

THE ANALYST.

OBITUARY.

ARTHUR GARFIELD LEVY.

ARTHUR GARFIELD LEVY was born in America on September 22, 1881. After two years at a *gymnasium* in Germany, he came to England and studied chemistry at Finsbury Technical College, taking his diploma in Applied Chemistry in 1899. For the next ten years he was assistant to Mr. Bertram Blount. Then he returned to America and took a position with Emeritus Professor Henry M. Howe, in the Department of Metallurgy, School of Mines, Columbia University, New York.

He was a Fellow of the Institute of Chemistry, and Bachelor of Science (First Class Honours) of London University. He was elected a member of this Society in April, 1903. From 1900 to 1909 he was an abstractor for the ANALYST, in which capacity his knowledge of German, which he spoke fluently and read easily, proved very useful. He also contributed several useful and important papers to the Society: "On the Analysis of Samarskite" (ANALYST, 1901); "A Rapid Method for the Determination of Tin in Copper-Tin Alloys" (1905); "Water from the Simplon Tunnel" (1905); and, with Mr. Bertram Blount, "The Use of Quartz Combustion Tubes, especially for the Direct Determination of Carbon in Steel" (1909). He returned to the last subject in a later paper, "The Estimation of Carbon in Steel" (1912). Under the Society's Analytical Investigation Scheme, he carried out an investigation on "The Estimation of Niobium in Presence of Tantalum, and some Reactions of Tantalum Compounds" (ANALYST, 1915). The care and labour he expended on this proved that absence from England had in no way lessened his affection for the Society.

Levy's knowledge of inorganic chemistry was extensive and coherent, his memory was retentive, and his aptitude for concentration was remarkable. Socially, his charm of manner and unflinching courtesy endeared him to all his contemporaries. The cordial remarks from the Chair and other speakers that invariably followed a paper from him are eloquent testimony of the confidence of the older practitioners in him. To him was entrusted the preparation of the second edition of Blount and Bloxam's "Chemistry for Engineers and Manufacturers" (Part II.).

To the deep personal regret that we feel at his death, which took place on July 12, 1916, is added the knowledge that professional chemistry is much the poorer through the loss of one for whom the future held such promise.

W. PARTRIDGE.

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

THE ESTIMATION OF PENTOSE OR PENTOSANS BY MEANS OF FEHLING'S SOLUTION.

By JULIAN L. BAKER, F.I.C., AND H. F. E. HULTON, A.I.C.

(Read at the Meeting, June 7, 1916.)

SOME years ago J. T. Flohil (*Chem. Weekblad*, 1910, 7, 1057-1063; *ANALYST*, 1911, 36, 161) described a method dealing with the estimation of pentose and pentosans involving the use of Fehling's solution. In a note appended to an abstract of the paper (*loc. cit.*) the method was criticised somewhat severely, but, in our opinion, quite legitimately. The process was, however, of sufficient interest to merit further investigation, and Eynon and Lane communicated to this Society an interesting paper recounting their experiences with the method (*ANALYST*, 1912, 37, 41). A very full and careful study was made of the action of furfural solutions of different concentrations upon Fehling's solution, also the effect of varying quantities of sodium chloride, resulting from the neutralisation of the hydrochloric acid with soda, upon the amount of reduced copper oxide.

We have had occasion to make use of Eynon and Lane's method for the estimation of pentoses and pentosans in malt, barley, wort, beer, etc. A considerable number of results have been collected, and a consideration of some of the points connected with the method may not be without interest.

The ordinary procedure as described by Tollens and his pupils for the distillation of the raw material with hydrochloric acid was followed. An aliquot portion of the dilute solution of furfural in hydrochloric acid thus obtained is neutralised and then boiled with Fehling's solution. The cuprous oxide, after being filtered on to a Soxhlet tube, is either dried and weighed as Cu, Cu₂O, or as CuO.

We are able to confirm the values found by Eynon and Lane for the copper oxide equivalent of pure furfural, but do not quite agree with their figures for the copper oxide yielded from 20 c.c. of Fehling's solution when heated with salt alone. The determination of this value, which is employed as a "blank," is of considerable importance, since, when the quantity of furfural present is small, it constitutes a considerable percentage of the total copper obtained; and while we agree fairly well with Eynon and Lane's figures for the lower quantities of salt, we diverge considerably when the amount of salt present is that usually obtained by the neutralisation of the acid distillate. The following values were obtained:

		SALT (NaCl) IN GRMS.												
		0	4	5	6	7	8	9	10	11	12	13	14	
CuO in mgrms.	{	6	9.3	10	10.6	11.3	11.7	12.2	12.7	13.3	13.8	14.2	14.8	Eynon and Lane Baker and Hulton
		9	10	10.5	11	12.5	14	16	20	20	20	21	22	

It must be remembered that the estimations made by Eynon and Lane were all obtained by diluting an aqueous solution of pure furfural to varying degrees, and then boiling such solutions with Fehling's solution and known amounts of salt. They cite values for the copper oxide obtained when the weight of furfural used varied from 10 to 50 mgrms. When, however, the furfural solution to be estimated has been obtained by distilling a material with hydrochloric acid the concentration of furfural in the distillate is usually very low, and in no case have we found such concentration of furfural to exceed 50 mgrms. per 100 c.c., and it may be as low as 10. Since only 60 or 70 c.c. is actually used for the analysis, it will be seen that the higher values which these chemists have recorded are not required.

The distillation of the material with hydrochloric acid (sp. gr. 1060) is carried out in the customary manner in a Wurtz flask of about 300 to 350 c.c. capacity, until 30 c.c. have passed over. Another 30 c.c. of the acid is then introduced through a tapped funnel, and a further 30 c.c. distilled, and the distillation continued until the whole of the furfural is collected, the concentration of acid in the flask being thus kept constant. The usual volume collected when the furfural is intended to be precipitated by phloroglucinol or phenylhydrazine is 400 c.c., and Flohil (*loc. cit.*) recommends that this volume be collected; but in our practice we have usually been able to stop the distillation short of this—in fact, as soon as a few drops of the distillate fail to give the coloration, characteristic of furfural, with aniline acetate.* It may be pointed out that in testing the distillate with the aniline acetate solution it is essential to have sufficient sodium acetate present to insure the only free acid being acetic, the red coloration not being produced in the presence of strong hydrochloric acid. Working in this way with about 1 to 2 grms. of material, or 20 to 30 c.c. of solution, it is usually found that the whole of the furfural is distilled when some 200 to 300 c.c. of the distillate have been collected, but the rate of liberation of furfural would appear to vary not only with the actual amount of furfural-yielding material, but also with its nature, and in the case of some aqueous extracts of malt and barley a somewhat larger volume of distillate than this may have to be collected. It is advisable to collect the distillate in a graduated cylinder having a small funnel, so as to remove traces of solidified volatile fatty acids which pass over. Towards the end of the distillation the aniline acetate test must be given time to develop,

* Pure aniline 10 c.c., glacial acetic acid 10 c.c., alcohol (80 per cent.) 80 c.c.

nor should it be concluded that the whole of the furfural is over unless the distillate gives no coloration after standing for at least five minutes.

When the whole of the distillate is collected the acidity is estimated on 10 c.c., using $\frac{N}{2}$ caustic soda. Usually between 8 and 12 per cent. hydrochloric acid is present, and from the acidity the amount of sodium chloride which will be present in the aliquot portion of the neutralised distillate used for the Fehling estimation may be calculated. As 20 c.c. of Fehling's solution in a total volume of 100 c.c. are used for reduction, a certain margin for variation in the amount of the distillate to be analysed is possible.

A suitable amount will usually be found to be from 50 to 70 c.c., the volume of which when neutralised with saturated caustic soda solution will generally be about 75 c.c., thus leaving 5 c.c. to be made up with water and 20 c.c. of Fehling's solution.* This mixture is then placed in a conical flask of about 250 c.c. capacity, connected with a vertical condenser, and heated at such a rate on a small Fletcher burner that the solution will boil in from twelve to fifteen minutes. Boiling is continued for exactly thirty-five minutes from the beginning of ebullition; at the end of this time the liquid is filtered through a Soxhlet tube and the cuprous oxide washed and dried at 100° and weighed as Cu_2O , or reduced to copper as in Eynon and Lane's paper, or, as we prefer, ignited in a current of air and weighed as CuO . The boiling must not be done on wire gauze, but on a piece of asbestos millboard or uralite at least 6 or 7 inches square. Even with this precaution it often happens that a brown film forms on the inner side of the flask above the level of the solution, and this is removable with difficulty.

Limits of Accuracy.—The ratio $\frac{\text{Furfural}}{\text{Copper}}$ ranges from .3 at the lower end of the scale to about .32 at the upper; that is to say, 3 mgrms. CuO represent approximately 1 mgrm. of furfural.

It has been found that working on the same material the variation in the weight of CuO obtainable will not usually exceed 2 to 3 mgrms. This is equivalent to about 1 mgrm. of furfural, and if, for example, 1 gm. of material is being used, and a quarter of the total distillate be employed for the copper determination, the furfural obtained will require multiplying by 400 in order to obtain the percentage on the material. If, as usually happens, about 50 mgrms. of CuO are weighed, an error of 5 per cent. on the final value may be introduced.

Suggestion for the Improvement of the Method.—There are two directions in which we think the method is capable of distinct improvement. (1) The employment of a much larger volume of Fehling's solution, in order that a considerably larger fraction of the total distillate may be treated with Fehling's solution, thus increasing the weight of CuO obtained, and so decreasing the error. (2) In the method of heating the Fehling solution. If instead of boiling the solution, as already described, the conical flask is immersed in a boiling water-bath, it will be found that the copper oxide obtained from salt and Fehling solution alone (the "blank") falls in a remarkable manner, thus:

* Eynon and Lane recommend neutralisation with sodium carbonate, but owing to local heating and effervescence serious loss of the extremely volatile furfural may take place. The calculated volume of saturated caustic soda solution is added in small quantities, care being taken to keep the solution cool.

Grms. Salt.	Mgms. CuO.		Water-bath Method.
	Eynon and Lane.	Baker and Hulton.	
0	6	9	2
7	11.3	12.5	3
10	13	20	4

The furfural-copper value *per se* is unaffected, and confirms the finding of Eynon and Lane, who state that the action of furfural on Fehling's solution is independent of the salt concentration. It also shows that the reaction with furfural proceeds to the same extent whether Fehling's solution is actually boiled or is merely heated to 100° C. The advantage of employing the boiling water-bath hardly needs emphasising, if for no other reason than the great reduction obtained in the "blank" correction, as when only a small weight of copper oxide is obtained the "blank" copper may amount to as much as 25 or 30 per cent. of all the copper weighed. It would appear probable that the large spontaneous reduction of Cu_2O when the salt and Fehling's solution are boiled over a flame alone is due to the raising of the boiling-point of the mixture. This, of course, is avoided in the water-bath method.

DISCUSSION.

Dr. DYER said, in reply to a question, that such work as had been done on the subject of the digestibility and food value of pentosans and pentoses indicated that such bodies had a food value as oxidisable or heat-giving matter, but he did not think there was any evidence of their functioning as direct builders of fat in the way that sugar and starch did.

Mr. C. REVIS said that soon after the publication of Messrs. Eynon and Lane's paper he had made some experiments which bore out the present authors' good opinion of the method. The boiling was carried out in a water-bath, which certainly seemed preferable to using a plate.

Mr. E. R. BOLTON asked if there was any special reason for using a Soxhlet tube in preference to a Gooch crucible, in which the oxidation could be carried out much more conveniently.

The PRESIDENT remarked that the difficulty due to heating of the upper part of the flask might be overcome by dropping a ring of asbestos over the neck of the flask.



SOME NEW OIL-SEEDS DERIVED FROM AMERICAN PALMS.

BY G. T. BRAY AND F. L. ELLIOTT.

DURING recent years considerable interest has been aroused from time to time in certain oil-seeds indigenous to South and Central America. These seeds are most often derived from one or other of the numerous species of palms. The Cohune nut (*Attalea Cohune*) has probably received most attention, and numerous attempts have been made to solve the difficult problem of shelling the nuts by machinery, so as to obtain the kernels in sufficiently large quantities for use as a source of oil on a commercial scale.

Samples of a number of these American palm kernels have been examined recently at the Imperial Institute, and in view of their probable importance in the future as sources of edible fats and feeding cakes, the results are now published.

"*Babassu*" Kernels.—These are derived from a species of *Attalea*, possibly *A. funifera*, Mart., allied to the Cohune palm (*Attalea Cohune*, Mart.), and have been received from Brazil under the above name and also under the names "Coco Babassu" and "Bassoba." Considerable quantities of the kernels have been exported lately from Para in the state of Maranhão, where the tree is abundant (*Dipl. and Cons. Repts., Ann. Series, Cd. 5526: Para Consular District, 1914, p. 26*). In 1915 nearly 1,200 tons of kernels were exported; of these, the greater part appears to have been obtained by hand shelling, the nuts being placed on end in a hole in a board and struck with an axe; but machinery for shelling the nuts is also being used.

The kernels have been crushed on a fairly large scale, and there seems to be every prospect of increased supplies being brought to this country. The kernels are stated to have been sold at about two pounds per ton less than copra.

The fruit is very similar in appearance to Cohune palm fruit (see *Bulletin of the Imperial Institute, 1913, 11, 226*), weighs on the average about 45 grms., and consists of an outer fibrous pericarp, enclosing a hard-shelled nut containing several kernels. A single fruit examined at the Imperial Institute contained five kernels, but from the size and formation of the kernels as exported from Brazil, it seems that this is an unusually large number.

The kernels are reddish-brown, and of a characteristic elongated shape pointed bluntly at the ends; they weigh on an average about 3 grms., and are 40-50 mm. long and about 13 mm. broad. The kernels are easily distinguishable from Cohune kernels, which are shorter and rounder.

The fat is fairly hard, cream-coloured, and resembles palm kernel and coconut oils in general appearance; in chemical character it is most closely allied to palm-kernel oil, and is similar, as would be expected, to Cohune kernel oil, although its iodine value is somewhat higher. The figures obtained from the examination of several samples of Cohune oil are quoted in the table of analyses for purposes of comparison. The results of examination of the residual meal show that it should possess a feeding value about equal to that of coconut cake and somewhat superior to that of palm-kernel cake.

“*Tucan*” or “*Large Panama*” Nuts.—“*Tucan*” or “*Tucum*” nuts are probably derived from *Astrocaryum vulgare*, Mart., and the “*Large Panama*” nuts appear to be identical. Another sample of nuts evidently derived from a different species of *Astrocaryum* was received under the name of “*Tucuma assu*,” but was unfortunately too small for examination.

The “*Tucan*” kernels weighed on an average 3.6 grms. and measured 18.25 mm. in length and 13.18 mm. in diameter; the outer skin was smooth and brown, and the white flesh harder and tougher than that of palm kernels or copra; in fact, the kernels were so hard that extraction of the oil was slow and difficult, and it seems quite possible that the kernels may prove troublesome to work on account of their hardness.

The fat extracted from “*Tucan*” kernels was cream-coloured and fairly hard; in chemical character it resembled palm-kernel oil, but contained less soluble volatile acids and had a somewhat higher melting-point. The residual meal contains only 10 per cent. of protein, and is obviously inferior to coconut or palm-kernel meal; although the meal yields only about 10 per cent. of “*crude*” fibre, the tough nature of the kernels seems likely to render the residual meal somewhat indigestible. The kernels are stated to have sold at from one pound to two pounds less per ton than fine palm kernels.

“*Paraguay*” Kernels.—These kernels appear to be derived from a species of *Acrocomia*, and are probably the source of “*Mocaya*” or “*Mbocaya*” oil, which, according to Lewkowitsch (“*Chemistry and Technology of Oils, etc.*,” [1914], vol. ii., p. 607), is derived from the kernels of *Acrocomia sclerocarpa* in Paraguay; “*Paraguay*” kernels are smaller than, but otherwise indistinguishable in appearance from, “*Gru-gru*” kernels (*Acrocomia sclerocarpa*), from the West Indies, but the Paraguay kernels contain a somewhat higher percentage of fat of decidedly softer consistency and higher iodine value. In view of our limited knowledge of South American palms and the difficulty of identifying the species from the seeds or fruits alone, it is quite probable that “*Gru-gru*” and “*Paraguay*” kernels are not identical in origin.

The “*Paraguay*” kernels are roughly spherical, about 12 mm. in diameter, and weigh about 1 gm. each; the skin is almost black and the flesh much softer than that of ordinary oil-palm kernels (*Elæis* sp.). The fat is decidedly softer than either coconut or palm-kernel oil, being only semi-solid at ordinary temperature, and the iodine value is higher than that of either of these oils, or than that of the oils derived from the other kinds of palm-kernels examined. The residual cake of “*Paraguay*” kernels is even richer in protein than coconut cake, and should have therefore a high feeding value. The kernels are stated to have sold recently in Liverpool at prices between those of fine palm kernels and copra.

“*Cokerite*” Kernels from *British Guiana*.—These are derived from *Maximiliana regia*, Mart. The fruits are somewhat similar to Cohune fruits, but more pointed and smaller, and had the following dimensions:

	Fruits.	Nuts.	Kernels.
Average weight, grms.	10.6	7.5	1.3
„ length, mm.	40-50	40-60	25
„ diameter, mm.	20-25	12-20	12

The fruits consisted of fibrous pericarp and bracts 29; shell 54; kernel 17 per cent.

	<i>Attalea</i> Species.	<i>Attalea Cohune</i> , Mart.	<i>Astrocaryum</i> Species. <i>A. vulgare</i> , Mart. (?)		<i>Acrocomia</i> Species.	<i>Acrocomia sclerocarpa</i> , Mart.	<i>Macramitana regia</i> , Mart.	For Comparison.*	
			<i>A. vulgare</i> , Mart. (?)	"Large Panama" nuts				<i>Elaeis guineensis</i> .	<i>Cocos nucifera</i> .
<i>Popular or native names</i>	"Bassaba" "Babassu" "Coco" Babassu	"Cohune" "Coyun" "Cahoun"	"Tucan" "Large Panama" nuts	Paraguay kernels	"Gru-gru" kernels	"Cokerite" kernels	Palm kernels	Coconut (Copra)	
<i>Origin</i>	Brazil	British Honduras	South America	South America	West Indies	British Guiana	West Africa	Tropics generally	
<i>Composition of kernels:</i>									
Moisture, per cent. . .	4.2	4.5	6.5	7.1	6.0	11.3	6.8	Generally about 5	
Oil in kernels as received, per cent.	67.2	65.4-71.6	48.6	37.6	65.2	56.9	48-51	63-70	
Oil in kernels dried at 100° C., per cent.	70.1	68.4-74.9	52.0	40.5	69.4	64.1	52-54	66-74	
<i>Constants of the oil:</i>									
Melting point ° C. † . .	26°	22°-24°	30.5°	—	—	27.8	26.29	23-26	
Titer test ° C. . .	23°	19.7° to 21.0°	27°	—	21°	24.2°	20.0-25.5	21.2-25.2	
Specific gravity at 100°/15° C.	0.868	0.868 to 0.871	0.867	—	0.865	0.867	0.873	0.874	
Acid value . . .	5.5	1.2-20.4	2.9	—	26.1	3.1	—	—	
Saponification value	249	252.4 to 256.5	249	250	247	253	245-248	260-262	
Iodine value, per cent. (Hübl, 17 hrs.)	15.6	11.0-13.7	11.6	—	28.5	13.0	14-17.5	7.9	
Unsaponifiable matter, per cent.	0.3	0.2-0.3	0.3	—	0.3	0.3	—	—	
Reichert-Wollny value	5.8	6.8-8.3	3.8	—	6.5	3.0	5.0-7.6	6.65-8.0	
Polenske value . .	10.2	12.5-15.4	5.9	—	10.2	7.0	10-12	15-20	

* Figures quoted are from Lewkowitzsch, "Oils and Fats", Bolton and Revis, "Fatty Foods", and from Imperial Institute Records.

† Open tube method.

‡ A sample of oil prepared in British Guiana gave similar results.

|| Five samples of oil examined.

||| Capillary tube method—complete fusion.

	<i>Attalea</i> Species.	<i>Attalea Cohuna</i> , Mart.	<i>Astrocaryum</i> Species. <i>A. vulgare</i> , Mart. (?)		<i>Acrocomia</i> Species.	<i>Maximiliana regia</i> .	For Comparison.		
			"Tucan"	"Large Panama" nuts			English-made Palm Kernel Cake.	English-made Coconut Cake.	
<i>Popular or native names</i> ..	"Bassoba" "Babassu" "Coco" "Babassu"	"Cohune" "Coyun" "Cahoun"	"Tucan"	"Large Panama" nuts	Paraguay kernels	"Cokerite" kernels			
<i>Origin</i> ..	Brazil	British Honduras	South America	South America	South America	British Guiana			
<i>Composition of residual meal (calculated to contain 7 per cent. of fat):</i>									
Moisture, per cent. ..	8.5	9.5	8.4	—	8.7	8.6	9.4	10.7	11.9
Crude proteins, per cent. ..	23.2	22.4	10.0	—	31.6	15.0	17.8	17.1	21.8
True proteins, per cent. ..	22.0	20.9	10.0	—	31.4	14.7	—	—	—
Other nitrogenous substances, per cent. ..	1.2	1.5	nil	—	0.2	0.3	—	—	—
Fat, per cent. ..	7.0	7.0	7.0	—	7.0	7.0	8.2	8.2	8.4
Carbohydrates, per cent. ..	45.9	40.0	62.9	—	35.5	52.5	50.6	51.1	42.6
Fibre, per cent. ..	10.6	16.1	9.5	—	11.7	12.6	10.1	9.5	9.4
Ash, per cent. ..	4.8	5.0	2.2	—	5.5	4.3	3.9	3.4	5.9
Nutrient ratio ..	1 : 2.7	1 : 2.5	1 : 7.9	—	1 : 1.6	1 : 4.4	1 : 3.9	1 : 4.1	1 : 2.8
Food units ..	121.4	113.0	105.4	—	132.0	108.0	116	114	118

The fibrous pericarp contained 12 per cent. of moisture and 15 per cent. of semi-solid oil resembling palm oil (*Elæis guineensis*) in character; this oil had saponification value 212, iodine value 51.4 per cent., and titer test 25.5° C.

The nuts usually contain two or three kernels, but sometimes only one is present.

The kernels are covered with a greyish-brown mottled skin, the flesh is whitish and resembles that of West African palm kernels in consistency. The kernel oil is cream-coloured and fairly hard, and resembles palm-kernel oil in its chemical constants, but yields smaller quantities of soluble and insoluble volatile acids. The residual cake contains only 15 per cent. of protein, and would have a feeding value inferior to that of palm-kernel cake.

Conclusions.—The kernels of the various species of palms dealt with in the present paper are, with the exception of those derived from a species of *Astrocaryum*, as rich in fat or even richer than those of ordinary West African palm kernels, enormous quantities of which are used as a source of oil and oil cake. They seem destined, therefore, to form valuable additions to the oil-seeds now utilised as sources of fat in the edible fat industry. The nuts of *Acrocomia* species and *Astrocaryum* species do not offer any particular difficulty in the way of exploitation, as the shells can be cracked by machinery such as is already used for ordinary palm nuts in West Africa (*Elæis guineensis*) (see *Bulletin of the Imperial Institute*, 1909, 7, 385).

In the case of Babassu and Cohune nuts the problem of extracting the kernels from the fruits is a difficult one. The fruits have a fibrous pericarp, the removal of which is generally regarded as necessary before the nuts can be cracked; further, the nut shell is thick and hard, and the usual presence of several kernels in Babassu nuts and the occasional presence of more than one kernel in Cohune nuts also tends to render cracking difficult. Several machines have been designed and patented both for removing the fibrous pericarp and for cracking the nuts, but comparatively little is known about their efficiency.

Hand-cracking is slow and laborious, but is, nevertheless, a method which has been employed for obtaining large quantities of Babassu kernels in Brazil, while enormous quantities of West African palm kernels are still obtained in this way.

The authors desire to express their thanks to Mr. R. G. Pelly and to Dr. J. R. Furlong for advice and assistance received in carrying out the work described in this paper.

SCIENTIFIC AND TECHNICAL RESEARCH DEPARTMENT,
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ANCIENT IRISH OAK.

By P. A. ELLIS RICHARDS, F.I.C.

By the courtesy of Professor A. Henry of the Royal College of Science for Ireland I have been enabled to examine some specimens of ancient Irish oak that in certain respects resemble the Russian oak described in my former note (ANALYST, 1916, 169). The portions of wood analysed were taken from various districts in Ireland, and their origin may be briefly described as follows:

The Kerry and Sligo oaks were taken from trees of which stumps exist *in situ* in margins of peat masses, the latter being several feet in thickness.

The Roscommon oak was obtained from a peat bog in that county.

The Annagasan specimen came from a log of oak in the county of Louth. It had been swept down by the river and was buried in sand and mud close to the sea near the river mouth.

Nothing is known of the age of the last-named oak, but the others in all probability date back to the Neolithic period, when, no doubt, wide tracts of peat-moss were covered with forest trees.

A comparison of the figures obtained from analyses of these specimens with those of the Russian oaks previously described shows that very similar changes in the mineral constituents originally present in the wood have taken place. The Kerry and Roscommon oaks are specially interesting in this respect, as it will be seen that in each of these cases the salts of the alkali metals have been almost entirely replaced by iron, calcium, and magnesium salts, whilst the mineral matter as a whole has largely increased. In no case was more than a trace of phosphate present.

In the Sligo oak the changes had evidently not proceeded so far, whilst the Annagasan sample was heavily impregnated with salt from its long contact with sea-water.

COMPOSITION OF WOOD.

Constituents.	Kerry (Black).	Roscommon (Black).	Roscommon (Grey).	Sligo (Brown).	Annagasan (from sea).
Total mineral matter ..	2.31	1.46	1.09	0.278	6.33
Per cent. soluble ..	13	8	6	56	90
Per cent. insoluble ..	87	92	94	44	10
Ether and alcohol extract	4.37	7.68	7.24	5.5	5.02
Water and total ex- tractives*	44.3	40.5	36.2	39.6	38.7
Cellulose	27.3	34.4	37.6	39.3	44.4
Lignin, etc.	28.4	25.1	26.2	21.1	15.9

* This figure includes moisture in addition to matter extracted by ether, alcohol, acid, and alkali.

COMPOSITION OF MINERAL MATTER.

Fe ₂ O ₃	62·70	33·90	25·80	3·95	0·37
Al ₂ O ₃	Trace	Trace	Trace	1·65	0·30
CaO	16·24	34·22	38·61	16·42	4·10
MgO	Nil.	0·80	1·02	15·28	7·27
SiO ₂	2·30	0·70	0·80	7·80	1·80
SO ₃	5·83	4·65	3·42	30·80	14·58
Cl	1·10	Trace	Trace	0·70	38·20
Total Na and K salts . .	3·30	Traces	Traces	11·70	68·20
CO ₂ (present as insoluble carbonates)	9·53	25·20	28·40	13·70	3·40

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Composition of the Fruit of *Cicer Arietinum L.* (Chick Pea). A. Zlatároff. (*Zeitsch. Unters. Nahr. Genussm.*, 1916, **31**, 180-183.)—The dry substance of the chick pea contains: Fat, 6·30; starch, 50·32; crude fibre, 3·62; total nitrogen, 3·34; protein nitrogen, 2·11; nuclein nitrogen, 0·10; ammonia nitrogen, 0·10; amide nitrogen, 0·01; amino-acid nitrogen, 0·12; total phosphoric acid (P₂O₅), 0·998; lecithin phosphoric acid, 0·142; protein phosphoric acid, 0·486; inorganic phosphoric acid, 0·118; organic soluble phosphoric acid, 0·244; total ash, 2·87 per cent. Oxalic acid (0·007 per cent.), citric acid, betain and choline (0·02 per cent. together), adenine, inositol, and an isomer of phytosterol, are also present. This isomer, which is termed *slanutosterol*, is present in the fat to the extent of about 0·3 per cent.; it yields an acetate melting at 128° C. W. P. S.

Analysis of Rancid Fats. G. Issoglio. (*Annali Chim. Applic.*, 1916, **6**, 1-18.)—A method of estimating the degree of rancidity of oils and fats is based upon the proportion of aldehydes, etc., liberated on distillation with steam under constant conditions, and measured by titration with permanganate solution. From 20 to 25 grms. of the sample are mixed with 100 c.c. of water and distilled in a current of steam, so that 100 c.c. of distillate are collected in ten minutes. Ten c.c. of the homogeneous distillate are then mixed with 50 c.c. of water, 10 c.c. of 20 per cent. sulphuric acid, and 50 c.c. of $\frac{N}{100}$ potassium permanganate solution, the mixture heated to the boiling point and kept boiling for five minutes in a flask connected with a ground-in condenser. After cooling, the liquid is treated with 50 c.c. of $\frac{N}{100}$ oxalic acid solution and titrated with $\frac{N}{100}$ potassium permanganate solution. If N represents the amount of permanganate required for the oxidation and *n* that

required in a blank test, and P the weight of fat taken, the *oxidisability value* of the fat may be expressed by the equation

$$X = \frac{(N-n) 80}{P}$$

Hence the *oxidisability value* represents the mgrms. of oxygen required to oxidise the organic compounds separated under constant conditions from the fat.

Speaking generally, the oxidisability value of sound fresh fats varies from about 3 to 10, while rancid fats show much higher values. The acid value does not stand in any definite relationship to the oxidisability value. For example, fifteen samples of fresh sound olive oils of different origin gave acid values ranging from 1.88 to 8.59, and oxidisability values of 3.20 to 10.45; while six samples of rancid olive oil showed acid values of 6.51 to 18.56, and oxidisability values of 14.62 to 59.10. When the oxidisability value exceeds 15 it will usually be found that the fat is rancid or has undergone some other change.

C. A. M.

Estimation of the Degree of Bolting of Flour. C. L. Spica. (*Annali Chim. Applic.*, 1916, 6, 26-28.)—Ten grms. of the dry fat-free flour are hydrolysed, the insoluble residue is distilled with hydrochloric acid of sp. gr. 1.06, and the whole of the furfural is collected. The distillate is mixed with 95 per cent. alcohol, so as to obtain a solution containing 50 per cent. of alcohol, and this is treated with an acetic acid solution of aniline acetate. The coloration produced is compared with that of a solution of 0.05 gm. of furfural in 1,000 c.c. of 50 per cent. alcohol. Practical tests with flour bolted to yield 60, 80, and 100 per cent. yielded solutions with colorations corresponding to 0.025, 0.1520, and 0.225 gm. of furfural respectively.

C. A. M.

Ester Acids of Lemon Juice. L. Wolfrum and J. Pinnow. (*Zeitsch. Unters. Nahr. Genussm.*, 1915, 30, 144-156.)—The authors have detected the presence of ethyl citric acid in lemon juice, the acid having been separated by fractional extraction in a Partheil-Rose apparatus and then precipitated as its calcium salt. The acid was also prepared by boiling together alcohol and citric acid (without a catalyst), and using the above-mentioned means for its isolation; the acid obtained in this way was identical with that isolated from lemon juice. The calcium, lead, and silver salts of the acid are but slightly soluble, and the acid itself saponifies at a very slow rate. The presence of an anhydride of the acid could not be detected.

W. P. S.

Estimation of Essential Oils (Essences) in Liqueurs. C. F. Muttelet. (*Ann. Falsific.*, 1916, 9, 70-73; 134-143.)—The quantity of essential oil in peppermint liqueur is best estimated gravimetrically. The liqueur is distilled, the distillate saturated with sodium chloride, extracted with petroleum spirit, and the residue of essential oil weighed after the solvent has been evaporated at ordinary temperature. The official (French) method, in which the quantity of essential oil is deduced from the iodine absorption of the sample, is not applicable to peppermint liqueur, since

it will not indicate the presence of added menthol; the latter does not absorb iodine. If, however, the essential oils are separated by means of petroleum spirit a determination of their iodine absorption will show whether or not added menthol is present. One grm. of oil of peppermint absorbs 0.450 grm. of iodine. The author has also estimated the essential oils in a number of other liqueurs by both the gravimetric and volumetric (iodine) methods; only in the cases of aniseed liqueur and caraway liqueur (Kummel) were agreeing results obtained by the two methods. With Curaçao, Bénédictine, Chartreuse, Angostura, and other bitters, the volumetric (iodine) method yielded untrustworthy results, as it was impossible to fix an average iodine value for the various liqueurs. Some of the liqueurs were coloured with caramel, and this introduced an additional difficulty in applying the volumetric method; the caramel absorbed iodine, and even when the samples were distilled and the method applied to the distillate, the results obtained were inaccurate owing to the presence of unsaturated (furfuraldehydic) compounds formed from the caramel.

W. P. S.

Estimation of Essential Oils (Essences) in Liqueurs. X. Rocques. (*Ann. Falsific.*, 1916, 9, 127-134.)—Methods which have been proposed for the estimation of essential oils in liqueurs were investigated by a Commission appointed by the Société des Experts-Chimistes de France. The volumetric method, which depends on the iodine value of the distillate from the liqueur, was found to yield accurate results only in the case of aniseed and caraway (kummel) liqueurs; as determined by the Hübl method, 1 grm. of aniseed oil absorbs 1.45 grms. of iodine, and 1 grm. of caraway oil 2.40 grms. of iodine. For all other liqueurs (Chartreuse, Bénédictine, mint, Curaçoa, orange bitters, etc.) the use of the gravimetric method is recommended; in this method the distillate from the liqueur is saturated with sodium chloride, and the essential oil extracted with petroleum spirit and weighed after the solvent has been evaporated at a low temperature. Well-known brands of liqueurs examined generally contained more than 0.5 grm. of total essential oil per litre, the maximum limit fixed by a recent French law; Curaçoa liqueur contained more than 2 grms. per litre, but vermouth was found to be free from any appreciable quantity of essential oil.

W. P. S.

Estimation of Essential Oils (Essences) in Vermouth. L. Ronnet. (*Ann. Falsific.*, 1916, 9, 144-145.)—Experiments carried out by the author show that vermouth is practically free from essential oils (see also *ANALYST*, 1916, 89). W. P. S.

Analysis of Maple Products. VII. J. F. Snell. (*J. Ind. and Eng. Chem.*, 1916, 8, 331.)—In continuation of previous work (*ANALYST*, 1914, 39, 85; 1916, 207) the author describes the conductivity test in its application to over two hundred samples of genuine syrups covering five seasons. Conductivity values ranging from 96 to 230 have been met with in genuine syrups, but the limits of percentage variation of the conductivity value in such syrups are much narrower than those of the older analytical values, with the exception of the volumetrical lead number. Conductivity values above 200 have only been met with in the

case of boiled syrups, while values below 110 have been found in genuine syrups of the year 1915, but not in the years 1911-1914. A new type of electrode (Van Zoeren, *J. Amer. Chem. Soc.*, 1916, **38**, 652) is now employed, necessitating the use of 20 c.c. of syrup in place of the 15 c.c. previously employed; this is mixed with 40 c.c. of distilled water and the resistance measured at 25° C. exactly with the dip electrode. The constant of the cell divided by the observed number of ohms and multiplied by 100,000 furnishes the conductivity number. H. F. E. H.

Fat of Egyptian Buffalo Milk. G. Hogan and E. Griffiths-Jones. (*Egyptian Dept. Public Health, Publication No. 5, 1916, 1-3.*)—Results of the examination of sixty-nine samples of buffalo butter-fat are recorded. The milk from which the butter was prepared was the mixed milk of five or six buffaloes, the animals being milked dry. Twenty-eight samples were taken during 1913, and the remainder during 1915. The maximum, minimum, and average analytical values obtained were as follows:

	Reichert-Meissl Value.	Polenske Value.	Saponification Value.	Iodine Value (Hübl).	Refractometer Value at 40° C.
Maximum	37.0	2.8	235	39.7	44.0
Minimum	24.5	1.0	218	23.0	40.4
Average	31.2	1.5	229	31.4	42.5

No seasonal variation was observed in any of the values (see also *ANALYST*, 1892, **17**, 46; 1913, **38**, 242). W. P. S.

Composition of Egyptian Buffalo Milk. A. Pappel and G. Hogan. (*Egyptian Dept. Public Health, 1914, 1-12.*)—Sixty-one samples taken from different herds were examined; each sample represented the mixed milk from six animals. The maximum, minimum, and average percentage results obtained are given below:

	Minimum.	Maximum.	Average.
Sp. gr. at 15°/4° C.	1.0294	1.0343	1.0324
Total solids	15.81	19.75	17.91
Fat (Adams' method)	6.05	9.75	7.95
Solids-not-fat	9.42	10.40	9.95
Lactose	4.65	5.19	4.86
Proteins	3.57	4.80	4.16
Ash	0.70	0.84	0.78
Chlorine	0.04	0.10	0.08

W. P. S.

Method for Detecting the Admixture of Goat's Milk with Cow's Milk. N. A. Brodrick-Pittari. (*Schweiz. Zentralbl. Milchw.*, 1915, 4, 413-414; 1916, 5, 3-4; through *Int. Rev. of the Science and Practice of Agriculture*, 1916, 7, 431-433.)—A method proposed by Steinegger is based on the fact that the casein of cow's milk dissolves readily in ammonia at 50° C., whilst the casein of goat's milk remains insoluble; Gabathuler and Pritzker have improved the method by separating the insoluble casein of goat's milk by centrifugal action. The author has investigated the method, and finds that—(1) The same amount of goat's milk, when mixed in equal quantities with milk from different cows, produces variable quantities of sediment. (2) The amount of sediment formed in a mixture of goat's milk and colloidal solutions decreases with a decrease in the concentration of the solution. (3) Decrease of the colloidal concentration of cow's milk by the addition of water diminishes the amount of sediment which may be produced by the presence of goat's milk. (4) When absolutely fresh goat's milk is added, the increase in the albumin content of cow's milk causes an increase in the amount of sediment of insoluble casein. (5) The amount of sediment diminishes with the age of the goat's milk. (6) Formaldehyde interferes with the formation of the sediment, whilst potassium bichromate promotes its formation and causes a marked separation of the sediment from the serum. (7) The formation of the sediment of insoluble casein is promoted by a complete skimming of the mixed milk under examination. On the basis of these results, the author recommends the following procedure in carrying out the test: 100 c.c. of the milk are treated with 1.5 c.c. of saturated potassium bichromate solution and then skimmed completely; 20 c.c. of the skimmed milk are now placed in a tube, 2 c.c. of 20 per cent. ammonia are added, the mixture is heated at 50° C. for one hour, and then submitted to centrifugal action. The formation of a sediment is a certain indication that the sample contains goat's milk. The minimum quantity of goat's milk that can be detected by the test is about 10 per cent., but the absence of a sediment does not prove the entire absence of goat's milk.

W. P. S.

Methyl Red as an Indicator in the Estimation of Nicotine by Toth's Method. P. Schick and G. Hatos. (*Zeitsch. Unters. Nahr. Genussm.*, 1914, 28, 269-270.)—In Toth's method (*ANALYST*, 1902, 27, 12) the nicotine is extracted by means of a mixture of ether and petroleum ether from the tobacco after the latter has been treated with sodium hydroxide; an aliquot portion of the ethereal solution is then titrated. The author finds that methyl red may be used as the indicator in this titration, and that it serves equally as well as the iodeosin recommended by Toth.

W. P. S.

Detection and Estimation of Nitrites and Nitrates in Meats, Sausages, etc. D. Acél. (*Zeitsch. Unters. Nahr. Genussm.*, 1916, 31, 332-341.)—Nitrites in meat preparations may be detected and estimated colorimetrically with the Griess-Ilosvay reagent; nitrates may be estimated in the same way after they have been reduced to nitrites by means of zinc and acetic acid. The presence of soluble starch, salicylic acid, sugar, vinegar, formaldehyde, or boric acid, does not interfere with the reaction.

Nitrites are produced during the burning of wood, and nitrites may be thus introduced into smoked meats, but it is found that the quantity is too small to have any material effect on the results obtained by the above method. Attention is drawn to the fact that potassium nitrate in meat is reduced rapidly to nitrite; for instance, if meat treated with potassium nitrate is kept at 10° C. for four days, about 50 per cent. of the quantity of nitrate present is reduced to nitrite. W. P. S.

Oil from the Fruit of the Canadian Vine. S. Fachini and G. Dorta. (*Annali Chim. Applic.*, 1916, 6, 301-304.)—The pulp of the fruit of the Canadian vine (*Parthenocissus quinquefolia*) yielded 3.3 per cent. of oil, while the kernels yielded 11.8 per cent. The fruit oil was a semi-solid mass, with a green colour and an astringent taste. It had the following constants: Refractive index at 15° C., 1.4722; saponification value, 192.3-193.3; iodine value, 90.3; Hehner value, 94.0; unsaponifiable matter, 1.67 per cent. *Fatty acids*: Iodine value, 94.4-94.6; molecular equivalent, 278.8; and iodine value of liquid fatty acids, 110.2. The solid fatty acids (about 10 per cent.) consisted of palmitic acid, while the liquid fatty acids contained oleic and linolic acids. The oil from the seeds was greenish-yellow and had a pleasant odour and taste. Its constants were: Sp. gr. at 15° C., 0.9215; refractive index at 15° C., 1.4778; saponification value, 189.2; iodine value, 141.4; Hehner value, 93.97; unsaponifiable matter, 1.44 per cent. *Fatty acids*: Iodine value, 144.6; molecular equivalent, 281.2; and iodine value of liquid fatty acids, 148.8. The solid fatty acids consisted of palmitic acid, while the liquid fatty acids contained oleic and linolic acids. The seed oil belonged to the semi-drying class. C. A. M.

Volatile Oil of *Calycanthus Occidentalis*. C. C. Scalione. (*J. Ind. and Eng. Chem.*, 1916, 8, 729-731.)—The leaves and twigs of the spice bush *Calycanthus occidentalis*, which grows in California and Oregon, yielded, when distilled with steam under pressure, from 0.15 to 0.37 per cent. of a greenish-yellow oil, with a bitter taste and an odour of camphor. It had the following constants: Sp. gr. at 25° C., 0.9295; optical rotation, +7° 28'; refractive index at 20° C., 1.4713; free acid, 0.05 per cent.; saponification value, 54.3; and acetyl value, 33.5. It was soluble in all proportions in 90 per cent. alcohol, and in 15 to 16 volumes of 70 per cent. alcohol. It had the following composition: Pinene (*d* and *l*), 8.30; cineol, 60.32; borneol, 9.21; and linalyl acetate, 18.99 per cent., with traces of camphor, methyl salicylate, and sesquiterpene alcohols. C. A. M.

Detection of Saccharin and its Estimation. M. Klostermann and K. Scholta. (*Zeitsch. Unters. Nahr. Genussm.*, 1916, 31, 67-78.)—Many of the tests which have been proposed for the detection and identification of saccharin are rendered useless when applied to food substances owing to the interfering action of other substances which may be present. For instance, clove oil, cinnamon oil, salicylaldehyde, gum benzoin, tannin, benzaldehyde, and vanillin, all give a reaction with the phenol-sulphuric acid test (formation of a red coloration when the

substance is heated with phenol and sulphuric acid, and then treated with sodium hydroxide): The following method is suggested with the object of overcoming these difficulties. The material to be tested is rendered acid and the saccharin extracted with a mixture of ether and petroleum spirit; the solvent is evaporated, the residue obtained is boiled for ten minutes with 10 per cent. hydrochloric acid, and the solution again evaporated to dryness. If the residue now obtained has an odour of vanillin it is washed several times with a mixture of equal volumes of ether and chloroform; saccharin is insoluble in this mixture. The residue is now treated with liquid phenol and the solution poured on to phosphoric anhydride. If saccharin is present the mixture exhibits a red colour which, on the addition of water, changes to yellow; this yellow solution gives a blue-red coloration when rendered alkaline, and the colour is discharged by ammonium sulphide. Besides vanillin (which is eliminated as described), benzaldehyde is the only other substance which gives a reaction similar to saccharin, and this aldehyde is expelled during heating with hydrochloric acid or it is converted into benzoic acid, which does not interfere. In methods for the estimation of saccharin, and which depend on the estimation of the sulphur content, the substance should be hydrolysed with hydrochloric acid and extracted with a mixture of ether and chloroform, in order to remove any *p*-benzoic sulphinide; otherwise, the sulphur contained in the latter would be included in the quantity yielded by the saccharin.

W. P. S.

Analysis of Soap Powders. L. Rosenberg and V. Lenher. (*J. Ind. and Eng. Chem.*, 1916, 8, 716-719.)—*Moisture*: 2 grms. of the sample are dried in a vacuum oven at 65° C. until constant in weight (about twenty hours). *Fat*: The residue from the moisture estimation is extracted with petroleum spirit, the solvent evaporated, and the residue dried at 110° C. *Soap and free alkali*: The fat-free residue containing the soap and mineral constituents is extracted for thirty minutes with 95 per cent. alcohol, which dissolves the soap and free alkali. The alcoholic extract is titrated with $\frac{N}{10}$ acid, with phenolphthalein as indicator, and the amount of caustic alkali found calculated as Na₂O. The solution is then diluted with a large amount of water, the alcohol expelled by evaporation, and the liquid boiled with a measured excess of $\frac{N}{1}$ nitric acid. An addition of 4 grms. of stearic acid is made, the liquid again boiled, then cooled, and the cake of fatty acids and added stearic acid separated and weighed. The excess of acid in the solution is titrated back with $\frac{N}{2}$ sodium hydroxide solution, and the amount of acid which had been used calculated into the amount of sodium oxide in combination in the soap. *Mineral constituents and insoluble matter*: The residue from the alcoholic extraction may contain sodium carbonate, chloride, silicate, sulphate or borate, starch, etc. It is dissolved in hot water, and any insoluble residue of starch, etc., separated and weighed. The solution is divided into five equal portions, and one of each taken for the estimation of the different salts of sodium by the usual methods. The recent introduction of continuous drying apparatus by the larger manufacturers has changed the relative composition of these powders. Commercial samples typical of those on the market gave the following results:

	Moisture.	Fatty Anhydride.	Na ₂ O as Soap.	Total Soap.	Sodium Carbonate.	Sodium Silicate.	Sodium Chloride.	Unsaponified Matter.	Total.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
1	37.82	18.39	2.63	21.02	39.37	0.45	0.55	0.69	99.90
2	22.06	19.44	3.58	23.02	53.53	0.99	0.75	0.24	100.59
3	23.75	21.63	2.71	24.34	50.35	0.72	0.35	0.85	100.36
4	20.86	18.93	2.65	21.58	55.38	0.90	0.86	0.45	100.03
5	18.52	20.34	2.68	23.02	56.45	0.36	1.32	0.74	100.41
6	43.38	16.74	2.23	18.97	36.30	0.72	0.34	0.87	100.58
7	30.40	21.46	2.86	24.32	40.00	0.99	0.72	0.79	97.22

Estimations of sugar, resin, glycerol, fluorides, and sodium bicarbonate, are not included in this scheme of analysis. In the authors' experience these powders are of simple composition.

C. A. M.

Studies of Soap Solutions. V. Lenher and M. V. R. Buell. (*J. Ind. and Eng. Chem.*, 1916, 8, 701-703.)—*Surface tension*: Pure sodium oleate solutions were delivered from a modification of Traube's stalagmometer provided with a device to regulate the pressure. The surface tension towards air at 25° C. expressed in dynes per cm. fell from 28.20 with $\frac{N}{10}$ solutions to 24.17 with $\frac{N}{80}$ solutions, and then rose to 24.91 with $\frac{N}{320}$ solutions and 41.28 with $\frac{N}{1280}$ solutions. Under the same conditions water gave 71.78 towards air. Frothing of the soap was most pronounced in the concentrations between $\frac{N}{80}$ and $\frac{N}{320}$. *Emulsifying action*: The emulsification of soap solution and immiscible liquid was effected by means of a thin metal paddle containing numerous perforations, which was driven by an electric motor. The liquids were agitated for five minutes and an emulsion regarded as satisfactory if it did not separate within five minutes. A difference of 0.1 c.c. of $\frac{N}{20}$ sodium oleate solution (=0.0015 gm.) could be detected by this method. The following results were obtained with pure water at 20° C.:

Substance.	Water.	Sodium Oleate required for Emulsification.
	c.c.	grms.
Turpentine, 50 c.c.	50	0.6331
Carbon tetrachloride, 50 c.c.	50	0.1140
Chloroform, 50 c.c.	50	0.7824
Benzene, 50	50	0.9804
Vaseline, 2.5 grms.	50	1.5200

Weak solutions of sodium oleate were more economical in the emulsification of turpentine and water. At higher temperatures a definite quantity of sodium oleate was able to emulsify greater amounts of the immiscible liquids, but the emulsifications were less stable than those made at lower temperatures. The emulsifying power of sodium oleate solutions was only very slightly reduced by the hydrolysis which had taken place after four and a half months. Hence a soap solution which has

stood for a long time appears to be as effective for washing purposes as a freshly made solution. At 100° C. sodium palmitate emulsified olive oil much more efficiently than did sodium oleate or stearate. *Suspensions*: Sifted ferric oxide and manganese dioxide were shaken with sodium oleate solutions of concentrations from $\frac{N}{30}$ to $\frac{N}{1280}$, and the mixtures allowed to stand for five days to three weeks. The maximum suspension invariably occurred with solutions of about $\frac{N}{320}$ strength.

C. A. M.

Colorimetric Method for the Detection of Potato Starch. G. Blunck. (*Zeitsch. Unters. Nahr. Genussm.*, 1915, **29**, 246-247.)—Potato starch and cell tissues are coloured a bright golden-yellow by the dye called "metachrome red G Agfa," whilst cereal starches are not coloured. The presence of potato starch in ordinary flour may be detected by treating a portion of the sample on a microscope slide with a drop of water, drying it, adding a drop of the dye solution, washing off the excess of the latter, and examining the slide under the microscope. The dye solution is prepared by saturating boiling 30 per cent. alcohol with the dye, filtering the cooled solution, and diluting it with 25 per cent. of its volume of water. When it is desired to apply the test to bread, a quantity of the crumb must be treated with dilute alkali solution and then washed before the dye solution is added. The treatment with alkali removes any acidity, and is an essential procedure, since in the presence of acids the dye colours wheat starch as well as potato starch.

W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

The Enzymes of Honey. F. Gothe. (*Zeitsch. Unters. Nahr. Genussm.*, 1914, **28**, 273-286.)—Lactase, proteases, and lipases are not present in honey, and the occurrence of inulase is doubtful. Invertase is present, a larger quantity being found in natural honey than in honey produced by bees which have been fed on sugar. The activity of the invertase is at a maximum at 40° C., and is destroyed when the honey is heated at 60° C. for one hour. Honey also contains catalase and diastase; the diastase content often increases as the quantity of catalase decreases. Filtration of the honey solution causes a considerable decrease in the activity of these two enzymes. A dirty or impure honey has a very high catalase value, but a low catalase value does not indicate that a honey is of inferior quality or that it has been heated. At the same time, the activity of the catalase is considerably decreased when the honey solution is heated at 60° C. for one hour, but the honey itself may be heated up to 70° C. before the activity of the catalase is affected by temperature.

W. P. S.

Properties of Honey Diastase. F. Gothe. (*Zeitsch. Unters. Nahr. Genussm.*, 1914, **28**, 286-321.)—An estimation of the activity of the diastase in honey gives an indication as to whether the honey is genuine. A high diastase value (Wohlgemuth's method, *ANALYST*, 1908, **33**, 362) shows that the honey is pure and in its natural state; if a low value is found, Fiehe's test (*ANALYST*, 1908, **33**, 397) and the pre-

cipitin reaction should be applied to the sample in order to obtain confirmatory evidence as to the presence of adulterants. The activity of honey diastase is destroyed when the honey is heated for some time at 60° C.; a honey so treated loses its aroma and biological properties and is a de-natured product. The author is of opinion that heated honey should not be called natural honey. The presence of acids or alkalis in any considerable quantity, prolonged contact with alcohol, filtration, and dialysis, all interfere with the activity of the diastase. Freshly prepared starch solution should be used in the estimation of the activity of the diastase, and the conditions prescribed by Wohlgemuth should be followed throughout. W. P. S.

Detection of Picric Acid in Urine and Viscera. E. Kohn-Abrest. (*Ann. Falsific.*, 1916, 9, 63-68.)—Twenty c.c. of the urine under examination are treated with 10 c.c. of mercuric sulphate reagent (mercuric oxide, 10 grms.; sulphuric acid, 40 c.c.; and water, 200 c.c.), the mixture is filtered after five minutes, and the filtrate is extracted with chloroform. After the addition of 0.5 c.c. of water the chloroform solution is evaporated, and the residual aqueous solution tested for the presence of picric acid. If picric acid is present in the urine this aqueous solution will have a yellow colour, dye wool or silk, and yield the isopurpurate reaction when treated with alkaline potassium cyanide solution. The method will detect as little as 0.005 gm. of picric acid per litre of urine. For the detection of picric acid in viscera the substance is extracted at 60° C. with 90 per cent. alcohol containing 1 per cent. of tartaric acid, the alcoholic extract evaporated under reduced pressure, and the residue thus obtained extracted twice with alcohol, the alcoholic solutions being evaporated each time. The final alcoholic solution of the residue is mixed with a small quantity of water, evaporated under reduced pressure, the residual solution treated with dilute sulphuric acid so that the acidity of the mixture shall be about 2 per cent., and extracted with ether. The ethereal solution is evaporated, the residue dissolved in a small quantity of 1 per cent. tartaric acid solution, and extracted with petroleum spirit and then with ether. These two solvents remove certain substances other than picric acid. The acid aqueous solution is now acidified with sulphuric acid, the picric acid is extracted with ether and identified by means of the usual reactions. W. P. S.

ORGANIC ANALYSIS.

Estimation of Benzol in Coke-Oven Gas by Burrell's Vapour Apparatus. L. C. Whiton. (*J. Ind. and Eng. Chem.*, 1916, 8, 733-734.)—Burrell and Robertson's apparatus (*ANALYST*, 1915, 40, 162) is not suitable for estimating benzol in coke-oven gas. The manometer readings range from 10 mm. for raw gas to 1 mm. for de-benzolised gas, and with an ordinary manometer it is difficult to read closer than 1 mm. In a vertical position it is impossible to surround the apparatus with CO₂-alcohol slush with economy of this material. Again, the phosphoric anhydride used requires frequent renewal, which is impossible with the apparatus of Burrell and Robertson. The principle of their method, however, is excellent, and, with modifications of the apparatus, has proved of practical service. The author gives

the manometer an inclination of 1 in 10 from the horizontal, and thus gets large readings. The bulb is laid horizontally in a trough, with a notch at the end through which the stem of the apparatus could pass. In this position it could be more economically surrounded with carbon dioxide slush. The trough, $8 \times 2\frac{1}{2} \times 2\frac{1}{2}$ inches, has a hinged cover and double walls with the air evacuated. As it is difficult to make this vacuum chamber absolutely tight, it is equipped with a cock by means of which it can be put in connection with a pump from time to time. The phosphoric anhydride is contained in a separate tube connected to the rest of the apparatus by stout rubber tubing. The method is substantially exact for benzol, but underestimates toluol, xylol, etc. The total of these substances may be underestimated by almost 25 per cent., so that the method cannot properly be spoken of as a means of estimating benzol and its homologues. As a means of controlling benzol scrubber efficiency, however, it is of some value. Finally, it is stated that the method is more difficult of operation than it appears and requires an operator skilled in handling such apparatus.

G. C. J.

Some Properties of the Water in Coal. H. C. Porter and O. C. Ralston. (*U.S. Bureau of Mines, Technical Paper 113, p. 28.*)—Air-dried coal may contain from 1 to 25 per cent. of moisture, according to the source, origin, and size of the sample. It can be substantially deprived of this moisture by heating for a short time in a current of dry air at 100° C. or by prolonged exposure to dry air at lower temperatures. The use of reduced pressure hastens the process, but does not carry it farther. The dry coal on exposure to ordinary atmospheric conditions reabsorbs moisture, in amount depending on the character of the coal and the humidity of the atmosphere. If the atmospheric conditions are identical with those under which the coal was air-dried, it will reabsorb moisture until it has substantially the same water-content as the original air-dried coal. In the experiments described in the paper, the final moisture content was always slightly less than the initial moisture content, but this may well be due to the facts that the attainment of final equilibrium is very slow and that the experiments were not indefinitely prolonged. It is shown that the vapour-pressure of the water in air-dried coal is below normal and that the degree of deficiency in this respect varies widely among different coals.

Heat is produced by wetting dry coal or partly dry coal containing less than its normal percentage of inherent water. The relative quantity of heat generated depends on the kind of coal and its relative deficiency in moisture as referred to its maximum normal content. In other words, the thermal effect of wetting varies directly as some function of the relative vapour-pressure deficiency in the coal.

The above facts are discussed from the standpoint of colloidal theory, which in the authors' opinion affords a ready explanation of them.

G. C. J.

Preparation of Diphenylamine-Sulphuric Acid Reagent. W. Tönies. (*Zeitsch. Unters. Nahr. Genussm.*, 1916, 31, 322.)—The diphenylamine-sulphuric acid reagent used in Tillmans' method (*ANALYST*, 1911, 36, 67) for the detection and estimation of nitrates may be prepared with ordinary sulphuric acid. If any blue

coloration appears, due to the presence of nitric acid in the sulphuric acid, it may be discharged by heating the reagent at 110° C.; this heating expels the nitric acid and does not injure the reagent.

W. P. S.

Relationship between the Physical and Chemical Constants of Fats.

H. J. Backer. (*Chem. Weekblad*, 1916, **35**, 954-967.)—The relationship between the refractive index, n , specific gravity, d , and the saponification value, V , and iodine value, I , of ordinary fats, may be expressed by the following equation:

$$\frac{n^2t - 1}{n^2t + 2} \times \frac{100}{d_4^{20}} = 33.07 + 0.00075I - 0.01375V + 0.002(t - 15).$$

In the presence of oxy-acids the first figure of the equation is lower. For example, the calculated and observed figures for linseed oil were 30.56 and 30.66 respectively, while in the case of castor oil the calculated index was 30.63 and the observed value 29.51 at 15° C. The relationship also holds good in the case of hydrogenated oils.

C. A. H.

Sensitive Indicator for Acidimetry and Alkalimetry and for Determination of Hydrogen Ion Concentration. **M. T. Bogert and G. Scatchard.** (*J. Amer. Chem. Soc.*, 1916, **38**, 1606-1615.)—A dinitrobenzoylene urea has been discovered whose monosodium salt is a very sensitive indicator for hydrogen ion concentrations between 10^{-6} and 10^{-8} , changing from colourless to greenish-yellow. In its behaviour as an indicator it resembles *p*-nitrophenol more closely than any other well-known indicator. Like *p*-nitrophenol, its yellow colour makes it unsuitable for work by artificial light. It is but slightly affected by neutral salts, not at all by chloroform or toluene, and proteins have no more influence upon it than upon *p*-nitrophenol. Its colour is unchanged by nitrous acid. It can be used in the cold or at 100° C. It gives a sharp end-point with ammonia and hydrochloric acid, but cannot be used to titrate carbonates. It should be superior to rosolic acid for many purposes for which that indicator is used. It may be prepared as follows: Twenty grms. anthranilic acid are dissolved in 700 c.c. water and 15 c.c. hydrochloric acid by warming. To the filtered and cooled solution, a solution of 15 grms. potassium cyanate in 50 c.c. water is added slowly with continuous stirring, this stirring being continued for twenty minutes after all the cyanate solution has been added. The uraminobenzoic acid precipitates as a pasty white mass of microscopic needles. Sodium hydroxide (300 grms.) is added, the solution being kept cool. The uramino acid dissolves and the sodium salt of benzoylene urea soon separates. The latter is filtered off, dissolved in 1,000 c.c. of boiling water, precipitated with acetic acid and the free benzoylene urea filtered off, washed with water and dried at 120° C. About 22 grms. are obtained. Of this product, 10 grms. are heated on the water-bath with 100 c.c. sulphuric acid and 12 c.c. nitric acid are added. Heat is evolved and the mixture becomes at first bright red, but soon changes to bright yellow. After digesting for an hour at 100° C., the mixture is cooled and poured into 1,000 c.c. of ice and water, the precipitate filtered off, washed with water and recrystallised from 1,000 c.c. of 50 per cent. acetic acid. The yield of

dinitrobenzoylene urea is about 14.5 grms. For the preparation of the sodium salt, 25 grms. are dissolved in 115 c.c. $\frac{N}{4}$ sodium hydroxide and 500 c.c. boiling water and the solution is filtered and cooled. A mass of long, bright yellow needles crystallises out. These are dried between filter paper and finally in a vacuum desiccator. For use as an indicator, a 0.3 per cent. aqueous solution is made.

G. C. J.

Determination of Very Small Quantities of Water in Alcohol by Means of the Critical Solution Temperature. V. Rodt. (*Mitt. K. Materialprüf.*, 1915, **33**, 426-433; through *J. Soc. Chem. Ind.*, 1916, **35**, 938.)—The method devised by Crismer has been reinvestigated and proved to be capable of giving extremely accurate results. A suitable petroleum oil giving sharp indications without artificial cooling may be prepared by mixing transformer oil with commercial paraffin oil. For standardising the oil, perfectly anhydrous alcohol must be prepared. About 2 litres of 99 per cent. alcohol are placed in a large flask with 300 grms. of quicklime, and the flask is suspended in a water-bath which can be quickly removed. The alcohol is gently boiled for some days under a reflux condenser protected by a phosphorous pentoxide guard-tube. The condenser is then reversed and arranged to deliver the distillate into a wide, corked burette, also guarded with moisture-absorption tubes. Successive portions of alcohol are distilled over into the burette and tested with the petroleum until a constant minimum value is attained. For the test, all the apparatus must be dried in an oven at 150° C. and every precaution taken against access of atmospheric moisture. An Erlenmeyer flask of about 50 c.c. capacity fitted with rubber stopper is accurately tared, a few drops of water are introduced rapidly from a pipette and the weight noted, then 15-25 c.c. of alcohol are drawn from the distillation burette and the flask is weighed again, so that the gravimetric composition of the aqueous alcohol is accurately known. The rubber stopper is replaced by another, also dried in the oven, having two holes, through one of which passes a standardised thermometer, reading to 0.2° C., and through the other a short glass rod. The glass rod is withdrawn, the point of a burette being inserted through the hole, and a measured volume of petroleum is run in, calculated from its sp. gr. to amount to 48/52 by weight of the alcohol employed; the measurement should be accurate to 0.1 c.c. The rod is replaced, the contents of the flask are warmed with gentle rotation until the liquid is perfectly clear, and rotation is continued while cooling, until a sudden sharp clouding of the solution appears. The critical temperature may be noted with an accuracy of 0.2° C., corresponding to 0.01 per cent. of water in the alcohol. A standardisation curve is plotted for a given petroleum oil which may be used subsequently for rapid and simple estimations of water in strong spirits; the rise of critical temperature is almost a straight line function of the percentage of water within the limits of 0.2-5 per cent.

Rinck's Method for the Detection of Methyl Alcohol. G. Fendler. (*Zeitsch. Unters. Nahr. Genussm.*, 1916, **30**, 228-230.)—This method (*ANALYST*, 1914, **39**, 407) was found to be unreliable owing to the fact that methyl alcohol yields traces of formaldehyde under the conditions of the test.

W. P. S.

Titration with Oxalic Acid, using Methyl Orange as Indicator. G. Bruhns. (*Zeitsch. anal. Chem.*, 1916, **55**, 321-340.)—Calcium, barium, strontium, and magnesium bicarbonate solutions may be titrated directly with oxalic acid solution, using methylorange as the indicator. Alkali solutions containing carbonate or borate can be titrated in the same way provided that calcium chloride, strontium chloride, barium chloride, zinc sulphate, cadmium sulphate, manganous sulphate, silver nitrate, or lead nitrate be added to the solution previous to the titration. In the titration of ammonia solution, the addition of calcium chloride or barium chloride is necessary. It is mentioned that the addition of 35 grms. of crystallised boric acid per litre of $\frac{N}{10}$ ammonia solution renders the latter so stable that it may be kept in an open vessel for several days without losing in strength as determined by titration with oxalic acid solution. The standard oxalic acid solution used in the titrations should be prepared with water free from carbon dioxide.

W. P. S.

Precipitation of Cholesterol and Phytosterol by Digitonin. O. Pfeffer. (*Zeitsch. Unters. Nahr. Genussm.*, 1916, **31**, 38-40.)—The following procedure is recommended for the rapid collection (by filtration) of the precipitate obtained when fatty acids from a fat or oil are treated with digitonin: The fatty acids are melted, digitonin solution is added, and the hot mixture poured on a filter which is placed in a water-oven until the fatty acids have passed through; chloroform should not be added, as its presence retards the rate of filtration by destroying the granular state of the precipitate. The precipitate and filter are then washed with hot chloroform, next with ether, and the filter is dried; the film of precipitate can now be removed readily from the filter and treated with acetic anhydride, etc. A specimen of hydrogenised fish oil (m.-pt. 52° C.) examined by the author yielded a cholesteryl acetate which melted at 112° C.

W. P. S.

Detection of Phytosterol by Precipitation with Digitonin. A. Olig. (*Zeitsch. Unters. Nahr. Genussm.*, 1914, **28**, 129-138.)—The method described by Marcusson and Schilling (*ANALYST*, 1913, **38**, 458) was applied to numerous mixtures of animal and vegetable fats and oils, and it was found that the melting-point of the acetate, after it had been recrystallised once or twice, almost always indicated whether phytosteryl acetate was present with the cholesteryl acetate. The presence of paraffin and of waxy or resinous constituents of shea and mowrah fats did not interfere with the test. The author agrees that the cholesterol and phytosterol can be precipitated with digitonin directly from the fats, and it is only when an inconclusive result is obtained, which is seldom, that it is necessary to saponify the fat and make the precipitation from the fatty acids.

W. P. S.

Estimation of Phytosterol in Vegetable Oils. M. Klostermann and H. Opitz. (*Zeitsch. Unters. Nahr. Genussm.*, 1914, **28**, 138-145.)—The quantity of phytosterol present in a vegetable oil depends to a certain extent on the method by which the oil has been obtained from the seeds, etc. Usually, the quantity varies from 0.133 per cent. in olive oil to 0.549 per cent. in sesame oil. The greater

part of the phytosterol occurs in the oil in the form of its ester (*cf.* ANALYST, 1914, 39, 310), and consequently, in the detection of vegetable oils in animal fats, it is advisable to saponify the fat before the cholesterol and phytosterol are precipitated with digitonin. The latter precipitates the alcohols only and does not react with the esters.

W. P. S.

Estimation of Volatile "Thinner" in Oil Varnish. E. W. Boughton. (*Chem. News*, 1916, 114, 75-77.)—The following methods were found to yield satisfactory results, the maximum error being about 1 per cent. in the case of a varnish containing 50 per cent. of thinner. The evaporation of the latter (turpentine or petroleum spirit) from drying films of freshly prepared varnishes is practically complete in two hours at 30° C. *Method 1*: One hundred c.c. of the varnish are steam-distilled, the vessel containing the varnish being maintained at 115°-120° C. until from 550 to 700 c.c. of aqueous distillate has been collected; this requires about ninety minutes. The water is then separated and the oily portion of the distillate weighed. To the weight is added 0.3 gm. for each 100 c.c. of water collected in the distillate. *Method 2*: About 1.5 grms. of the varnish is poured over the bottom of a flat-bottomed metal basin 8 cm. in diameter, and the basin is then heated in an air-bath at 110°-115° C. until its contents cease to decrease in weight. *Method 3*: Brown's method (see ANALYST, 1914, 39, 560). *Method 4*: A small stoppered weighing bottle, containing about 1 gm. of the varnish and a small camel's-hair brush, is weighed. The varnish is then poured on to a glass plate, 10 by 15 cm., and brushed over the surface in a thin film, and the bottle and brush are re-weighed. The plate is now placed in a cabinet with free access of air until no further loss of weight takes place; about two hours' drying is required.

W. P. S.

INORGANIC ANALYSIS.

Estimation of Air, Water-Vapour, and Nitrous Oxide in Mixtures of these Three Constituents. G. A. Burrell and G. W. Jones. (*J. Ind. and Eng. Chem.*, 1916, 8, 735.)—An application of a method already described by Burrell and Robertson (ANALYST, 1915, 40, 162) to this particular purpose. The gas is admitted to a vacuum bulb connected to a manometer and provided with a stop-cock. The mixture is liquified by immersing the bulb in liquid air, and the air is withdrawn by means of a Töpler pump and measured. The residual gas is subjected to a temperature of -78° C. (CO₂-alcohol slush) and the nitrous oxide withdrawn and measured. Finally the partial pressure of the water-vapour is measured. The ratio of this pressure to the barometric pressure multiplied by 100 grms. gives the percentage of water-vapour in the original sample. Three samples of American "laughing gas" analysed in this way showed from 95.6 to 96.2 per cent. of nitrous oxide, air 2.0 to 2.1 per cent., and water-vapour 2 per cent.

G. C. J.

Estimation of Aluminium. C. F. Sidener and E. Pettijohn. (*J. Ind. and Eng. Chem.*, 1916, 8, 714-716.)—As the result of experiments described in the paper, it is shown that boiling for one minute precipitates aluminium quantitatively,

whilst longer boiling may lead to partial re-resolution. Aluminium hydroxide need not be washed free from ammonium chloride before ignition. The excess of ammonia present when precipitation is made should be as small as possible, and never more than 1 or 2 c.c. in 250 c.c. of solution. Aluminium hydroxide, freshly precipitated, is soluble to a slight extent in water and to about the same extent in dilute ammonium nitrate solution. Precipitates of alumina, when large, require to be heated with the blowpipe for as long as forty minutes, and the ignited alumina is strongly hygroscopic.

G. C. J.

Iodimetric Estimation of Gold. L. Vanino and F. Hartwagner. (*Zeitsch. anal. Chem.*, 1916, 55, 377-388.)—The method described by Peterson (*ANALYST*, 1899, 24, 108) is modified by the author in order to render it capable of yielding more accurate results. The gold chloride ($\text{AuCl}_3 \cdot \text{HCl}$) is treated with a mixture of potassium iodide and potassium iodate, and the liberated iodine is titrated with thiosulphate solution. The reactions taking place are shown by the following equations: (1) $6\text{AuCl}_3 \cdot \text{HCl} + 18\text{KI} = 6\text{AuI} + 12\text{I} + 18\text{KCl} + 6\text{HCl}$; (2) $6\text{HCl} + 5\text{KI} + \text{KIO}_3 = 6\text{I} + 6\text{KCl} + 3\text{H}_2\text{O}$; (3) $18\text{I} + 18\text{Na}_2\text{S}_2\text{O}_3 = 18\text{NaI} + 9\text{Na}_2\text{S}_4\text{O}_6$. Three molecules of thiosulphate correspond to 1 atom of gold. Using quantities of gold chloride equivalent to 2–10 mgrms. of gold, the error in the results obtained varies from -0.9 to $+4.5$ per cent. Free acid must not be present in the salt. The method may be applied to the analysis of the salt $\text{AuCl}_3 \cdot 2\text{H}_2\text{O}$; the quantity of iodine liberated by the salt from potassium iodide (Peterson's method) is a measure of the quantity of gold present. The difference between this quantity of iodine and that liberated when the salt is treated with a mixture of iodate and iodide gives the amount of hydrochloric acid present.

W. P. S.

Precipitation of Magnesium Salts by Ammonium Carbonate. F. Fichter and R. Osterwalder. (*Zeitsch. anal. Chem.*, 1916, 55, 389-392.)—The authors have investigated the nature of the precipitate obtained when a soluble magnesium salt is treated with ammonium carbonate. Ordinary ammonium carbonate when dissolved yields a solution containing a mixture of ammonium carbonate and ammonium hydrogen carbonate. If this solution is added to magnesium sulphate solution a precipitate of hydrated magnesium carbonate, $\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$, forms gradually, the precipitation being accelerated by warming the mixture. Crystals of hydrated magnesium carbonate appear within about thirty minutes when magnesium sulphate solution is mixed with ammonium carbonate solution to which ammonia has been added previously in such quantity that the proportion of ammonia to carbon dioxide in the solution corresponds with the formula $(\text{NH}_4)_2\text{CO}_3$; if this mixture is heated to 40°C ., an amorphous precipitate of basic magnesium carbonate (*magnesia alba*) forms, which changes into the hydrated carbonate when the mixture is cooled. Magnesium sulphate solution treated with ammonium carbonate solution containing an excess of ammonia yields a precipitate of the basic carbonate, and this, after the lapse of a few hours, changes into the hydrated carbonate; if, however, the precipitation is made from a hot solution, the basic carbonate remains as such when the mixture is cooled. A double carbonate, having the composition

$(\text{NH}_4)_2\text{CO}_3 \cdot \text{MgCO}_3 \cdot 4\text{H}_2\text{O}$, is obtained when one equivalent of magnesium sulphate (in solution) is treated with ten equivalents of ammonium carbonate, $(\text{NH}_4)_2\text{CO}_3$ (also in solution).
W. P. S.

Volumetric Estimation of Thiosulphate in the Presence of Sulphite. A. Sander. (*Zeitsch. anal. Chem.*, 1916, **55**, 340-342.)—Bodnár has described a method (*ANALYST*, 1914, **39**, 101) for the estimation of thiosulphate in the presence of sulphite which depends on the reaction of the thiosulphate with silver nitrate. This method can be rendered much more simple and rapid by substituting mercuric chloride for the silver nitrate. The sulphite forms a soluble complex with the mercuric chloride, $\text{Na}_2\text{SO}_3 + \text{HgCl}_2 = \text{HgCl} \cdot \text{NaSO}_3 + \text{NaCl}$, whilst the thiosulphate reacts according to the equations: $\text{Na}_2\text{S}_2\text{O}_3 + \text{HgCl}_2 = \text{HgS}_2\text{O}_3 + 2\text{NaCl}$; $\text{HgS}_2\text{O}_3 + \text{H}_2\text{O} = \text{HgS} + \text{H}_2\text{SO}_4$; and $2\text{HgS} + \text{HgCl}_2 = \text{Hg}_3\text{S}_2\text{Cl}_2$. The solution containing the sulphite and thiosulphate is simply treated with an excess of mercuric chloride solution, and the mixture titrated with $\frac{N}{10}$ sodium hydroxide solution, using methyl-orange as indicator. The acidity of the mixture is due to the liberated sulphuric acid, which in turn is a measure of the thiosulphate present. A quantity of ammonium chloride should be added to the mixture before the titration is commenced in order to prevent the precipitation of mercuric oxide by the added alkali. (See also *ANALYST*, 1916, 84.)
W. P. S.

Detection of Tungsten. M. L. Hartman. (*Chem. News*, 1916, **114**, 45-46.)—The reduction test and especially its limit of sensitiveness have been investigated. In solutions containing only tungsten salts the test is very sensitive, and as little as 1 mgrm. of tungstic acid may readily be detected. The most satisfactory reducing agent is metallic tin, and at least 10 per cent. of concentrated hydrochloric acid should be added to the liquid to be tested. For testing ores, the following procedure is recommended: About 0.2 gm. or more of the finely powdered ore is boiled with concentrated hydrochloric acid until half the acid has evaporated. The solution is diluted with an equal volume of water, a piece of mossy tin added, and the mixture warmed unless a blue colour develops in the cold. If this test gives negative results, about 0.5 gm. of the ore is fused with 4 grms. sodium carbonate, the melt dissolved by boiling water in the crucible, the solution acidified with an equal volume of concentrated hydrochloric acid, a piece of tin added, and the mixture warmed if necessary. This test will detect 2 per cent. of tungsten with certainty. Vanadium gives a blue colour on reduction, but tartaric acid will effect the reduction in the case of vanadium, whereas it will not reduce tungstic acid.
G. C. J.

APPARATUS, ETC.

An Adiabatic Calorimeter. F. Danills. (*J. Amer. Chem. Soc.*, 1916, **38**, 1473-1480.)—Richards' adiabatic calorimeter (*J. Amer. Chem. Soc.*, 1909, **31**, 1275; 1910, **32**, 268, 431, 1176; 1911, **33**, 863) works excellently, but the use of strong alkali is expensive and otherwise objectionable. Instead of relying on the heat of reaction between alkali and acid to control the temperature of

the jacket, electrical heating may be resorted to, but electric heating with coils is inadmissible because of the time lag. By making the whole water-jacket the conductor, however, its temperature rises instantly and uniformly throughout the passage of the current, nor is rapid stirring necessary, for the heat is not localised. A calorimeter designed on this principle is figured in the paper. The inner can or calorimeter proper is of thin copper, plated with silver filled with water (500 c.c.). It is set in a slightly larger nickel-plated vessel and supported on short pieces of very small glass tubing, so that there is everywhere an air gap of 0.5 cm. between the vessels. Six similar glass supports soldered to the sides of the inner can serve to centre it within the outer one. A brass collar is soldered to the outer vessel, and is tapered so that a brass cover sets down into it as a ground-glass stopper fits into its bottle. The outer vessel in its turn is supported within a third large copper water-jacket by nine glass tubes fitting snugly over short brass rods. A still larger vessel and packing of kieselguhr diminish the radiation. The water-jacket is provided with a stirrer of the propeller type, as is the calorimeter vessel itself. The vessel constituting the water-jacket and the intermediate can within it are connected with the terminals of a 100-volt alternating current through a knife switch, so that the current may be passed directly through the water of the jacket. All tubes leading into the calorimeter, whether of glass or of metal, are soldered to the cover, and there is, accordingly, no danger from leakage. In order to cut down the space into which the liquid can vaporise, a thin strip of mica is wired round the top of the inner can and projects above it so as to touch the cover. With this arrangement, liquid cannot evaporate to fill the air space at the sides and bottom, but only at the top. In most adiabatic calorimetry, two thermometers have been used, one in the calorimeter and one in the jacket. Rapid readings entail a considerable strain on the observer, so a thermocouple is substituted for them in the instrument now described. The filament of an ordinary tungsten lamp is focussed through a reading lens to a galvanometer on to a ground-glass scale or a white paper scale in a dark box. The galvanometer is placed about 5 metres from the scale and calorimeter. A difference in temperature of 0.01° C. between the inner can and the jacket gives then a deflection of about 1 cm. When the outer jacket is too cold the switch is closed, the current passes through the water and the temperature rises. When the thermocouple registers zero, the switch is opened and heating ceases at once. Means for the automatic control of the heating circuit by a selenium cell are also described, but, because the hand regulation is so simple, this selenium cell regulator is not recommended unless a large number of calorimetric measurements are to be made.

G. C. J.

Automatic Pipette. A. Lowy. (*J. Ind. and Eng. Chem.*, 1916, 8, 734-735.)—The pipette has a stopcock of special construction above the bulb. Fig. 2 shows the position of the cock while the liquid is being drawn up. Line *AB* (the lower limit of the plug, the letters *AB* being scarcely legible in the illustration) is the graduation mark. *F* is a cylindrical bore through the plug connecting *G* and *H* during suction. *E* is a cylindrical bore ending at the back of *F* at an angle of 90°, of which *C* is a continuation and ends at a slight elevation at *D*. Fig. 3 shows the

cock, with the plug turned clockwise 90° from position shown in Fig 2. *D* shows the place closed by thumb. In this position, *C* is connected with *H* through *E*, whilst *G* is shut off from *H*. With suction applied above *G* (Fig. 2) the liquid is drawn up above the cock. The thumb is then placed on *D*, the plug rotated clock-

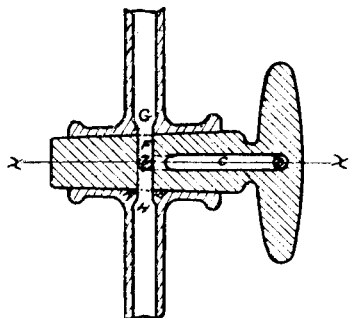


FIG. 2

Section at Y-Y

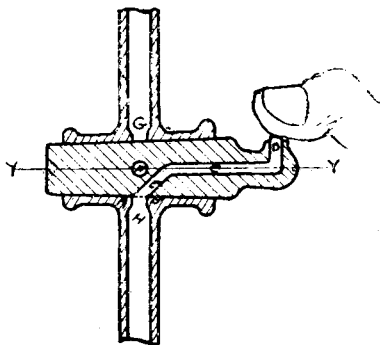
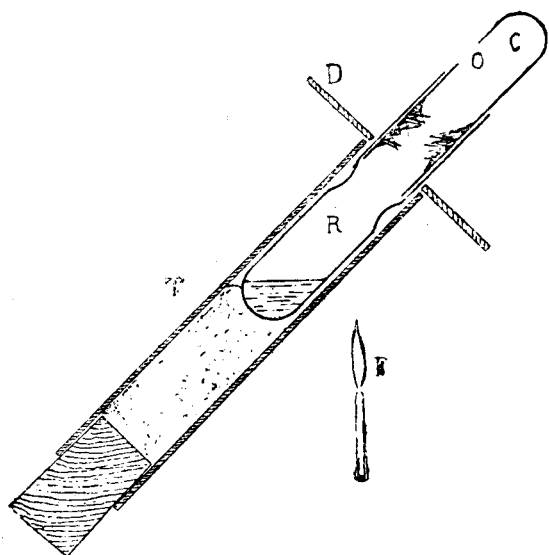


FIG. 3 - Section at X-X

wise 90° , and the thumb released. The pipette then delivers its contents up to, but not including the bore of plug. The excess in and above the plug is released by rotating the plug counter-clockwise through 90° , and is then ready for use again.

G. C. J.

Device for Subliming and Weighing Small Quantities of Iodine. G. Fouque. (*Bull. Soc. Chim.*, 1916, 19, 270-272.)—The iodine is introduced into a

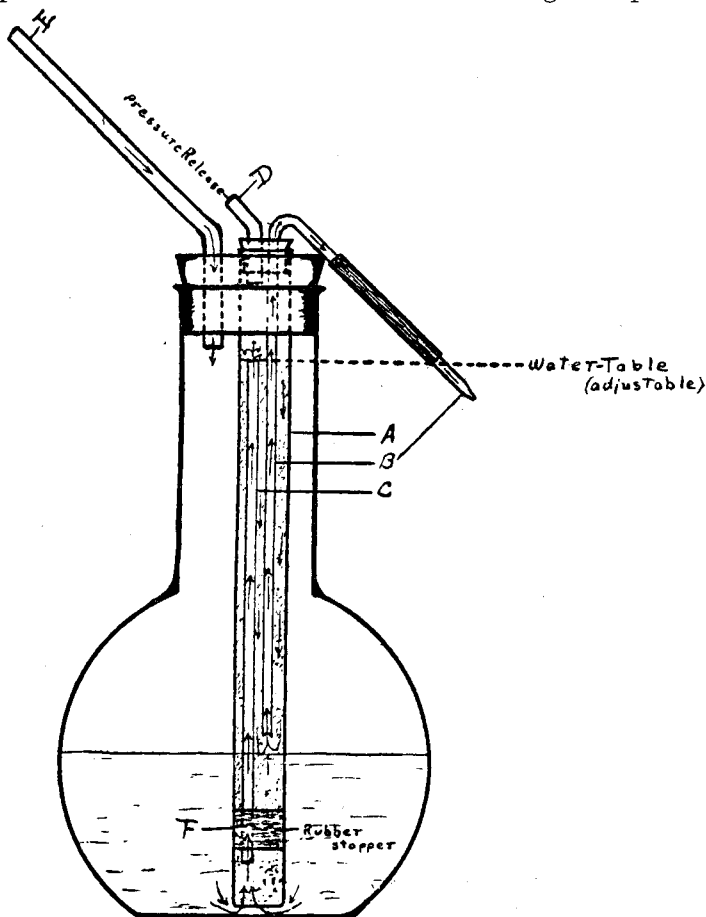


test-tube *R*, which has a constriction about 4 cm. from the bottom. Into this tube slides easily another tube *C*, with a small opening *O*, about 15 mm. from the end. The tube *R* is placed in a tube *T*, about 10 to 15 cm. in length, which is partially filled with sand and is inclined at an angle of 45° , and this tube is heated by means of a small flame about 15 mm. high which is directed toward a point just below the constriction in the tube *R*. The sublimation is effected at about the rate of 1 grm. per hour, and the iodine condenses in *C* at the other side of the asbestos guard *D*. Subsequently the tube *C* is withdrawn, covered by another tared

tube which encloses the opening *O*, left to cool in a desiccator and weighed. The yield of iodine recovered in test experiments with quantities of 2 to 6 grms. was, as a rule, over 98 per cent.

C. A. M.

Non-Spattering Wash-Bottle. F. C. Clapp. (*J. Amer. Chem. Soc.*, 1916, 38, 1502-1503.)—To avoid spattering, the water-table of the wash-bottle is raised above the level of the tip of the nozzle. "Back action" is thus prevented and the nozzle tube remains full of water. While in use, the thumb is held over *D* and water is forced up through *C* until the water-table indicated has been reached, the water-level being maintained by the stopper *F*. The nozzle works as though the bottle were full up to the raised water-level. Before releasing the pressure from the



mouthpiece, the thumb is taken off *D*, so that the height of the water-table in *A* may be insured and to prevent "back action" through *B*. *D* should be of large diameter (5 mm.) and project only to the bottom of the small cork in *A*. The size and length of *B* are not important so long as the bottom projects an inch or so below the water-table. The stopper *F* should be near the bottom of *A*, which should be 12 mm. in diameter and project up through the stopper of the wash-bottle and reach nearly to the bottom. *C* should be of small diameter (4 mm.). Its height determines the height of the water-table. G. C. J.

NOTE.—The water-table is the level of the water in the wide tube, the effective level from which the jet is fed, much above the water-level in the flask and high above the jet.

REVIEWS.

VINEGAR: ITS MANUFACTURE AND EXAMINATION. By C. AINSWORTH MITCHELL.
Charles Griffin and Co., Ltd. 1916. Price 8s. 6d. net.

Having regard to the great age and importance of the vinegar industry, it is strange, as the author remarks, that the literature should be so meagre and that there should not be in existence a single work dealing with the subject from the point of view of English practice. Brannt's "Treatise on the Manufacture of Vinegar and Acetates" has long occupied the position of a standard work of reference, but it is in many respects greatly behind the times, and there was unquestionably an opening for a book dealing more adequately with the scientific aspects of the industry and embodying the experience of a technologist acquainted with the manufacturing conditions obtaining in this country. Mr. Mitchell's qualifications for the task are of the highest order, and, in view of his exceptional experience, it is to be regretted that he has not seen his way to deal a little more comprehensively with the practical side of the manufacturing operations—particularly those concerned in the various methods of acetification.

As might have been anticipated, the book is written in a clear and attractive style, and whilst, on the whole, it affords evidence of careful revision, the reviewer has noticed a few errors to which attention is drawn with a view to their rectification in future editions.

Thus, on p. 87, it is stated that the object of flaking maize and rice is to gelatinise the starch granules, and it is added that, since "to some extent the heating does the work of diastase," such products have become known as "malts" in the brewing industry. The main object of steaming the grain in the manufacture of the products to which the author alludes is, of course, to break up the cell structures of the grain and so to facilitate the action of diastase on the contained starch granules. Gelatinisation of the starch is only an incidental effect, and one which the manufacturer endeavours not to carry too far. The term "malt" was adopted a good many years ago by certain manufacturers, and is not at the present time generally employed in the brewing industry. It was, of course, an entirely incorrect designation.

The statement that if the vinegar-maker were to boil his worts he would "convert dextrin into an insoluble form," is obviously incorrect. The statement that worts containing commercial glucose attenuate very far as compared with those containing molasses needs some qualification, since most samples of commercial solid glucose contain from 15 to 20 per cent. of unfermentable matters.

The analyses of prepared grain given on p. 177 are incorrect and very misleading. Thus, ordinary torrefied barley would contain an average of 55 to 60 per cent. of starch instead of 71 per cent., and over 20 per cent. of fibre instead of 6 per cent. as stated. On the other hand, two analyses of flaked maize are given, the one showing 15 per cent. of woody fibre and the other 0.8 per cent. Both these numbers are wrong, since flaked maize with an average moisture percentage would contain about 7 or 8 per cent. of fibre and about 70 per cent. of starch. The author states that

in the process of torrefying barley "the fat is lowered by about 50 per cent." This statement is not correct, nor is it borne out by the analysis on p. 177.

The section dealing with the acetic acid bacteria is well written, but in the present condition of the vinegar industry is of comparatively little practical value, since vinegar-makers rarely, if ever, employ pure species or even know the biological characters of the organisms they are using. The author, in fact, concludes this chapter by the statement: "It is to be feared that too little stress is laid upon the aroma of the vinegar brewed in England to make the use of pure cultures of bacteria appreciated from this point of view. On the other hand, it is possible that by the use of cultures of special species the loss of acid during acetification might be materially reduced." There is, in fact, considerable scope for the zymotechnologist in connection with the manufacture of vinegar.

The chapter headed "Methods of Examination" deals in the main with well-known methods, and calls for little criticism.

It is well recognised that in the colorimetric estimation of traces of metallic impurities, strict attention to details is essential if accurate results are to be obtained. In this connection the author might well have been more explicit. The experienced analyst is scarcely likely to refer to this book for assistance, whilst the inexperienced operator who did so would find, instead of detailed instructions, but little more than hints as to methods of procedure. The statement in the preface that the author has assumed "a general knowledge of analytical chemistry" on the part of his readers and has "omitted details to be found in any ordinary textbook" scarcely covers this point, since details of methods such as I refer to are not, as a rule, to be found in such textbooks.

For the detection of tin the author might well have referred to Schryver's colorimetric method, which is particularly well suited both for qualitative and quantitative purposes.

In dealing with arsenic, the author reprints in a condensed form the report of the Conjoint Committee of the Society of Chemical Industry and of the Society of Public Analysts, but does not refer to any of the work which has been done since, either in the direction of purifying the materials or of obtaining greater sensitiveness and increased certainty in the working of the process.

The interpretation of analytical results, the analytical standards for vinegar, and the so-called "malt vinegar question," are dealt with clearly and concisely. There are few who will not echo the author's complaint that the present position is unsatisfactory in the extreme and his hope that before long something may be done to "put an end to the present state of uncertainty and confusion." It may perhaps be suggested to the author that in future editions he might omit reference to the earlier theories of acetification, since these are only of historical interest and can be found at much greater length in many of the well-known standard works by those who are interested in the subject.

The illustrations and the print are good, and there is an adequate index.

A. CHASTON CHAPMAN.

VOCABULARY OF INDIAN MEDICINAL SUBSTANCES AND DRUGS. By C. M. GUPTA, F.T.S. Khosla Brothers, Lahore, India. Price Rs. 8 as. 8 net.

The work in question is of a nature which few persons could or would attempt to compile. It is not really a medical work and does not claim to be one, and the author admits that he has had no medical training, or such training as is regarded as necessary among Western nations. Indeed, the author would not have attempted to compile the work if he had been a medical man or with a Western training.

The book is an attempt to enumerate in alphabetical order the drugs and other materials used in medicine in India. In Section A the names are given in Urdu, which is largely used as a vernacular in India; in Sanskrit, in which the most ancient and learned writings of the East are to be found; in Arabic, in which the ancient knowledge of the Muhammadans is to be found; in Persian, used largely by present-day Muhammadans in India; in English and in Latin, and sometimes in Greek, Hebrew, etc. The medicinal properties and uses are also given, as described apparently in very numerous works, ancient and modern, and in the languages named, to which the author has referred, and also the doses or methods of application. As an indication of the nature of the work, under the word "Sona" in Urdu, which means "gold," and under which the author includes "gold calx" (!) and gold chloride, under very slightly modified Urdu names, the remarks given are as follows:

"A valuable tonic and alterative, increases strength and beauty, improves intellect and memory, clears the voice and increases sexual powers; it is used in fevers, consumption, insanity, epilepsy, syphilis, hysteria, impotency, etc., etc., B. P."

Part B contains details of the species and parts of some plants used in medicine; Part C gives the "forms into which each drug or medicinal substance can be prepared under certain processes"; and Part D is a "Statement showing Terminology of the Medical Science or Therapeutical Classification of Medicinal Properties of Remedies."

The author of this work, who states that he is a Government Pensioner, and formerly an assistant to the Director of Public Instruction, Punjab, has evidently in his leisure time tried to fit himself as one of a class of unofficial practitioners practising medicine in India without any licence, who in certain parts would be called a "kobiraj," and in whom a large number of the poorer and simple or less educated natives of India have much faith. Indeed, up to the present time in India, Government has not found it possible to require legal qualifications of training from such persons, though vernacular schools of medicine are being established and encouraged in various parts, which in time will make it possible to have regulations in India similar to those of Europe, but that time is still distant.

The value of such a work as this can be clearly seen from the extract on the medicinal uses of gold, gold calx, and gold chloride (all under one heading). On the other hand, from the fact that the author has apparently taken the trouble to learn a good many languages and has, so far as he could, studied several systems of medicine and read many books, it is clear that he has laboured hard to produce a valuable book. The corresponding names of the drugs, etc., in different languages may be useful in certain cases, but as the author does not appear to have had any scientific or medical training, such lists even may have a limited value.

ALEXANDER PEDLER.

ELEMENTS OF MINERALOGY. By FRANK RUTLEY. Nineteenth edition, revised by H. H. READ, with an introduction by G. T. HOLLOWAY. London: T. Murby. 1916. Price 3s. 6d. net.

That this is a useful little book is proved by the facts of its having reached a nineteenth edition, and of the last six editions having been run through in fourteen years. The reviser, while retaining in general the descriptive portion of the book as it was, has endeavoured to make it more useful to the prospector and the economic mineralogist, as distinguished from the purely scientific collector and classifier—the need for and desirability of doing which are argued with much skill and great clearness in Mr. Holloway's admirable introduction. Accordingly, we find a good deal of space in the aggregate devoted to the economic uses of minerals and metallic ores, and the properties of metals and other substances derived from them. All this, with the short accounts of methods of smelting and manufacture, and the statistical information inserted here and there, gives a sense of reality to the work which must be a healthy stimulant to the student, and deserves a hearty welcome.

The descriptions are terse and clear, and the classification, especially that of the silicate minerals, lends itself well to the picturesque spacing out of the whole domain, and hence to the acquirement and remembrance of its main features; and two most useful characteristics of the book are its description of the occurrence of minerals as original or derived constituents of their matrices, and of the usual association of other minerals with that momentarily under consideration. Could the student, along with this book, have access to a collection of minerals containing not only the finely crystallised specimens with which we are familiar in collections, but also the massive minerals as they occur with their associates in rocks, and ores as they are actually seen in mining and metallurgical industries, his progress would be both rapid and sure.

In the introductory portion of the book, the chapter on physical properties of minerals is, on the whole, clearly written, and those on crystallography are unusually good for so short a summary; but when one comes to the chemistry and the optical properties of minerals, one is tempted to ask whether the insertion of these chapters is not simply an old tradition which would best be honoured in its breach; whether they can be of any possible use to those to whom they are not superfluous. Let anyone honestly try to form a clear idea of what is likely to be the picture of things in the mind of the student who comes to this book ignorant of chemistry, when, after having absorbed in four pages verbal definitions of atoms, molecules, and valency, and a table of elements with their symbols and atomic weights, he then learns that an acid "is formed by the combination of hydrogen with a non-metal, or with a group of non-metallic elements, the hydrogen being capable of replacement by a metal. The group of molecules [*sic*] combined with the hydrogen is termed the 'acid radicle.'" Or let him try to put himself into the mental position of that same student, assumed to have had no teaching whatever in physics, when, opening the book hopefully at Chapter V., on the optical properties of minerals, he plunges into the statement that "the sensation of light is given to the retina of the eye by vibrations in the ether transverse to the direction of propagation. These vibrations are believed to be due to the rapid periodic change of the electromagnetic

condition of the ether." Would it not be better that he should simply take for granted the statement that muscovite is a silicate of aluminium, potassium, and hydrogen, or the fact that light which has passed through the polariser of a microscope cannot get through the analyser in certain positions, till he has elsewhere really learnt the meaning of the statement and the accepted explanation of the fact? These dogmatic summaries are, in my opinion, at the best useless, and from their very brevity often misleading, and wasteful of an earnest student's time. For instance, on p. 96 the student is told that the ratio of the sines of the angles of incidence and refraction never varies for the same two media, *no matter what* may be the obliquity with which the light falls on the surface; but on p. 101 he finds without warning that a ray, meeting a surface at an angle greater than the critical angle, undergoes total reflexion; on p. 94 he may work out for himself what *frequency* means, but he gets no assistance; and on p. 109 he will be a very extraordinary student if he does not suppose there is some mystic connection between crystal edges and the cross-wires of the eyepiece.

If reviser and publisher would have the courage in the next edition to break with the bad old custom of endeavouring to make every textbook of a branch of science a pocket encyclopædia of the related branches, expunge these chapters, tell the student to go to the chemists for his chemistry, the physicists for his physics, and devote the space to an expansion of the descriptive section, or of the purely manipulative side of the chemical and physical examination of minerals, this very good book would be of still greater value.

A casual run through the book discloses a few misstatements, ambiguities, and cases of doubtful English, which in the next edition might be amended. At the top of p. 45 it is the specific gravity of the pieces of mineral as an aggregate which is obtained, not of the several pieces "simultaneously"; limonite will certainly not be converted into magnetite (p. 21) by heating in a glass tube; manganese peroxide is not sold for chemical uses on a basis of 80 per cent. of Mn (p. 276), an obviously impossible percentage, but of MnO_2 ; and the day has surely gone by when gypsum is to be looked for as an adulterant of flour. Again, on p. 132 we find that sulphur "is insoluble, but is dissolved by carbon bisulphide," and on p. 161 the book sins with many others in using the word "weight" instead of "density." Lastly, a protest may be entered against the use of "somewhere round" in place of the ordinary English word "about." A careful reading of the next edition, which one may hope will be wanted after the usual interval, will remove these and similar blemishes.

J. T. DUNN.
