THE ANALYST

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

An Ordinary Meeting of the Society was held at the Chemical Society's Rooms, Burlington House, on Wednesday, April 3rd, the President, Mr. Edward Hinks, being in the chair.

Certificates were read for the first time in favour of:—Alfred Norman Leather, B.Sc., F.I.C., Richard Harold Morgan, B.Sc., A.I.C., and William George Painton, B.Sc., A.I.C.

Certificates were read for the second time in favour of:—Peter Trevisa Clarke, B.A., Alfred Clive James, B.Sc., A.I.C., Herman Lee, B.Sc., A.I.C., James Frederick Morse, Lawrence John Odling, Willie Horner Wilkinson.

The following were elected Members of the Society:—Frank Atkins, Edmund Baron Bennion, M.Sc., A.I.C., John Haslam, M.Sc., A.I.C., Stanley Gordon Kendrick, B.Sc., A.I.C., Bryn Jones, B.Sc., A.I.C., John Upton Lewin, B.Sc., A.I.C., and Leslie John Walker.

The following papers were read and discussed:—"Furfural and Diastase in Heated Honey," by L. H. Lampitt, D.Sc., F.I.C., E. B. Hughes, M.Sc., F.I.C., and H. S. Rooke, M.Sc., A.I.C.; "Further Notes on Methods of Sewage and Water Analysis; Anti-Oxidation and Stabilisation of Pollution," by J. W. Haigh Johnson, M.Sc., F.I.C.; and "Potassium Cyanate as a Reagent for the Detection of Cobalt," by B. J. F. Dorrington, B.Sc., A.I.C., and A. M. Ward, B.Sc., Ph.D., A.I.C.

Obituary.

GEORGE WATSON GRAY.

By the death of George Watson Gray, which occurred on the 12th February last, at the age of 66, Liverpool has lost one of its best known consulting chemists. He received his early training at the Rutherford Technical College, Newcastle-on-Tyne, and in the laboratories of Mr. John Pattinson, and came to Liverpool in 1883 as

assistant to Mr. A. Norman Tate. Ten years later Mr. Watson Gray set up his own laboratories and continued in practice until a short time before his death.

Among the branches of analytical work to which he devoted special attention may be mentioned the rarer constituents of alloy steels, ferro-silicon, and tanning materials, and he was the author of many papers dealing with these matters, latterly in collaboration with his partner, Mr. James Smith.

Mr. Watson Gray took an active part in the establishment in Liverpool of the first provincial Section of the Institute of Chemistry, and occupied the chair for the first few years. Until ill-health interfered he was an extremely hard worker; he used to say that his work was his hobby, but he found time occasionally for a tramp in the Lake District, of which he was very fond.

He was a Member of the Society of Public Analysts for over forty years.

E. GABRIEL JONES.

The Freezing Point of Milk.

By A. VAN RAALTE, D.Sc.

For more than twenty years we have made use of the freezing point of milk to determine with certainty its adulteration with water. Milk which has a freezing point nearer to zero than -0.53° C. is, as we are convinced, adulterated.

So far back as 1898 the late Dr. A. Lam regularly used this method in his laboratory in Rotterdam, so that the method has been in use more than 30 years in this country. In the Journal of the Dutch Society for the Investigation of Milk he published, in 1909, an article on the freezing point of milk. He cited there 33 different articles, published between 1892 and 1909, dealing with this method; all the authors gave for the freezing point of milk figures varying between -0.54° and -0.59° C.; only Messrs. Bordas and Genin (1896) gave figures between -0.46° C. and -0.56° C., but they analysed—without knowing it—some adulterated samples. The figures, given by Mr. Winter in 1895, viz. -0.54° C. to -0.57° C., have proved to be correct.

Dr. Lam came to the conclusion that mixed milk with a freezing point nearer to zero than -0.54° C. must contain added water; and that milk with a freezing point lower than -0.59° C. must be considered as unsatisfactory.

The milk of some individual cows may sometimes give abnormal figures, but the determination of the freezing point in thousands of samples of mixed milk, taken in the presence of inspectors, has shown that this point lies between -0.54° C. and -0.57° C. I, myself, in 1909, when analysing 155 samples of milk, taken in the presence of inspectors, obtained figures for the freezing point varying only between -0.54° and -0.57° C.

Even in 1914, however, there was still a difference of opinion between some Dutch experts on this subject. But after the polemical discussions of that year

all Public Analysts in Holland agree that normal mixed milk has a freezing point between -0.54° and -0.57° C.

Ever since, all competent judges in Holland have regarded milk with a freezing point nearer to zero than -0.54° C. as being adulterated with water, and the Dutch Government has fixed in its Milk Decree the maximum for the freezing point at not higher than -0.53° C., to be absolutely on the safe side.*

It is, of course, understood that the acidity of milk, when being analysed, may not exceed 9 (c.c. of N/4 alkali for 100 c.c. of milk, with phenolphthalein as an indicator).

Milk obtained from cows with diseased udders can have a freezing point below -0.57° C. Such milk contains, while still in the udder, a great number of lactic acid germs; so long as the milk remains in the udder all lactic acid will be neutralised, as was proved by Mr. Straub in my laboratory. Immediately after milking lactic acid will be formed. This lactic acid does not manifest itself in a high acidity; the acidity of this milk is normal, because milk of such cows has a very low acidity (± 4) immediately after milking.

In most cases the low freezing point makes it unnecessary to take samples in the presence of inspectors, and this method is very rapid in the hands of a skilled analyst. This is a great advantage. The results of the control of milk are largely dependent upon close supervision.

It is for this reason that, in Amsterdam, we analyse every year about 30,000 samples of milk, *i.e.* about one sample for every thirty inhabitants.

Thanks to this strict control it has been found possible to obtain a figure of only 2 per cent. of milk samples adulterated with water, as was the case in 1927. In that year we analysed 29,124 samples, of which 596 contained added water.

In order not to give an advantage to the skilled adulterator it is necessary to determine the freezing point of any milk with a percentage of non-fatty-solids of 8.2 and less, excepting during summer, when we take 8 per cent. as the limit.

This made it necessary in 1927 to determine the freezing point of 1876 samples, an average of 6 a day.

This number, of course, may vary; we determined the freezing point of a maximum of 12 samples a day, and this was done by the same chemist, with an assistant, who analysed 100 samples of milk, and generally had to analyse several samples of butter-milk on the same day.

* The following note from the Report of Dr. Monier-Williams deals with the confusion which may arise from the use of the terms "higher" and "lower" as applied to freezing points below zero:—"A freezing point of -0.520° is obviously higher than one of -0.550° , but it is apparently the usual custom among writers on the subject to express the results obtained in terms of the depression of the freezing point with reference to water, generally expressed by the symbol Δ . Thus a value of 0.550° would be higher than one of 0.520° , as representing a greater depression of the freezing point. The adoption of the latter mode of expression has much to recommend it and tends to avoid confusion as the terms 'higher' and 'lower' correspond with the actual figures and the constant repetition of the minus sign is avoided. In the present report therefore the symbol Δ is used throughout to indicate the difference between the freezing point of milk and that of water."—Editor.

With reference to the two excellent papers published in the March issue of THE ANALYST, it may be mentioned that some of the questions raised in the discussion have been answered in the following contributions from my laboratory: J. Straub, "Milchsäurebestimmungen in Milch" Rec. Trav. Chim. Pays Bas, 1927, 46, 866), and J. Straub and L. Soep, "Recherches sur la Concentration osmotique des Lumeurs" (Arch. néerland. de Physiol. de l'Homme et des Animaux, 1928, 12, 346).

The freezing point method is now coming into use in the United States and in Germany. Figures found in both countries are in absolute conformity with the figures we find in Holland.

The method of the freezing point of milk therefore deserves international acceptance.

KEURINGSDIENST VAN WAREN, 732 KEIZERSGRACHT, AMSTERDAM.

The Determination of Small Quantities of Beryllium in Rocks.

By B. E. DIXON, M.Sc., A.I.C.

(Read at the Meeting, February 6, 1929.)

There is a good deal of negative evidence for the supposition that the occurrence of beryllium in small quantities is much more extensive than appears from its infrequent mention in mineral and rock analyses. Normally, beryllium is not tested for in these circumstances, and is then probably included in the figure for aluminium. Absence of distinctive qualitative tests for beryllium to some extent accounts for this. Examination of those analyses which are provided with a description of the method employed has led to the conclusion that the beryllium figure is, in many cases, untrustworthy. This is chiefly due to the lack of an accurate and suitable method for separating beryllium from titanium; this problem seems to have been overlooked, although there are a number of excellent methods available for separating beryllium from aluminium. In view of this fact, and because it is highly probable, from mineralogical considerations, that titanium will be present in appreciable quantities in rocks containing small quantities of beryllium, an attempt has been made to devise a suitable separation of these two elements.

Most treatises on mineral analysis recommend that beryllium should be separated by the method of Parsons and Barnes (J. Amer. Chem. Soc., 1906, 28, 1589), which depends upon the total solubility of beryllium hydroxide, and the

total insolubility of ferric and aluminium hydroxides in boiling 10 per cent. sodium bicarbonate solution. In these circumstances, however, titanium is not completely precipitated. For example, a solution containing 0.0252 grm. of titanium chloride and 0.0248 grm. of beryllium chloride was treated according to the method of Parsons and Barnes; only 0.0219 grm. TiO2 was precipitated, the remainder being found with the beryllium. This is probably due to the formation of a soluble double alkali titanium carbonate (Auger, Compt. rend., 1923, 177, 1302), which is not wholly reprecipitated under the conditions of the experiment.

In other cases the titanium has been separated from, inter alia, beryllium, by precipitating the titanium either with hydrogen sulphide in boiling acid solution (e.g. Glaser, J. Amer. Chem. Soc., 1896, 18, 782), or by pouring into sodium hydroxide solution and boiling (Wenger and Wuhrmann, Ann. Chim. anal., 1919, 1, 337). Both methods are open to grave risk of contamination of the titanium precipitate with beryllium, and the latter method has the further disadvantage that titanium hydroxide is markedly soluble in the alkaline solution (Hillebrand, "Analysis of Silicate and Carbonate Rocks," p. 132). Nor are these objections removed if sodium peroxide is used in conjunction with the alkali (Noyes and Bray, "Qualitative Analysis of the Rare Elements," p. 165).

USE OF ORGANIC BASES AS REAGENTS.—After some preliminary search, it was decided that the most promising method of accomplishing a separation of titanium and beryllium seemed to be one involving the use of organic bases. The use of various organic bases for the precipitation of hydroxides of some of the rarer elements has been described by Jefferson (J. Amer. Chem. Soc., 1902, 24, 540), and Hartwell (id., 1903, 25, 1128). Hess and Campbell (id., 1899, 21, 776) introduced the use of phenylhydrazine for the quantitative precipitation of aluminium, and the scope of this reagent was widened by Allen (id., 1903, 25, 421), who worked out separations of aluminium, titanium, zirconium, and thorium from ferrous iron and beryllium, which are not precipitated by phenylhydrazine. It was found (see later) that phenylhydrazine was not suited to the separation of beryllium and titanium, chiefly because some of the beryllium tends to be precipitated with the titanium. It was thought that, if a base could be found, sufficiently weak to avoid causing any precipitation of the beryllium, whilst still strong enough to precipitate completely the very weak hydroxide of titanium, a successful separation might be worked out. After numerous trials such a reagent was found in p-chloroaniline, which possessed certain other advantages. A series of tests was carried out to ascertain how far the separation was complete, and for this purpose specially pure standard solutions of beryllium and titanium chlorides were prepared.

Separation by means of p-Chloroaniline.—Known volumes of standard titanium and beryllium chloride solutions were measured out into a beaker and the strongly acid mixture diluted to 250 ml. and heated nearly to boiling point. Ammonium hydroxide was then added cautiously with constant stirring until the solution acquired a turbid appearance, but was still distinctly acid to litmus.

The process of neutralisation, which is very critical, should not be carried to the point where a perceptible flocculation takes place. From 1 to 1.5 grm. of pchloroaniline were then added, the cover-glass replaced to avoid loss by spraying, and the solution carefully brought to boiling point and maintained at this temperature for three minutes. The solution was filtered, the precipitate washed with hot water until free from chloride, ignited and weighed. The filtrate from the titanium was heated, a slight excess of ammonium hydroxide added,* the solution boiled for a moment and filtered. The precipitate of beryllium hydroxide was washed with dilute, slightly ammoniacal ammonium nitrate solution until free from chlorides, ignited and weighed. Any deposit adhering to the sides of the beaker was dissolved in dilute nitric acid, precipitated with ammonia, and added to the main beryllium precipitate.

TABLE I.

	Amounts taken (in grm.)		Amounts found (in grm.)		Error (in grm.)	
No.	BeO.	TiO ₂ .	BeO.	TiO ₂ .	BeO.	TiO2.
1	0.0056	0.0020	0.0057	0.0019	+0.0001	-0.0001
2	0.0052	0.0471	0.0052	0.0473	nil	+0.0002
3	0.0052	0.0471	0.0051	0.0472	-0.0001	+0.0001
4	0.0052	0.0471	0.0050	0.0475	-0.0002	+0.0004
5	0.0052	0.0471	0.0051	0.0470	-0.0001	-0.0001
6	0.0212	0.0056	0.0212	0.0055	nil	-0.0001
7	0.0212	0.0056	0.0213	0.0055	+0.0001	-0.0001
8	0.0198	0.0107	0.0201	0.0106	+0.0003	-0.0001
9	0.0198	0.0107	0.0198	0.0107	nil	nil
10	0.0409	0.0056		0.0061	-	+0.0005
11	0.0409	0.0056		0.0060		+0.0004
12	0.0409	0.0056		0.0056		nil
13	0.0409	0.0056		0.0058		+0.0002
14	0.0409	0.0056		0.0058	•	+0.0002
15	0.0409	0.0056		0.0055		-0.0001

It is evident that, with quantities of beryllium and titanium of the order indicated in Table I., a second precipitation with p-chloroaniline is unnecessary. It should also be avoided, if possible, owing to the difficulty of redissolving in acid the precipitate of titanium after it has been boiled for three minutes. Moreover, no titanium could be detected by means of hydrogen peroxide in the beryllium residues.

If the separation of titanium and beryllium by means of p-chloroaniline is carried out in a solution containing a considerable quantity of sulphuric acid instead of hydrochloric acid, the same accuracy is not attained. The error is insignificant when only small quantities of beryllium are present, but if the beryllium content is increased it is found that the titanium figure is too high (Table II). Since precautions were taken to decompose by ignition with ammonium carbonate

^{*} At this point, if the correct acidity for the titanium separation had been attained, at least $1.5\,$ ml. $4\,$ N-ammonium hydroxide solution were required before the first appearance of a precipitate.

any sulphate that might have been present in the titanium precipitate, this increase in weight must be due to beryllium.

TABLE II.

	Amounts taken.			Amounts found.		Error.	
No.	BeO. Grm.	TiO ₂ . Grm.	Total. Grm.	BeO. Grm.	TiO ₂ Grm.	BeO. Grm.	TiO ₂ .
. 1	0.0058	0.0481	0.0539	0.0058	0.0482	nil	+0.0001
$\frac{2}{3}$	$0.0058 \\ 0.0483$	$0.0481 \\ 0.0502$	$0.0539 \\ 0.0985$	$0.0052 \\ 0.0463$	$0.0488 \\ 0.0518$	-0.0006 -0.0020	$+0.0007 \\ +0.0016$

Use of Phenylhydrazine Precipitant.—In order to compare the value of the two organic precipitants for this purpose, a series of tests was carried out as described above, but with the substitution of phenylhydrazine for p-chloroaniline. The results are tabulated in Table III.

TABLE III.

	Amounts taken.			Amounts found.		Error.	
No.	BeO. Orm.	TiO ₂ . Grm.	Total. Grm.	BeO. Grm.	TiO ₂ . Grm.	Beo. Grm.	TiO ₂ . Grm.
1	0.0058	0.0460.	0.0518	0.0050	0.0467	-0.0008	+0.0007
2	0.0058	0.0460	0.0518	0.0052	0.0465	-0.0006	+0.0005
3	0.0061	0.0471	0.0532	0.0056	0.0477	-0.0005	+0.0006
4	0.0061	0.0471	0.0532	0.0055	0.0478	-0.0006	+0.0007

Comparing these figures with the corresponding tests 2-5, Table I, with p-chloroaniline, it is seen that, when phenylhydrazine is used, the error involved is appreciable. This probably indicates that phenylhydrazine is a sufficiently strong base to precipitate some beryllium with the titanium. A second precipitation, which might eliminate this beryllium, has the drawbacks of unduly lengthening the time of analysis and the difficulty of redissolving the titanium precipitate already referred to. Further, even when freshly made, phenylhydrazine forms an appreciable amount of tarry matter which may occlude beryllium compounds that cannot then be washed out. This tar formation can be decreased by maintaining the solution at a lower temperature, but only at the sacrifice of further contamination of the titanium hydroxide. p-Chloroaniline is free from these disadvantages.

OBJECTIONS TO THE USE OF TANNIC ACID.—Moser and Singer (Monatsh., 1927, 48, 673) state that titanium can be quantitatively separated from beryllium by precipitation with tannic acid in the presence of acetic acid and large quantities of ammonium acetate and nitrate. Iron can be separated from beryllium in the same manner, but in this case a little hydrogen peroxide is added to the solution because the tannic acid always reduces some of the iron to the ferrous state. It is doubtful, however, if hydrogen peroxide could be employed in the presence of titanium. Moreover, when iron is present, the acidity of the solution has to be

reduced to such an extent as to favour co-precipitation of some of the beryllium with the iron (and titanium, if this be present). Since a small quantity of iron almost invariably accompanies the beryllium and titanium in the final stages of the method of analysis to be described, the use of tannic acid here is excluded for the reasons just mentioned. The use of cupferron (Lundell and Knowles, J. Amer. Chem. Soc., 1920, 42, 1439) in this connection is limited, because of the errors and loss of time introduced by the complete destruction of the excess reagent that is necessary before the small quantity of beryllium present can be determined in the filtrate from the titanium.

DETERMINATION OF SMALL AMOUNTS OF BERYLLIUM.—When it is of importance to make an exact determination of small amounts of beryllium in, for example, a silicate rock, the following procedure is suggested as an alternative to that usually followed in the treatment of the precipitate formed by ammonia in the presence of ammonium chloride. This precipitate should contain the iron, aluminium, titanium, beryllium and phosphorus, with small amounts of chromium, zirconium, vanadium, if present, and residual silica. The use of sodium carbonate in the fusion of the mixed oxides at this stage (Wenger and Wuhrmann, loc. cit., and Britton, ANALYST, 1922, 47, 50) has the advantage of (1) avoiding the introduction of the sulphate ion, (2) removing the residual silica in a form in which it can be readily estimated, and (3) eliminating phosphorus, which might be troublesome later. The subsequent treatment by sodium bicarbonate removes the iron, and much of the titanium, and the final separation by p-chloroaniline removes the remainder of the titanium, leaving the beryllium in solution. If present, chromium dissolves in the extract of the sodium carbonate melt together with the aluminium; vanadium, which would behave similarly, is not likely to be present in rocks containing beryllium. Traces of zirconium which may dissolve in the sodium bicarbonate solution are completely precipitated by p-chloroaniline. If much iron is present in the original mixed oxides, traces may escape the double sodium bicarbonate precipitation, but these also are completely precipitated by ϕ -chloroaniline.

Method.—The hydroxides of iron, aluminium, etc., are separated from the solution of chlorides by two precipitations of the boiling solution with ammonia, followed by an evaporation of the ammoniacal filtrate to recover traces of these metals which have escaped precipitation. The united precipitates are placed in the weighed platinum crucible containing the residue from the silica determination, dried, ignited and heated until of constant weight. The contents of the crucible are ground with a platinum rod and intimately mixed with 5 grms. of sodium carbonate (prepared by heating pure sodium hydrogen carbonate), a thin layer of carbonate being spread on the top. The crucible is then heated for $2\frac{1}{2}$ —3 hours at a temperature just high enough to keep the contents molten. The cooled melt is left to digest overnight in 500 ml. water, passed through a fine filter and washed with dilute sodium carbonate solution. If it is intended to estimate the aluminium gravimetrically, the fusion process is now repeated.

If chromium is present, it can be estimated colorimetrically in the filtrate after the bulk of the solution has been somewhat reduced by evaporation. The solution is then carefully acidified with 15 ml. of hydrochloric acid, and evaporated to dryness in a platinum basin to render the small amount of silica present insoluble. The basin is drenched with a few ml. of hydrochloric acid, 100 ml. hot water added and the solution filtered. The precipitate is ignited and weighed in a platinum crucible and the weight of silica determined by the loss in weight after evaporation with hydrofluoric and sulphuric acids. If desired, aluminium can be determined in the filtrate by a double precipitation with ammonia; correction must be made for the chromium and phosphorus which are also precipitated.

Ten ml. of hydrochloric acid are poured into the crucible in which the sodium carbonate fusion took place, in order to extract any adhering matter, and the extract is used to dissolve the precipitate of iron, titanium, and beryllium on the filter paper. The filter paper is ashed and any residue formed dissolved in hydrochloric acid and added to the main solution. The solution is now treated according to Parsons' and Barnes' process (loc. cit.). The solution is neutralised with ammonia and 10 grms. of solid sodium bicarbonate (free from sodium carbonate) per 100 ml. of solution are added to the cold solution. The beaker is covered with a clock-glass and the solution heated to boiling as quickly as possible and maintained at boiling point for one minute. The solution is quickly cooled and filtered. The residue is washed with 50 ml. of hot 10 per cent. sodium bicarbonate solution, redissolved in hydrochloric acid, neutralised as before, and the precipitation with sodium bicarbonate repeated. The final precipitate of ferric and titanium hydroxides is ignited in the platinum crucible which was used for the sodium carbonate fusion.

The united filtrates from the precipitations by sodium bicarbonate are carefully acidified with 30 ml. hydrochloric acid, and the solution boiled to expel carbon dioxide. In view of the large amount of sodium salt present, a preliminary separation of the beryllium and titanium hydroxides from the solution is advisable. The precipitate is dissolved in hydrochloric acid and the treatment with p-chloroaniline as described on page 270 is applied. The precipitate from the p-chloroaniline is redissolved in hydrochloric acid and the precipitation process is repeated. The beryllium is determined in the united filtrates by precipitation with ammonia, and the precipitate, after ignition and weighing, is tested for contamination with iron or titanium.

The precipitate produced by the p-chloroaniline is added to the platinum crucible containing the bulk of the iron and titanium and ignited. The contents of the crucible are brought into solution by fusion with potassium pyrosulphate and subsequent leaching with dilute sulphuric acid. In this solution the titanium and iron can be estimated by suitable methods, for example, the titanium colorimetrically and the iron by means of titanous chloride. The crucible is weighed empty, and dissolved platinum is separated from the solution by means of sulphuretted hydrogen, in order to arrive at the correct total weight of oxides precipitated by ammonia. The weight of alumina, if it has not been directly

determined, is found by subtracting from the total the weights of all other oxides.

Table IV shows the results obtained in the determination of beryllium by the method just described in mixtures of known amounts of iron, aluminium, titanium, and beryllium chlorides. In the case of numbers 3 and 4 the precipitate from the p-chloroaniline was not subjected to a second precipitation by that base. With these two exceptions it is seen that the results are in excellent agreement with the theoretical. Examination of the beryllium residues showed them to be free in every case from titanium and iron.

TABLE IV.

•	Amount	ts taken.		Found	
Fe ₂ O ₃ . Grm.	Al ₂ O ₃ . Grm.	TiO ₂ . Grm.	BeO. Grm.	BeO. Grm.	Error. Grm.
0.0270	0.0258	0.0248	0.0250	0.0248	0.0002
,,	,,	,,	,,	$0.0254 \\ 0.0243$	+0.0004 -0.0007
,,	,,	,,	,,	0.0233	-0.0017
		,,	,,	$0.0252 \\ 0.0249$	+0.0002 -0.0001
	Grm. 0·0270	Fe ₂ O ₃ . Al ₂ O ₃ . Grm. Grm. 0·0270 0·0258 '' '' 0·1082 0·1032	Grm. Grm. Grm. 0·0270 0·0258 0·0248	$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$

SUMMARY.—(1) It is suggested that the chief obstacle to the accurate determination of small quantities of beryllium in silicate rocks lies in the difficulty of its separation from titanium.

- (2) A method is described for the separation of beryllium and titanium by means of p-chloroaniline.
- (3) Details are given of a method for the analysis of a silicate rock with especial reference to the accurate estimation of beryllium in small quantities.

The author desires to express his thanks to Sir Robert Robertson for permission to publish this paper, and also to Dr. J. J. Fox for his valuable criticism.

GOVERNMENT LABORATORY, LONDON, W.C.2.

New Apparatus for Electrolytic Analysis.

By HENRY J. S. SAND, D.Sc., Ph.D., F.I.C.

THE apparatus to be described is based, in general purpose and design, on that used by me in 1907 (J. Chem. Soc., 1907, 91, 374), and in later years for the deposition and separation of metals by electrolysis. The apparatus has been modified by several users of the methods described by me and of similar ones (Fischer, Z. Elektrochem., 1907, 13, 469; and Elektroanalytische Schnellmethoden, Stuttgart, 1926; Lassieur, Electroanalyse Rapide, Paris, 1927). The subsequent designs have been superior in economy of platinum, but I believe have been inferior in ease

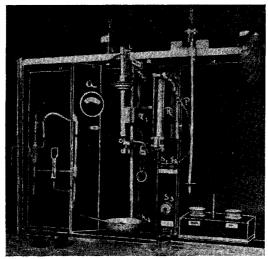


Fig. 1.

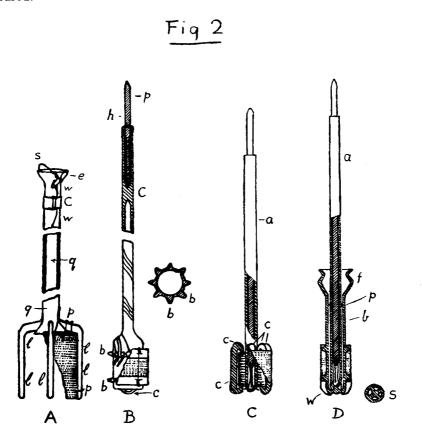
and rapidity of working. In 1911 (Trans. Faraday Soc., 1911, 6, 205) I also described apparatus for the deposition of certain metals, in which the anode was mounted on glass and the cathode constructed of metals such as silver and nickel. The designs of electrodes now submitted are developments of these, having as their main object a reduction in the weight of platinum required. The stand and other auxiliary apparatus have also been completely redesigned in detail, but the following distinctive features, to which importance is attached, are either the same as, or developments of, those to be found in the old design.

The outer electrode surrounds the inner everywhere, except below, being placed as close as possible to the bottom of the beaker. This is of importance when it is desired to confine the lines of flow of the current to the space between the electrodes for the purpose of controlling the potential of the outer. The stem of the

inner electrode revolves inside a quartz glass guide tube which forms an integral part of the outer, thus ensuring correct alignment, even when the clearance between the two electrodes is reduced to a minimum. The process of washing and disconnecting is made as rapid as possible by the following features. The support for the beaker can be removed without interfering with the position or connections of the electrodes, and the outer may be washed with a spray of water while the beaker is being lowered. Disconnection is carried out by loosening a clamp which holds the outer electrode and simply pulling the inner out of a specially designed clutch which forms part of a flexible rubber spindle. The two electrodes may be removed together without a short-circuit between them, if only the inner one is held by its stem, and they are suitable for washing by immersion in alcohol or other drying liquids contained in jars. Since the quartz guide tube now forms part of the outer electrode, a fixture must be provided for drying it with hot air. This is illustrated in Figure 5B and also in Figure 1, and consists of a quartz drying tube which swings in a brass fork f attached to the back of the board a (Fig. 1). electrode is placed on the end a, while the portion b is heated by means of a Bunsen flame, air being blown at the same time through the end c by means of a hand spray bellows. A preliminary drying of the gauze of the electrode thus also takes place, which is then completed by holding the burner under the electrode itself at some distance from it.

THE ELECTRODES.—Both electrodes are usually mounted on quartz glass frames, but ordinary glass may often be used for the inner one, when it has not to be weighed. In some cases a frame constructed of rubber and glass has likewise been found useful. A rotating anode is also described which is designed to be used in conjunction with a revolving partition consisting of a parchment paper thimble. This is a development of the anode described by me (J. Chem. Soc., 1908, 93, 1589). The electrodes are shown in Figure 2, A representing the outer, B the inner mounted on a quartz glass frame, C the same mounted on a rubber plus glass frame, whereas D represents the revolving anode designed for use with a parchment diaphragm. The frame of the outer electrode is built up on a quartz glass tube q, of about 7 mm. bore, which is flared at both ends; about 2 mm. from the lower end are sealed four legs l, of which three are shown. These are made from 4 mm. rod, the horizontal portions being flattened to give additional strength. The top of the tube q is provided with two eyelets e, which serve as supports for a suspension wire s, and also provide a means for holding the leading-in wire in position. The platinum gauze jacket, which is shown partly cut away, is provided with a small slit at the top to allow it to be drawn over the flange, left at the bottom of the flared quartz tube, and is then fastened firmly to the frame by means of thin platinum wire. In addition, clips of thin platinum wire ϕ , twisted together over the legs of the frame, hold the jacket firmly in position. A leading-in wire w, of 0.4 to 0.6 mm. diameter, connects the gauze jacket in a steep spiral with the collar c, and is held in position by the eyelet e. The collar, which is designed to be held by a clamp, is made of thin foil or gauze, the ends being folded together as shown.

If from one cause or another one of the legs of this electrode should become fractured, it is a simple matter to remove the platinum and repair the frame in the oxy-gas blowpipe. For this purpose a small stand should be improvised. It should be built up on a suitable base to have a central rod for holding the tube, and outside this a block of wood covered with uralite, having a hole of the correct size to take the leg. By successively adjusting all the legs to fit into this hole, while the tube is supported by the central rod, a true position for all the legs may be ensured.



The frame of the inner electrode B is a closed pipette-shaped vessel provided with two rows of eight beads, b, on the body of the frame (shown also in section). The stem is a tube which is closed at C, its diameter being about 1.5 mm. smaller than the bore of the tube q of the outer electrode, in which it is designed to revolve. About three cm. of the stem are left open at the top, to provide a seating for the leading-in peg p of the electrode, made of non-corrodible metal, preferably of silver. The latter is partly cut away to provide space for it to be cemented to the tube, ridges equal to the bore of the tube being left to ensure a central position.

It has been found that if lead (grain) is melted in the tube, heated to dull red heat, and the peg, likewise heated to dull red heat, is then introduced, a perfectly satisfactory joint is made on cooling. The peg has a hole of about 1 mm. diameter immediately above the quartz tube. The connection between the platinum gauze jacket of the electrode and the peg is made by 0.3 mm. platinum wire, which is wound upwards on the exterior of the stem in four steep spirals. For this purpose the wire is first wound round the body of the frame below the top row of beads, two short branch wires from it having been previously provided by twisting it on itself for about 1 cm. From the loop formed below the beads the wire is taken past one of the beads in a steep spiral to the top of the stem. It is then threaded through the hole h in the peg, and taken down again in a similar parallel spiral. At the foot it is taken past one of the beads and twisted to the free end of the loop or to a branch wire, then taken spirally again to the top, and back as before, being finally secured to one of the branch wires. In order to ensure good contact between the platinum wire and the silver peg, a small silver wire wedge is driven into each end of the hole h. The jacket consists of a piece of gauze which is strengthened by means of foil at the top and bottom edges, where it is to be supported on the beads. The other edges are finished off in such a way as to allow the jacket to be pulled tightly over the beads as a cylinder by means of pieces of platinum wire. The jacket is also provided with two wires which are brought into contact with the leading-in wires, by being twisted to the free branches mentioned above. The electrode can be hung from a balance by means of the quartz glass loop.

When the electrode is made of ordinary glass the construction is somewhat modified. The leading-in rod, usually of aluminium, is taken down the whole of the stem into the body of the frame, and is cemented in position with a red lead cement. Four platinum wires are fused into the aluminium and sealed through the body of the frame, which resembles that described for quartz. Also, for glass it was sometimes found useful to take a single wire spirally up the outside of the stem to prevent it from being chafed by contact with the guide tube of the outer electrode.

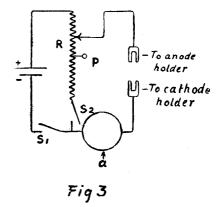
The electrode C was found very useful as an anode for zinc and similar determinations. It is built up on a glass tube into which an aluminium rod carrying four platinum wires is cemented. These wires are taken through the glass tube at the bottom, as shown. This is most simply accomplished by cutting the tube at the place where the wires leave it, placing these in position, and then sealing on the cut portion again, drawing off, and finally closing the tube at the place shown. Over the glass tube is slipped a piece of thick-walled rubber tubing or a rubber plug, made of black rubber (i.e. free from metal oxides). This serves as a support for eight hooks, c, made of glass rod. On these is mounted the jacket of platinum gauze, which is fitted with short wires that are connected by twisting to the leading-in wires proceeding from the aluminium rod. This electrode, which can be easily constructed in the laboratory, is very resistant to fracture owing to its resilience, and can, if necessary, be readily repaired. The weight of platinum

on the electrode A is about 7 grms., that on the electrodes B and C about 3 or 4 grms.

The electrode D is intended for use as an anode when the electrolyte to be analysed must not come in contact with it, so that oxidation may be avoided. For this purpose it is designed to hold a parchment thimble diaphragm, which revolves with it and holds an auxiliary electrolyte, say, of dilute acid or a salt. The thimble, of 16 mm. diameter, is cut to the correct length and provided with two slots at the top, slipped over the electrode after soaking in water and fastened to the funnel-shaped portion by means of thread. Since a small amount of the ions to be estimated will always diffuse through the parchment, the electrode is so designed that the whole of the liquid contained in the anode chamber formed by it may be forced into the cathode chamber towards the end of a determination, and be displaced by fresh liquid. For this purpose the electrode is constructed of an inner tube, a, with aluminium rod and four platinum leading-in wires similar to that used in electrode C. To the bottom of this tube is fused a protrusion carrying four arms to which the outer tube b, with a funnel top, is sealed. The bottom of the electrode thus represents the appearance shown by s. The tube a has four beads p, of such a size as to keep the tube b in position and thus strengthen the electrode, whereas the tube b carries two rows of eight beads for holding the platinum jacket j, the construction being similar to that described for electrode B. The leading-in wires are taken through the bottom of the electrode and twisted to short wires welded to the platinum jacket. The cathode used with this anode must be open at the top, the gauze usually rising above the level of the electrolyte. It will be seen that when fresh electrolyte is poured into the funnel f towards the end of a determination, the original electrolyte is forced down the annular space between a and b, and finally pushed upwards and into the cathode chamber through

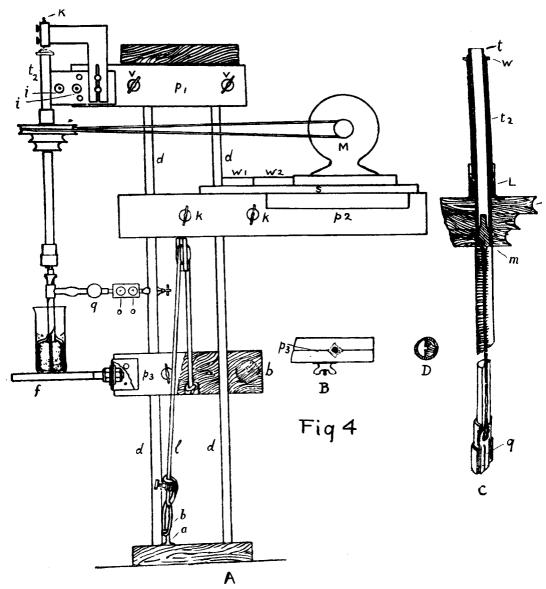
the slits at the top of the parchment. This electrode has been found to give good results in the rapid separation of copper from sulphate solutions containing large amounts of iron salts. It is obvious that good results can only be obtained in such solutions if the formation of ferric salts, which would dissolve the copper, is inhibited.

THE STAND AND ELECTRICAL CONNECTIONS.—Fig. 1 is a photograph of a complete stand, being one of four which are permanently set up for use on the same base. Fig. 3 is a diagram of the electrical connections, the same letters being used in these



figures to represent corresponding parts. The figures are self-explanatory, but the following remarks may be made. The resistance R shown is of 2.7 ohms and 14 ampères carrying capacity, and is arranged so that by alteration of the switch s_2 it may be used either in series or in shunt with the electrolytic

apparatus. Further, the latter is so arranged that by the interchange of two connections either the outer or the inner electrode may be made the cathode. The terminal P is fitted to allow the apparatus to be used for potentiometric



titrations according to the method of Roberts (J. Amer. Chem. Soc., 1919, 41, 1358). A scale is fitted to the rheostat for convenience during potentiometric titrations.

Figs. 4A to 4D represent the central portion of the stand, giving details of

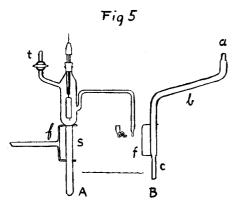
the stirring arrangement and the supports for beaker and electrodes. Two rods, d, d, of rustless steel, are fitted at each end to the wooden stand and serve as supports for the wooden crossbars, p_1 , p_2 , p_3 . Of these, p_1 and p_2 are screwed firmly to the rods, whereas p_3 slides readily on them, being provided with a hole of quadratic section which is lubricated with vaseline (see Fig. 4B). The motor is represented by M. It is controlled by the switch s_3 (Fig. 1), and a regulating resistance of 590 ohms and 0.7 amp, carrying capacity which is shunted across the 200 volt mains, the motor being connected with one end and the slider. The resistance is not visible in Fig. 1, being fitted to the back of the board holding the motor switch The position of the motor and hence the tension on the pulley is controlled by two wedges, w_1 , w_2 , or else by screws. The clutch holding the inner revolving electrode and the connection with it are shown in detail in Fig. 4C. A steel tube t_1 is suspended by means of a washer w inside a brass tube t_2 , which acts as a bearing to it. The steel tube also holds the oil trap l and the pulley p. It is closed at the bottom by a steel plug, m, of the shape shown, which is screwed and cemented into it. Electrical connection between m and the stationary terminals i i (Fig. 4A) is made by means of mercury on which a drop of oil floats, and the steel wire k. The further flexible connection to the inner electrode is constructed of a strand of flexible copper wire, let into the plug m, and a rubber tube of 3/32 in. bore. The clutch is novel and requires detailed description. A segment of the rubber tube is removed by a razor at the bottom. It has a section of the relative size apparent from the blank portion shown in the cross section in Fig. 4D, and a length in the direction of the tube, of about one inch. The piece of rubber thus removed is replaced by one of metal of similar shape, preferably of silver, which is soldered or otherwise fastened to the lower end of the strand of flexible wire. Over the bottom of the rubber tube and the metal segment a tightly fitting rubber tube jacket is pushed to a height of about two inches. This rubber tube is turned on itself at the bottom, or else another piece of its own diameter of about one inch length is pulled over it, in order to produce some pressure on the metal segment. The latter projects about one-sixteenth inch below the bottom and has a V-groove filed into it. The rubber tube is cut so that about seven-eighths of the original interior is left at the bottom, and this is lubricated with common chalk. be seen that the whole arrangement forms a very simple clutch for the inner electrode, which is connected or disconnected by pushing its stem into, or pulling it out of, the rubber tube.

The clamp q holds the outer electrode. As in previous designs, the V-shaped jaw offers a metallic contact and may, if desired, be coated with silver foil, cork being left on the opposite jaw. The clamp is provided with two terminals, o, o, for connection with the source of current and the potentiometer.

The ring f for holding the beaker is fastened to the sliding bar p_3 , which has already been referred to; b is a counterpoise of lead. This bar is controlled by the leather pulley cord l. The brass ring b engages in the dresser hook a, which is cut off short, so that b may be removed from it without raising the beaker. The length of the cord l is made adjustable by the slip-knot arrangement provided at the

bottom, the loop of the slip-knot being held in position, as shown, by a screw clip fastened to the cord.

THE AUXILIARY ELECTRODE VESSEL.—In the auxiliary electrode formerly used, connection with the liquid under examination was made through a film of



electrolyte held round the barrel of a closed tap. This introduces a considerable resistance of uncertain magnitude, and is sometimes objectionable. It has been recently criticised by T. B. Smith (Trans. Faraday Soc., 1928, 24, 216), who describes a number of new designs. For some time past I have reverted to the form shown in Fig. 5A, which illustrates the vessel fitted up as a quinhydrone electrode. A novel feature claimed for this electrode is the manner in which it is held, a matter which is by no means of negligible interest. A hollow stem s is sealed to the bottom of

the vessel, and this is dropped through two holes provided in the prongs of the fork f, which is adjustably fitted to the stand. The electrode and its support are also visible in Fig. 1. Connection with a reservoir bottle placed on the top of the stand is made through a piece of thick-walled rubber tubing via the tap t.

SIR JOHN CASS TECHNICAL INSTITUTE, E.C.3.

A New Test for Boric Acid and Borates.

By A. SCOTT DODD, B.Sc., F.I.C., F.R.S.E.

(Read at the Meeting, February 6, 1929.)

VARIOUS methods have been suggested for the detection of boric acid. Of these, the best known is the turmeric test with its modifications. Probably the most delicate test is that with tincture of mimosa flowers (L. Robin, ANALYST, 1904, 29, 330), while for larger quantities the presence of boric acid is indicated by a green flame produced on igniting the substance with methyl alcohol or ethyl alcohol and glycerin.

In a previous publication (ANALYST, 1929, 19) the author mentioned the pink coloration produced by the addition of mannitol and Sofnol Indicator No. I as characteristic of boric acid. Since then further investigations have been carried out to ascertain the soundness of the test, and to determine how it might be adapted for wider application.

As a test for borates in mixtures containing salts of various metals and borates, it was found to work very satisfactorily. Phosphates, arsenates, chromates, and tungstates appeared to be the only substances, which caused any interference with the distinctness of the reaction. The details of the test are as follows:

Place about 10 c.c. of the unknown substance in acid aqueous solution in a test tube. Add several drops of a solution of methyl red or Sofnol Indicator No. 1 and neutralise with caustic soda solution. Boil and filter into another test tube, if necessary; cool, add a drop or two of dilute sulphuric acid until the liquid is distinctly acid, and neutralise by dropping in $0.1\ N$ sodium hydroxide solution until the pink colour just disappears. Add about $0.5\ \mathrm{grm}$ of mannitol and shake the mixture. A distinct reddish-pink coloration is given if borates are present. The reaction is probably as expressed by the following equations:

$$\begin{split} \text{Na}_2 \text{B}_4 \text{O}_7 + 3 \text{H}_2 \text{O} &= 2 \text{NaOH} + 4 \text{HBO}_2 \\ (2 \text{NaOH} + \text{H}_2 \text{SO}_4 &= \text{Na}_2 \text{SO}_4 + \text{H}_2 \text{O}) \\ \text{HBO}_2 + \text{C}_6 \text{H}_{14} \text{O}_6 &= \text{C}_6 \text{H}_{12} \text{O}_6 \text{BO}^{\prime} + \text{H}^{\bullet} + \text{H}_2 \text{O}. \end{split}$$

The mannitol and boric acid complex is much more highly ionised than metaboric acid, and renders the solution distinctly acid and gives the characteristic reddish colour with the indicator employed.

It was found that the presence of small quantities of carbonic acid did not cause any appreciable interference with the test if 0.01 grm. of boric acid was present. It has been stated in some text-books that the reaction given with turmeric paper requires to be considered with caution, as "acid solutions of zirconic, titanic, tantalic, niobic and molybdic acids also colour turmeric paper brown," and may therefore be mistaken for boric acid. These acids, however, are not usually likely to be found. The author made careful comparisons of the reactions of two of the most common, namely, titanic and molybdic acids, with those of boric acid.

It was found that, when using turmeric either in solution or on paper, confusion is likely to arise only when the quantity of these acids is large. With small quantities of the acids the stain on turmeric paper is merely brown in the case of molybdic acid, and dull reddish brown in the case of titanic acid, whilst the stain given by boric acid is a bright rose pink. Further, when these stains are touched with a solution of caustic soda, the boric acid stain only is turned green.

The mannitol and Sofnol No. 1 test was also tried with molybdic acid and titanic acid in the following manner:

A solution containing about 0.015 grm. of molybdic acid (MoO₃) and nitric acid was placed in a titrating basin, together with 2 drops of a solution of Sofnol Indicator No. 1, and neutralised with 0.1~N NaOH. When 0.5 grm. of mannitol was added no pink coloration was given, but when 3 c.c. 0.1~N boric acid solution, or 0.0186 grm. of boric acid was added, the solution became very distinctly bright pink. This solution was then titrated after addition of phenolphthalein, and was found to require 3.00 c.c. of 0.1~N sodium hydroxide solution, thus showing

that the presence of molybdic acid does not interfere with either the reaction or the determination of boric acid.

A similar test was tried with titanous chloride. As, however, this removed the colour from the Sofnol Indicator No. 1, even when acid was present, the following treatment was employed. A solution containing about 0.015 grm. of titanous chloride was placed in a titrating basin, and excess of caustic soda solution run in until a precipitate was formed. Then N sulphuric acid was added, drop by drop, until the solution was slightly, but distinctly, acid to litmus paper. The solution was boiled for 5 minutes, cooled and neutralised, two drops of Sofnol Indicator No. 1 being used. The results obtained were exactly similar to those given with molybdic acid, and showed also that the presence of titanic acid does not interfere with either the reaction or the determination of boric acid.

Solutions of titanous chloride and molybdic acid were also found to give negative reactions with mannitol and methyl red solution. Therefore, when dealing with mixtures containing large quantities of salts the mannitol and methyl red test appears to be at least as characteristic of boric acid as the turmeric test, and in some instances less liable to lead to erroneous conclusions.

The following substances were tested and were found to give negative results with the mannitol and methyl red test, and also did not interfere with the boric acid reaction when present along with borates:

Metallic Radicals.—Aluminium, ammonium, antimony, barium, bismuth, cadmium, calcium, cobalt, copper, iron, lead, lithium, magnesium, manganese, mercury, molybdenum, nickel, potassium, silver, sodium, strontium, tin, titanium, and zinc.

Acid Radicals.—Acetates, benzoates, bromates, bromides, chlorates, chlorides, citrates, formates, iodates, iodides, lactates, molybdates, nitrates, nitrites, oxalates, salicylates, sulphates, sulphides, sulphites, tartrates, and tannates.

In the case of arsenates, phosphates, chromates, and tungstates difficulty was experienced in ascertaining the exact point of neutrality, as the change in colour from red to yellow was not at all sharp. Tungstates differed from all the other substances examined, and gave a distinct reddish pink colour similar to that given by boric acid. This shows that tungstic acid resembles boric acid in that it also forms a complex with mannitol. The reaction, however, is much slower than in the case of boric acid and mannitol, the reddish pink colour reaching its maximum depth only after being allowed to stand for several minutes.

Sensitiveness of the Mannitol and Methyl Red Test.—The sensitiveness of this test is not quite so great as that with turmeric paper. The latter is stated (Treadwell and Hall, Vol. I, p. 359) to give a visible reaction with 0.002 mgrm. of B_2O_3 , whereas this test was found to give a distinctly visible reaction with 0.2 mgrm., and a very distinct reaction with 0.3 mgrm. of boric acid (H_3BO_3) in 10 c.c.

The sensitiveness is increased by concentration, and 0.004 mgrm. of boric acid in 2 drops of liquid was found to give a visible reaction.

Glycerin or invert sugar may be used in place of mannitol, but the reaction is not quite so distinct with small quantities of boric acid. Glycerin was found to give a visible reaction with 0.3 mgrm. of boric acid in 10 c.c. of solution.

LABORATORY OF CITY ANALYST, EDINBURGH.

DISCUSSION.

The President said that this contribution arose out of another paper, recently read, which gave rise to much discussion. Unfortunately, the interfering substance was phosphate, which was generally present when one was troubled with boric acid.

Dr. B. S. Evans said that he would like to suggest as the method of neutralisation of the liquid in the case of phosphates the method which he always used, namely, the iodide and iodate method, which did not depend on any colour reaction at all, and which he had always found to be satisfactory. He would like to know whether Mr. Dodd's method had ever been applied to the detection of tungsten.

Dr. Cox, in the absence of the author, said that the reaction was certainly more sensitive than the ordinary test. Even in the presence of phosphates the colour was characteristic. He could not, of course, answer Dr. Evans's question with regard to tungsten.

Official Appointments.

THE Minister of Health has confirmed the following appointments:—

- Dr. H. E. Cox, M.Sc., F.I.C., as Public Analyst for the County of Cornwall (March 12th, 1929).
- Mr. STANLEY DIXON, M.Sc., F.I.C., as Public Analyst for the County Borough of Cardiff (April 10th, 1929).
- Mr. S. E. Melling, F.I.C., as Public Analyst for the Borough of Accrington (formerly Joint Public Analyst) (April 16th, 1929).
- Mr. J. H. Sugden, M.Sc., F.I.C., as Additional Public Analyst for the County Borough of Cardiff (April 19th, 1929).
- Mr. A. Lerrigo, B.Sc., F.I.C., as Acting Public Analyst for the City of Birmingham (April 21st, 1929).
- Mr. H. H. BAGNALL, B.Sc., F.I.C., as Public Analyst for the City of Birmingham (to date from July 1st, 1929).

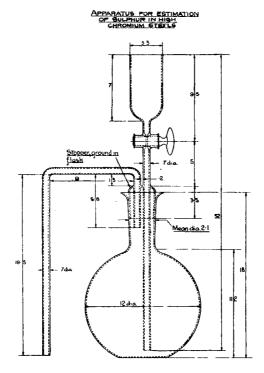
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Notes.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

A RAPID METHOD FOR DISSOLVING HIGH CHROMIUM STEELS FOR THE DETERMINATION OF SULPHUR.*

In the usual method for the gravimetric determination of sulphur in steel the sample is dissolved in aqua regia, which converts the sulphides present into sulphates; solution of an ordinary carbon steel is prompt, not to say violent. In the case of a high chromium steel, however, more especially of a stainless steel, the attack is very much less vigorous, and even when the metallic particles have



disappeared there usually remains a black sludge, presumably chromium carbide, on the bottom of the beaker, which often takes many hours' digestion on the hot plate to dispel it completely. The trouble appears to be bound up with the passivity induced by nitric acid, and the apparatus shown in the accompanying figure was devised in order to permit of the sample being dissolved first in hydrochloric acid alone, and afterwards oxidised with nitric acid.

^{*} Communication from the Research Department, Woolwich.

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The apparatus consists of a flask (cap. 700 c.c.) with a ground-in hollow stopper carrying a tapped funnel, the stem of which passes down to the bottom of the flask, and a leading tube whose short arm ends just below the stopper and whose long arm ends just above the level of the bottom of the flask.

Process.—Five grms. of the sample are weighed into the flask, the stopper is inserted, and 25 c.c. of water are run in through the tapped funnel. The outer end of the leading tube is placed dipping to the bottom of a cylinder containing 35 c.c. of concentrated nitric acid, and 25 c.c. of concentrated hydrochloric acid are run into the flask cautiously through the funnel, the tap being then closed. The apparatus is allowed to stand until the evolution of gas slackens somewhat, after which it is placed on a double asbestos pad on the plate, the end of the delivery tube being kept dipped to the bottom of the cylinder containing the nitric acid the whole time. When the evolution of gas, which is at first increased, finally becomes quite slow, owing to the sample being nearly all dissolved, the tap of the funnel is opened to obviate the danger of the nitric acid being sucked back owing to accidental cooling by draughts. When evolution of gas has entirely ceased, the apparatus is removed from the plate, the tap being left open and the cylinder in position, and allowed to cool completely; when quite cold the tap is closed, and the top of the flask is held under a stream of hot water for a few seconds. care being taken that the sudden expansion of the air does not fling any drops of nitric acid out of the cylinder. The flask is then immediately held under the cold tap, so that the sudden contraction of the air shall draw all the nitric acid back into the flask; the points to be aimed at are:—

- (a) As far as possible, heating only the air in the flask and not the liquid.
- (b) Drawing the nitric acid back so quickly that it has not time to react with the ferrous salts before the cylinder is empty.

A violent reaction due to the oxidation of the ferrous salts and the carbon compounds, ensues; when this is over the cylinder and leading tube are rinsed into the flask by running about 20 c.c. of water into the former and allowing it to suck back in the same way as was done for the nitric acid, this operation being repeated two or three times. Finally, the contents of the flask are poured into a beaker and the flask, funnel and stopper thoroughly rinsed in; 5 c.c. of 20 per cent. potassium nitrate solution are added, the solution evaporated to dryness, and the sulphur determined in the ordinary way.

The following results were obtained with Messrs. Ridsdale's British Chemical Standard Steels:

Standard.	Mean result on certificate.	Result obtained.
N.	0.034	0.034
0.1	0.032	0.031
R.	0.052	0.049
A.2.	0.020	0.020
H.	0.047	0.043
W.	0.075	0.071

B. S. Evans.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

NON-ALCOHOLIC PRODUCTS SOLD AS GINGER BRANDY, AND ORANGE AND QUININE WINE.

On March 6, a firm was summoned at Old Street Police Court, London, for selling ginger brandy which contained no brandy, and for selling orange and quinine

wine deficient in quinine to the extent of 95 per cent.

Mr. W. G. Jenkins, prosecuting, said that the proceedings were taken under Sec. 2 of the new Food and Drugs (Adulteration) Act, 1928. The inspector had taken samples from men who were selling bottles of the preparations from door to door in Old Ford Road. On analysis the ginger brandy was found to contain no alcohol.

The solicitor for the defence said that the firm manufactured non-alcoholic wines, and that everyone in the district knew that alcoholic wines could not be sold without a licence. The label on the bottle was "Ginger Brandy (flavour), superior Non-Alcoholic." It was perfectly clear that it was not intended to have any brandy in it, and it was well known that 1s. 9d. (the price paid) was not the price of real ginger brandy.

With respect to the second summons, evidence was given that this was labelled

"Orange Quinine Wine," and was being sold at 1s. 9d. per bottle.

Dr. F. L. Keith, Medical Officer for Bethnal Green, said that there was only a trace of quinine in the bottle. In cross-examination he agreed that there was an orange wine and a quinine wine in the British Pharmacopoeia, but not an orange and quinine wine.

Mr. A. È. Parkes, Public Analyst for Bethnal Green, gave evidence to the effect that the sample analysed by him was a solution of sugar containing a mere

trace of quinine, and was devoid of alcohol.

For the defence it was stated that the article was sold as a beverage rather than as a medicine. The amount of quinine was not equivalent to that required by the British Pharmacopoeia, but they were now using a greater proportion of quinine.

The Magistrate (Mr. Snell) dismissed the first summons. With regard to the second summons, he said that it was clear that in that particular bottle there was not anything like the quantity of quinine a person might expect to get. He therefore inflicted a penalty of £5 with £2 10s. costs, and on the first summons the Borough Council would have to pay £2 10s. costs.

LABELLING OF BUTTER CREAM SANDWICHES.

A GROCER was summoned on March 14, at Lodon, Norfolk, for selling, to the prejudice of the purchaser, butter cream sandwiches not of the nature, substance, and quality demanded. On analysis, the sandwiches were found to contain "foreign fat, other than butter fat, to the extent of 100 per cent."

The defendant relied upon a warranty from a London firm, whose explanation was that what they meant to imply by the label was that the sandwiches had a butter flavour. The firm now recognised that the label was apt to mislead the public, and gave an undertaking to revise the description of these particular goods.

In these circumstances the Bench decided to dismiss the case against the defendant under the Probation of Offenders Act, on payment of £3 19s. costs.

New Zealand.

SIXTY-FIRST ANNUAL REPORT OF THE DOMINION LABORATORY.

In his Report for the year 1927 the Dominion Analyst (Dr. J. S. Maclaurin) states that the total number of samples examined was 5086, of which 3420 were for the Public Health Department, 334 for the Customs, 666 for the Mines Department, and 24 for the Police. In addition, 2330 samples were analysed at the Auckland Branch Laboratory, and 2058 at the Christchurch Branch Laboratory.

MILK.—The number of samples taken in Wellington was 1708. Of these, 6 were watered, 8 had been skimmed, 2 were decidedly stale, and 15 were deficient in various ways. The use of an ice-chest by the inspector when taking samples in the summer months has proved effective in checking the sale of stale milk.

The milk samples (1607) examined at the Auckland Laboratory showed a considerable reduction in the amount of added water, and the cleanliness of the

milk showed definite improvement as the result of rigid inspection.

The number of samples examined at the Christchurch Laboratory (1324) was much more satisfactory than in the previous year, but in view of the fact that there are probably 500 to 600 milk vendors in the city, more samples should be taken, so as to ensure that every vendor's milk will be sampled at least four or five times a year. The percentage of samples adulterated was 9.6, as compared with 10.6 for 1926. At the present time the City Council is making efforts towards municipalising the city milk supply.

Salt in Beer.—In the early part of the year several samples contained more than the permissible amount of salt (50 grains per gallon), but later none of them exceeded the standard. Proceedings were taken against one brewer as the result of analyses in the Auckland Laboratory.

LIME WATER.—More than a third of the 49 samples examined were deficient in strength, and one contained lead. Of the 38 samples examined at the Christchurch Laboratory, nearly half were seriously deficient in lime, and a few had been made up with dirty water. In one or two cases common salt had been added. Sixty-five samples were examined at the Dunedin Branch Laboratory, and only 36 complied with the B.P. requirements.

Charred Notes.—A quantity of ashes from a fire were examined for the Police, for evidence of banknotes alleged to have been burnt, and by careful examination a large number of fragments of notes were obtained. By cautious calcination twenty of the largest fragments were identified as part of the Bank of New Zealand £1 note. In the course of the investigation it was found that a number of the double-sided notes on issue in New Zealand are fairly resistant to destruction.

TOXICOLOGICAL CASES.—In connection with the cases investigated at the Wellington Laboratory, specimens were found to contain aniline oil, cocaine and holocaine, morphine, strychnine, and veronal.

At the Auckland Laboratory strychnine in sufficient quantity to cause death was found in four cases. In one series of cases pyridine was also found in the stomach, indicating that the alkaloid had probably been dissolved in methylated spirits. In another case the strychnine had been placed in a bottle of beer by some person unknown, the bitter taste of the alkaloid being momentarily masked by the taste of the beer.

Federated Malay States.

ANNUAL REPORT OF THE INSTITUTE FOR MEDICAL RESEARCH FOR 1927.

THE total number of samples examined for the various Government Departments in the chemical laboratories was 13,555, as compared with 7756 in 1926. The increase was chiefly due to samples of chandu dross, waters, toddies and stained articles.

MILK.—Five hundred and eleven samples were analysed, of which 75 were unsatisfactory. Samples are submitted by officers of the Health Branch, and also by officers of Sanitary Boards, all samples having been taken under the provisions of the Sale of Food and Drugs Enactment, 1913. Judging by the results of the analyses in the laboratory at Kuala Lumpur, there has been a considerable improvement in the quality of the milk supply in the last seven years.

Toddy.—Owing to the increased supervision of toddy shops there was a large increase in the number of samples examined, viz. 849, as compared with 171 in 1926.

Under the present regulations it is an offence to sell toddy in which:—(a) The alcoholic strength exceeds 10 per cent. by volume; (b) the acidity exceeds 0.8 per cent. expressed as acetic acid. These regulations were adopted to prevent (a) the fortification of toddy with spirit; (b) the sale of very old toddy. A method of detecting the watering of toddy has now been devised, and 263 of the samples gave indications of this adulteration, although conforming to the regulations (a) and (b).

RADION ALFA.—A proprietary remedy for malaria sold under this name was analysed. It was found to consist of methylene blue and quinine, and to be radioactive. Similar pills devoid of radioactivity were prepared, and alternate cases of malaria admitted to the hospital were given two of the proprietary pills or two of the equivalent pills twice daily. The results indicated that the addition of the radio-active substance did not materially increase the therapeutic value.

VITAMIN B EXTRACT.—The preparation of this extract from rice polishings was continued throughout the year, 27,680 fluid ounces being issued, as compared with 8960 ounces in 1926. The increased demand was due to the loss of vegetables caused by the floods in December, 1926.

An extract of rice polishings is now prepared in Java, and the anti-beri-beri vitamin from this extract is adsorbed on acid clay obtained from cheribou. The expense of concentration is thus avoided, and the product is obtained as a powder, which is made into tabloids.

An attempt was made in the Kuala Lumpur laboratory to adsorb the vitamin on purified kaolin, but the resulting product was of little value. The experiments are to be continued with Japanese clay.

Beri-Beri and Rice "Toxin."—During the rains and floods practically all the rice stocks in two districts became sodden with water. Conditions should therefore have been suitable for the generation of rice "toxin," which of recent years has received attention as a possible factor in producing beri-beri. But an analysis of cases in one of the districts brought out the interesting facts that among the Chinese the maximum number of cases occurred during the month following the floods, whereas the maximum was not reached for the Malays and Tamils until the third and fourth months, respectively. This is to be attributed, not to national idiosyncrasy, but to the fact that the normal Chinese diet approaches more nearly to the starvation line for vitamin B.

Another observation tending to discredit the toxin theory was made during the Kelantan outbreak. Rice stocks on a rubber estate were sodden with water for several days, but immediately after the floods subsided supplies of vegetables were obtained from outside and retailed to the coolies at nominal charges. No cases of beri-beri occurred on the estate. On a second estate rice stocks were kept quite dry throughout the flood period, but the vegetable gardens were washed out, and no attempt was made to obtain supplies. Cases of beri-beri developed among the coolies.

VIABILITY OF Bacillus Typhosus AND V. Cholerae.—The viability of intestinal pathogenic bacteria in river water is of local importance, because many natives deposit excrement directly into the river, and effluents from septic tanks discharge into drains leading to a river.

Samples were therefore obtained from the three streams (I, II and III) entering Kuala Lumpur and from the outgoing river (IV). In each flask 250 c.c. of river water were placed and infected with heavy doses of 1000 millions of B. typhosus or 3000 millions of V. Cholerae. Sub-cultures were made every 12 hours. B. typhosus was recovered for 3 days from specimens I, II and III, and for 4 days from specimen IV. The cholera vibrio was recovered for 2 days from specimen IV, and for 3 days from specimens I, II and III. Although it is not claimed that natural river conditions were closely simulated in the experiment, these results probably indicate the serious menace to the public health caused by the pollution of rivers.

Meteorological Office, Air Ministry.

CHANGES OF ZERO IN SPIRIT THERMOMETERS.*

In an attempt to explain the fall of reading noted over a period of some years in the case of certain spirit thermometers, experiments have been carried out to ascertain the effect of the presence of acetone, in the filling liquid, upon the readings of spirit thermometers over a period of time. It is found that in the case of spirit thermometers containing acetone a marked fall of reading is obtained in course of time when the thermometers are exposed to light. It is suggested that the effect is due to the contraction of the liquid consequent upon the formation of condensation products from the acetone under the influence of light.

^{*} Professional Notes, No. 51. By W. F. Higgins, M.Sc., and G. G. Bilham, B.Sc., A.R.C.Sc. H.M. Stationery Office, 1929. Price 4d. net.

The following conclusions have been drawn from the experiments described:

(1) In selecting a liquid for filling spirit thermometers, the presence of acetone as an impurity in the spirit should be carefully guarded against. A guarantee of freedom from acetone should be demanded from the firm supplying the spirit. (2) Owing to the common presence of acetone as an impurity in methylated spirit obtained from the usual sources, the use of this material, either in its commercial form or when redistilled, should be avoided. (3) Thermometers filled with either pure methyl or ethyl alcohol are stable over long periods. (4) The use of a mixture of ethyl and methyl alcohol should be avoided. (5) The addition of aniline colouring matter to pure ethyl alcohol does not affect the stability of the zero. (6) The nature of the residual gas, whether air or nitrogen, does not appear to affect the subsequent behaviour of the instrument. (7) The depression of zero associated with the presence of acetone occurs only on exposure to light.

An Appendix contains a note by the Government Chemist on the methods used for purifying the ethyl and methyl alcohols and the acetone used in these experiments, together with the tests of purity applied.

The International Temperature Scale.*

In 1911 the directors of the national laboratories of Germany, Great Britain and the United States agreed to undertake the unification of the temperature scales used in their respective countries. This course was approved by the Fifth General Conference of Weights and Measures (1913), and at the Sixth General Conference (1921), it was decided to expand the field of activities of the International Committee and International Bureau, and to co-ordinate results obtained in other institutions. Finally, in 1927, the Seventh General Conference, representing 31 nations, adopted unanimously a resolution approving of the provisional adoption of an international scale, submitted for discussion by the Bureau of Standards, the National Physical Laboratory and the Physikalisch-Technische Reichsanstalt.

The English text of the Introduction and Definition of the Scale is as follows:

Introduction.—The experience of the Bureau of Standards, as of the National Physical Laboratory and of the Reichsanstalt, has for many years past indicated the necessity, for industrial purposes, of international agreement on a scale of temperatures ranging from that of liquid oxygen to that of luminous incandescent bodies. As a result of discussion extending over a considerable period, agreement has been reached by the three laboratories, subject to possible minor drafting amendments, on the attached specification for a practical scale, as affording a satisfactory basis on which uniformity in certification of temperature measurements for industrial purposes may be maintained.

It is to be understood that this proposal does not purport to replace the absolute temperature scale which it is recommended should be adopted, on principle, by the International Conference on Weights and Measures. It is intended merely to represent this scale in a practical manner with sufficient accuracy to serve the everyday needs of the laboratories for the purpose of industrial certifications, and is to be regarded as susceptible of revision and amendment as improved and more accurate methods of measurement are evolved.

It is anticipated that this scale will shortly be adopted by the three laboratories for the purposes indicated, and the attached draft is presented to the conference for consideration, with the recommendation that it should be officially adopted, with such amendments, if any, as may be agreed on, as the best practical realisation at the present time of the ideal thermometric scale.

^{*} Report of the National Physical Laboratory for the Year 1928, pp. 29-33.

PART I. DEFINITION OF THE INTERNATIONAL TEMPERATURE SCALE.

- 1. The Thermodynamic Centigrade Scale, on which the temperature of melting ice, and the temperature of condensing water vapour, both under the pressure of one standard atmosphere, are numbered 0° and 100°, respectively, is recognised as the fundamental scale to which all temperature measurements should ultimately be referable.
- 2. The experimental difficulties incident to the practical realisation of the thermodynamic scale have made it expedient to adopt for international use a practical scale designated as the International Temperature Scale. This scale conforms with the thermodynamic scale as closely as is possible with present knowledge, and is designed to be definite, conveniently and accurately reproducible, and to provide means for uniquely determining any temperature within the range of the scale, thus promoting uniformity in numerical statements of temperature.
- 3. Temperatures on the international scale will ordinarily be designated as "°C.," but may be designated as "°C.(Int.)" if it is desired to emphasise the fact that this scale is being used.
- 4. The International Temperature Scale is based upon a number of fixed and reproducible equilibrium temperatures to which numerical values are assigned, and upon the indications of interpolation instruments calibrated according to a specified procedure at the fixed temperatures.
- 5. The basic fixed points and the numerical values assigned to them for the pressure of one standard atmosphere are given in the following table, together with formulae which represent the temperature (t_p) as a function of vapour pressure (p) over the range 680 to 780 mm. of mercury.
 - 6. Basic fixed points of the International Temperature Scale-

(a)	Temperature of equilibrium between liquid and gaseous oxygen at the pressure of one standard atmosphere (oxygen point)	°C. 182·97
	$t_p = t_{760} + 0.0126 (p - 760) - 0.0000065 (p - 760)^2$	
(b)	Temperature of equilibrium between ice and air-saturated water at normal atmospheric pressure (ice point)	0.000
(c)	Temperature of equilibrium between liquid water and its vapour at the pressure of one standard atmosphere (steam point)	100.000
	$t_p = t_{760} + +0.0367 (p-760) - 0.000023 (p-760)^2$	
(d)	Temperature of equilibrium between liquid sulphur and its vapour at the pressure of one standard atmosphere (sulphur point)	444.60
	$t_p = t_{760} + 0.0909 \ (p - 760) - 0.000048 \ (p - 760)^2$	
(e)	Temperature of equilibrium between solid silver and liquid silver at normal atmospheric pressure (silver point)	960.5
(<i>f</i>)	Temperature of equilibrium between solid gold and liquid gold at normal atmospheric pressure (gold point)	1,063

Standard atmospheric pressure is defined as the pressure due to a column of mercury 760 mm. high, having a mass of 13.5951 g/cm.³, subject to a gravitational acceleration of 980.665 cm/sec.², and is equal to 1,013,250 dynes/cm².

It is an essential feature of a practical scale of temperature that definite numerical values shall be assigned to such fixed points as are chosen. It should be noted, however, that the last decimal place given for each of the values in the table is significant only as regards the degree of reproducibility of that fixed point on the International Temperature Scale. It is not to be understood that the values are necessarily known on the Thermodynamic Centigrade Scale to the corresponding degree of accuracy.

- 7. The means available for interpolation lead to a division of the scale into four parts.
- (a) From the ice point to 660° C. the temperature t is deduced from the resistance R_t of a standard platinum resistance thermometer by means of the formula

$$R_t = R_o (1 + A + Bt^2).$$

The constants R_0 , A, and B of this formula are to be determined by calibration at the ice, steam, and sulphur points, respectively.

The purity and physical condition of the platinum of which the thermometer is made should be such that the ratio R_t/R_0 shall not be less than 1.390 for $t=100^\circ$ and 2.645 for $t=444.6^\circ$.

(b) From -190° to the ice point, the temperature t is deduced from the resistance R_t of a standard platinum resistance thermometer by means of the formula,

$$R_t = R_o [1 + At + Bt^2 + C (t - 100) t^3].$$

The constants R_o , A, and B are to be determined as specified above, and the additional constant C is determined by calibration at the oxygen point.

The standard thermometer for use below 0° C. must, in addition, have a ratio R_t/R_0^{\bullet} less than 0.250 for $t=-183^{\circ}$.

(c) From 660° C. to the gold point, the temperature t is deduced from the electromotive force e of a standard platinum v. platinum-rhodium thermo-couple, one junction of which is kept at a constant temperature of 0° C., while the other is at the temperature t defined by the formula

$$e = a + bt + ct^2.$$

The constants a, b, and c are to be determined by calibration at the freezing point of antimony, and at the silver and gold points.

(d) Above the gold point the temperature t is determined by means of the ratio of the intensity J_2 of monochromatic visible radiation of wave length λ cm., emitted by a black body at the temperature t_2 , to the intensity J_1 of radiation of the same wave length emitted by a black body at the gold point, by means of the formula

$$\log_{\mathbf{c}} \frac{J_{2}}{J_{1}} = \frac{c_{2}}{\lambda} \left[\frac{1}{1,336} = \frac{1}{(t+273)} \right]$$

The constant c_2 is taken as 1.432 cm. degrees. The equation is valid if $\lambda(t+273)$ is less than 0.3 cm. degrees.

Part II deals with the recommended experimental procedure for determining: (1) The temperature of equilibrium of liquid and gaseous oxygen; (2) the temperature of melting ice; (3) the temperature of condensing water vapour; (4) the temperature of condensing sulphur vapour; (5), (6) and (7) for standardising a thermo-couple; (8) subsidiary points.

Revised Table of Atomic Weights for 1929.

The Council of the Chemical Society has ordered the following table to be published in their journal (*J. Chem. Soc.*, 1929, 216). In the accompanying Report, which is signed by Messrs. F. W. Aston, H. V. A. Briscoe, R. Whytlaw Gray, and E. K. Rideal, it is stated that Clarke's method of computation being considered trustworthy, his final values for the 36 elements for which no determinations have since been published have been adopted. In other cases Clarke's figures have been modified in accordance with more recent work.

For the nine "simple" elements, H, He, C, N, F, Na, P, As, and I, the values obtained by Aston, with his new mass-spectrograph, are adopted in preference to those obtained from "physical" or "chemical" data, as the Committee are of opinion that Aston's is less liable to error than any other method.

Where definite information is available the mass-numbers of the known isotopes of the elements are also given, in the order of their abundance, as deduced from the relative intensities of the lines in the mass-spectrum. In cases where the last figure may be in error by two or three units it is given as a subscript.

ATOMIC WEIGHTS. 1929.

Atomic number.	Name.	Symbol.	Atomic weight.	Mass-numbers of isotopes in order of intensity.
1	Hydrogen	H	1.0078	1
2	Helium	He	4.002_{2}	4
3	Lithium	Li	6.94	7, 6
4	Beryllium	Be	9.02	9
5	Boron	В	10.83	11, 10
6	Carbon	С	12.0036	12
7	Nitrogen	N	14.008	14
8	Oxygen	Ö,	16.0000	16
.9	Fluorine	F	19.00	19
10	Neon	Ne Na	20·18 23·000	20, 22, 21 23
11 12	Sodium	Mg	24.30	24, 25, 26
13	Magnesium Aluminium	Al	26.97	27
14	Silicon	Si	28.08	28, 29, 30
15	Phosphorus	P	30.98,	31
16	Sulphur	Ŝ	32.065	32, 33, 34
17	Chlorine	C1	35.457	35, 37
18	Argon	Α	39.94	40, 36
19	Potassium	K	39.105	39, 41
20	Calcium	Ca	40.09	40,44
21	Scandium	Sc	45·1 ₅	45
22	Titanium	Ti	47.90	48
23	Vanadium	V	50.95	51
24	Chromium	Cr	52.04	52
25	Manganese	Mn	54.95	55
26	Iron	Fe	55.84	56, 54
27	Cobalt	Co	58.95	59 58, 60
28	Nickel	Ni Cu	58·69 63·55	63, 65
29 30	Copper Zinc	Z _v	65.38	64, 66, 68, 67, 65, 70, 69
30 31	Gallium	Ga	69.72	69, 71
32	Germanium	Ge	72.60	74, 72, 70, 73, 75, 76, 71, 77
33	Arsenic	As	74.934	75
34	Selenium	Se	79.2	80, 78, 76, 82, 77, 74
35	Bromine	Br	79.91_{5}	79, 81
36	Krypton	Kr	82.9	84, 86, 82, 83, 80, 78
37	Rubidium	Rb	85.43	85, 87
38	Strontium	Sr	87.63	88, 86
39	Yttrium	Yt	88.9_{3}	89
40	Zirconium	Zr	91.2	90, 94, 92, (96)
41	Niobium	Nb	93.3	
40	(Columbium)	(Cb)	06.0	
$rac{42}{43}$	Molybdenum Masurium	Mo Ma	96.0	
43 44	Ruthenium	Ru	101.6	
45	Rhodium	Rh	102.9	
46	Palladium	Pd	106.7	
47	Silver	Ag	107-880	107,109
48	Cadmium	Cď	112.40	114, 112, 110, 113, 111, 116
49	Indium	In	114.8	115
50	Tin	Sn	118.70	120, 118, 116, 124, 119, 117, 122, 121, 112, 114, 115
51	Antimony	Sb	121.76	121, 123
52	Tellurium	Te	127.5	128, 130, 126
53	Iodine	I	126.93_{2}	127
54	Xenon	Xe	130.2	129, 132, 131, 134, 136, 128, 130 126, 124
55	Caesium	Cs	132.8_{1}	133
56	Barium	Ba	137.3_{6}	138
57	Lanthanum	La	138.90	139
58	Cerium	Се	140-2	140, 142

Atomic number.	Name.	Symbol.	Atomic weight.	Mass-numbers of isotopes in order of intensity.
59	Praseodymium	Pr	140-9	141
60	Neodymium	Nd	144.25	142, 144, 146, (145)
61	Illinium	Il	l – •	
62	Samarium	Sm	150.4,	
63	Europium	Eu	152.0	
64	Gadolinium	Gd	157.0	
65	Terbium	Tb	159-2	
66	Dysprosium	$\mathbf{D}\mathbf{y}$	162.4	
67	Holmium	Ho	163.5	
68	Erbium	Er	167-6	
69	Thulium	Tm	169-4	
70	Ytterbium	Yb	173.	
71	Lutecium	Lu	175.	
72	Hafnium	Hf	178.6	
73	Tantalum	Ta	181.3	
74	Tungsten	w	184-1	
75	Rhenium	Re		
76	Osmium	Os	191.	
77	Iridium	Ir	193.04	
78	Platinum	Pt	195.2	
79	Gold	Au	197.2,	
80	Mercury	Hg	200.6	202, 200, 199, 198, 201, 204, 196
81	Thallium	Tl	204.	
82	Lead	Pb	207.2	208, 206, 207
83	Bismuth	$\mathbf{B}\mathbf{i}$	209.00	
84	Polonium	Po		
85				
86	Niton	Nt	222	
	(Emanation)	(Em)		
87				
88	Radium	Ra	225.9_{5}	
89	Actinium	Ac		
90	Thorium	Th	232-15	
91	Proto-actinium	Pa		
92	Uranium	U	238-15	

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Difference in Osmotic Concentration between Yolk and White of Egg. J. Straub. (Rec. Trav. Chim. Pays-Bas, 1929, 48, 49-82.)—The difference in osmotic concentration between yolk and white of hens' eggs, indicated by the respective freezing points, about -0.6° C. and -0.45° C., is discussed. Results are given of calculations of the partial freezing point depressions, due to the known proportions of the various soluble constituents of yolk and white, and the sum of these is about 0.52° C. for the former, and about 0.27° C. for the latter, the divergences from the actual figures being thus 0.08° and 0.18° respectively. Since the skin of the living yolk is permeable by water, equilibrium between yolk and white would indicate an over-pressure of 1.8 atmos. in the yolk, and the conclusion is drawn that such equilibrium does not exist in the resting eggs. The maintenance of a stationary state other than one of equilibrium demands a continuous supply of energy, and the possible sources of this are considered.

T. H. P.

Comparison of the Monier-Williams and the A.O.A.C. Methods for Determination of Sulphurous Acid in Food Products. J. Fitelson. (1. Assoc. Off. Agric. Chem., 1929, 12, 120-129.)—A comparison of the Monier-Williams method for determining sulphurous acid (ANALYST, 1927, 52, 343, 415) with the A.O.A.C. method establishes the need of a more accurate method than that of the A.O.A.C. in the case of food products containing volatile sulphur compounds. The use of copper salts as a wash trap to remove sulphides from the distillate in the official method is shown to produce inaccuracies, and the accuracy and reliability of the Monier-Williams method under varied conditions is confirmed. In the course of a detailed study of brined onions it is conclusively shown that little or no sulphurous acid is developed during brining, so that where detected by the Monier-Williams method it is added, and that shown by the A.O.A.C. method can be taken as a rough indication of the quantities of volatile sulphur compounds present in the onions. The use of brine reduces the volatile sulphur, this being largely due to a leaching out by the brine. D. G. H.

Solubility Tests of Castor Oil. H. P. Trevithick and M. F. Lauro. (Oil and Fat Ind., 1929, 6, 27–29.)—Failure of castor oils to pass solubility tests, especially where alcohol of less than 95 per cent. strength is used, is not to be considered proof of adulteration without a further chemical investigation. Castor oil thickens on keeping, gravity and viscosity increasing without change in iodine value. The acetyl value furnishes one of the most useful indicative figures, and it is considered that the filtration method in determining the acetic acid liberated from the acetylated oil should not be used, but that distillation of the saponified acetylated fat with phosphoric acid is more trustworthy.

D. G. H.

Indian Ephedras. Their Extraction and Assay. S. Krishna and T. P. Ghose. (J. Soc. Chem. Ind., 1929, 48, 67-70T.)—The content of ephedrine in the Chinese plants "Mahuang" (Ephedra sinica and E. equisitina) does not depend on the altitude at which the plants are grown, but diminishes as the rainfall of the locality increases. It is determined as follows: 100 grms, of the air-dried (about 5 per cent. of moisture) and finely-powdered green stems are treated for 2 hours with 400 c.c. of a mixture of 3 parts of ether and 1 part of chloroform, the mass being then well shaken with 50 c.c. of ammonia (3 parts of 0.880 ammonia and 1 part of water), left overnight and filtered. The residue is treated twice in the same way. The combined extracts are distilled to remove the bulk of the solvent, and the residue is extracted with 75, 60, 60, and 50 c.c. of 1.5 per cent. hydrochloric acid. The total acid extract is filtered, made strongly alkaline with potassium carbonate and almost saturated with salt, the alkaloids thus liberated being extracted four times with ether. The bulk of the ether is distilled off and the rest allowed to evaporate at room temperature. Excess of 0.1 N hydrochloric acid is added, and the excess titrated with 0.1 N sodium hydroxide in presence of methyl orange, the total amount of alkaloid being calculated as ephedrine. titrated solutions are rendered alkaline, and the alkaloids again extracted with

ether and treated with alcoholic hydrochloric acid to convert them into the hydrochlorides, which are dried over calcium chloride and caustic potash in a vacuum desiccator. Finally, ephedrine hydrochloride is isolated from the mixed hydrochlorides by treatment with dry chloroform, in which it is practically insoluble: 100 c.c. of chloroform dissolve 0.02 grm. of ephedrine hydrochloride at 15°, 0.04 grm. at 30°, and 0.084 grm. at 60° C. The hydrochloride is dried and weighed. The large-scale extraction is also described.

Biochemical.

Bio-assay of Commercial Pituitary Powders. W. T. McClosky and J. C. Munch. (J. Assoc. Off. Agric. Chem., 1929, 12, 135–136.)—Analyses of commercial samples of pituitarium (cleaned dried powdered posterior pituitary lobes of domesticated animals used for food) showed activities which were generally between 30 and 50 per cent. of the standard posterior pituitary powder, U.S.P.(X.); commercial anterior powders were uniformly inactive by the U.S.P. method on guinea-pig uterus, and commercial whole powders showed a ratio of activity of 1 part of posterior substance in 8 parts of whole body. It is suggested that the standards for physiological activity should be; for pituitarium U.S.P. 50 per cent., and for desiccated whole pituitary powder 5 per cent. of the activity of the U.S.P. official standard posterior pituitary powder.

Comparison of the Oxytocic, Pressor and Anti-Diuretic Activities of Commercial Samples of Pituitary Extract. U. G. Bijlsma, J. H. Burn and J. H. Gaddum. (Quart. J. Pharm., 1928, 1, 493-508.)—The standardisation of pituitary extract is almost universally effected by comparison with the international standard on the isolated guinea-pig uterus. It has been suspected that this comparison is not always an accurate guide to the pressor potency of different extracts, and some clinical reports have indicated that it is not a guide to the anti-diuretic potency.* The recent separation of oxytocin and vasopressin by Kamm, Aldrich, Grote, Rowe, and Bugbee (J. Amer. Chem. Soc., 1928, 50, 573) has furnished final proof that the oxytocic and pressor properties are due to two different substances, and possibly the anti-diuretic effect is due to a third. It was therefore thought important to determine how far differences in anti-diuretic and pressor potency arise in commercial extracts of approximately equal oxytocic power. Four commercial extracts of the posterior lobe of the pituitary have thus been examined in comparison with the international standard for their oxytocic and pressor power by the three authors working entirely independently, and three separate reports are presented. One worker has also tested their anti-diuretic power on the unanaesthetised dog; another has devised and described a new method of testing for this action on a normal human being. In the discussion the average values of the investigators are compared, and the following conclusions are drawn:-The results of the different workers agree with one another, except that there are differences of opinion as to both the pressor and oxytocic powers of one of the

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extracts and as to the anti-diuretic power of another. The anti-diuretic effect is not due either to the pressor or to the oxytocic principle. The test for any one of these three active principles does not provide a reliable index of the concentration of any other of them in commercial extracts. The present international standard can only be used as a standard for anti-diuretic activity if it is found that different preparations of it contain the same concentration of the anti-diuretic principle; if different samples are alike, the existing standard will serve as a standard for all three properties.

P. H. P.

Creatine Content of the Muscles and some other Tissues in Fishes. A. Hunter. (I. Biol. Chem., 1929, 81, 513-523.)—The creatine content of the skeletal muscles has been determined in fifteen species of fishes from the coast of British Columbia. Each genus, with two exceptions, was represented by at least two individuals, some by as many as four. In several cases the heart or the testis or both were also analysed, and in two instances only (those of the dogfish and the skate) the brain was examined. It was not possible to obtain and examine each of the species under identical conditions, e.g., some had been kept in a pen and others were captured in the open. Considerable differences were found to exist between different species, and also between different individuals of the same species, yet each species presents a fairly characteristic range of creatine values. The results obtained are tabulated. In the mammal different muscles often show differences of creatine content, and thus a comparison of species must be based upon analyses of homologous muscles or muscle groups. In order to test whether this applied to fishes, three samples of flesh were taken from each of two ling cod (Ophiodon elongatus), (1) from the dorsal region just behind the head, (2) from the abdominal wall, midway between the head and tail, and (3) from the compact mass of muscular tissue posterior to the cloacal aperture. There was found to be a progressive and conspicuous increase of creatine concentration from before backward, i.e. the highest value was in the powerful propelling muscles of the tail. Therefore in most other cases samples were taken only from the caudal region, but in the case of the skate, the laterally disposed muscles of the pectoral fins, used for propulsion were found to contain as much creatine as those of the tail. The differences between species do not correspond in any obvious way with zoological subdivisions; there is no systematic difference, with respect to muscle creatine, between the Teleostomi and the Elasmobranchs. The highest figure found for tail muscle (0.74 per cent.) is in one of the Teleostomi (Clupea), the lowest (0.48) in a selachian (Raia); the ratfish (grouped as an elasmobranch), shows 0.55 per cent., and the dogfish, another Elasmobranch, 0.63 per cent. The elasmobranchs show a lower concentration of creatine in the heart than the Teleostomi. In general, the skeletal muscles of fish contain more creatine than those of mammals. Mammalian muscle exceeds 0.6 per cent. in a few isolated cases, the mixed flesh of the rabbit shows an average of 0.53, and the muscles of most other mammals yield less than 0.5 per cent. In seven out of the thirteen genera of fishes represented, the average concentration is close to 0.6 per cent., in three (Hydrolagus, Sebastodes and Leptocottus), it lies between 0.5 and 0.6; in one (*Phanerodon*) between 0.6 and 0.7. In one only (*Raia*), it falls below 0.5, and in *Clupea* it even exceeds 0.7. Mammals, however, show a higher concentration of creatine in the heart and in the testes. The two analyses made of selachian brain tissue indicate a content of creatine equal to that of mammalian brains. In fishes, as in mammals and birds, red muscles contain less creatine than pale, and foetal muscle less than the adult. It cannot safely be assumed that creatine exists in fish muscle in exactly the same state as in mammalian muscle.

P. H. P.

Colorimetric Determination of Total and Inorganic Sulphates in Blood Serum, Urine and other Body Fluids. E. G. Wakefield. (J. Biol. Chem., 1929, 81, 713-721.)—A colorimetric method for the determination of sulphates, the principle of which was originally described by Hubbard (J. Biol. Chem., 1927, 74, 5), has been checked after certain changes which gave most consistent results had been made, and has been found to be a microchemical method which is adaptable for clinical uses. Hubbard's process was for the determination of inorganic sulphates in blood serum. The reagents described by him are retained, but certain portions of the manipulative procedure and the strengths of some of the solutions have been modified, and the method has been extended to permit the determination of total and conjugated sulphates in the blood serum, and of total, inorganic and conjugated sulphates in the urine and in the fluids which in oedematous conditions collect in the peritoneal cavity, thorax and elsewhere. The method consists in treatment of the serum or urine with trichloroacetic acid to remove the proteins, centrifuging, and addition of the supernatant fluid to a solution of benzidine base. The precipitate of benzidine sulphate is washed in acetone, centrifuged, drained, dissolved in dilute hydrochloric acid, and treated with diluted hydrogen peroxide and 0.5 per cent. ferric chloride solution. The yellow colour thus formed is fully developed after about 5 minutes, and remains constant until about 10 minutes have passed; a few drops of concentrated hydrochloric acid added to the standard and the unknown will prevent the rapid fading of the colours if readings cannot be made during the second five minutes. The acidity must be the same in the standards and the unknown. Normal values for the total, inorganic and conjugated sulphate content of the blood serum, as determined by this method, are given. This study confirms the results of Denis and Reed (J. Biol. Chem., 1926, 71, 191; ANALYST, 1927, 52, 96), who demonstrated the presence of conjugated sulphates in human blood. P. H. P.

Association of Vitamin A with Greenness in Plant Tissue. II. Vitamin A content of Asparagus. J. W. Crist and M. Dye. (J. Biol. Chem., 1929, 81, 525-532).—It has previously been shown by Dye, Medlock and Crist (J. Biol. Chem., 1927, 74, 95; ANALYST, 1927, 52, 552) that the vitamin A content of head and leaf lettuce varies more or less directly with the greenness of the plant tissue. Since asparagus is offered for consumption in both the green and bleached state, and also as a canned product, an investigation has been made of its vitamin A content. Different types and varying amounts of asparagus were given to albino

rats, which had been placed on a vitamin A-free diet until their store of the vitamin was depleted, and growth was determined over a period of 8 weeks. Curves show the results. Green asparagus, whether fresh, freshly cooked, or canned, when given daily at the rate of 0.1 grm. per animal, contained vitamin A in sufficient quantities to promote health and growth in the rats. Fresh, bleached (term used where tissues have never been allowed to become green) asparagus, given daily at the rate of either 0·1 or 0·5 grm. per animal gave no stimulus to health and growth; the animals died as rapidly as the negative controls. Cooking in open kettle fashion effected an improvement in the nutritive quality of bleached asparagus, but did not render its value comparable to that of the green product cooked in the same manner. Green asparagus tissue had lower percentages of water and iron than the bleached tissue, but higher percentages of ash, nitrogen, sulphur, calcium, phosphorus, and possibly manganese. The quantities of manganese were so small that the figures could not be taken as significant. The data from these experiments support the conclusion that the vitamin A content of plant tissue is associated with its greenness. There is no direct evidence that the chlorophyll is the vitamin, but where the tissues are decidedly green the vitamin is abundant. It is an open question whether or not the chlorophyll or some part of the chlorophyll molecule, e.g., the phytol alcohol unit, is the vitamin, or functions in the production of the vitamin, or is merely a circumstance attendant upon the reactions of the plant when the environment is such as to effect the synthesis of the vitamin. It is possible that the poor quality of bleached asparagus as food for the animal may not be due alone to vitamin A deficiency, but also to a superabundance of deleterious chemical compounds. P. H. P.

The Vitamin A, B and C content of Artificially Versus Naturally Ripened Tomatoes. M. C. House, P. M. Nelson and E. S. Haber. (J. Biol. Chem., 1929, 81, 495-504.)—The use of ethylene in the ripening of fruits and vegetables (used to a limited extent commercially during the past 3 years) possesses distinct advantages over the older method. However, since the consumer is interested in the nutritive constituents as well as in the exterior appearance of the final product, and fruits and vegetables are eaten largely because of their vitamin value, it is of interest to know the effect this new commercial method of ripening has upon the vitamin content. Therefore the vitamin content of tomatoes, a foodstuff commonly subjected to this treatment, was investigated. A comparison was made of the vitamin A, B and C content of green, air-ripened, ethyleneripened and vine-ripened tomatoes. Twenty rats were used in each group for the vitamin A and B tests. Ten guinea pigs were used in each group for the vitamin C tests. Statistical treatment of the data has shown that:—The four lots of tomatoes showed no difference in their vitamin B content, and thus the methods of ripening used did not alter the amount of vitamin B present in the green mature fruit. The vitamin A content of ripened tomatoes was found to be greater than that of the green mature fruit. The same quantity of vitamin A was developed in the tomatoes regardless of the method of ripening used. The green tomatoes were

relatively poor in vitamin C. Air-ripened and ethylene-ripened tomatoes were richer in this vitamin than the green fruit, and vine-ripened tomatoes were superior to either the artificially ripened or the green tomatoes. Therefore the commercial method of ripening tomatoes in an ethylene-air mixture (1:800 was used) produces fruit which is equally as rich in the vitamins A, B and C as fruit which has been picked green and ripened in air.

P. H. P.

Variations in Amounts of the Antirachitic Vitamin in Different Samples of Cod-liver Oil, Milk and Butter. K. H. Coward. (Quart. J. Pharm., 1928, 1, 534-538.)—A large number of substances have been examined for their content of vitamin D by the method described by Coward (Quart. J. Pharm., 1928, 1, 27; Analyst, 1928, 53, 449), in which the amount of activity contained in 0.0001 mgrm. of a standard preparation of irradiated ergosterol is adopted as a unit of antirachitic activity. It is shown that different samples of cod-liver oil, milk, and butter may vary enormously in their antirachitic potency, and the object of the paper is to call attention to these variations. Four selected samples of cod-liver oil were found to contain 150, 100, 70-80, and 50 units of antirachitic activity per grm., respectively. So great a variation amongst selected samples suggests a still greater variation in a series bought in the open market. Probably samples of spring and summer high-priced butters are approximately equal in their content of vitamin D (some showed 0.8 to 1.0 unit per grm.), but in some instances the butter could not be expressed in units, as there was no comparison with the standard (margarine containing a vitamin concentrate). One sample was extremely poor in the antirachitic factor. Two out of three samples of milk were almost entirely lacking in vitamin D. Since one of these two was examined in midsummer, it seems probable that all winter milk will be found deficient. It is evident that the widespread belief that milk and butter are rich sources of the antirachitic vitamin is largely erroneous. Recent evidence does not challenge the view that a sufficiency of the fat soluble vitamins (A and D) will always be obtained in a diet containing plenty of milk and butter, so far as vitamin A is concerned, but it may well be that it is often not true for vitamin D, and that, in order to ensure the presence of enough of the latter, it should be added specifically in one of the available forms. It is suggested that the advertisement of preparations containing added vitamin D as substitutes for cod-liver oil will be misleading unless the amount of vitamin present in the daily dose for a child is 150 units, or for an adult 300 units. Substitutes for cod-liver oil must also contain vitamin A. If other laboratories in Great Britain desire to state their results in terms of the same units, the Pharmacological Laboratory is prepared to supply a small portion of the standard sample of irradiated ergosterol on request.

P. H. P.

Photochemical Action of Various Sterols. L. Hugounenq and E. Couture. (Comptes rend., 1929, 10, 742-743.)—Cholesterol from cod-liver oil gives, after 5 days, an impression on a sensitive plate through a quartz plate, but

loses its activity after a month in the dark. It appears to be a phenomenon of phosphorescence, and a sample kept for some months in diffuse light was more active than one freshly prepared. Sterols from cow's blood and from snails gave no impression after 15 days, but that from the silkworm gave a clear positive effect. Ergosterol from pure yeast gave a clear stain on sensitised gelatin either in direct contact or when separated by a layer of 3 mm. of air or a thin film of collophane. Samples from less pure yeasts under similar conditions showed more intense reactions.

D. G. H.

Agricultural.

Determination of Small Quantities of Nitrogen in Plant Materials. J. T. Sullivan and L. E. Horat. (J. Assoc. Off. Agric. Chem., 1929, 12, 133-135.) —The nitrogen in plant-materials rich in organic matter but low in nitrogen may be determined with as high an accuracy as the Kieldahl method gives with larger samples, by an application of the Folin and Farmer method. Plant extracts are treated in a Kjeldahl flask by boiling off the water or alcohol from the acidified extract and digesting with 1-2 grm. of copper sulphate and 3 c.c. of concentrated sulphuric acid. Superoxol may be added when fumes appear, or more sulphuric acid is used. Heating is continued for 1 hour after clearing of the solution, which, after cooling, is transferred to a test tube; washings are added followed by 5 c.c. of saturated sodium hydroxide, and, after again cooling, 10 c.c. of alkali completes neutralisation, and a 2 hours' aeration is carried out in the Van Slyke-Cullen urea apparatus with 0.02 N sulphuric acid. Methyl red is used in the back titration. The nitrogen in dry apple wood was found to average on 10 grms. by the Kjeldahl method 10.72 mgrms., and on 1 grm. by the micro method 1.065 mgrm.; and in apple wood extract 7.83 and 0.795 mgrm. respectively.

D. G. H.

Determination of Hoof Meal. W. F. Sterling. (J. Assoc. Off. Agric. Chem., 1929, 12, 129-132.)—The mixture of hoof and horn, dried and ground, is known as hoof meal, and the quantity of hoof meal present in a mixture may be determined by adding sufficient chloroform or carbon tetrachloride to 1 grm. of sample (ground to pass a 40-mesh sieve) to float the meaty portion. The sediment of bone is drawn off, and the portion that floats is poured on to a filter with the solvent used, washed and dried. To this is added 50 c.c. of a solution of 0.1 Nhydrochloric acid containing 1 grm. of pepsin U.S.P., and the mixture kept at 37-40° C. for 48 hours, after which 5 c.c. of 1 N sodium hydroxide is added and left for 10 minutes, when the mixture is centrifuged. The clear liquid is poured off, the residue washed with warm water and again centrifuged, and the process repeated several times, the final washing being with alcohol. The residue is dried and weighed and the weight multiplied by 1.54 gives the hoof meal. A microscopic examination of the residue should be made, and if vegetable tissue is present a correction is applied, if possible. D. G. H.

Organic Analysis.

Analysis by Means of the Thiocyanogen Value of Fats containing Linolenic Acid. Analysis of Linseed Oil. H. P. Kaufmann and M. Keller. (Z. angew. Chem., 1929, 42, 20-23; 73-76.)—Comparisons of the iodine and thiocyanogen values of linseed oils after various periods and in the presence of various amounts of reagent have enabled the authors to formulate equations connecting the percentage contents of oleic (O), linolic (L) and linolenic (Le) acids with the total saturated fatty acids (G), and the iodine (I) and thiocyanogen (T) values. Elimination of impossible equations led to the conclusion that two out of the three double linkages of the linolenic acid, corresponding with T=182.46, are satisfied during the determination of the thiocyanogen value (ANALYST, 1928, 53, 613). The linseed oil to be analysed is saponified in the absence of oxygen, the unsaponifiable matter removed, and the fatty acids liberated, removed in pentane and well dried over freshly ignited sodium sulphate. The iodine value is then determined by the bromine method (cf. following abstract) with an excess of bromine solution (100 to 200 per cent.) for periods of 2 and 24 hours. The thiocvanogen value (loc. cit.) is determined on about 0.1 grm. of the acids, and in the presence of linolenic acid it is preferable to use a large excess (200 per cent.) of a 0.13 N solution of reagent and to titrate after 24 hours. The composition of the fatty acids is best obtained by Bertram's method (loc. cit. and Z. Unters. Lebensm., 1928, 55, 179), which gives higher results than Twitchell's method. The following equations may then be solved:—(1) G+O+L+Le=100; (2) 89.93O+ $181 \cdot 14L + 273 \cdot 70Le = 100I$; (3) $89 \cdot 93O + 90 \cdot 57L + 182 \cdot 46Le = 100T$. The saturated portion of a mixture of the above acids is determined by the relation G=100-1.100T, but oleic acid may be replaced by other simple unsaturated acids (e.g. elaidic, erucic, petroselinic acids, etc.). A Calcutta linseed oil was found to have the following composition: - Saturated acids and unsaponifiable matter, 10-8; O, 11.9; L, 32.6; Le, 40.2; and glycerol residue 4.5 per cent. The literature of the subject is discussed critically, and the uses and advantages of the thiocyanogen value indicated. J. G.

Partial Halogen Addition to Unsaturated Fatty Acids. β -Elaeostearic Acid Glyceride and Wood Oil. H. P. Kaufmann and C. Lutenberg. (Ber., 1929, 62, 392–401.)— β -Elaeostearic acid glyceride (m.pt. 60 to 61° C.) was prepared by exposure to light from the sun or quartz lamp of a solution of Hankow wood oil in pentane in the presence of solid iodine, and recrystallised from acetone. The addition of bromine to two double bonds of the glyceride and of natural wood oil was determined at 18° C. in the absence of light by titration, after various periods of time of a mixture of 30 c.c. of a $0.1\ N$ solution of bromine and $10\ c.c.$ of a solution of sample (about $0.12\ grm.$), both in pure carbon tetrachloride, with $0.1\ N$ sodium thiosulphate solution. The iodine values, also, were determined by titration of the same mixtures in the presence of 25 c.c. of a 5 per cent. aqueous solution of potassium iodide, and it was shown that normally after 3 to 6 hours a

theoretical value was obtained in each case corresponding with the absorption of 2 mols. of bromine. For a normal oil variations in the amount of the excess of bromine has no influence. The "partial iodine value" was determined by the addition to about 0·1 grm. of sample in 15 c.c. of a mixture of equal volumes of chloroform and carbon tetrachloride of 20 c.c. of a 0·1 N solution of iodine in a 0·1 N solution of bromine in methyl alcohol saturated with sodium bromide. After 4 hours in the dark the mixture was titrated in the presence of potassium iodide. Comparative tests with the thiocyanogen value (Analyst, 1928, 53, 613) showed that both for wood oil and for the glyceride the absorption corresponded with the saturation of one double bond, though the thiocyanogen value was usually 1 to 2 units higher than the partial iodine value for the same sample. (Cf. Toms, Analyst, 1928, 53, 69.)

Constitution of α -Elaeostearic Acid, the most important Component of Chinese Wood Oil (Tung Oil). J. Böeseken. (J. Soc. Chem. Ind., 1929, 48, 71–72T.)—Objection is raised to Steger and van Loon's statement that uncertainty exists in our knowledge of α -elaeostearic acid (J. Soc. Chem. Ind., 1928, 47, 362T), the constitution of which has been proved, mainly by the investigations of the author and his collaborators, to be

 CH_3 . $(CH_2)_3$.CH:CH.CH:CH:CH:CH. $(CH_2)_7$. CO_2 H. T. H. P.

Determination of the Iodine Value. II. Action of Iodine Chloride Solutions on Fatty Acids with Conjugated Double Linkings. E. T. Gelber and J. Böeseken. (Rec. Trav. Chim. Pays-Bas, 1929, 48, 377-385.)—For linolic acid, iodine values obtained after 2 hours' absorption correspond with only one double linking, and even when the action of Wijs's solution is continued for 18 hours, the value for two double linkings is not quite reached. Investigation shows that the reaction takes place in two stages; in the first, which is comparatively rapid, chlorine only is added at one of the double linkings, free iodine being separated, whereas saturation of the second double linking occurs only slowly. This behaviour is not peculiar to linolic acid and its esters, but is typical for higher fatty acids with conjugated double linkings. Similarly, with elaeostearic acid, two of the three double linkings are saturated quickly, whilst saturation of the third, even when a seven-fold excess of reagent is used, occupies some days. These results indicate that elaeostearic acid is a linolenic acid with conjugated double linkings. above conclusions hold only for iodine chloride solutions, iodine bromide solutions reacting in a fundamentally different manner. T. H. P.

Insect Oils. M. Tsujimoto. (J. Soc. Chem. Ind. Japan, 1929, 32, 49B-54B.)—Firefly Oil.—The insects used were "Genji-hotaru" (Luciola vitticollis Kiesenwetter), and "Heike-hotaru" (Luciola parva Kiesenwetter), and after removal of the wings they yielded 4.8 per cent. of an orange-yellow viscous fat of iodine value 116. Of this, the fatty acids and unsaponifiable matter amounted to 85 per cent., consisting of 87 per cent. of fatty acids and 13 per cent. of unsaponifiable matter. The fatty acids had: M.pt., 36° C.; neutralisation value, 179.2; iodine value

(Wijs), 110.7; and ether-insoluble bromides, 17.9 per cent. The oil contained highly unsaturated acids. The unsaponifiable matter was a pale yellow soft crystalline mass, partly insoluble in petroleum spirit, the insoluble portion appearing to consist of an unknown higher alcohol. It was found that the head and thorax contained 1.5, and the abdomen 3.8 per cent. of oil (calculated on the whole insect), with iodine values 115 and 105, and $n_{\rm p}$ 1.4770 at 28.5° C. and 1.4732 at 29.5° C., respectively. The abdominal oil contained 18.65 per cent. of unsaponifiable matter.

Locust Oil.—On extraction with ether dried locusts (Oxya japonica Willemse) yielded 3 per cent. of dark greenish-yellow viscous oil having: Sp. gr. 18° C., 0.9688; acid value, 44.3; iodine value (Wijs), 122.6; saponif. value, 171.5; unsaponifiable matter, 15.75 per cent., of iodine value 91.8. The fatty acids (67.25 per cent.) showed: Neutralisation value, 196.0; iodine value, 150.8; ether-insoluble bromides, 37.4 per cent. (m.pt. 178° C. and containing 62.42 per cent. of bromine and therefore a hexabromostearic acid). The iodine value of the liquid acids (75 per cent.) was 187.7, and neutralisation value 189.2. The chief constituents of the fatty acids are C_{18} acids. By the digitonin method 44.1 per cent. of sterol was obtained from the unsaponifiable matter.

Cricket Oil.—The dried crickets (Acheta mitrata, Burmeister.) gave 2·4 per cent. of a dark brownish viscous oil showing: Sp. gr. at 19·5° C., 0·9312; acid value, 58·7; iodine value (Wijs), 116·0; sap. value, 181·5; unsaponifiable matter, 11·32 per cent. with iodine value 119·2. The fatty acids (78·73 per cent.) had iodine value, 124·3; neutralisation value, 202·4; ether-insoluble bromides, 6·9 per cent. (m.pt. 177–178° C. containing 62·13 per cent. bromine). By the digitonin method 45·45 per cent. of sterol (cholesterol) was obtained. D. G. H.

Determination of Neutral Fat in Sulphonated Oils. R. Hart. (J. Amer. Leather Chem. Assoc., 1929, 24, 120).—The proportion of neutral fat is at present usually determined gravimetrically, but the author suggests that it is much more convenient and accurate to determine, in the usual manner, the saponification value corresponding to the glyceride, provided that the alcoholic solution is neutral to phenolphthalein, and that allowance is made for the fact that ammonia soaps in solutions of alcoholic potash behave like free fatty acids. The saponification value of the glycerides may be expressed as mgrms. of KOH per grm. or as percentage of free fatty acid. The saponification value of the original oil before sulphonation must have been determined previously. Possible error due to splitting off of organically combined sulphur trioxide during saponification is considered negligible.

R. F. I.

Inorganic Analysis.

Absorption of Oxygen by Alkaline Pyrogallol. T. J. Drakeley and H. Nicol. (J. Soc. Chem. Ind., 1929, 48, 62T.)—The effect on the evolution of carbon monoxide of varying the concentration of potassium hydroxide from

0.05~N to 4 N, and the pyrogallol from 0.5 to 10 grms. per 100 c.c., is shown in this table:

Carbon	monoxide	evolved	on	oxvgen	absorbed	(without	agitation).
• • •				711.78-11		(** **********************************	

Normality of	Concentration of pyrogallol per 100 c.c.								
potassium hydroxide.	0.5 grm. Per Cent.	1·0 grm. Per Cent.	2·5 grms. Per Cent.	3·5 grms. Per Cent.	5·0 grms. Per. Cent.	10·0 grms. Per Cent.			
0.05		0.1	0.7		0.4				
0.10		0.6	0.4						
0.20	$3 \cdot 1$		0.55	0.4	0.4				
0.25			0.65						
0.40	3.5	3.3	0.95	0.75	0.5				
0.67			$3 \cdot 2$	$2 \cdot 65$					
0.80	3.75	3.65	$3 \cdot 4$	$3 \cdot 2$	0.9	0.55			
$1 \cdot 2$	_		$4 \cdot 6$	3.8		_			
1.5	6.5	6.0	$5\cdot 2$		3.2	0.85			
$2 \cdot 2$		8.5	$7 \cdot 1$						
2.5				7.0	_				
3.0		9.9	7.9	7.3	6.7				
4 ·0		8.5	7.5		5.9				

It is seen that the carbon monoxide evolved increases with the concentration of the alkali, but decreases with the concentration of the pyrogallol. If curves are plotted of percentage carbon monoxide evolved against normality of alkali, peculiar shapes of curve are obtained, which show no stoichiometrical relationship between the two functions. Agitation during oxygen absorption decreases the evolution of carbon monoxide in solutions of higher concentration, but not in concentrations below $0.6\ N$ alkali and $2.5\ grms$. of pyrogallol per $100\ c.c.$ An absorption made with agitation in a very old solution was always found to give a lower percentage of carbon monoxide than an absorption made without agitation in a fresh solution of the same composition.

Iodimetric Determination of Chromium (Chromic Oxide) in Chrome Alum. J. E. S. Han. (J. Amer. Leather Chem. Assoc., 1929, 24, 124.)—In the ordinary determination of chromium by oxidising the potash chrome alum solution with sodium peroxide, followed by the decomposition of excess and treatment with acid and potassium iodide, two sources of error are possible. One is the presence of iron in the chrome alum which will give results too high by liberating iodine, and the other is insoluble matter in the peroxide, which, on treatment with acid, will produce hydrogen peroxide, decomposing its equivalent of chromic acid, thus giving results too low. Both errors may be overcome by filtering the oxidised, well-boiled alkaline solution before acidification. With chrome alums containing as much as 0.5 per cent. of ferric oxide one filtration is found to be enough, the amount of chromium co-precipitated with the iron being negligible. The amount of iron in the precipitate may, if required, be readily determined after washing it free from sodium chromate.

R. F. I.

Determination of Bismuth. G. J. Hough. (Chemist Analyst, 1929, 18, 3-4.)—A solution in aqua regia of 0.5 grm. of the sample is neutralised with ammonia, an excess of ammonium sulphide added, and the precipitate filtered after 5 minutes on the water bath. It is then washed, dissolved in 10 c.c. of nitric acid and 50 c.c. of hot water, and the bismuth reprecipitated from the filtered solution by ammonia, filtered, redissolved in dilute sulphuric acid, and boiled for about 30 minutes with a small square of aluminium foil. When reduction is complete the aluminium is removed, the metallic bismuth dissolved in 10 to 15 c.c. of warm, saturated ferric chloride solution, and titrated in the presence of 200 c.c. of cold water and 5 c.c. of syrupy phosphoric acid (sp. gr. 1.7) with 0.1 N potassium permanganate solution standardised under similar conditions. Lead in small quantities may be removed as sulphate. Satisfactory agreement with the gravimetric and molybdate methods was obtained with ores containing antimony, arsenic, copper, and silver.

J. G.

Action between Copper Salts and Glycerol. B. K. Vaidya. (Nature, 1929, 123, 414.)—Vigorous action occurs when glycerol solutions of copper salts are heated to 150–200° C., the salts (except cupric chloride) being decomposed into fine metallic copper of over 99 per cent. purity and free acid, which may decompose further. In the case of cupric chloride, crystalline cuprous chloride is quantitatively formed, probably as the result of a secondary reaction. Almost the same result is obtained with other polyhydric alcohols, such as glycol, erythritol, and mannitol. It appears likely that a copper compound of the type $C_6H_{10}O_6Cu_3$ is first formed and later undergoes decomposition into copper, carbon dioxide, methane, and, possibly, ethane. The reaction serves for the preparation of copper highly suitable for catalytic purposes, even crude copper sulphate giving a good product.

T. H. P.

Sodium Alizarinsulphonate as a Reagent. F. G. Germuth and C. Mitchell. (Amer. J. Pharm., 1929, 101, 46-52.)—The reagent (0·3 c.c. of 0·5 per cent. solution) was added to 5 c.c.-portions of one per cent. solutions of metallic chlorides or nitrates. Precipitates of some shade of red were obtained in the case of lead, mercury", bismuth, copper, antimony, tin", iron", cobalt, magnesium, aluminium, and platinum". The precipitates with cadmium and tin were orange; chromium, yellow; iron", smoky black; uranium, deep violet; thallium, dark blue; and titanium", black. Soluble compounds of a red shade were obtained with barium, strontium, calcium, zinc, nickel, and gold solutions. The same precipitates were obtained at a dilution of 0·01 per cent. of the reacting salts, by addition of 0·1 c.c. of reagent and one drop of 5 per cent. ammonia. Most of the metals still gave a precipitate at a concentration of one part of salt in 1,000,000 of water.

Detection of Vanadium. A. Fölsner. (Chem. Zeit., 1929, 53, 250.)—
The hydrogen peroxide test was found to be unreliable for minute quantities (cf. ANALYST, 1926, 595). The author gives preference to lead acetate as a reagent.

For the detection of vanadium in steel, the aqua regia solution is evaporated to dryness, the silica filtered off, and the iron precipitated with an excess of caustic soda. The alkaline filtrate is acidified with acetic acid, and a solution of lead acetate added; a faint but distinct turbidity was obtained at a concentration of 0.03 grm. V. per litre (but cf. Evans and Clarke, ANALYST, 1928, 53, 475).

W. R. S.

Physical Methods, Apparatus, etc.

Absorption Spectra and Fluorescence of Fats. H. P. Kaufmann. (Chem. Umschau, 1929, 36, 34-35.)—The recent advances in the subject are discussed with special reference to a paper by Sproesser (id., 1928, 35, 325) and to the author's step photometer (Z. angew. Chem., 1928, 41, 1123), which is recommended for the quantitative measurement of luminescence. It is concluded that absorption spectra measurements are of little use for the identification of foreign substances in cacao butter, though they may prove of service with pure substances. For example, it was shown that the selective absorption of the elaeostearic acids was of a distinctly different type from that of linolic acid; therefore the former does not contain two unsaturated linkages (cf. Andant, Compt. rend., 1927, 184, 1068; and Weiss, Analyst, 1929, 178).

J. G.

Fluorescence of Colouring Matters in Ultra-Violet Light. A. Seyewetz and J. Blanc. (Comptes rend., 1929, 10, 714-715.)—Colours showing in aqueous or aqueous-alcoholic solutions a clear and characteristic fluorescence fall into the following classes: diphenyl methane derivatives; auramines and pyronines; triphenylmethane derivatives; phthaleins and some rosaniline derivatives; thio-benzenylic derivatives; primuline and thioflavines; quinoline and acridine derivatives; quinoline yellow and red, and acridine yellow and orange; fluorescent compounds resulting from diazotising and interacting of already fluorescent substances. Fluorescence of halogen derivatives of fluorescein diminishes from chloride to iodide, and with the number of substituted halogen groups. For a given colour fluorescence varies according to the solvent. The fluorescence of dyed fibres diminishes rapidly with increase of colour fixed, and varies in colour with the nature of the fibre.

D. G. H.

New Melting-point Apparatus. F. Kerchow. (Chem. Zeit., 1929, 53, 219.)—This is a modification of Hosking and Short's apparatus (ANALYST, 1926, 51, 270). The bulb of the thermometer, with the capillary-tube containing the substance, dips into a small paraffin-oil bath, as only in this way can identity of temperature between substance and thermometer be ensured. The vertical tube in which the thermometer is suspended is surrounded by an air-jacket open at the bottom, and the steady air-current is heated by a coil of resistance wire, having a resistance of about 7 ohms, inserted in the horizontal portion of the tube. The heating current is controlled with the help of an ammeter and an external resistance,

so that, when the melting-point is approached, the rate of temperature-rise may be adjusted to 1° in one or two minutes. With an air-current of 950 litres per hour and a heating current of 6 amperes, 100° C. is reached after 4 minutes and 200° C. after 10 minutes; with 7 amperes, 300° C. is reached in 16 minutes. After shutting off the heating current, the temperature rises a further 30° from 100°, 11° from 200°, or 5° from 300° C. The current necessary to cause a rise in temperature of 1° in 1–2 minutes is 1·5 amperes at 50°, 2·8 at 100°, 4 at 200°, or 6 at 300° C. These data should be determined for the particular apparatus used, so that the initial current of 6 or 7 amperes may be shut off at the right time and the proper current to give the slow temperature-rise started when the afterheating has nearly ceased.

T. H. P.

Reviews.

DIE CHEMISCHE ANALYSE. Edited by Dr. B. M. MARGOSCHES. Vol. XXVI. DIE VISUELLE LEITFÄHIGKEITSTITRATION UND IHRE PRAKTISCHEN ANWENDUNGEN. Prof. Dr. G. JANDER and Dr. O. PFUNDT. Pp. 64+viii. Stuttgart: Ferdinand Enke. 1929. Price 9s.

There are two classes of electrometric titration, the potentiometric method and the conductometric method. In the former the change in potential of a suitable electrode immersed in a solution containing its ions is determined after the addition of various amounts of reagent, whilst in the latter it is the change in resistance or conductivity of the solution that is measured. Potentiometric titration is of course widely used, but its limitations and pitfalls are only too familiar to those who use it, and it is surprising that conductometric measurements are not more extensively adopted as supplementary methods, or, in certain cases, even as alternatives. This apparent neglect is probably due to the fact that since, in order to avoid electrode polarisation, alternating currents must be used for conductivity measurements by the Wheatstone bridge method, an A.C. instrument is required to indicate the point of zero current. A.C. indicating instruments are usually less sensitive than the D.C. type, and though the ordinary telephone may give quite accurate results it has certain obvious disadvantages, particularly when rapidity is required. The popularisation of wireless, however, has recently led to the production of crystals and rectifying valves as commercial articles, and by rectification of the current by these or similar means the ordinary sensitive D.C. indicating instruments may be used. Hence the so-called "visual method."

This small volume, which is the twenty-sixth of the series on Chemical Analysis edited by Dr. B. M. Margosches, deals with this important method in a clear and concise manner. The advantages of conductometric over potentiometric titration in certain cases and the limitations of the telephone method are

indicated, and useful hints are provided for the construction of an apparatus with the maximum sensitiveness. The author favours the iron-constantan thermocouple by means of which the heating effect of an A.C. current is translated into millivolts. Nevertheless, more space might well have been devoted to the valve rectifier, as this is widely used. The crystal detector, also, in spite of its instability, provides a cheap and efficient method for rectification, and some recommendation as to the best type for the purpose would have been welcome. Carborundum is preferred by many workers. An induction coil is, of course, the obvious means of providing A.C. current, but where a steady supply is required an oscillator is very suitable, and a description of one of these instruments would also have been very useful.

After a short chapter on the choice of reagents and the influence of impurities, a number of typical titrations are described. These include acidimetry, alkalimetry, the determination of iron, and of chlorides and sulphates (e.g. in drinking water). Ammonium salts (e.g. in fertilisers) may be titrated with a strong base by this method, and conversely, salts of weak acids may be displaced by strong acids. The determination of potassium is dealt with at length, and the method appears rapid and sensitive, and may be used in the presence of sodium. An attempt is also made to apply the method to the routine testing of milk, though the more familiar determination of the ash content of sugar solutions is not mentioned.

On the whole, it seems that, from an analytical point of view, great possibilities exist for applications of conductometric titration. At present, however, apart from straightforward determinations of single substances, the analyst will usually have to work out the procedure or standardise the apparatus for a particular case. The volume will perform a service in bringing before the analyst a somewhat neglected aspect of electrometric titration.

The book is rather expensive for its size, though the high standard of production of the other volumes of the series is maintained. A misprint occurs on p. 43, where the ferric ion is represented as Fe...

Julius Grant.

APPLIED CHEMISTRY. By C. K. TINKLER and H. MASTERS. Volume I. WATER, DETERGENTS, TEXTILES, FUELS, ETC. Second edition, revised. xi+296. With 34 illustrations and 2 plates. London: Crosby, Lockwood & Son. 1929. Price 15s. net.

This volume, the previous edition of which was reviewed in this Journal (1920, 45, 346), is intended as a textbook principally for the use of third-year students of chemistry who propose taking a degree in public health or household and social science. The principal title is perhaps somewhat misleading, since it is suggestive of an enormously greater field than that covered by the text.

The subject-matter comprises an admirable selection of methods for sampling, and for the chemical and physical analysis of a variety of materials from a hygienic

and economical standpoint, together with concise theoretical explanations where necessary. That the authors possess a wide knowledge of the students' requirements is evident from their judicious experimental instructions, which are so complete that but little assistance from the demonstrator should be necessary, even in such a determination as the calorific value of coal.

Among other notable features of the volume are the numerous references to other pages and to various textbooks and reports in which fuller information may be found.

Much care has been expended on the elimination of errors both from the text and the index, with the result that very few adverse criticisms are called for, but in practice little difference in the result is obtained whether the temporary hardness of a water is determined by soap solution or by titration with standard acid, although a sentence on p. 25 tends to contradict this. The estimation on p. 40 is liable to yield low results owing to partial solution of the silica on addition of water after evaporation to dryness. Heating of the residue to 250° C. is essential if this is to be avoided. In Fig. 3 the small tube C would hardly contain sufficient dilute acid to decompose the 1 grm. of solid used for the estimation, unless the remainder of the apparatus was inconveniently bulky. Apart from a very few insignificant typographic errors, the remainder of the volume is free from fault, although some readers may take exception to the term "microphotographs" being used for the very excellent series of photomicrographs depicting textile fibres on the two plates. The former expression is generally applied to minute photographs of large objects. The volume is altogether an extremely useful and valuable production, the typescript, illustrations, and index being complete and reliable, whilst the general style throughout is a testimony to the care expended upon it by both the authors and publishers. To the reviewer the only undesirable and practically useless feature present is the 16 pages of advertising matter inserted at the end of the volume where the index should be placed.

T. J. WARD.

CHEMISTRY IN MEDICINE. Pp. xxii+757. 8vo. New York: The Chemical Foundation, Inc. 1928. Price \$2.

This book is the result of a co-operative effort among nearly 50 biologists, chemists and medical men, to present to the American public "the great possibilities for advance in medical science through further intensive co-operation between chemistry and medicine." It is hoped by this means to produce chemical uplift in the non-Elect and that the uplifted will duly see to it that still more dollars are devoted to this kind of research. It may seem odd to men of science in poverty-stricken Europe, who read so frequently of what seem to them magnificent donations to universities and similar bodies in the United States, that these institutions should need more money, but Dr. J. Stieglitz, the editor of this volume, and his five associate editors, assure the reader that this is the case.

This is by far the best of a number of books written in recent years to explain to the ordinary man what science, and especially chemistry, has done for him. In the reviewer's experience the ordinary man remains blissfully ignorant of the existence of such literature, even when he is "high-brow" enough to read and appreciate H. G. Wells, de Kruif, Sinclair Lewis, Pierre Hamp, or Aldous Huxley, whose works sometimes carry a scientific atmosphere, and even upon occasion bristle with scientific terms.

One can imagine those figments of Mr. Sinclair Lewis's imagination, "Mr. Babbitt" and "The Man who knew Coolidge," struck by the caption "Heredity and Development" in this volume, starting to read Prof. Alexander Weinstein's interesting, but rather technical article, on this subject. In the fifteenth line they meet the word "cytoplasm," and in the 17th the term "chromosomes." Will they consult the glossary, which the editors have thoughtfully provided, and carry on, or will they give up in despair? This is not a criticism of Dr. Weinstein's article; it is a mere statement of the difficulty he and his colleagues have made a gallant effort to overcome. This effort may fail in its immediate objective of reaching the general public, but it may still produce great effects indirectly, for the book can hardly fail to interest chemists, biologists and medical men in the necessity of somehow or other finding means of awakening public concern in these matters.

The book is no mere recital of the successes achieved by chemists in the production of synthetic drugs, though these receive due attention, but covers such fundamental matters as the work of the physiologist and the biochemist on heredity and development, metabolism of the body, problems of nutrition, dietary diseases, and hormones; and the work of the chemist in safeguarding water and food supplies, devising means for the disposal of sewage and providing protection against industrial disease. In a series of fascinating articles constituting the last two sections of the book, the present position of work on the great parasitic diseases, malaria, amoebic dysentery, leprosy, hookworm, syphilis and tuberculosis is outlined, and Dr. Voegtlin explains why chemotherapy is a hope of mankind.

The articles are not all of equal merit; they overlap here and there, and some of their authors are unduly impressed with American achievements which do not look quite so important on this side of the Atlantic, but taken altogether, the editors and contributors have done their work admirably, and if they fail to reach and interest the general public it is not for want of earnest endeavour on their part.

British readers will note with pleasure the tributes paid to the work of many of their countrymen throughout the book, and when Dr. Voegtlin points out that chemistry is called upon to save Africa they will no doubt remember that a good slice of Africa is included in the British Empire, and that it is their job to supply the chemistry.

T. A. HENRY.

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