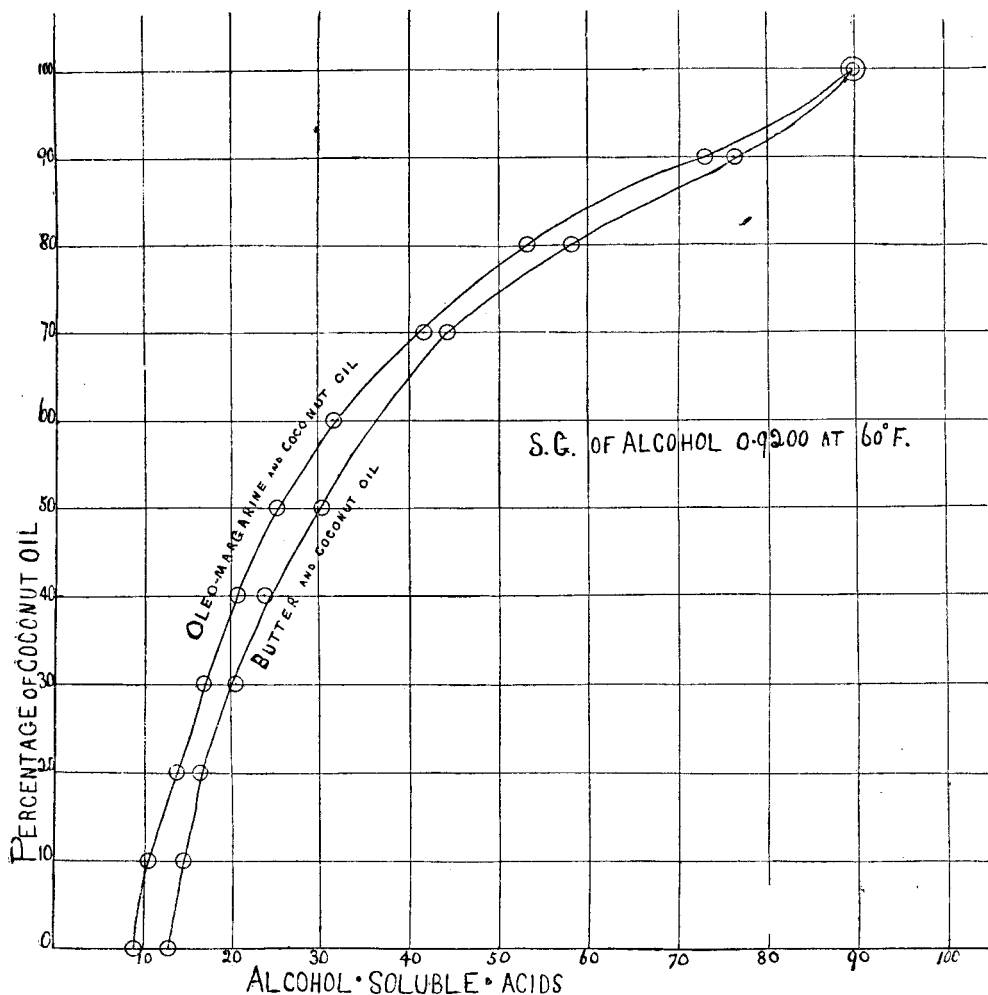
The figures obtained for the alcohol soluble acids by using this combined process on butters have always been within the limits 9·0 to 14·0. The higher figures have so far always been found in conjunction with high Reichert values; thus, with a single exception where the figure was 29·1, all those butters which have given a a higher alcohol soluble acid figure than 12·0 have had a Reichert value of 30·0 or more. This fact may be extremely useful in finding small percentages of coconut oil in some butters. The following example may make this clear: A certain sample of butter gave the following values: Reichert 25·3, Polenske 1·5, alcohol soluble

acid figure 11·5. When mixed with 5 per cent. of coconut oil the figures became 24·6, 2·2, 12·9 respectively. Neither the Reichert nor the Polenske figure would cause suspicion in this case, yet the alcohol soluble acid figure, taken in conjunction with the low Reichert value, would at once indicate the presence of coconut oil. All references to the alcohol soluble acid figure are, of course, to the combined method suggested in this paper.

The average figure obtained on samples of commercial margarines containing no coconut oil has been 6·5, whilst lards have given figures varying from 4·2 to 6·0, with 5·0 as about the average.

I desire to acknowledge the valuable assistance which I have received from my assistant, Mr. Cyril R. Bagshawe, the greater part of the experimental work in the above paper having been carried out by him.

MUNICIPAL LABORATORY,
    SALFORD.

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THE DIFFERENTIATION OF COCONUT OIL AND PALM-KERNEL OIL IN MIXTURES.

BY G. D. ELSDON, B.Sc., F.I.C.

(Read at the Meeting, June 6, 1917.)

IN previous papers the present author has stated that where coconut oil occurs in simple mixtures with either butter or oleo-margarine the combined Reichert-Polenske process affords a rapid and accurate method of analysis. It is obvious, however, that if the oil actually present be palm-kernel oil, which is now largely used in the manufacture of margarine, and if the amount present be worked out, using the usual Polenske figure for coconut oil, the amount of palm-kernel oil will not only be erroneously returned as coconut oil, but will also be seriously underestimated.

In the following table the amount of coconut oil present in various commercial margarines is shown worked out both from the Polenske figure and also from the method recently described (ANALYST, 1917, 72), and it will be seen that the figures (which have been obtained on samples taken in the usual way under the Food and Drugs Acts) often disagree, indicating the presence of more or less palm-kernel oil or similar product.

The amount of coconut oil has been calculated from the Polenske by the methods of Cribb and Richards (ANALYST, 1911, 36, 327) and of Arnaud and Hawley (Ibid., 1912, 37, 122), and from the Shrewsbury-Knapp by the use of the curve given in a previous paper (Ibid., 1917, 76), taking 4·0 as the average figure for the non-coconut basis.

It would thus appear possible to distinguish between coconut and palm-kernel oils in mixtures of various fats such as margarine, by obtaining both the Polenske and Shrewsbury-Knapp values and assuming the presence of palm-kernel oil in those

.cases where the percentage of coconut oil calculated from the Polenske figure was less than that calculated from the Shrewsbury-Knapp figure. Further, knowing the sum of the coconut and palm-kernel oils present, it should be possible to calculate from the Polenske value of the mixture the relative amounts of coconut and palm kernel oils present; the Polenske value of palm-kernel oil being about 11·0. From the table it would appear that samples 1, 2, 4, 6, and 8, contain more or less palm kernel oil, although in each case there is also a proportion of coconut oil. This is, of course, based on the fact that, although the Polenske figures for coconut and palm-kernel oils are very different (about 17·0 and 11·0 respectively), the Shrewsbury-Knapp figures are practically identical.

COCONUT OIL IN COMMERCIAL MARGARINES.

No. of Samples.	Reichert.	Polenske.	Modified Shrewsbury-Knapp.	Coconut Oil per Cent.	
				From P.	From S.K.
1	6·0	8·2	76·0	52	88
2	8·3	6·3	29·0	40	50
3	3·2	2·5	11·5	21	18
4	12·6	9·3	43·9	54	68
5	3·8	2·3	12·5	19	20
6	5·2	5·0	27·9	34	46
7	5·0	4·7	16·9	33	31
8	5·4	6·9	37·5	45	61
9	2·4	1·3	8·2	11	10
10	5·1	4·7	17·3	32	32
11	4·6	3·2	14·1	24	24
12	5·4	6·1	22·7	40	41

It may be argued that in those cases where coconut stearin is used which has a lower Polenske value than coconut oil, the presence of palm-kernel oil would be deduced. This is unfortunately true, but the same objection can be brought against any process that will ever be suggested, for coconut stearin and palm-kernel oil have (within the usual natural limits), practically identical compositions. The main difference between the composition of coconut and palm-kernel oils is the occurrence of a greater proportion of the glycerides of the lower fatty acids in the former: when a portion of these is mechanically removed for the formation of the stearin of coconut oil, it will be seen that the composition of the coconut stearin will become more or less identical with palm-kernel oil. Even in this case, however, the use of the Shrewsbury-Knapp process would reveal the presence of either coconut stearin or palm-kernel oil in their correct proportion, whilst if relying on the Polenske alone the amount of such oil would be underestimated. Figures for coconut stearin are given by Shrewsbury and Knapp (ANALYST, 1912, **37,** 4), and by Knapp (*Ibid.*, 1917, 83).

MUNICIPAL LABORATORY,
    SALFORD.

DISCUSSION.

Mr. REVIS said that he still thought that the author had scarcely succeeded in proving that the Shrewsbury-Knapp process was superior in usefulness to the Polenske process. The Reichert-Meissl figure of 25, upon which Mr. Elsdon had based his calculation, had a corresponding maximum Polenske figure of 2·0, and the figure of 2·2 would therefore afford ground for suspecting the presence of coconut oil. In the case of the alcohol-soluble acids, however, if a figure of 12 usually went with a Reichert-Meissl figure of 30, then a figure of 11·5 would scarcely correspond with a Reichert-Meissl of 25; so that, if a figure, say of 10 were taken, in that case the addition of 1·4 would have produced an alcohol-solubility figure of less than 12 and so be insufficiently high to be regarded as evidence of coconut oil. With regard to the estimation of the percentages of coconut and palm-kernel oils in a mixture, this might be arrived at successfully enough, and in more than one way, when one knew the total percentages of the two oils; but in many cases in practice the total had to be found also, and it was to meet the difficulties of such conditions that, in conjunction with Mr. Burnett, he had suggested a method (ANALYST, 1913, **38,** 255) by which, within certain limits, one could arrive at the respective proportions of the two oils without first knowing the total quantity.

In reply to Mr. Revis's criticism, the author would like to point out that no claim is made for the greater usefulness of the Shrewsbury-Knapp process as compared with the Polenske process. In his opinion the data from the former process are necessary to prevent incorrect conclusions being drawn from the latter. As regards the relationship between the Reichert-Meissl and Shrewsbury-Knapp values, no general connection has yet been noticed beyond that stated above; it is quite possible for a butter to have a Reichert-Meissl figure of 25 and a Shrewsbury-Knapp figure of 11·5, this having been noticed in other cases besides the one mentioned. The difficulties that Mr. Revis finds in the estimation of the total coconut and palm-kernel oils in a mixture would be abolished by the use of the Shrewsbury-Knapp process.

ERRATUM.

Page 76, on the graph, *for* s.g. 0·091 *read* s.g. 0·910.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Oxidisability Values of Medicinal Fats. G. Issoglio. (*Annali Chim. Applic.*, 1917, **7,** 187-199.)—The method of estimating the oxidisability of oils and fats (ANALYST, 1916, **41,** 304) affords a means of judging as to their suitability for medicinal purposes. From the examination of a large number of samples of the oils ordinarily used in medicine, including olive, almond, and cod-liver oils and lard, the conclusion is drawn that their oxidisability value should not exceed 10. In the case of castor oil, however, the oxidisability value does not afford the same information as to the age of the oil, since even when the oil has a high acid value and is old, only relatively small amounts of volatile aldehydic and ketonic compounds may be formed. For example, it was found that castor oils of various origin, free from alcohol, showed acid values ranging from 7·52 to 18·23, whilst the oxidisability values varied from 0·85 to 3·18. The presence of traces of alcohol will affect the results. Under the conditions of the estimation 1 mgrm. of alcohol would reduce 1·738 c.c. of $\frac{N}{100}$ potassium permanganate solution. Samples of fresh olive oil from different parts of Italy showed acid values of 1·06 to 1·85 and oxidisability values of 2·85 to 3·18, whilst fresh Italian almond oils had acid values of 1·35 to 4·12 and oxidisability values of 1·18 to 3·15. Cod-liver oil which is of a reddish or brownish yellow colour should not be used in medicine, since it will almost certainly have undergone some decomposition, and will show a high oxidisability value. For example, the following results were obtained with commercial samples of cod-liver oil:

Origin.	Colour.	Acid Value.	Oxidisability Value.	Iodine Value.
Labrador	Reddish	4·06	28·06	140·8
Newfoundland	Brown-red	20·42	43·96	157·15
Unknown (old)	Reddish	37·42	69·72	138·84
,,	Brown-red	28·47	51·22	132·3
Hamburg	Light red	38·42	67·12	135·4
Japan	Nearly colourless	2·35	6·41	145·8
Norway	Pale yellow	1·84	6·30	157·4
Hamburg	,,	8·07	12·3	153·8

If mercurial ointment contains fat with a high oxidisability value (*e.g.*, 18 to 19) the use of rancid fat is indicated, whilst an abnormally high value (*e.g.*, 74 to 100) points to the presence of oil of turpentine. C. A. M.

Correction Required in applying the Babcock Formula to the Estimation of Total Solids in Evaporated Milk. O. L. Evenson. (*J. Ind. and Eng. Chem.*, 1917, **9**, 499-501.)—That the sp. gr. of freshly drawn milk, when measured at a definite temperature, say 15°/15° C., gradually increases on standing was observed many years ago by Quevenne, and was shown by Fleischmann to be due to solidification or change in the physical state of the fat. When milk is cooled below the solidification point of the fat, the latter begins to solidify and increases the sp. gr. Conversely, when milk which has been cooled to its maximum sp. gr. is heated, its sp. gr. decreases until the fat is again melted. The change in sp. gr. increases with the percentage of fat, and is consequently marked in the case of evaporated milk. The purpose of this investigation was to determine the error of the Babcock short formula when applied to evaporated milk, so that an average correction might be made and more accurate results obtained when the formula is applied to estimate total solids in milk at temperature of 5° and 37° to 40° C. It has been found that results, comparable with those obtained with the Babcock formula on whole milk, may be obtained if the formula $T = \dfrac{G - 1\cdot8}{4} + 1\cdot2\ F.$ is applied to evaporated milk cooled to its maximum gravity, or if the formula $T = \dfrac{G - 0\cdot5}{4} + 1\cdot2\ F.$ is applied to milk kept at 37° to 40° C. for one and a half hours or until no further change in sp. gr. occurs. G. C. J.

Detection of Added Water in Milk. H. Durand. (*J. Ind. and Eng. Chem.*, 1917, **9**, 44-45.)—Methods depending on determination of the refractive index of the serum are found to be untrustworthy for the detection of less than 10 per cent. of added water. The cryoscopic method, with slight modifications of Henderson and Merton's procedure (*Chem. News*, 1914, **112**, 259), devised by the author, gave excellent results in his hands, but is slow. A method devised by the author depends on the addition of 2 grms. of finely divided silver nitrate to 100 c.c. of milk, and the estimation of the silver in an aliquot part of the serum by titration with $\frac{N}{10}$ thiocyanate, using ferric alum as indicator. This method has proved rapid, but a sufficient number of estimations have not yet been made to establish its applicability. G. C. J.

Report on Preservatives: Formic Acid. A. F. Seeker. (*J. Assoc. Off. Agric. Chemists*, 1917, **3**, 33.)—The directions given by Röhrig (*Zeitsch. Nahr. Genussm.*, 1909, **19**, 4) for Wegner's method were employed, in which the material under examination previously acidified with tartaric acid is distilled in a current of steam. The distillate, after being rendered alkaline, is evaporated to dryness, and any sodium formate present decomposed at a temperature of 170° C. in a current of carbon dioxide with sulphuric acid, the carbon monoxide generated being collected over caustic soda solution in a eudiometer tube. Then if X = weight of formic acid, v = observed volume of carbon monoxide, p = barometric pressure in millimetres corrected for vapour tension of water at t° temperature, and t = absolute temperature (273 plus

observed temperature in degrees Centigrade), then $X = \frac{vp}{t} \times \cdot 0007359$. Samples of jam and fruit pulp to which known amounts of formic acid had been added were examined by eight observers by this method, and it was generally agreed that the Fincke method (ANALYST, 1911, **36**, 103, 496) was superior in every way, far more time and attention being required for the Wegner method. It was recommended that the Fincke method be made official. H. F. E. H.

Detection and Estimation of Saccharin in Alimentary Substances.
A. Bonis. (*Ann. Falsif.*, 1917, **10**, 210-218.)—The saccharin is best separated by extracting the acidified substance with ether; in the case of alcoholic liquids the alcohol must be removed previous to the extraction, and in many instances a preliminary treatment with lead acetate and subsequent removal of the excess of lead will be found advantageous in eliminating substances which might contaminate the extracted saccharin. To separate saccharin from fatty substances (saccharin is soluble in fats) the sample is shaken with 5 per cent. sodium carbonate solution, the aqueous portion is filtered, acidified, again filtered, and the filtrate extracted with ether. The residue obtained on evaporating the ether will consist of saccharin still contaminated with traces of tannin, colouring matters, salicylic acid, etc.; it is purified by dissolving in a few c.c. of dilute sulphuric acid, heating the solution on a water-bath and adding concentrated permanganate solution, drop by drop, until a slight excess is present. The mixture is then cooled, extracted with petroleum spirit, and the extract evaporated. The saccharin thus separated may be identified as such by fusing a portion of it with sodium hydroxide for one minute at 270° C., dissolving the mass in water, acidifying the solut'on with sulphuric acid, and extracting it with petroleum spirit. The latter extracts the salicylic acid formed from the saccharin, and the salicylic acid may be identified by the ferric chloride test. Several confirmatory tests may be applied. Saccharin yields a purple coloration when heated with phenol and concentrated sulphuric acid and the mixture then rendered alkaline with sodium hydroxide. A blue coloration is obtained when saccharin is heated with sulphuric acid and the solution then treated with sodium phenolate solution and sodium hypochlorite solution.

The weight of the purified saccharin residue gives approximately the quantity of the substance present. If desired, the amount of saccharin may be estimated in this residue by fusing it with sodium hydroxide, extracting the salicylic acid formed, and estimating this colorimetrically with ferric chloride. A control test must, however, be carried out at the same time and under exactly similar conditions, using a known quantity of pure saccharin. Other methods for the estimation of saccharin consist in hydrolysing it by boiling with hydrochloric acid and estimating the ammonia formed; converting the saccharin into its ammonium salt and estimating the nitrogen liberated when this salt is treated with hypobromite; converting the saccharin into its silver compound and weighing this, etc. As, however, these latter methods require a fairly large amount of saccharin on which to operate, whilst the quantity of the substance separated from foods is usually very small, the colorimetric method is to be preferred. W. P. S.

Estimation of Saccharin in Foods. C. B. Gnadinger. (*J. Assoc. Off. Agric. Chemists*, 1917, **5**, 25-32.)—The solubility of saccharin in different solvents was determined with the following results, the figure being the weight in grms. of saccharin dissolved by 100 grms. of solvent at 25° C.: Acetone, 14·37; methyl acetate, 8·09; ethyl acetate, 4·51; ethyl alcohol (99·5 per cent.), 3·84; amyl acetate, 1·69; ethyl ether (" over sodium "), 1·56; amyl alcohol, 1·22; distilled water, 0·4; chloroform, 0·259; benzol, toluol, xylol, carbon tetrachloride, and carbon disulphide, under 0·13; and petroleum ether (B.P. 30 to 65° C.), 0·0009. Of the foregoing it was found that ether and ethyl acetate are suitable solvents for saccharin extraction; chloroform should not be used. Saccharin is more readily extracted from hydrochloric acid solution than from dilute acetic acid solution. Continual washing of the ether extract is undesirable and unnecessary. The slight solubility of saccharin in petroleum ether offers a means of separation from fat, benzoic acid, and salicylic acid. The estimation of saccharin in the presence of mustard by the usual methods is worthless, since the sulphur in the mustard oils is included with that from the saccharin. When mustard is absent, preliminary clarification with lead acetate is recommended as preventing emulsification; while if mustard is present, the solvent used must be evaporated and the residue treated with petroleum ether, which removes most of the pungent oily sulphur-bearing substance. By further treatment of the residue with bromine, practically all the interfering substances are removed. The fusion method for determining the sulphur in saccharin, and thus indirectly the saccharin itself, was found satisfactory, but care is needed to prevent loss of sulphur by burning it off before oxidation can take place. An electrolytic method for determining sulphur in saccharin was worked out, and is described in detail. It is accurate and easier to operate than the fusion method. In the case of alcoholic liquids, the alcohol should be distilled off after rendering the solution alkaline with caustic soda; they may then be acidified, clarified with lead acetate, and extracted as described.

H. F. E. H.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Use of Potassium Persulphate in the Estimation of Total Nitrogen in Urine. L. C. Scott and R. G. Myers. (*J. Amer. Chem. Soc.*, 1917, **39**, 1044-1051.)—By the use of potassium persulphate, which must be obtained free from nitrogen, the time of digestion, as compared with the ordinary Kjeldahl method, is considerably shortened and losses of nitrogen thereby decreased. Oxidation is rapidly completed at a lower temperature, and the accuracy of the results is not inferior to those obtained by the Arnold-Gunning method. The procedure is as follows: 5 c.c. of urine are measured into a 500 c.c. Kjeldahl flask, 10 c.c. of sulphuric acid and 10 c.c. of a 2·5 per cent. solution of copper sulphate are added, and the mixture is boiled until the mass is light brown or yellow. The flask is allowed to cool until it is possible to touch the glass without inconvenience, and 1·5 to 2·0 grms. of potassium persulphate is added, depending on whether the colour is yellow or brown. The contents of the flask are heated until the reaction begins, the flask is removed from the flame and the contents are rotated until the solution is colourless.

The mixture is again heated until the evolution of sulphur trioxide fumes has practically ceased. If necessary, the flask may be again cooled and more persulphate added. When permanently colourless, the solution is boiled vigorously until all the trioxide is expelled and the acid begins to condense on the sides of the flask. When cold, 150 c.c. of water, 40 to 45 c.c. of 30 per cent. sodium hydroxide, a few pieces of granulated zinc, and a little paraffin wax are added, and the ammonia is distilled off in the ordinary way. The time necessary for the digestion is about fifteen minutes, as compared with two and a half hours by the ordinary Kjeldahl method. The completeness of the oxidation with small quantities of sulphuric acid and salts, when using the persulphate method, makes it eminently suitable for the microchemical estimation of nitrogen in urine by direct Nesslerisation of the diluted reaction product. Five c.c. of the urine are diluted to 50 c.c. and 1 or 2 c.c. of the diluted urine is taken, according to whether the original sp. gr. is above or below 1·018. The dilute urine is introduced into tubes of Jena glass, 200 mm. long and 25 mm. wide, on which lips have been moulded to facilitate pouring. Then 0·3 c.c. of sulphuric acid is added and a glass bead to prevent bumping. The tubes are clamped in a slanting position and heated with a small burner. When the water has evaporated and the contents have become black the acid begins to condense on the sides of the tube. The tube is then allowed to cool thoroughly, and 0·3 to 0·4 grm. of potassium persulphate is added. The tube is held over the flame until the reaction commences, then removed, and the oxidation allowed to proceed until the liquid becomes colourless. Finally, the liquid is heated until the fumes of sulphur trioxide are expelled and then allowed to cool; when cold the liquid should not solidify. The liquid is diluted in the tube and washed into a 250 c.c. flask. The volume is made up to about 200 c.c., then 18 to 19 c.c. of $\frac{N}{1}$ potassium hydroxide are added, the liquid is rapidly rotated, and, while in motion, diluted Nessler reagent (5 c.c. to 25 c.c. of water) is quickly run in. It is important that no trace of calcium or magnesium be present in the water, otherwise the test will be cloudy. The test solution is compared with a solution made by diluting 5 c.c. of standard ammonium sulphate (5 c.c. = 1 mgrm. of nitrogen) to 250 c.c.; the maximum colour develops in about thirty minutes. The results by the microchemical method agree well with those obtained by the usual method.   J. F. B.

ORGANIC ANALYSIS.

Examination of Acetic Anhydride. **L. G. Radcliffe and S. Medofski.** (*J. Soc. Chem. Ind.*, 1917, **36,** 628-630.)—Commercial acetic anhydride usually contains from 70 to 90 per cent. of actual anhydride. The yellow coloration of many commercial samples may be removed by distillation with a little phosphorus pentoxide, which yields a colourless distillate containing less anhydride than the original product. For example, in the case of one sample, the amount of acetic anhydride was reduced from 85 to 77 per cent. A comparative study of the published methods of estimating the amount of anhydride in commercial products was made. The methods included the following: (A) Direct titration after hydrolysis with

water.   (B) Treadwell's method, in which a known quantity of the sample is heated with an excess of barium hydroxide beneath a reflux condenser until the anhydride is completely hydrolysed, and the resulting acetic acid is then titrated with standard alkali.   (C) The method of Menschutkin and Vasilieff, in which the anhydride is mixed with aniline, with which it forms acetanilide and acetic acid, and titration of the latter.   (D) The International Standard Method of the Committee on Glycerol Estimation (ANALYST, 1911, **36**, 318).   (E) A modification of (C) in which 20 c.c. of aniline are used instead of 2 or 3 c.c., with the object of absorbing the heat liberated in the reaction and thus preventing the secondary reactions.   The following results were thus obtained in the analysis of a commercial and a "pure" sample of acetic anhydride:

	A. Per Cent.	B. Per Cent.	C. Per Cent.	D. Per Cent.	E. Per Cent.
Commercial sample, yellow..	84·92	85·61	98·42	85·30	84·80
Ditto, after distillation over P_2O_5	84·90	—	—	85·20	85·40
"Pure" acetic anhydride, colourless	92·48	94·35	109·23	92·72	93·17

The general conclusion drawn from the results is that while the method of direct titration is the most rapid, that of the Glycerol Committee (D) is the most trust-worthy.   C. A. M.

Identification of Alcohols.   E. E. Reid.   (*J. Amer. Chem. Soc.*, 1917, **39**, 1249-1245.)—Alcohols, when heated with phthalic anhydride, yield acid phthalic esters, and the sodium salts of these heated with p-nitrobenzyl bromide give mixed phthalic esters having definite melting-points.   These reactions may be used for the identification of many different alcohols.   A moderate excess of the alcohol is heated for one hour with 1 grm. of phthalic anhydride; for a primary alcohol the temperature used is 100° C., for a secondary 140° C.   In the case of the lower alcohols, the mixture is sealed up in a tube and heated in a boiling water-bath, whilst with the less volatile alcohols, the mixture may be heated in an open tube.   The product is then mixed with 10 c.c. of water and shaken with 15 c.c. of ether and 5 c.c. of $\frac{N}{10}$ sodium hydroxide solution; the aqueous layer is drawn off, extracted with ether, and then evaporated to dryness.   The residue thus obtained is heated for one hour under a reflux condenser with 5 c.c. of water, 10 c.c. of 95 per cent. alcohol, and 1 grm. of p-nitrobenzyl bromide, and the product is recrystallised from 63 per cent. alcohol. The following are the melting-points of some mixed phthalic esters which have been prepared: Methyl p-nitrobenzyl phthalate, 105·7° C.; ethyl p-nitrobenzyl phtha-late, 80° C.; propyl p-nitrobenzyl phthalate, 53° C.; isopropyl p-nitrobenzyl phtha-late, 74° C.; allyl p-nitrobenzyl phthalate, 61·5° C.; n-butyl p-nitrobenzyl phtha-late, 62° C.; n-octyl p-nitrobenzyl phthalate, 41° C.; benzyl p-nitrobenzyl phthalate, 83° C.; phenylethyl p-nitrobenzyl phthalate, 84·3° C.; bornyl p-nitrobenzyl phtha-late, 100° C.; isobornyl p-nitrobenzyl phthalate, 87° C.   W. P. S.

New Method for the Estimation of Aldose Sugars. J. Bougault. (*Compt. rend.*, 1917, **164,** 1008-1011.)—The aldose sugars are oxidised quantitatively to the corresponding monobasic acids by a solution of iodine in sodium carbonate (hypo-iodite). It is best to use about three times the theoretical quantity of the oxidising agent; the excess is then titrated back with thiosulphate after acidification. The oxidation of the aldose group is accompanied by a secondary reaction, small but not negligible, on the alcoholic groups of the sugar, and it is necessary to apply a correction for this reaction based on the increment of the reduction of iodine with increase of time. For instance, 25 c.c. of a solution containing 0·25 grm. of dextrose were mixed with 50 c.c. of iodine solution (1 c.c. =0·020 grm. of iodine) and 50 c.c. of a 15 per cent. solution of crystallised sodium carbonate. The mixture was divided into portions of 25 c.c. each, in closed flasks, and titrations were made at intervals of thirty minutes after acidification with hydrochloric acid. The quantity of iodine absorbed after thirty minutes was 71·00 mgrms. (theory 70·56). The oxidation of dextrose was therefore complete after thirty minutes under the conditions adopted, but the consumption of iodine increased slightly with the time. This increase amounted to 0·40 mgrm. in the second half-hour, but since the increments were found to become smaller for each succeeding period, it was considered legitimate to estimate the excessive consumption of iodine in the first thirty minutes at 0·48 mgrm., which being deducted from the total value found gave the quantity of iodine consumed by the aldose group itself as 70·52 mgrms., equivalent to 0·2498 grm. of dextrose in the original solution. Under the conditions cited, all the aldose sugars—pentose, hexoses, and bioses—were completely oxidised in half an hour, with the exception of mannose, which required ninety minutes before the results of successive titrations showed constant differences. Ketose sugars and non-reducing disaccharides are not oxidised by hypoiodite, but they show the small secondary reaction due to the slow oxidation of their alcoholic groups. For this reason the estimation of aldose sugars in mixtures containing non-reducing sugars is not very accurate, but the method may be usefully applied by determining the approximate correction required or by comparison with solutions of pure sugars. The method is also restricted by the fact that many organic substances other than sugars possess the property of reducing hypoiodite. (*Cf.* Romiju *Zeitsch. Anal. Chem.*, 1897, **36**, 349.) J. F. B.

Optical Activity of Essential Oils from Various Species of Pine. D. E. Tsakalotos. (*Gazz. Chim. Ital.*, 1917, **47**, 285-287.)—Oil of turpentine obtained from the Aleppo pine (*Pinus halepensis*) is composed in the main of dextrorotory pinene, whilst the oil derived from the maritime pine (*Pinus maritima*) contains lævorotatory pinene as its main constituent. According to Belloni, however (*Sull' essenza di gemme di P. maritima*, 1905), the oil derived from the buds of the Aleppo pine is lævorotatory $[a_D] = -26·5°$. To settle this point the author has examined samples of oil distilled from the buds, and has found it to have a specific rotation of $[a_D] = +39·4°$. He finds that this pine produces oil which contains principally dextrorotory pinene from whatever part of the plant the oil is derived, whatever the locality (Greece, France, Algiers), or the season when the oil is distilled. C. A. M.

Mechanism of the Ninhydrin Reaction. J. M. Retinger. (*J. Amer. Chem. Soc.*, 1917, **39,** 1059-1066.)—Ninhydrin is the name applied to triketohydrindene hydrate, which gives a characteristic colour reaction with amino-acids and amines. The triketohydrindene hydrate hydrolyses during boiling, giving *o*-carboxylglyoxal, which reduces part of the triketohydrindene to dioxindone, which in turn combines with another molecule of triketohydrindene to give hydrindantine. The amino-acid or amine derived from enzyme action gives first, as shown in the alloxantine series on alkaline salts, a monobasic salt which is colourless; further boiling produces a dibasic neutralisation and the molecule splits into equal parts with trivalent carbon—a free valency—as cause of the absorption in the visible spectrum. Exposure to air in aqueous solution decomposes the split molecules further, giving *o*-carboxylmandelic acid, ammonia, carbon dioxide, water, and an aldehyde which in turn is partly used up for reducing the other part of the molecule to dioxindone.

J. F. B.

Reducing Matter Extractable from Filter-Paper. R. S. McBride and J. A. Scherrer. (*J. Amer. Chem. Soc.*, 1917, **39,** 928-933.)—The filter-papers in general use contain reducing substances which are extracted by the solutions passed through the paper and which cause the filtrate to decolorise permanganate more readily than the unfiltered liquid. When the titration is made in the cold with $\frac{N}{10}$ permanganate the effect on the result in the ordinary way is practically negligible, but when using $\frac{N}{50}$ or $\frac{N}{100}$ permanganate, and especially when the titration is carried out hot, a relatively considerable error may be introduced by the reducing matter extracted from the filter-paper. The magnitude of the error has been investigated with various makes of standard commercial filter-papers by passing 250 c.c. each of simple reagents—*e.g.*, water, dilute sulphuric acid, dilute sodium hydroxide and carbonate—through the filters and adding sufficient $\frac{N}{10}$ permanganate to the acidified filtrates to produce a tint equivalent to that of 1 drop (0·03 c.c.) of the same permanganate in an equal volume of pure water. From the quantity of permanganate required and from the rate at which the pink colour faded away, as compared with blank tests of the same reagents unfiltered, a measure was obtained of the amount of reducing matter extracted from the filter-paper. The maceration of filter-paper pulp with the reagent gave results very similar to those obtained when the reagent was passed through the paper in the ordinary way. The temperature at which the reagent was filtered had very little effect on the amount of reducing matter extracted, but the temperature at which the permanganate was subsequently added influenced the results considerably, a larger and more rapid consumption of permanganate being recorded with hot titrations at 70° to 80° C. than in the cold. The experiments indicated that the main bulk of the reducing matter is extracted by the first portion of the reagent passed through, and that if the filter be washed with 25 c.c. of the reagent before use, filtration and titration may be carried out, even hot, without sensible error. Comparatively long exposure of the paper or titration in presence of the paper has no abnormal influence.

J. F. B.

Varnish Analysis and Varnish Control. II. Viscosity of Varnishes. M. N. Seaton, E. J. Probeck, and G. B. Sawyer. (*J. Ind. and Eng. Chem.*, 1917,

9, 35-40.)—From the manufacturers' standpoint it is important to be able to determine viscosity before the varnish has been filtered. Generally at this stage it will contain a certain amount of more or less finely divided solids in suspension. In a viscometer which depends on measurement of the rate of flow through a small orifice, "flock" of this kind is almost certain to make the results useless. Instruments which depend on observation of the rotation of a disc, sphere, or cylinder, however, serve much better. Of two instruments of this type, the Stormer has not been found to give results as accurate as those desired in work with varnishes, and, moreover, it is slow in use. The Doolittle viscometer, on the other hand, gives excellent results, and by a proper arrangement of heating baths is capable of rapid manipulation, it being possible to examine a dozen samples within the hour. The authors work at a temperature of 40° C., as this is well above any normal room temperature, and it is easier to run heating baths than cooling baths. Examples are given of the use of such determinations to the manufacturer in controlling his processes, varying his products to meet special requirements, and classifying an unknown varnish put up to him to match. G. C. J.

INORGANIC ANALYSIS.

Analysis of Antimonial Lead. C. R. McCabe. (*J. Ind. and Eng. Chem.*, 1917, **9,** 42-44.)—Demorest's method (*J. Ind. and Eng. Chem.*, 1913, **5,** 842) serves well, with one important modification. This consists in dissolving the alloy completely in sulphuric acid, mere decomposition leaving nearly 10 per cent. of the antimony occluded by the lead sulphate. The alloy (1 grm.) is dissolved in 50 c.c. of sulphuric acid, the solution diluted to 100 c.c. and boiled, diluted to 150 c.c., boiled and decanted through a Gooch crucible, and the flask, precipitate and filter washed with two portions of 25 c.c. of water. The lead sulphate in the flask is dissolved in 50 c.c. of 20 per cent. ammonium acetate, reprecipitated with 25 c.c. of sulphuric acid and filtered. washed by filling the crucible once, dried, ignited, and weighed. This second filtrate, which contains a little antimony but no tin, is titrated with $\frac{N}{10}$ permanganate by adding an excess and titrating back with $\frac{N}{10}$ ferrous solution. The antimony in the first and principal filtrate is estimated in a similar manner. The permanganate is standardised on pure antimony dissolved in sulphuric acid and diluted. After the permanganate titration, the tin in the first filtrate is reduced by boiling with iron nails after addition of 25 c.c. sulphuric acid, in which, if necessary, enough antimony has been dissolved to bring the total amount present up to 0·1 grm. The stannous solution is then diluted, filtered through cotton to eliminate particles of iron, and titrated against iodine solution which has been standardised by means of a solution of tin in sulphuric acid. G. C. J.

Application of the Gutzeit Test to Works' Estimations of Arsenic. C. Hollins. (*J. Soc. Chem. Ind.*, 1917, **36,** 576-577.)—A modification of an apparatus used by Bird (*Chem. and Drugg.,* 1901, 600) has been found very useful

for the estimation of arsenic in de-arsenicated vitriols, phosphates, phosphoric acid, and other substances containing only small quantities of arsenic. A wide-mouthed bottle is fitted with a two-holed rubber stopper. Through one hole is fitted a glass tube bent at a right angle and closed by a glass-rod stopper; this tube is used for clearing out the gas after an unfinished experiment. A set of three washing bulbs are blown on a piece of glass tubing, 4 to 5 mm. bore; the diameter of each bulb is about 2 cm. The two lower bulbs contain a little lead acetate solution (10 to 20 per cent.); the upper bulb is tightly packed with glass-wool and the tube is constricted below this bulb to facilitate packing. Above the third bulb the tube is also constricted and then terminates in a straight portion, 10 cm. long, at an angle of 45°, in which a strip of mercuric chloride paper is placed and shielded from strong daylight. The test paper is made by steeping thin drawing paper in 1 per cent. mercuric chloride solution; it is stable in the dark for 2 to 3 months. A standard arsenic solution containing 0·001 mgrm. of As_2O_3 per c.c. is made from $\frac{N}{10}$ sodium arsenite solution. Standard stains are made by placing in the bottle x c.c. of arsenic solution, 40-x c.c. of water, and 2 c.c. of sulphuric acid free from arsenic, and, when the mercuric chloride paper is in position, introducing 3 to 4 grms. of arsenic-free granulated zinc, so that the stream of gas is maintained at 2 to 4 bubbles per second. After half an hour the stain is complete and the paper is sealed up in a bulb-tube in presence of phosphorus pentoxide. The standard stains should be kept in the dark and will be reliable for about three months. For testing de-arsenicated vitriol, the mixture is made up with 40 c.c. of water, a measured volume (0·5 to 2·0 c.c.) of the vitriol, and sufficient pure sulphuric acid to make up the total acid to 2 c.c. of 1·84 sp. gr. Three drops of stannous chloride solution are added to reduce arsenic compounds. The best stains are produced when the gas is evolved at the rate of 2 bubbles per second. The most accurate results are obtained with stains corresponding to 7 to 20 micromgrms. of As_2O_3, and the accuracy is within 5 per cent. A stain which is otherwise invisible may be developed by touching the paper with a drop of concentrated hydrochloric acid; a stain which is just visible on development represents about 0·5 micromgrm. of As_2O_3; on 1 c.c. of concentrated vitriol the normal range of the test is from 0·3 to 27 parts per million.

J. F. B.

Dissolved Oxygen in Rain-Water. **E. H. Richards.** (*J. Agric. Sci.*, 1917, **8**, 331-337.)—Oxygen dissolved in the rain-water carried into the soil is an important factor in the root aëration of growing plants and in the regulation of the bacterial activity of soils. The results recorded by the author refer to the year 1915, the values found being expressed as a percentage of saturation when compared with the figures found by Dittmar ("*Challenger* Reports: Physics and Chemistry," vol. i., p. 160) for distilled water at similar temperatures. The method employed was that of Winkler (*Berichte*, 1888, **21**, 2,843), and no determination was made on rainfalls under 0·3 inch. The saturation percentage was found to vary between 78 per cent. and complete saturation, the average value for the year being 91 per cent. It was observed that summer rain, unlike that which falls during the rest of the year, is not fully saturated with oxygen—a fact for which it is difficult to find a reason, as

the exact opposite of this would be the more probable. Rain-water is very nearly saturated with oxygen when its temperature as collected is below 15° C., as is the case for about nine months in the year in this climate. When the temperature of the rain is above 15° C. the dissolved oxygen is always below saturation, occasionally as much as 25 per cent. H. F. E. H.

Microchemical Reactions of the Perchloric Ion. G. Denigès. (*Ann. Chim. anal. appl.*, 1917, 22, 127-128.)

—Many alkaloids, and notably strychnine, brucine and morphine, form insoluble perchlorates which may be recognised under the microscope by their crystalline form. Conversely, these alkaloids may be used for the detection of traces of the perchloric ion. A drop of the liquid under examination is placed on a glass slip and stirred with a drawn-out glass rod which has previously been dipped into an aqueous solution of strychnine sulphate (1 : 100) or a 2 per cent. solution of brucine in 1 per cent. acetic acid, or a 2 per cent. aqueous solution of morphine hydrochloride. Should a turbidity appear, the drop is examined under the microscope. Strychnine sulphate will detect 1 grm. of perchloric ion per litre, whilst brucine acetate will detect 2 to 3 grms., and morphine hydrochloride 5 grms. per litre. C. A. M.

Solubility of Calcium Phosphates in Citric Acid. A. A. Ramsay. (*J. Agric. Sci.*, 1917, 8, 277-298.)

—It is customarily held that, excluding basic slag, calcium and phosphoric acid exist in three states of combination—namely, mono-, di- and tricalcic phosphate, and that the former is soluble in water, the second is insoluble in water but soluble in ammonium citrate (or citric acid), while the third form is insoluble both in water and in ammonium citrate (or citric acid). That this is so is further confirmed by the general acceptance of the use of ammonium citrate solution, to differentiate between " reverted phosphate " (dicalcic phosphate) and tricalcic phosphate.

The following conclusions are supported by the experimental evidence adduced by the author. The substances sold as " phosphate of lime " and " calcii phosphas, B.P.," are not pure tricalcic phosphate, but are mixtures of di- and tricalcic phosphates. Sodium phosphate (Na_2HPO_4) added to ammoniacal calcium chloride and the resulting precipitate washed with water yields a mixture of di- and tricalcic phosphate and calcium hydrate. Bone ash dissolved in hydrochloric acid and precipitated with ammonia, the precipitate being well washed, yields also a mixture of di- and tricalcic phosphate and calcium hydrate. When three equivalents of lime (3CaO) are made to act on one equivalent of phosphoric acid (P_2O_5) and the resulting precipitate removed with little delay, pure tricalcium phosphate is obtained. When two equivalents of lime are made to act on one equivalent of phosphoric acid the product is not dicalcic phosphate, but is a mixture of di- and tricalcium phosphate. In the case of pure tricalcium phosphate, about 91 per cent. of the total phosphoric acid is soluble in the prescribed 2 per cent. citric acid solution in thirty minutes when following the method adopted for the estimation of " citrate soluble " phosphoric acid. By the simple addition of calcium

carbonate to a pure tricalcic phosphate, the " citrate solubility " of the phosphoric acid is reduced from 91 to 84 per cent. The availability of the phosphoric acid as judged by the extraction with 2 per cent. citric acid solution is inexact, since the solubility of the phosphoric acid in pure tricalcium phosphate is reduced from 91 per cent. to 84·5 per cent. by the addition of 14 per cent. of lime as carbonate, and to 84·3 by the further addition of 14 per cent. lime as carbonate. The prescribed 2 per cent. citric acid solution is more correctly a solvent for lime than for phosphoric acid, since, broadly speaking, the whole of the excess of lime beyond that present as tricalcic phosphate goes into solution in the first thirty minutes' extraction. Pure tricalcic phosphate is largely soluble in 2 per cent. citric acid solution, as are also the so-called tricalcic phosphates produced by the addition of ammonia to acid solutions of tricalcic phosphate, or by the mixing of disodium hydrogen phosphate with ammoniacal calcium chloride. (These are mixtures of di- and tricalcium phosphate in varying amounts.) Since tricalcic phosphate and dicalcic phosphate are both soluble in the prescribed 2 per cent. citric acid solution, the statement that dicalcic phosphate can be differentiated from tricalcic phosphate, by means of the selective action of this solvent, is untenable.

It follows that the manurial value of phosphates cannot be determined by a 2 per cent. citric acid solvent in the method prescribed, and it therefore is a matter for consideration whether or not the further use of this method should be continued.

<div align="right">H. F. E. H.</div>

Solubility of Mineral Phosphates and Superphosphates in Dilute Mineral and Organic Acids. A. Aita.

(*Annali Chim. Applic.*, 1917, **9,** 200-210.)—Comparative estimations were made of the solubility of mineral phosphates and superphosphates in dilute sulphuric, hydrochloric, formic, acetic, oxalic, citric, and tartaric acid solutions of $\frac{1}{100}$ to 1 grm. mol. per litre in strength. After making allowance for such factors as the physical condition and proportion of lime in the samples, it was found that the amount of phosphoric anhydride dissolved by the mineral acids from superphosphates was comparable with that dissolved from mineral phosphates. When extracted with the organic acid solutions mineral phosphates behaved in the same way as when treated with inorganic acids, except that there was less dissociation. In the case of oxalic acid similar amounts of phosphoric acid were extracted from superphosphates and mineral phosphates, but the other organic acids extracted much higher yields from the superphosphates. For example, the following amounts of phosphoric anhydride were dissolved in comparative estimations: By formic acid, 73·28; acetic acid, 62·61; oxalic acid, 24·44; tartaric acid, 66·43; and citric acid, 77·87 per cent. The high citric-solubility of phosphoric acid is due to the formation of complex citrophosphates in combination with aluminium and ferric iron, and it is probable that similar complex compounds are formed with other organic acids.

<div align="right">C. A. M.</div>

Estimation of Potash by the Lindo-Gladding Method. P. L. Hibbard.

(*J. Ind. and Eng. Chem.*, 1917, **9,** 504-513.)—In the preliminary treatment of the solution to remove lime, phosphates, etc., ammonium oxalate must not be added until after

the solution has been made alkaline with ammonia, and, if there is more than enough phosphoric acid present to form insoluble compounds with the lime, etc., present, ammonium oxalate should not be used, because it increases the amount of phosphate remaining in solution. If much soluble phosphate is present, phosphoric acid should first be removed by addition of a soluble calcium salt, the gelatinous precipitate (which occludes potash) washed, redissolved in acid, calcium phosphate reprecipitated by ammonia and filtered off, and the filtrate combined with the first filtrate. From this mixed filtrate, excess of lime is removed by means of ammonium oxalate.

Ignition of the residue is probably the chief source of the low results obtained by the method. About 10 mgrms. of sugar added during the evaporation with sulphuric acid greatly reduces the risk of mechanical loss.

In the final precipitation as potassium platinichloride, concentration of potassium should be so low that there is no immediate precipitate, and the author states that, when there is much sodium present, not much more platinum solution than is necessary to combine with the potash should be added, as it is much easier to remove sodium as chloride or sulphate by means of the ammonium chloride wash liquor than it is to remove it as platinichloride by washing with alcohol. G. C. J.

Analysis of Pyrolusite and Other Oxidised Manganese Ores. O. L. Barnebey and G. M. Bishop. (*J. Amer. Chem. Soc.*, 1917, **39**, 1235-1237.)—Ferric iron is usually present in manganese ores and causes an error in the estimation of the available oxygen of the ore by the iodimetric method, since ferric salts in acid solution liberate an equivalent amount of iodine from potassium iodide. In neutral tartrate solution, however, ferrous iron is oxidised completely to the ferric state by an excess of iodine, whilst manganous salts are not affected. These reactions may be applied to the estimation of available oxygen in pyrolusite. The sample is ground finely and dried at 110° C. for one hour; 0·2 grm. of the powder is then treated in a flask with 10 c.c. of $\frac{N}{1}$ sodium iodide solution and 5 c.c. of concentrated hydrochloric acid. When the reaction is completed, 0·5 grm. of powdered sodium tartrate is added, the mixture diluted to 150 c.c., and an excess of sodium bicarbonate is introduced. The free iodine is then titrated with standard arsenite solution. The use of sodium salts is recommended in the method, as potassium salts tend to yield a precipitate of potassium acid tartrate. W. P. S.

British Sands Suitable for Glass-Making. P. G. H. Boswell, H. F. Harwood, and A. A. Eldridge. (*Memoir published at the Instruction of the Ministry of Munitions of War, by the Imperial College of Science and Technology, 1916, pp. 1-92, with plates and microphotographs.*)—A sand composed of grains of one grade, which should not be a coarse one, yields the best material for glass-making provided that its chemical composition is satisfactory. The silica content must be high and the quantity of iron oxide present should be less than 1 per cent., the higher limit being permissible only for glass used in making the cheapest kind of bottles. A sand used in the manufacture of the best varieties of glass (optical glass, flint and sheet glass, etc.) should not contain more than 0·08 per cent. of iron oxide. Alumina, magnesia, and lime may be present as felspars, ferromagnesian and lime-bearing minerals, and calcareous cement, but are required only for certain glasses; these

bases are refractory and lengthen the time required for melting. Sands free from them are preferable, and it is better to rely on the sand only as a source of pure silica, and to add other bases for the purpose of making various kinds of glass. The sand should consist of angular grains. Fontainebleau sand and Lippe (Germany) sand are the most perfect sands for glass-making, and a sand equal in all respects to these does not appear to exist in the British Isles. Sand from Aylesbury, in Buckinghamshire, is, however, practically equal in quality to Fontainebleau sand, but is of less extent; sands from Huttons Ambo and Burythorpe, Yorkshire, and other places, are as good as the Belgian sand imported. The best Lynn sand is also equal to much of the Belgian sand and is superior to some of the Dutch sand. The same applies to Godstone and Reigate sands, but these deposits are irregular. A large number of British sands are less pure, but are well suited to the making of common bottle-glass.

Sands may be improved by washing, burning, extraction with hydrochloric acid, magnetic separation treatment, and grinding, but these operations entail expense and can only be adopted for sands used in making the best kinds of glass.

Analyses of glass-making sands are given in the following table:

	SiO_2	Al_2O_3	Fe_2O_3	CaO	Loss on Ignition.
	%	%	%	%	%
Lippe..	99·88	0·18	None	None	0·21
Fontainebleau	99·80	0·13	0·006	Trace	0·18
Belgian	99·38	0·30	0·02	None	0·23
Dutch	99·23	0·50	0·02	None	0·22
Sandymount Strand, Dublin	84·24	3·61	0·47	4·65	4·81
Jura, shore-sand	97·89	1·19	0·07	0·18	0·42
Parsley Hay, High Peak	74·54	18·04	0·05·	0·19	7·24
Brassington, High Peak	90·40	6·56	0·18	0·16	2·48
Abergele	99·35	0·54	0·04	None	0·36
Charlton, Thanet Beds	95·21	2·43	0·42	0·19	0·88
Alum Bay, Headon Hill Sands	96·96	1·90	0·11	0·34	0·64
Aylesbury, Lower Greensand	99·80	0·32	0·03	None	0·22
Leighton Buzzard, Lower Greensand	99·59	0·25	0·21	None	0·27
Reigate, Lower Greensand	98·93	0·67	0·02	Trace	0·28
Aylesford, Lower Greensand	99·06	0·56	0·04	0·17	0·22
Lynn, Lower Greensand	99·23	0·59	0·04	0·11	0·25
Flitwich, Lower Greensand	98·30	0·87	0·18	None	0·47
Godstone, Lower Greensand	99·56	0·26	0·06	None	0·24
Burythorpe Fox Cover Pit, Helloway Beds ..	96·79	1·63	0·22	None	0·60
Burythorpe Park, Helloway Beds	97·73	1·41	0·06	Trace	0·32
South Cave, Helloway Beds	94·80	2·93	0·13	0·14	0·47
Huttons Ambo, Upper Estuarine ..	99·04	0·84	0·03	0·10	0·19
Gurseley, Coal Measures	97·45	1·76	0·09	0·13	0·78
Crofthead, Carboniferous ..	97·00	2·02	0·20	0·14	0·76
Muckish Mountain, Dalradian	99·37	0·36	0·05	None	0·12

Mechanical analyses of the best sands indicate that a sand for use in glass-making should have at least 70 per cent., and, if possible, more than 90 per cent. of its grains of one grade, and that this grade should be in most cases medium sand— *i.e.*, with a diameter between 0·25 and 0·5 mm.; if the main grade has a diameter of 0·1 to 0·25 mm., so much the better, but such sands are not abundant in the British Isles. W. P. S.

Estimation of Silica in Commercial Analysis. F. G. Hawley. (*Chem. Engineer*, 1917, **25**, 100-103.)—The principal source of error in the estimation of silica is due to the solution of the precipitate in hydrochloric acid. The degree of solubility of freshly precipitated silica depends upon the amount of acid present, the strength of the acid, the temperature, and the time of contact. In some of the methods of estimation in which from 50 to 100 c.c. of dilute hydrochloric acid are used to dissolve the bases as much as 2 per cent. of silica may be dissolved from a sample containing less than 20 per cent. When the volume of the acid solution is large, considerable time is required to saturate it with silica, but with a small volume of acid the saturation-point is rapidly reached. Hence the length of time of boiling is of less importance under the latter conditions. For the same reason dilution of the liquid with boiling water must be avoided. The experiments cited indicate that the most satisfactory method of dehydrating the silica is to heat it for thirty minutes at 120° to 150° C. As a rule, boiling for three minutes with 60 per cent. hydrochloric acid yields as pure a silica as is given by boiling for a longer time or with stronger acid. The following modification of the peroxide fusion method embodies the results of these experiments. A mixture of 0·5 grm. of the sample with about 4 grms. of a flux composed of equal parts of sodium peroxide and sodium hydroxide is fused in a nickel crucible at a temperature which is graduall increased to a dull red heat. After the fusion the crucible is covered with a watch-glass and placed in a 4-inch basin, and 2 to 3 c.c. of warm water introduced from a wash-bottle, more water being subsequently added until the mass is disintegrated. About 10 c.c. of 60 per cent. hydrochloric acid are then introduced little by little, followed by 90 per cent. acid until it is in excess, after which the basin is placed on a hot plate. When the liquid has evaporated the dry residue is covered with a watch-glass, and heated for thirty minutes at about 135° C., and then cooled and boiled for three minutes with 15 c.c. of 60 per cent. hydrochloric acid. The sides of the basin are rinsed with warm water, and the contents transferred to a filter and washed twice with warm water and once with dilute hydrochloric acid, the basin being also rinsed with the acid whilst its sides are rubbed with a rod to remove adhering silica. The filter is again washed twice with water, partly dried and ignited in a crucible for about ten minutes, and weighed as soon as cool. A correction of 0·4 per cent. is made for the silica lost by solution under these conditions, whilst a deduction is made for impurities in the silica and silica in the flux, the amount of the gain from these sources approximately balancing the solubility loss.

C. A. M.

APPARATUS, ETC.

Measurement of Absolute Viscosity of very Viscous Media.   S. E. Sheppard. (*J. Ind. and Eng. Chem.*, 1917, **9**, 523-527.)—For the measurement of the viscosity of extremely viscous fluids, the most convenient method is that based on the observation of the velocity of fall of a heavier spherical body through the liquid and the application of Stokes' law:

$$K = \frac{2g R^2 (s - s')}{9V},$$

where K is the coefficient of viscosity, g the gravitational constant, R the radius of the sphere, V the velocity of fall, s the density of the sphere, and s' that of the liquid.

Stokes' law only holds exactly when (1) the fluid is infinite in extent, (2) the sphere is smooth and rigid, and (3) the velocity of fall is small.   With the highly viscous liquids under consideration, condition (3) is readily satisfied, whilst steel balls, such as are now manufactured for bearings but were unknown in Stokes' day, satisfy condition (2).   Condition (1) can never be perfectly fulfilled.   The author has, however, determined experimentally a series of corrections to correct for this.   The observed time of fall (T) is corrected to what it would be under Stokes' conditions of infinite extent of fluid (T∞) by an equation of the type:

$$T = T\infty + \frac{C}{(r - 1)^2},$$

where r is the ratio of the diameter of the cylinder used for the experiment to that of the sphere and C a constant which has the value 268 for a $\frac{1}{4}$-inch sphere, 72 for a $\frac{3}{8}$-inch sphere, and 46 for a $\frac{1}{2}$-inch sphere.

A further small correction is necessary because the depth of the liquid is not infinite.   When a depth of 40 cm. of liquid is available and the velocity is calculated from the time of fall through the middle 20 cm. or thereabouts, the value of K found by application of Stokes' law and the correction previously described should be multiplied by $(1 + 11 \cdot 6 \, ^R/_L)$, where R is the radius of the sphere and L the total height of the liquid in the cylinder.   G. C. J.

Improved Compensator for Gas Analysis.   E. T. Gregg. (*J. Ind. and Eng. Chem.*, 1917, **9**, 528.)—Two platinum wires are sealed into the compensator side of the mercury manometer.   The upper wire, which is sealed about $\frac{3}{8}$ inch above the mercury when level, is bent downwards at right angles and just touches the surface of the mercury.   The lower wire enters far enough to make electrical contact with the mercury.   In series with the two wires are a dry cell, switch and lamp.   To use the compensator for adjusting the volume of a gas before making a reading, the contact between the mercury and upper platinum wire is broken by means of the levelling bulb containing mercury; the switch is then closed and the compensator adjusted until the break in the circuit at the mercury-platinum contact is just closed, whereupon the lamp lights and the compensation is completed.   Rough adjustment is made by sliding the mercury bulb and its support upon the iron rod by hand.   The fine adjustment is made by taking up the sag in the arm supporting the bulb

by means of a thumb-screw. The switch is then opened to prevent sparking at the mercury surface when breaking the circuit and to prevent deterioration of the battery. Compensation should always be made with a rising mercury column at point of contact. In practice this compensator checks the gas volume consistently and accurately and does it in less time than the old optical method of adjusting the height of the mercury to a mark. It removes the difficulty of adjustment due to poor or changing light and decreases the strain on the eyes of the operator. Where many analyses are made, it removes one of the chief sources of fatigue.

G. C. J.

Measurement of Cryoscopic Constants at Elevated Temperatures.
J. H. Mathews. (*J. Amer. Chem. Soc.*, 1917, **39**, 1125-1133.)—An apparatus is described for controlling the temperature of the jacket while freezing-points are being determined with the use of solvents having relatively high melting-points. The freezing-point tube proper is placed in a larger tube which serves as an air-jacket, and the two tubes are suspended in the neck of a round-bottomed flask provided with a side-tube. A suitable liquid, usually some hydrocarbon, having a boiling-point a few degrees above the freezing-point of the solvent to be used, is placed in this flask and boiled. The vapour completely envelops the freezing-point tube, and then passes into an inclined condenser attached to the side-tube; the condensed liquid returns to the lower part of the flask through a by-pass. The other end of the condenser is connected with a large reservoir (*e.g.*, a five-gallon bottle), which in turn is connected with a suction pump and a manometer. The temperature of the vapour surrounding the freezing-point tube can be adjusted to any desired point by regulating the pressure in the system, and is subject to both easy and accurate control. When naphthalene is used as the solvent, sublimation may be prevented by passing a feeble electric current through a coil of fine platinum wire placed in the upper part of the freezing-point tube. W. P. S.

Device for Automatic and Intermittent Washing of Precipitates.
E. C. Lathrop. (*J. Ind. and Eng. Chem.*, 1917, **9**, 527-528.)—The device differs in only one respect from the old one of an inverted flask with two tubes of moderately small bore passing through the cock. The difference lies in the use for the air inlet tube of a capillary tube. The result is intermittent action, which results in much more efficient washing with a definite quantity of water. With a capillary tube, water does not begin to flow from the flask immediately the lower end of the air inlet tube ceases to be submerged, but only when the level of liquid in the funnel has fallen to a point such that the resulting differences of hydrostatic pressure in the two tubes is sufficient to overcome the force of capillarity which tends to keep the capillary tube full of water. Experiment has shown that the vertical distance from the end of the capillary tube to the point on the other tube at which the pressure just overbalances the capillarity is just a little greater than the height of the capillary rise. It is clear that by having the outlet tube nearly touching the precipitate, the air inlet tube of suitable bore and ending a little below the top edge of the paper, a precipitate may be washed automatically almost as well as by hand.

G. C. J.

Contamination of Precipitates in Gravimetric Analysis. Solid Solution and Adsorption *v.* **Higher-Order Compounds. G. McP. Smith.** (*J. Amer. Chem. Soc.*, 1917, **39**, 1152-1173.)—As regards the contamination of barium sulphate by iron, it is shown that barium sulphate, which has been precipitated from solutions containing ferric salts, carries down the latter in varying quantities, not in the form of a solid solution of basic ferric sulphate or other compound, nor adsorbed on the surface of the grains, but in the form of a hydrated complex barium sulphato-ferriate which is present in intimate mechanical admixture with the barium sulphate.

W. P. S.
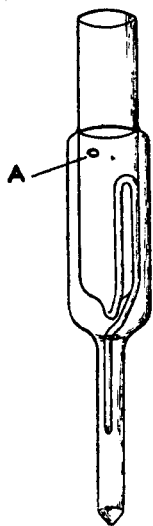
Modified Soxhlet Extractor. D. F. Twiss and W. McCowan. (*J. Soc. Chem. Ind.*, 1917, **36**, 692.)—The apparatus is based upon the same principle as that devised by Blount (ANALYST, 1888, **13**, 127). In this modification, however, there is only one aperture, *A*, about 0·7 cm. in diameter, through which the vapour passes into the inner compartment, and this restriction causes sufficient increase in the temperature and pressure to keep the solvent gently boiling. A further advantage is that the double jacket is shorter than in Blount's extractor, which adds to the strength of the apparatus, and reduces the air-cooling surface with which the vapour comes in contact before entering the aperture. C. A. M.

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REVIEWS.

ANNUAL REPORTS OF THE SOCIETY OF CHEMICAL INDUSTRY ON THE PROGRESS OF APPLIED CHEMISTRY. Vol. I., 315 pp. and Indices. 1917. Price 3s. 6d.

The custom of issuing annual reports on the progress of the various sciences is one of such obvious utility that it is not surprising to find that the Society of Chemical Industry has followed the example of similar institutions.

In applied chemistry a special case may easily be made out for the appearance of a report such as this. It is given to few to be personally and practically familiar with more than one or two of its branches, while there are many to whom a general knowledge of them is desirable and necessary, but on whose time the constant stream of literature dealing with pure chemistry or with their own special department of the science makes only too ample a demand.

The present report does not cover all the sections dealt with in the Society's journal, partly because those on fibres, dyeing, metallurgy, electro-chemistry, and sugar were not completed in time, and partly because agricultural and analytical chemistry and the chemistry of food and drugs, having been dealt with in the Annual Report issued by the Chemical Society, were purposely omitted. In refraining from

issuing a separate report of its own on these subjects, the Society of Chemical Industry has set an excellent example, which it is to be hoped will ere long be taken to heart by similar societies, so that the waste of labour involved in the translation, abstracting, and printing, by three or four different scientific bodies, of the same set of papers will no longer be a reproach to the common sense of scientific men.

Nominally the Report for the year 1916, the volume before us, actually deals with 1914 and 1915 as well, thus covering the whole period since the commencement of the war.

The following fifteen sections, as classified in the Society's journals, are dealt with: Fuel and Heating (J. W. Cobb); Gas, Destructive Distillation, Tar Products (E. V. Evans); Mineral Oils (J. W. Cobb); Colouring Matters and Dyes (G. T. Morgan); Acids, Alkalis, and Salts (H. A. Auden); Glass and Ceramics (J. A. Audley); Building Materials (J. A. Audley); Oils, Fats, and Waxes (G. H. Warburton); Paints, Varnishes, and Resins (R. S. Morrell); Indiarubber (H. P. Stevens); Leather and Glue (J. T. Wood); Fermentation Industries (A. R. Ling); Water Purification, etc. (F. R. O'Shaughnessy); Fine Chemicals (F. L. Pyman); Photographic Materials and Processes (B. V. Storr).

In a series of articles by different authors, and on such widely different subjects, uniformity of treatment is hardly to be expected, but, taking these facts into account, it may be said that this new undertaking has been very successfully carried out. One or two of the articles are unnecessarily diffuse, but most of them are models of what such reports should be, and give in the minimum of space a clear and comprehensive statement of the progress of each branch during the period under review; and the copious references to original papers and to patent specifications render it easy for the reader to refer to the original sources. These references are conveniently placed at the bottom of each page, except in the case of the article on indiarubber, where they are at the end of the article—a plan of which the advantage is not apparent.

Printer's errors and similar oversights are remarkably few. On page 25 we note that the new form of bolometer is in the text attributed to Callender, whereas we believe it was the joint production of Bone, Callender, and Yates, to whose paper on the subject reference is made.

The type and paper are excellent, and the volume is very conveniently of the same size as those of the Annual Reports of the Chemical Society. The Society of Chemical Industry is to be congratulated on having initiated a new and valuable departure, and one which is especially opportune at the present time.

CECIL H. CRIBB.

BAYLEY'S CHEMISTS' POCKET BOOK. Eighth Edition, edited by R. ENSOLLO, E. and F. N. SPON, 1917. Price 7s. 6d. net.

The publication of this useful little volume in the form of a handbook has afforded an opportunity for the recasting of the whole work and the inclusion of many new tables. The International atomic weights for 1915 and 1916 have been used for the factors and tables in Sections V. and VI., which deal with gravimetric

and volumetric analysis, while other sections include valuable data referring to Weights and Measures, Physics, Mathematics, General Analysis, Photography, and numerous other branches of Chemistry. In some few cases the author's name is appended to the tables quoted, but it would have been no more trouble and distinctly more useful if, in addition, the place and date of publication had been indicated. Thus the present Official Original Gravity Tables, which have superseded those contained in the first schedule of the Inland Revenue Act of 1880, are quite rightly given in the section on Beer, but are simply headed " Thorpe and Brown," while a table giving the " divisors " or solution factors for certain sugars is credited to " Brown and Jones "; both of these tables are to be found in the *Journal of the Institute of Brewing*, 1914, vol. 20. The printing and general get-up of the book is good, and considering the amount of matter compressed into its 420 pages, and the obvious care which has been given to insure accuracy, the price (7s. 6d.) is certainly not excessive.   H. F. E. HULTON.

LABORATORY MANUAL OF BITUMINOUS MATERIALS FOR THE USE OF STUDENTS IN HIGHWAY ENGINEERING. By PRÉVOST HUBBARD. Pp. 153. New York: John Wiley and Sons. London: Chapman and Hall, Ltd., 1916. Price 7s. net.

The progressive spirit of American Universities is shown by the fact that several have inaugurated courses of instruction in Highway Engineering, and the author of this excellent little manual, who is chief of that section of the U.S. Department of Agriculture which deals with Road Material Tests and Research, is lecturer in the graduate course of Highway Engineering in Colombia University.

The book is entirely American, and describes American products and American testing methods exclusively. There is no reference in it to the work or specifications of the English Road Board, or to the methods of testing used here. It is arranged in three parts. The first, occupying twenty-seven pages, gives a list of definitions of materials and tests which have become standard in the United States, and describes the various types of bituminous materials used by American highway engineers. The second part, occupying the major portion of the work, is devoted to a description of methods of testing, and the third part shows how these tests are applied and their results interpreted. Many of the testing methods are taken verbatim from the U.S. Department of Agriculture bulletins and other American publications, and represent the standard methods which have come into use in the States. As a rule they are good and well described, some of them perhaps rather more delicate and elaborate than appears necessary for the purpose. Specific gravity is determined by hydrometer at the standard temperature of 25° C., the Westphal balance and Sprengel tube methods being given as alternatives. Hubbard's specially designed pycnometer is used for very viscous and semi-solid bitumens, and the displacement method for solid materials and mineral aggregates. Having described all which methods, the author then shows the use to be made of the determination for identifying a material, and how by means of it the coefficient of expansion is determined. This plan is followed throughout the book. Under consistency tests, viscosity is measured by the Engler viscometer, which the author

admits is " far from ideal " for the wide range of bituminous materials used. Pre-
sumably it is only used for the more fluid types. A " float test " is next described,
which appears to be a weird combination of melting-point, fluidity, and heat-con-
ductivity tests, and is certainly not a viscosity test. The penetration tests which
follow seem much more useful. Then follow methods of determining melting and
softening points, ductility, flash and burning points, volatility, methods for estima-
tion of water, distillation and solubility tests for estimating total bitumen and
separating this into asphaltenes and carbenes, etc., methods for ash, fixed carbon,
paraffin scale, and for the analysis of emulsions, creosote oils, and bituminous aggre-
gates. In the third section of the book complete series of tests of fluid petroleum
products, emulsions, asphalts, asphalt cements and fillers, refined tars and wood
preservatives and bituminous aggregates, are arranged in tables, with copious notes
showing how the results are to be interpreted. This section of the book is interleaved
with blank pages for notes. There is a fairly complete table of contents, but no index.
Notwithstanding the absence of any reference to English practice, the book should
prove extremely useful to all analysts who have to advise on highway matters. If,
in a future edition, English practice and testing methods were also described and an
index added, the general usefulness of the book to English chemists and engineers
would be greatly enhanced.   L. ARCHBUTT.

' EXPLOSIVES," Vol. II., by ARTHUR MARSHALL. Second Edition in Two Volumes.
    Churchill, 1917. Price £3 3s. 0d. (*Cf.* ANALYST, 1917, 259.)

The second volume of the above work, which has just appeared in the new
edition, is of special interest to analytical chemists, as it deals very fully with the
analysis of explosives. To those occupied in explosive work the chapters on the
physical properties and tests, the stability tests, and the ballistic properties, will
also be found useful. Many advances have been made in these directions in recent
years, and the appearance of this comprehensive review of the subject is very oppor-
tune at present, as the attention of most chemists has been directed to the study of
explosives. Whilst recognising the importance of naval and military explosives,
however, Mr. Marshall has dealt also very fully with industrial explosives, which play
a most important part in mining, road construction, etc. The safety precautions
necessary in the construction of buildings, the transport and handling of explosives,
the gases evolved, and the special dangers of coal-mines, are of the greatest import-
ance in the explosives industry, and the information given by Mr. Marshall will be
found very useful to any chemists who are concerned with this branch of the subject.
The section on " Materials and their Analysis " will be of chief interest to
analytical chemists. Explosives form a specialised branch which has in the past
remained in the hands of comparatively few chemists, as the handling of such com-
pounds requires very special care. The textbooks on the subject have remained
somewhat scanty, and have not kept pace with the developments in explosive
research. For this reason the collection of information on the analytical methods
used in explosive work meets a distinct want. Information is given as to sampling,
and the chemical reactions are given for a large number of explosives and their

ingredients. It way, perhaps, be criticised that propellants and their ingredients have received more than their share of notice. Thus, ten pages are allotted to nitro-cellulose, whilst such important materials as trinitrotoluene, perchlorates, aluminium, and others, receive little or no notice.

A systematic scheme for the analysis of unknown mixtures might with advantage have been added, and the methods for the quantitative analysis of cordite and similar mixtures would be helpful to many chemists. The whole compilation cannot, however, fail to be very useful for reference, and will undoubtedly lighten the labour of many analysts.   R. C. FARMER.

INTRODUCTION TO THE RARER ELEMENTS. By PHILIP E. BROWNING. Fourth
    Edition. London: Chapman and Hall, Ltd., 1917. Price 7s. net.

The author states in his preface " that this little work is intended not to treat exhaustively the subject of the rarer elements, but rather to serve as an introduction to the study of an interesting group which the chemist in the inorganic field cannot afford to neglect. When one considers that more than fifty elements are included under the heading " Rarer Elements," and that of these a number such as the elements of the platinum group, vanadium, molybdenum, and tungsten form more numerous and complex series of compounds than the more common elements, it is clear that anything approaching an exhaustive treatment is impossible in a work of this size, and much must necessarily be omitted. For this reason discrimination is most important in the compilation of a work of this kind; unfortunately there is not sufficient evidence of the exercise of this quality on the part of the author. Lists of methods of estimation and separation are given, which include some that are standard methods and in general use, others that have been merely described or suggested, and others, again, that are known to contain sources of error, without a word or sign to assist the student in his selection. As an example of the brevity of treatment one may mention the gravimetric estimation of gold, which is disposed of in four lines, yet considerably more space is devoted to the estimation of tellurium. These defects notwithstanding, the book has its value: it contains a surprising amount of information in a very condensed form, with comparatively few errors. The experienced reader will realise its limitations, but will find it of service as a small book of reference before having recourse to larger works.

The book, however, was written for students, and prepared from material used by the author in a course of lectures at Yale University. Regarded from the educational point of view, the book possesses a peculiar interest, and some reference may be made to the purely tutorial chapters dealing with experimental work and review questions. With the exception of those elements which are unavailable because of their scarcity, lists of experiments are given beginning with the extraction of the salts of the element from a mineral and followed by a series of experiments illustrating the qualitative analytical reactions. In no case is a quantitative exercise set, an omission which seems questionable in an experimental course for students. A similar attitude is also noticeable in the review questions; to answer these the student must have committed to memory almost all the reactions mentioned in the text, for questions are to be found such as, " How may Ru and Os be detected when

present together ?" "How does a mixture of H_2O_2 and HCl act upon Nb_2O_5 and Ta_2O_5 ?" yet no question is set on the quantitative estimation of even the more common amongst the rarer elements such as titanium, lithium, or thorium. While agreeing with the author as to the importance of the rarer elements, one may yet doubt whether it is wise to ask a student to burden his memory with such a huge collection of facts regarding substances which he may never meet in practice. If inquiry be made of the experience of successful analysts, it will be found that they have been able to detect these rare elements when necessary, and even the much more troublesome and evasive organic substances, without having memorised all their reactions; analysts have even discovered and characterised new substances in the course of their analytical practice, as instance the recent discovery of spinacene in fish oils, an achievement which evidently cannot be attributed to a knowledge of the reactions of the substance discovered. The secret of such success appears to depend less upon an extensive knowledge of the rare than upon a more profound knowledge of, and familiar acquaintance with, the common, by means of which any abnormality is recognised and the presence of the rare inferred. This stage having been reached, it is more usual to refer to books than to rely upon memory. If this experience can serve as a guide to teachers, it would point to the practice of giving rare substances for analysis to advanced students, but permitting the use of books rather than attempting to impose a tax upon the memory which but few could endure.

E. WECHSLER.

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LAW REPORT.

The sale of Milk: *Need of a New Act. Grigg v. Smith. King's Bench Division, before the Lord Chief Justice, Mr. Justice Ridley, and Mr. Justice Atkin. (*The Times,* July 27, 1917.)—This appeal raised a point of great public interest as to the sale of milk. It was brought on a case stated by the Justices of Stratford-on-Avon upon an information against the respondent Smith, under Section 6 of the Food and Drugs Act, 1875, for selling milk which was deficient in fat to the extent of 13 per cent., and which was not of the nature, substance, and quality demanded by the purchaser.

The respondent was the owner of only one cow, which had recently calved. He occasionally sold milk to a milk retailer, and the appellant purchased from the respondent's wife half a pint of milk. On the morning of the purchase the cow had not been fully milked, and the respondent stated that he had left some milk in the cow for the calf. Nothing was added to or abstracted from the milk. The analyst certified that the milk was deficient in fat to the extent of 13 per cent., the actual percentage being 2·6 instead of 3 per cent., as fixed by the Sale of Milk Regulations, 1901. This was found as a fact by the Justices. Four days after, when the cow was completely milked, a sample taken contained 3·9 per cent. in milk fat. The Justices held that the deficiency was due to the manner in which the respondent had milked his cow. They held, however, that as the milk had been

sold as it came from the cow, without abstraction or addition, it was of the nature, substance, and quality demanded by the appellant, and they dismissed the information.

Mr. C. F. Lowenthal appeared for the appellant. The respondent did not appear.

Mr. LOWENTHAL referred to Hunt v. Richardson (32 *The Times* Law Reports, 560; [1916], 2 K.B., 446); Marshall v. Skeft ([1913], 29 *The Times* Law Reports, 152); 108 *L.T.*, 1,001, 636; and Anness v. Grivell ([1915], 3 K.B., 685).

The LORD CHIEF JUSTICE, in giving judgment, said that the question for decision was whether the view of the law taken by the Justices was correct as based on the facts found which bound that Court. The Court had had the advantage of an able and interesting argument from Mr. Lowenthal, but it was impossible to get over the decision in Hunt v. Richardson (*sup.*). The magistrates had before them one standard as to milk—namely, that it must contain not less than 3 per cent. of milk fat, and the authority for that was to be found in the regulations of 1901. The Justices thought that, though the milk did not contain the full percentage, yet 'as it was sold as it came from the cow the presumption of adulteration was rebutted by the evidence, and no offence had been committed. In his opinion this was the true effect of the decision of the Hunt v. Richardson (*sup.*). He thought that the majority of the Court decided that when the product was sold as it came from the cow it was milk, and being so there was no offence on its sale. In that case, as in this, there was an attempt to show that the milk must contain 3 per cent. of milk fat; but that was not so. What was said was that if it did not contain that percentage there should be a presumption that something had been added or abstracted; but that presumption could be met by evidence; and if the Court were satisfied that the presumption had been met by evidence then no offence was committed. In his opinion, Hunt v. Richardson (*sup.*) could not be distinguished from the present case. The Justices here were right, and the appeal must be dismissed.

He wished to add that the arguments had convinced him that it was desirable for the authorities to reconsider the position, and to determine whether it was the intention of the Legislature, or of the departments which had the means of introducing amended Acts of Parliament, that milk should be sold to the public as it was in this case, with an undoubted deficiency in milk fat, as compared with milk usually sold; the result being that the farmer could retain for himself the better quality milk, leaving to the public the inferior quality. He did not think that he should express any opinion on the policy to be adopted, but he wished to say that the effect of Hunt v. Richardson and the case before the Court made it desirable that the matter should be considered.

Mr. JUSTICE RIDLEY and Mr. JUSTICE ATKIN gave judgment to the same effect, and expressed similar opinions as to legislation.

Mr. JUSTICE ATKIN added that a farmer was now entitled by law to give a preference to his own calves over the babies of his customers.

Solicitors: Messrs. Ward, Bowie, and Co., for Messrs. T. W. Walthall and Pritchard, Birmingham.