

# THE ANALYST

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

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AN Ordinary Meeting of the Society was held on Wednesday, June 7, 1922, at the Chemical Society's Rooms, Burlington House. The President, Mr. P. A. Ellis Richards, F.I.C., was in the Chair.

Certificates were read for the first time in favour of Messrs. George Scott Robertson, D.Sc. (Dun.), F.I.C., Frederick John Martin, M.A. (Cantab), A.I.C., and Frederick Stanley Shadbolt, A.I.C.

Certificates were read for the second time in favour of Messrs. Archibald Steele Whamond and Thomas John Ward.

Mr. Frederick Major, B.Sc. (Lond.), A.I.C., was elected a member of the Society.

The following papers were read:—"The Action of Natural Waters on Lead," by John C. Thresh, M.D., D.Sc., F.I.C.; "The Estimation of Meconic Acid in Opium," by H. E. Annett, D.Sc., F.I.C., and M. N. Bose, M.A.; "The Composition of Cows' Milk in the Sudan," by A. F. Joseph, D.Sc., F.I.C., and F. J. Martin, M.A., A.I.C.; and "The Use of the Daylight Lamp in Volumetric and Colorimetric Analysis," by W. Singleton.

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## "Certain Tropical Oilseeds."

BY E. R. BOLTON, F.I.C., AND D. G. HEWER, B.Sc.

(Read at the Meeting, April 5, 1922.)

**BACURY KERNELS.**—These kernels, which have been brought to our notice from several quarters, are likely to be of some importance in the future owing to the fact that they are available in large quantities in South America, and their collection on a commercial scale is said to be quite practicable. At least two firms have been prepared to ship considerable quantities, but, owing to the dulness of the markets and lack of demand, have not, to our knowledge, yet done so.

The seeds are the product of *Platonia insignis* (Natural Order *Guttiferæ*), and, although they contain a very large proportion of oil, the oil has unfortunately certain drawbacks which have hindered its coming into use, as it might otherwise have done.

The kernels are flattened and ovoid in shape, about  $1\frac{3}{4}$  in. long by 1 in. broad, and are surrounded by pulp. Five or six are contained in the pod-like structure, which is about the size and shape of a large apple. The seeds are soft, covered by a thin brown skin and pitted all over with small red-brown ducts. These ducts contain a resin-like substance which causes the extracted oil to be of a dark brown colour. The pulp surrounding the kernels is said to be eaten by the natives of Brazil.

The fat is firm in consistence, although not brittle, and owing to the difficulty of removing the brown pigment and traces of resinous matter, does not readily lend itself to the production of an edible oil. The high "titer" of the fatty acids suggests utility for candle-making, and after bleaching by chemical means the fat would be suitable for soap.

**ANDIROBINHA OR MAPIA SEEDS.**—The botanical origin of these seeds, which come from Brazil, has not so far been traced, but owing to the large proportion of oil which they contain, and the exceptional character of this oil, a description of them would appear to be of some interest.

Unfortunately the seeds are possessed of very hard thick shells which make up some 83 per cent. of the whole, and which would necessitate the use of a special machine for their removal. This operation should, for obvious reasons, be carried out in the country of origin.

The whole nuts are about 2 ins. long by  $1\frac{1}{2}$  in. wide, and contain a kernel  $\frac{3}{4}$  in. wide by 1 in. long. These kernels are covered by a reddish black skin which makes up about 15 per cent. of the weight of the seed. Each seed is characterised by two blunt points and a large light yellow scar.

The oil, which is a cream to green liquid, is readily extracted by means of petroleum spirit, and it will be seen that its analytical constants are very characteristic, *e.g.* a very high saponification value simulating those of the kernel oils of the Palm group; very high Reichert-Meissl and Kirschner values combined with an almost negligible Polenske value. In fact, the Reichert-Meissl value, with the one exception of Malukang Oil cited by Lewkowitsch, appears to be the highest recorded figure for a vegetable oil. These abnormal figures appear to be due to a large proportion of valeric acid.

The oil itself is somewhat bitter, and is unlikely to prove suitable for refining for edible purposes, but possessing as it does a high "titer," should be valuable for soap.

**BEY BEANS.**—These seeds, which outwardly bear some resemblance to *Mimusops Djave*, have been identified as a species of *Baillonella*. They are smaller than the common *Djave* nuts, and the shells are thinner, but the kernels contain about the same proportion of oil.

It will be noticed from the analytical figures that the oil has the same analytical characteristics as *Mimusops* oil, the figures for which are given for comparison, *e.g.* both are semi-solid oils of low saponification values and high unsaponifiable matter. The oil from *Bey* beans, however, has a very exceptionally high "titer." In neither case is the non-fatty portion of the seed likely to prove useful, since it is alleged to contain cyanogenetic substances, but provided the seeds are obtained fresh, the oils after suitable refining would serve for edible purposes.

**PARINARIUM SEEDS.**—Various types of *Parinarium* seeds have been described from time to time (*Bull. Imp. Inst.*, 1918, 16, 38; ANALYST, 1921, 46, 326), the oils of which appear to have widely different properties and consequently uses. It appears to be an exceedingly difficult matter to identify the various *Parinarium* species, and it may well turn out that some samples described under different native names may eventually prove to be identical.

The two samples here described were received under the name of "Pajura" and "Jaboty" respectively. "Pajura" consisted of reddish brown nuts weighing about 45 grms. each, having thick tough shells making up  $\frac{3}{4}$  of the total weight, and containing very oily kernels surrounded by husks. "Jaboty" was of quite a different appearance, and had dark grey irregular, somewhat friable, shells surrounding kernels not unlike ordinary Brazil nut kernels, and which made up nearly half the whole weight.

"Pajura" yielded a viscous yellow oil with drying properties, while "Jaboty" gave a firm yellow fat which is most probably edible. Both oils were of low acidity, and in the case of *Jaboty* fat a sample of seed in a very mouldy and decomposed condition gave a fat with an acidity of only  $6\frac{1}{2}$  per cent. The keeping properties would therefore seem to be good.

SEEDS OF THEOBROMA SPECIES.—Two species of seeds were examined:—

- (1) *Theobroma grandifolia*, "Cupu Assu."
- (2) *Theobroma bicolor*, "Lupu."

Both these are of the Natural Order *Sterculiaceae* and are closely related to the ordinary Cocoa Bean.

Cupu Assu has already been described by Bray and Islip (ANALYST, 1921, 46, 325), and the Lupu seeds were rather larger and flatter. Both seeds, in common with the cocoa bean, are characterised by the tightly folded cotyledons. The only real difference between the two samples under consideration is in the proportion of fat they contain. This fat is in both cases a creamy white solid, more or less lard-like in consistency, and there is little doubt that it could be refined for edible purposes, although the present samples, not being very fresh, gave fats of rather high acidity for this purpose..

	(1) <i>Platonia insignis</i>	(2) Andiro- binha	(3) <i>Baillonella</i> sp. Bey Beans	(4) Mimuseops Djave for comparison with (3) Pajura	(5) <i>Parinari- um</i> sp. Jaboty	(6) <i>Theobroma grandifolia</i> Cupu Assu	(7) <i>Theobroma bicolor</i> Lupu	
Shell	—	83	13	50 75 husk 6	50	20	25	
Kernel	—	17	87	50	19	50	75	
Per cent. of oil on kernel	70.0	70.8	63.0	65-70	76.5	42-53	47	62
Melting point	{ incip. °C. { complete °C.	34.4	—	33-9	—	40.6	34.3	30.0
		51.7	—	44-1	—	45.0	45.5	42.1
Setting point, °C.	43.3	—	—	—	38.9	—	25.6	
Saponification value	191.8	236.8	180.3	184.2	194.5	227.3	189.2	189.0
Unsaponifiable matter, per cent.	4.2	2-3	6.2	4.0	0.6	0.5	—	0.9
Iodine value	63.3	52.7	60.7	65.1	98.0	5-9	44.3	44.4
Butyrefractometer at 40°C. (Zeiss)	60.0	51.9	51.7	51.8	82.4	37.0	45.6	45.8
Free fatty acids as oleic acid, p.c.	13-30	4-16	30	9.3	0.3	1-7	22	11
"Titer", °C.	—	31.2	—	52.8	—	—	—	—
Reichert-Meissl. value	—	45.0	—	—	—	—	—	—
Polenske value	—	0.5	—	—	—	—	—	—
Kirschner value	—	26.7	—	—	—	—	—	—

## Investigation of Atkinson's Process for the Estimation of Potassium in the presence of Sodium, Magnesium, Sulphates and Phosphates.

BY S. J. WATSON, B.Sc.

(Under the Analytical Investigation Scheme.)

THIS process depends upon the comparative insolubility of perchlorate of potassium in methyl alcohol as compared with the sulphates, phosphates and perchlorates of sodium and magnesium. The procedure adopted by Atkinson (ANALYST, 1921, 46, 354) is as follows:—

“The mixture of salts is evaporated in a 300 c.c. beaker with perchloric acid until fumes of the acid are evolved, and to ensure complete removal of hydrochloric acid, if present, the evaporation with perchloric acid is repeated; 100 c.c. of acidified methyl alcohol are added, and the solution is maintained at the boiling point, with occasional stirring, for an hour. It is then allowed to stand overnight and filtered through a Gooch crucible, the transference and washing being effected with 50 c.c. of acidified methyl alcohol, and finally with about 20 c.c. of ethyl alcohol.”

(The alcohol was acidified with perchloric acid (sp. gr. 1.12) in the ratio of 95 of alcohol to 5 of perchloric acid.)

SALTS USED.—The potassium salt used for testing this method was the nitrate, which can easily be obtained pure. The other salts used were sodium sulphate and magnesium phosphate.

To obviate the necessity for numerous weighings, standard solutions were made up as follows:—

A. *Solution of Potassium Nitrate.*—This contained 10.7447 grms. per litre, and was prepared from potassium nitrate which had been heated at 100° C. in an air oven for some time, and allowed to cool in a desiccator. The strength of this solution was equivalent to 5 grms. of  $K_2O$  per litre.

B. *Solution of Sodium Sulphate,  $Na_2SO_4$ .*—This contained 10 grms. of the anhydrous salt per litre.

C. *Solution of Magnesium Phosphate,  $Mg_3(PO_4)_2$ .*—Ten grms. of the salt were dissolved in the least possible quantity of strong hydrochloric acid, and the solution made up to a litre.

PROCEDURE.—Every detail of the method described above was followed; the methyl alcohol used had a specific gravity of 0.82, and 95 per cent. by volume of it was acidified with 5 per cent. of perchloric acid of specific gravity 1.1. The Gooch crucibles, containing the usual layer of asbestos fibre, were dried to constant weight at 100° C.

EXPERIMENTAL RESULTS.—Blank tests were made with pure potassium perchlorate to find out the loss due to solution during the process:—

(1) Pure potassium perchlorate (2.005 grms.) was treated as in the actual method. At the conclusion the increase in the weight of the Gooch crucible, due to the potassium perchlorate, was 1.984 grms., so that the loss due to solution was 0.021 gm.

(2) In the second case, 1.997 grms. of potassium perchlorate were taken, and 1.976 grms. were obtained at the close, representing a loss, due to solution, of 0.021 gm. Experiments were then made with varying quantities of the three above-named solutions, and the results are tabulated below:—

No.	Potassium Nitrate taken = Grms. $K_2O$	Sodium Sulphate taken Grms.	Magnesium Phosphate taken Grms.	Potassium Perchlorate found Grms.	Potassium Nitrate calculated Per Cent.
1	0.250	0.200	0.200	0.700	95.02
2	0.250	0.200	0.500	0.700	95.02
3	0.250	0.500	0.200	0.693	94.06
4	0.250	—	—	0.708	96.10
5	0.250	—	—	0.711	96.52
6	0.250	—	—	0.713	96.78
7	0.250	0.500	0.500	0.728	98.82
8	0.250	0.500	0.500	0.710	96.38
9	0.100	—	—	0.276	93.66
10	0.100	0.200	0.200	0.278	94.34
11	0.100	0.100	0.100	0.279	94.68
12	0.100	0.200	0.100	0.280	95.02
13	0.100	0.100	0.200	0.281	95.36
14	0.050	0.100	0.100	0.133	90.26

In addition, the following two tests were made with pure potassium sulphate:—

No.	Potassium Sulphate taken Grms.	Magnesium Phosphate taken Grms.	Potassium Perchlorate found Grms.	Equivalent to Potassium Sulphate Grms.	Purity of Potassium Sulphate Per Cent.
15	0.504	0.500	0.791	0.497	98.61
16	0.508	0.500	0.798	0.501	98.62

If to the amount of potassium perchlorate found in Nos. 4, 5, 6, and 9, we add the amount lost by solution, *i.e.* 0.021 gm., we get the following figures:—

No.	Potassium Perchlorate found Grms.	Potassium Perchlorate found + 0.021 Grms.	Purity of Potassium Nitrate Per Cent.
4	0.708	0.729	98.60
5	0.711	0.732	99.03
6	0.713	0.734	99.53
9	0.276	0.297	100.45

From this it would appear that the low result obtained where the potassium salt alone is used is due to the loss by solution of some potassium perchlorate.

If this loss is to be compensated, at least 0.021 grm. of some other substance must be held out of solution by the potassium perchlorate.

In order to find out to what extent the compensating errors occurred, the potassium perchlorate obtained in some of the experiments was dissolved in 20 c.c. of hot water. One drop of barium chloride solution was added, and the precipitate produced was compared with some previously prepared standards containing from 1 up to 5 mgrms. of sulphuric acid in 20 c.c., to which had also been added 1 drop of the barium chloride solution.

Nos. 3, 7, 8, and 15 were thus dealt with, and the resulting precipitates were, in all cases, most nearly comparable with the standard containing the 5 mgrms. of sulphuric acid; three of them lay between the standards containing 4 and 5 mgrms., the fourth being slightly higher.

Standards containing 1 up to 5 mgrms. of phosphate (as  $P_2O_5$ ) were prepared, and the potassium perchlorate from Nos. 2 and 16 dissolved in 20 c.c. hot water and treated with a drop of ammonium nitro-molybdate (freshly made). In both cases only the merest trace of phosphate was noticeable.

In all probability the sodium and magnesium would only be held out of the solution together with the sulphate or the phosphate radicle.

The presence of sodium or magnesium as perchlorate is hardly likely, since both perchlorates are soluble in alcohol, as was shown by Kreider (*cf.* H. W. Wiley, *Principles and Practice of Agricultural Analysis*, Vol. II., pp. 584 and 588).

From the fact, pointed out by Caspari (Wiley, *loc. cit.*, Vol. II., p. 588), that if a fair excess of perchloric acid is left on the potassium perchlorate before the addition of the alcohol, it will ensure complete separation of phosphoric acid from the potassium, the absence of phosphoric acid from the precipitate is to be expected; since 15 c.c. of perchloric acid were used for the precipitation of the potassium, which represents about eight times the theoretical amount required; the acid being of sp. gr. 1.1.

As it is impossible to state with which bases the sulphate radicle was combined, all that can be inferred is that at least 5 mgrms. of sulphate (expressed as  $H_2SO_4$ ), were held out of solution by the potassium perchlorate, together with an almost negligible trace of phosphate.

This explains the low results, for the loss due to solution of the potassium perchlorate has not been compensated to any sufficient degree by substances held out of solution.

EVAPORATION WITH PERCHLORIC ACID.—Nos. 1, 2 and 3 were evaporated down twice, 10 c.c. of perchloric acid being used each time, whereas all the other experiments were carried out with only one evaporation after the addition of 15 c.c. of the acid.

It will be noticed that, despite the presence of sodium sulphate and magnesium phosphate, these three are at least a full 1 per cent. lower than in all other cases where the same quantity of the potassium salt was taken, even where it was used alone. Why this should be so I cannot say, but it is a point worthy of notice.

CONCLUSIONS.—The process in my opinion is unsatisfactory:

(1) Because the compensating errors necessary to overcome the loss due to the solution of some potassium perchlorate do not occur to an extent sufficient to balance this loss.

(2) Because the quantity of alcohol and perchloric acid used is too large for making the process economical, in view of the price of methyl alcohol and perchloric acid.

In the process the contents of the beaker are kept at the boiling point for an hour, during which time a large amount of the alcohol is lost by evaporation, even when the beakers are covered with watch glasses.

The recovery of the methyl alcohol from the waste, after filtration and washing, involves a considerable amount of trouble. This is due to the fact that the only satisfactory way would be by distillation in steam. In view of the large amount of acid which must find its way into the filtrate, a certain amount of esterification would take place on heating; and to make direct distillation possible, dilution of the waste with water would be necessary for safety; which, in fact, would involve the treatment of large volumes of liquid.

(3) Keeping the contents of the beaker at the boiling point for an hour, whilst perhaps necessary to ensure complete solution of undesirable substances, must also favour the solution of potassium perchlorate itself, although this would be, to some extent, diminished by allowing the beaker to stand overnight.

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## A Tropical Milk Supply.

By ALEXANDER BRUCE.

WITH a view to establishing standards for milk produced under tropical conditions, a series of analyses has been made of the milk of cows and buffaloes in the city of Colombo. Five hundred genuine samples of milk from cows and 500 from buffaloes, 1000 in all, were examined to give the necessary data.

In going over different dairies it was found impossible to get 250 individuals; hence the milk from some dairies, representing the milk from animals previously examined, had to be re-examined. The question of seasonal variations is eliminated, as there are no seasons in the Tropics as in the temperate climates.

Cows' MILK.—The samples examined were drawn from 16 dairies. The breeds found were Scind, Nellore, Cross Bred, Cape and Nellore, Australian and South Indian, Native and Coast; of these the Scind animal is the favourite with the dairies, and 101 animals of this class were selected. The South Indian also seems fairly common, and of these 67 were chosen. Of the others there were 4 Australian, 12 Nellore, 23 Cross Bred, 25 Native, and 17 Coast. The condition of all was generally good. The usual times for milking in Colombo are between 4 and 5 a.m., and between 1 and 2 p.m., so that usually 16 hours intervene between



evening and morning milking. Owing to these unequal intervals between milking, the yield of the morning milk was greater than that of evening milk, and the milk was of poorer quality.

The average composition of the different breeds is given in the following table:

TABLE I. (AVERAGE OF ALL SAMPLES.)

Breed	No. of Cows	Average Daily Yield in Pints	Specific Gravity	Total Solids Per Cent.	Fat Per Cent.	Solids not Fat Per Cent.	Ash Per Cent.
Scind .. ..	101	7.34	1030.2	13.48	4.92	8.56	0.73
South India .. ..	90	6.46	1030.0	13.72	5.02	8.70	0.68
Native .. ..	18	2.92	1029.1	12.95	4.49	8.46	0.70
Nellore .. ..	14	5.86	1029.2	13.45	4.85	8.60	0.73
Cross-Bred Coast .. ..	12	6.54	1031.7	14.25	5.12	9.13	0.72

The averages of the 250 morning and 250 evening samples were found to be as follows:—

	Morning	Evening	Total Average
Specific gravity .. ..	1030.5	1028.4	1029.5
Total solids, per cent. .. ..	12.81	14.45	13.63
Fat .. ..	4.16	5.89	5.03
Solids not fat .. ..	8.65	8.58	8.60
Water .. ..	87.19	85.55	86.37
Ash .. ..	0.71	0.69	0.70

All the estimations were made directly by standard methods, as the formula worked out by Richmond might not have been found to be accurate for the tropics; although only in a few cases has there been found a difference between the results calculated by Richmond's formula and those obtained by actual estimation.

If we collate the results with those obtained by Richmond for the Aylesbury Dairy Co. (*Dairy Chemistry*, 3rd ed., p. 306), we get the following comparative figures:—

	Morning Aylesbury	Evening Aylesbury	Morning Colombo	Evening Colombo
Total solids, per cent. .. ..	12.47	12.81	12.81	14.45
Fat .. ..	3.57	3.92	4.16	5.89
Solids not fat .. ..	8.90	8.89	8.65	8.56
Ash .. ..	—	—	0.71	0.69
Specific gravity .. ..	1.0322	1.0319	1030.5	1028.4

and the average is :

	Aylesbury Per Cent.	Colombo Per Cent.
Total solids .. ..	12.64	13.63
Fat .. ..	3.75	5.03
Solids not fat .. ..	8.89	8.60
Ash .. ..	0.75	0.70
Specific gravity .. ..	1.0320	1.0295

It will be seen from these results that Colombo milk is very markedly richer in fat than English milk.

On the general results of the analyses one may say that the high percentage of fat in the milk is due to the breed and the richer feeding. Poonacs (crushed oil cakes), and such like oily foods, being common to the country, are easily and cheaply obtained.

The specific gravity is a little lower than that of English milk, this being due to the large percentage of fat present. The total solids, however, are in excess of those of English milk, owing to the high percentage of fat present in the milk. From analyses not given in detail it has been found that the proteins are slightly higher than in English milk, and the sugar is a little less, whilst the average ash is also slightly less.

The ash of the milk was also analysed in order to find if it differed much from that of English milk; the following are the results:—

## ANALYSES OF MILK ASH.

	Morning Per Cent.	Evening Per Cent.	Average English Analysis Per Cent.
Iron .. ..	Trace	Trace	0·53
Lime .. ..	21·45	23·04	22·0
Magnesia .. ..	3·46	3·31	3·05
Potassium oxide ..	14·98	14·36	24·67
Sodium oxide .. ..	13·48	13·56	9·70
Phosphoric acid ..	39·04	38·40	28·45
Sulphuric anhydride ..	Trace	Trace	0·30
Chlorine .. ..	9·70	10·00	14·28

The figures represent the average of a number of samples. As each sample of milk came in and was tested, the ash left from the ash estimation was brushed into a bottle and reserved for the ash analyses. The analyses, it will be seen, agree fairly well with those obtained with English milk, but the phosphoric acid and sodium oxide are higher, potassium oxide and chlorine lower. As a means of forming an opinion as to the genuineness of milk, the hydrometer is of little use in this country, as cows' milk can be adjusted by the use of water, coconut milk or buffalo milk. The percentage of fat is of interest, but cannot be relied upon without confirmation, as milks have been examined which would pass the fat standard, but which, nevertheless, were not genuine. The most important figure is "solids not fat," and this falls whenever water is added. In some cases jaggery sugar is added, but this is easily detected by the usual methods used for recognising cane sugar. Coconut milk is used as an adulterant, the high percentage of fat being the useful constituent. If the fat is separated and tested in the refractometer, any considerable admixture should be detected.\*

\* NOTE.—Coconut oil gives butyro-refractometer readings of 33·5 to 35·5 at 40 C. and butter fat readings of 40 to 44, whereas on the oleo-refractometer butter gives readings of -29 to -35 at 45 C. and coconut oil about -59. Adulteration is generally "added water"; rarely does a case of "cream removed" occur.

BUFFALO MILK.—The average composition of 500 samples of buffalo milk was as follows:—

TABLE II.

	Yield in Pints	Specific Gravity	Total Solids Per Cent.	Fat Per Cent.	Solids not Fat Per Cent.	Sugar and Protein Per Cent.	Ash Per Cent.
Morning	4.54	1030.7	17.31	7.96	9.35	8.53	0.82
Evening	3.20	1029.4	18.43	8.97	9.46	8.66	0.80
Average	3.82	1030.0	17.87	8.47	9.40	8.59	0.81

Some of the fats ran as high as 12 per cent. and solids-not-fat to 15 per cent. The analytical methods used were those adopted for the cows' milks, and the results may be compared with some of the few data given by other workers:—

	Colombo (Cochrane)	Indian (Barry)	Transylvanian (Strohmer)	Egyptian		Average 500 Col.
				(Pappel & Richmond)	(Pappel & Hogan)	
Specific gr.	1.0275	1.031	1.0319	1.0354	1032.4	1.030
Total solids, per cent.	17.18	18.67	18.28	15.95	17.91	17.87
Fat	7.86	7.78	9.02	5.56	7.95	8.47
Solids not fat	9.32	10.89	9.26	10.39	9.95	9.40
Ash	0.82	—	0.77	—	0.78	0.81

It will be seen from these figures that buffalo milk is much richer than cows' milk, and a comparison is not out of place.

Average of 500 Samples	Specific Gravity	Total Solids Per Cent.	Fat Per Cent.	Solids not Fat Per Cent.	Sugar and Casein Per Cent.	Ash Per Cent.
Cows	1029.5	13.65	5.03	8.62	7.92	0.70
Buffaloes	1030.0	17.87	8.47	9.40	8.59	0.81

A series of nitrogen determinations was also made, the results being:—

	Nitrogen Per Cent.	Proteins Per Cent.
Morning	0.73	4.56 (average of 18 samples)
Evening	0.74	4.63 ( „ „ 27 „ )
Average	0.735	4.59 ( „ „ 45 „ )
Pappel & Hogan (Egyptian Buffalo)	—	4.16 ( „ „ 16 „ )

It will be seen that the percentage of nitrogen is higher than that in either English or Colombo cows' milk, which usually contain 0.5 and 0.6 per cent. respectively. Richmond considers that English milk should never contain less than 0.5 per cent. of nitrogen.

RATIONS.—The foods and rations used during the investigation are of interest, as they show a difference on comparison with the usual English Dairy ration:—

## ANALYSES OF CATTLE FOOD.

	Rice Bran Per Cent.	Dhall Husk Per Cent.	Rice Conjee Per Cent.	Wheat Pollard "A" Per Cent.	Wheat Pollard "B" Per Cent.
Moisture (loss at 100° C.)	10.00	11.30	95.74	12.50	11.80
Ether extract	7.60	2.30	0.66	3.50	3.80
Proteins	13.81	8.12	0.10	13.62	13.81
Carbohydrates	52.20	41.43	2.81	61.48	58.59
Fibre	6.75	29.65	0.07	6.20	8.40
Ash	9.64	7.20	0.62	2.70	3.60
	100.00	100.00	100.00	100.00	100.00
Soluble ash (water)	2.44	1.80	0.57	1.20	1.66
Containing nitrogen	2.21	1.30	0.016	2.16	2.21
Food units	105.7	67.4	3.2	104.3	102.6
Nutritive ratio	1.5	1.5.7	1.29.5	1.5.1	1.5

	Cotton Seed Cake Per Cent.	Poloondu Per Cent.	Gingelly Poonac Per Cent.	Mauritius Grass Per Cent.
Moisture loss at 100° C.	9.50	11.60	10.80	83.00
Ether extract	17.90	1.50	13.40	0.21
Proteins	15.75	22.75	31.75	2.01
Carbohydrates	32.74	53.59	29.00	7.67
Fibre	20.35	4.80	3.65	5.61
Ash	3.76	5.76	11.40	1.50
	100.00	100.00	100.00	100.00
Soluble ash (water)	1.36	1.32	1.50	0.89
Containing nitrogen	2.52	3.64	5.08	0.33
Food units	116.8	114.2	141.9	13.2
Nutritive ratio	1.4.7	1.2.5	1.1.9	1.4

It will be seen that the Ceylon ration is richer in fat and protein matter and poorer in carbohydrates than the English ration. In the latter the fat is about 0.4 lb. per day, proteins 2.5 lbs., carbohydrates 12.5 lbs., with a nutritive ratio of 1.5.4, whilst the Ceylon ration is about:—Fat, 2.5 lbs., per day, proteins 2.75 lbs., and carbohydrates 6.2 lbs., with a nutritive ratio of 1.4.2.

No inference can be drawn from the variation of ration on the yield and quality of milk, as the animals differed in breed, and conditions generally varied.



## DISCUSSION.

Mr. H. DROOP RICHMOND said that in his opinion the specific gravities of milk in tropical climates were slightly lower than they would be if determined in England. With his brother he had established the fact that the fat in milk, when drawn, was liquid and of lower specific gravity than when it solidified, and also that the solidification was a slow process when the milk was cooled, and *a fortiori* it would be still slower when the atmospheric temperature was high. In a tropical climate the fat would therefore be largely retained in the liquid state, and the maximum specific gravity which was usually accepted as the normal would not be attained. Mr. Bruce's figures for ash and for the composition of the ash would appear to indicate that the estimation was made at a somewhat high temperature, with consequent volatilisation of chlorides of the alkalis; the percentages of ash given were rather below those found in cows' milk elsewhere, and both the alkalis and the chlorine were lower in the ash than in English milk, and roughly to the equivalent extent. The higher phosphoric acid was in accord with the higher amount of proteins found, as the chief protein (casein) contained phosphorus which was found in the ash as phosphoric acid. It was to be regretted that Mr. Bruce had given neither his minimum percentages, nor the standards he deduced.

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 Note.

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

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## FORMATION OF CARBOLIC ACID DURING PUTREFACTION.

LAST year I had occasion to examine portions of an exhumed body for poisons. The body had been taken out of a river, but a postmortem examination showed that death was not due to drowning. Subsequently an insurance company, in which the deceased had heavily insured his life, refused to pay on the ground that deceased had committed suicide. The body was then exhumed (51 days after the post mortem), and portions sent to me for examination. I found no poison, with the exception of carbolic acid, which was present in appreciable amounts. It was estimated by distillation with dilute sulphuric acid and water, the distillate being redistilled and the carbolic acid estimated by means of bromine by comparison with a standard solution of pure phenol. The following results were obtained:—Kidney, 0.039 grm.; small intestine, 0.021 grm. per 100 grms.

Judging by the text books at my disposal, these amounts appeared to be far too large to have been produced by putrefaction, and I therefore tested the point by making the following experiments:—

Twelve sheep's kidneys were purchased. Four of these (A 1-4) were inoculated with liquid from the decomposing spleen from body of deceased; four (B 1-4) were inoculated with broth supplied by the Government Bacteriologist, Wellington, and said to contain *B. coli communis*, *B. lactis aerogenes*,

*Friedlander's bacillus*, *B. mucosus capsulatis*, and *Morgan's Bacillus*; and four (C 1-4) were untreated. Each kidney was then placed in a pint glass jar loosely covered with a glass lid, and kept in a dark cupboard at ordinary room temperature. After standing for various periods of from 47 to 94 days, the amount of carbolic acid that had been produced was estimated by the same methods that had been employed on portions of the body of the deceased.

The following results expressed as grms. per 100 grms. of kidney were obtained:—

		A1.	B1.	C1.
After	47 days	0·011	0·010	0·021
	57 „	0·015	0·017	0·022
		A2.	B2.	C2.
	74 days	0·025	0·012	0·005
		A3.	B3.	C3.
	84 days	0·018	0·028	trace
		A4.	B4.	C4.
	94 days	0·018	0·027	0·014

These results show that in the untreated kidneys (C 1-4) the amount of carbolic acid formed varied from a trace up to 0·022 gm. per 100 grms., and in the inoculated kidneys from 0·010 to 0·028 gm. per 100 grms. The experiments were started on the 2nd of June, and had they been conducted in warmer weather, the results, no doubt, would have been appreciably higher. The body of the deceased lay for 51 days at a temperature of probably five to ten degrees higher than the temperatures during the experiments.

The results are sufficient to prove that the amounts of carbolic acid found in the body of the deceased could have been formed by bacterial action.

In view of the fact that information in regard to the amount of carbolic acid that may be formed during putrefaction is difficult to obtain, I am of the opinion that the above results should be of value.

DOMINION LABORATORY,  
(DEPARTMENT OF INTERNAL AFFAIRS),  
WELLINGTON, N.Z.

J. S. MACLAURIN  
(*Dominion Analyst*).

## Notes from the Reports of Public Analysts.

*The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.*

### CITY OF BIRMINGHAM.

#### REPORT OF THE CITY ANALYST FOR THE FIRST QUARTER, 1922.

DURING the quarter 1412 samples were submitted for analysis, 1266 of which were analysed under the Sale of Food and Drugs Acts. Of the latter samples 1108 were bought informally, and of these 53 were adulterated. Of the 158 samples formally bought, 22 were adulterated.

**MILK.**—The average composition of the 565 samples examined was: Fat, 3·63; and solids not fat, 8·69 per cent. The proportion adulterated was 9·4 per cent.

**RICE.**—A formal sample contained 0·7 per cent. of mineral facing, and the vendor was cautioned.

**SPONGE CAKES.**—Samples contained 0·60 and 0·36 per cent. of boric acid respectively. The first vendor stated that he had used liquid eggs in preparing the cake, and the invoice for these eggs stated that they were preserved with a small percentage of boric acid, and should only be used as an ingredient for food products. The composition of the sponge cakes showed that about 2 per cent. of boric acid must have been present in the liquid eggs.

**CUSTARD POWDER.**—A formal sample contained 4·6 per cent. of protein, whilst the label stated that 24·37 per cent. was present. The vendor was cautioned for giving a false label in contravention of Sec. 27 of the Food and Drugs Acts.

**SEIDLITZ POWDERS.**—Of 12 informal samples 10 had been carefully prepared. One sample, marked "extra strong," contained twice the amount of Rochelle salt required by the B.P., instead of only  $1\frac{1}{2}$  times the amount.

**COLOURLESS TINCTURE OF IODINE.**—Nine of the 18 informal samples were prepared in accordance with the British Pharmaceutical Codex, 1911, and ten large wholesale drug houses stated, in reply to an enquiry, that they accepted the authority of the Codex for the preparation of this tincture. Six samples were practically free from ammonia, and contained from 0·8 to 4·9 per cent. of ash, which contained sulphates, showing that the iodine had been decolorised by thiosulphate instead of ammonia. One of the samples contained only 43 grms. (instead of 67 grms.) of alcohol per 100 c.c. The vendors of these samples were cautioned. One sample contained about 2 per cent. of potassium iodide; it had been incorrectly prepared by adding ammonia to ordinary tincture of iodine.

**GREGORY'S POWDER.**—Five informal samples were of the correct composition, but one sample was deficient of about 4 per cent. of rhubarb.

J. F. LIVERSEEGE.

## COUNTY OF LANCASTER.

### ANNUAL REPORT OF THE COUNTY ANALYST FOR THE YEAR 1921.

Of the 4919 samples, 378 (8·22 per cent.) submitted by the police and 22 (6·9 per cent.) submitted by inspectors of local authorities were adulterated. In most cases the adulteration was of a slight character, the percentage of serious cases being only 3·07 per cent., as compared with 3·34 per cent. in 1920.

**MILK.**—The number of adulterated, deficient or dirty samples of the 2484 samples taken was 297, or 11·96 per cent. Some milk sellers still have the idea that, if they have not wilfully abstracted cream the milk is "as it came from the cow." The proportion of cow dung exceeded 5 parts per 100,000 in the case of 0·48 per cent. of the samples, and exceeded 2 parts per 100,000 in 2·85 per cent.

**MARGARINE.**—One sample had an enclosed ticket bearing a picture of a cow. In future legislation it would be desirable to adopt the language of the Merchandise Marks Acts, which prohibits not merely false description in *words*, but "any other (false) indication, direct or indirect."

**GINGER.**—One sample consisted of the Japanese variety. This is usually so poor in all the essential properties of ginger, and frequently has such a disagreeable flavour peculiar to itself, that it is desirable that its sale as "ginger" without further description should not be allowed. Similar instances occur in the case



of "wild" nutmeg and "wild" mace, which are botanically similar to the genuine products, and, although commonly described as "wild," are cultivated as adulterants (*cf.* ANALYST, 1921, 46, 139).

**CORNFLOUR AND ARROWROOT.**—A sample of cornflour consisted of sweet potato starch. An undertaking was given not to sell it any longer as "cornflour," but the same variety of starch subsequently appeared in a sample of arrowroot, which in practice is always maranta starch.

**SAGO.**—In 24 cases tapioca was supplied where sago was asked for, and in no case was real sago obtained. This practice was upheld in the case of *Sandys v. Rhodes*, and, as a consequence, true sago has almost disappeared from retail shops.

**BORAX AND TARTARIC ACID.**—A number of samples of borax, cream of tartar and tartaric acid were found to contain lead or arsenic in excess of the B.P. limits. All the samples of arsenical borax were sold by grocers; but, although it might be contended that it was sold for household purposes, and not as a drug or preservative, one of the samples contained as much as 7 grains of arsenic per lb.

**GREGORY POWDER AND MAGNESIA.**—Several samples were found to contain or consist of magnesium carbonate instead of calcined magnesia. Proceedings were instituted in two cases. In the case of 5 samples where "magnesia" was asked for, the parcel was labelled "carbonate of magnesia." Two samples of "magnesia" consisted of magnesium sulphate, tartaric acid, sodium carbonate, and sugar, which was obviously a misconception of what the purchaser required.

**CHLORODYNE LOZENGES.**—A sample contained 1.65 per cent. of chloroform, but none of the other usual ingredients of chlorodyne. The proper description of such goods is "chloroform" lozenges, and they should not be sold to children.

**SWEET SPIRIT OF NITRE.**—A number of samples were deficient in strength. As the weather was warm at the time, no legal proceedings were taken, but the vendors were cautioned.

W. COLLINGWOOD WILLIAMS.

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## MEMORANDUM FROM ASSOCIATION OF PUBLIC ANALYSTS OF SCOTLAND

### ON ADMINISTRATION OF FOOD AND DRUGS ACTS.

At a meeting of the Association of Public Analysts of Scotland, held on December 16th, 1921, Messrs. R. M. Clark (the President) and R. T. Thomson were instructed to prepare a memorandum giving the views of the members of the Association (representative of practically all the districts administered under the Food and Drugs Acts) on points on which the Consultative Council on Medical and Allied services had been requested to report to the Scottish Board of Health. The following is an outline of this Memorandum, issued on February 14th, 1922.

**INADEQUATE PROTECTION OF THE PUBLIC AGAINST THE USE OF IMPURE FOOD AND DRUGS.**—In the unanimous opinion of the members of the Association the public is inadequately protected, the causes for this including the following: (1) Lack of uniformity in administration, and, in many cases, failure of local authorities to utilise their powers. (2) Inequality of sampling and inadequate number of samples taken. (3) Omission to take samples other than milk, butter and a few other articles. (4) Unsatisfactory sampling, due to multiplication of sampling officers, and lack of experience of many of them. (5) Ineffective control of administration by the Board. (6) Absence of legal standards for Foods and Drugs. (7) Lack of contact between Public Analysts and the Board. (8) Want of chemical officer at the Board.

**LACK OF UNIFORMITY IN ADMINISTRATION.**—In some districts there is adequate supervision and control; in others the administration is more or less a dead letter. Tables prepared by Dr. Tocher, showing the number of samples taken in every local area, indicate that this statement does not exaggerate the position. In 1913 the numbers of samples (per 10,000 of population) ranged from 4·8 to 36 in the burghs and groups of burghs, and from 0· to 28 in the counties, whilst in 1920 the corresponding figures were 2·9 to 42·1 in the burghs, and 0· to 23 in the counties. In Kilmarnock (population 34,729) only 4 samples (all milk) were taken in 1920.

The Board in a circular, dated April 1, 1920, attributed this want of uniformity to shortage of staff and pressure of special work during the war, but Dr. Tocher's tables show that many local authorities completely failed to exercise their powers prior to the war. In the burgh of Helensburgh, for instance, not a single sample of milk was taken during the years 1907 to 1913. During the four years 1910–1913 there were 38 informal samples of butter taken, 25 per cent. of which were reported to be margarine, but, in spite of these reports, not a single formal sample was taken, to enable proceedings to be instituted.

**INEQUALITY OF SAMPLING.**—The failure in this particular is attributable (a) to the sampling officer, and (b) to the local authority. As the food and drugs work is subsidiary to the usual work of Sanitary and Police Inspectors (the usual sampling officers), and there is no control, there is little incentive in the direction of efficient sampling. On the other hand, an inspector gets credit from his Health Committee for keeping down expenses, especially in the small burghs.

Each local authority is also anxious to keep the expenses down, and, as in most cases the public analyst is paid a fixed fee for each sample analysed, reduction of the number of samples also reduces the expense. This is not possible in the case of public analysts who are paid a fixed salary.

The influence of this factor upon the number of samples is clearly shown by the following figures for two neighbouring burghs:—

Burgh (Population 32,895). *Analyst paid by salary for 50 samples per annum.*

	1912	1913	1914	1915	1916	1917	1918	1919	1920	1921
No. of samples submitted	44	48	58	51	48	44	46	54	42	48

Burgh (Population 34,729). *No salary. Fixed fee per sample.*

	1912	1913	1914	1915	1916	1917	1918	1919	1920	1921
No. of samples submitted	49	47	29	19	9	6	22	30	4	28

**INEFFECTIVE CONTROL BY THE BOARD.**—As the Board receives a quarterly report from the Analyst, the failure of certain local authorities to administer the Acts properly must be within the Board's cognisance. It is suggested as a remedy that the Board should fix the minimum number of samples to be taken per 1000 of the population per annum, and that there should be some official at the Board to see that these instructions are carried out.

**OMISSION TO TAKE SAMPLES OTHER THAN MILK, BUTTER, ETC.**—According to Dr. Tocher's tables, only 3 per cent. of the samples taken in Scotland during 1913 were drugs, and only 8 per cent. articles of food other than 14 articles (of which the chief were milk and butter). The results for 1920 were very similar.

This is attributed partly to omission of instructions from the Board as to the nature of the samples to be taken. It should be the duty of an official of the Board to digest information collected from various sources, and thereafter advise local authorities as to matters of importance happening in other areas.

**IMPROVED METHODS OF SAMPLING AND APPOINTMENT OF WHOLE-TIME SAMPLING OFFICERS.**—As there are several objections to the Sanitary Officers of

small burghs being the sampling officers, it is suggested that whole-time Sampling Officers, or Food Inspectors, should be appointed for each large town or county, and that smaller counties and burghs should jointly appoint such an official. Lack of experience on the part of Sampling Officers has, in many cases, been the cause of samples not being uniformly divided, with the result of differences between the analyses of the Official Analyst and the analyst for the defence.

**POWERS OF THE BOARD OF HEALTH.**—So far back as 1898-9, the English Local Government Board gave an indication as to the number of samples which should be taken in relation to the population. In the Board of Agriculture (Intelligence Division) Annual Report, 1900, it was found necessary to remove the supposition that a number of samples equivalent to 1 per 1000 of the population was officially recognised as sufficient for the purposes of the Acts. In the Report of the Food Products Committee, 1894, the view was expressed that a proportion of samples much in excess of 1 per 1000 should be taken in the district of every Local Authority.

It is suggested in this Memorandum that the Scottish Board of Health have powers to fix the minimum number of samples to be taken per annum, and to compel each Local Authority to conform to this standard.

**THE ABSENCE OF STANDARDS.**—In the case of most articles the Public Analyst is expected to fix a standard based largely on his own experience. This is unsatisfactory and costly both to the Public Authority and to the defence, and smaller authorities are not prepared to incur the expense. There should be legal standards for all foods and drugs.

The standards recommended in certain Reports issued by the English Local Government Board seem to have been accepted in the English Courts, but are of no value in Scotland. For example, in the Lanarkshire bleached flour case (1913) no reference was allowed by the Sheriff to the English Local Government Report on the subject, or to any information contained therein. Should the Scottish Board of Health approve of the standards suggested in the English Reports, definite regulations embodying those standards would have to be issued by the Board before convictions could be obtained and the public protected.

The articles for which standards are urgently required include: Fat in cheese, non-fatty solids in buttermilks, sand in cinnamon, starch and glucose in jams and jellies, saponin and preservatives in aerated water and non-alcoholic wines, boric acid in sausages, etc.

**LACK OF CONTACT BETWEEN PUBLIC ANALYSTS AND THE BOARD.**—Public Analysts are not consulted when new regulations are to be issued. They are anxious to co-operate with the Board, but get no encouragement to do so.

**LACK OF CHEMICAL OFFICER AT THE BOARD.**—A chemical authority at headquarters is needed. Such an official should advise the Board on all chemical matters, and be responsible for the proper administration of the Acts by the Local Authorities.

His work could also include: (1) Working out uniform methods of analysis. (2) Recording and collecting data for establishing standards. (3) Drafting regulations to deal with certain impure foods and drugs which at present evade the Acts.

His presence would also ensure that no Regulations (*e.g.* Ministry of Food Orders: Bread and Flour) were issued when it was not possible to ascertain whether the articles referred to complied with those regulations.

**REMEDIES FOR THE DEFECTS IN ADMINISTRATION.**—Based on the foregoing considerations the following recommendations are made:—(1) Appointment of whole-time Sampling Officers or Food Inspectors. (2) Issue of instructions by the Board as to the number and nature of samples to be taken, with guidance as to the manner and methods of sampling to be employed. (3) Legal standards

fixed for all food and drugs (after consultation with advisory committee consisting of manufacturers, analysts, medical officers, and other parties. (4) Standard methods of analysis established for all foods and drugs. (5) More effective control of administration by the Board. (6) Appointment of Chemical Officer at the Board.

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### GERMAN REGULATIONS FOR THE DENATURATION OF SPIRITS OF WINE.

In accordance with Sec. 13 of the regulations of the German Spirit Monopoly, April 8, 1922 (*Zentralbl. für das Deutsche Reich*, 1922, p. 166; *Reichszollbl.*, 1922, p. 110; *Chem. Zeit.*, 1922, 46, 476), the following substances may be added to those permitted to be used for "denaturing" spirit intended for use in the manufacture of perfumery or cosmetics: Two litres of the diethyl ester of phthalic acid, or 1 litre of oil of turpentine, or 0.5 kilo. of thymol per 100 litres of spirit.

These substances must have the following characteristics: *Diethyl ester of phthalic acid* must be a colourless to yellowish liquid, with a slight odour of an essential oil, and must boil between 282° and 295° C. *Oil of turpentine* must answer to the requirements given under I, B, V, of Part II. of "Technical Estimations" in the Regulations. *Thymol* must consist of colourless, nearly transparent crystals or a crystalline mass, and when heated must volatilise without leaving an appreciable residue: it must melt between 45° and 51° C.; it must sink in water, but when melted must float on the surface. When a few granules are dissolved in 1 c.c. of glacial acetic acid and the solution treated with 6 drops of concentrated sulphuric acid and 1 drop of concentrated nitric acid a green coloration must be produced.

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## ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

### Food and Drugs Analysis.

**Composition of Meat Products, especially of those rich in Water. E. Feder.** (*Zeitsch. Unters. Nahr. Genussm.*, 1922, 43, 193-199.)—Analyses have been made of the flesh from oxen suffering from intense malnutrition or from disease. Marked increase in the absolute water-content and a high value of the ratio of water to organic non-fat is accompanied by a very small proportion of fat. The constants of the latter, particularly the saponification value, which is distinctly low with the samples examined, serve to indicate the condition of the slaughtered animal as regards nutrition. Although in cases of extreme malnutrition the fat almost completely disappears from the whole body, cholesterol is still found in considerable quantities in the ethereal extracts.

Estimations have also been made in samples of meat of the water-soluble nitrogen and of the "residual nitrogen" remaining dissolved after precipitation by means of trichloroacetic acid. With six samples of beef obtained from healthy

animals and having values of the ratio, water : organic non-fat, below 4, the residual nitrogen amounted to more than 45 per cent. of the soluble nitrogen, whereas with various samples showing high values of this ratio it fell to 33.1 per cent. Relations similar to that existing between the soluble and residual nitrogen are found in some cases between the refraction of the aqueous extract of meat and that of the filtrate obtained after heating this extract. Instances have recently been observed in which the numbers for organic non-fat and nitrogenous constituents exhibit considerable divergence, and it appears inadmissible, especially with samples of meat poor in fat, to estimate the fat by calculation. If the fat is calculated according to Grossfeld's suggestion, the ash-content must be estimated, as well as the proportions of water and nitrogenous substances, the method being more tedious and more expensive than that now proposed. T. H. P.

**Detection of Fructose in the presence of Aldoses.** I. M. Kolthoff. *Chem. Weekblad*, 1922, 19, 1-2.)—Two c.c. of a 1 per cent. solution of the mixed sugars are treated with 4 c.c. of 0.1 *N* iodine solution and then with 5 c.c. of 2 *N* sodium hydroxide solution, the tube shaken and left for 1 to 5 hours, and the excess of iodine removed by adding a few drops of *N* sodium thiosulphate solution. Two c.c. each of Fehling solution A and B are now added, and the tube heated in boiling water for not longer than 5 minutes. In the presence of fructose a precipitate of cuprous oxide will be obtained, and the amount may be approximately estimated by the time taken for the precipitate to form. Thus 5 per cent. of fructose produces a precipitate within 1 minute, 2.5 per cent. within 2 minutes, and 1 per cent. in 4 minutes. If the heating is continued longer than 5 minutes, reduction occurs, even in the absence of fructose. The method enables 1 per cent. of fructose to be detected in the presence of sucrose or dextrin; 0.25 mgrm. of fructose in presence of 20 mgrms. each of dextrose and sucrose; and 0.2 mgrm. of fructose mixed with 10 mgrms. each of lactose, dextrose and sucrose.

**Clarification of Solutions containing Reducing Sugars by Means of Basic Lead Acetate. Effect of Different Agents for Removing Lead.** D. T. Englis and C. Y. Tsang. (*J. Amer. Chem. Soc.*, 1922, 44, 865-867.)—The loss of reducing sugars varies when lead is precipitated in different forms; it is primarily caused by occlusion of sugar in the lead precipitate, and is much reduced by washing the precipitate. The loss with fructose is much greater than with dextrose, and carbonates, sulphates and oxalates cause considerable loss, whereas with sodium phosphate the loss is almost negligible, and there is the advantage that a clear filtrate is easily obtained. Tannic acid also causes but little loss. Sodium phosphate is therefore the best agent for removing lead. H. E. C.

**Preparation of Pure Sucrose for Standardisation of the Saccharimeter.** A. Kraisy. (*Zeitsch. Ver. deut. Zuckerind.*, 1921, 71, 785-797; *Chem. Abstr.*, 1922, 16, 1022-1023.)—Pure sucrose, for use in re-determining the one hundred point of the saccharimeter, is best prepared by precipitation of the sugar with alcohol and re-crystallisation from a slightly alkaline solution. From 1 to 1.5 kilos.

of sugar is dissolved at 65° C. in recently boiled water containing 80 to 100 mgrms. of sodium carbonate per 500 c.c., to form a 70 to 73 per cent. solution. This is filtered through ordinary filter paper in a Büchner funnel, if necessary after the addition of some alum in a cream. The filtrate is cooled nearly to the temperature of the room and treated with 1.5 vols. of freshly prepared absolute alcohol, which is added in small portions at a time, with frequent stirring. The supersaturated solution is filtered through hardened paper with the aid of slight suction, and the first half of the filtrate returned. The filtrate is collected in a large separating funnel (to protect it from dust), and is afterwards transferred to a large porcelain mortar, where it is allowed to crystallise, being meanwhile rubbed, from time to time, with a pestle. After 2 to 3 hours the crystals are drained by suction on a hardened filter in a Büchner funnel, washed several times with hot alcohol, and dried, first on porcelain plates to a moisture content of 0.03 per cent., and finally *in vacuo* at 30° to 50° C. To obtain sucrose of the highest degree of purity the whole operation must be repeated two or three times. A specimen thus prepared contained 0.006 per cent. of ash, and 10 grms. of it reduced 1.5 to 1.8 mgrms. of copper. It was therefore sufficiently pure for standardising the saccharimeter.

**Composition of Lees Wine and Wine Lees. L. Semichon.** (*Comptes rend.*, 1922, 174, 1179-1182.)—The turbid wine separated from the marc gradually clarifies and, after a few weeks, is separated into a racked wine and into a thick residue of lees wine, which is again separated by decantation a few weeks later, the sludge then yielding further wine when pressed. The decanted wine and the pressed wine closely resemble one another in composition. When compared with the original racked wine they show the following characteristics: About 2 per cent. of alcohol less by volume; identical total, fixed and volatile acidity; at least 50 per cent. more dry extract; slight increase in the total ash, the soluble ash being diminished and the insoluble ash increased; diminution by about one-third of the alkalinity of the ash; diminution of the cream of tartar and of the total tartaric acid by more than 50 per cent., the total potassium oxide being diminished by scarcely 10 per cent.; increase of the phosphoric acid by more than 100 per cent.; appreciable diminution in the sum of the acid and alcohol, the ratio (acid: alcohol) showing an increase; decrease of the ratio (alcohol: extract) by 50 per cent. These results show that the potassium bitartrate which disappears during the conversion of the original wine into lees wine is replaced by a virtually equivalent proportion of potassium biphosphate. Such distinctive properties of lees wine originate in the digestion, during several weeks, of the old yeast cells which comprise the bulk of the lees, and serve for the identification of such wine. It would obviously be advantageous to filter the lees from the first racking in a press since the wine thus separated would have sensibly the same composition as the racked wine itself.

T. H. P.

**Estimation of Esters in Imitation Flavouring Extracts. G. F. Beyer.** (*J. Ind. Eng. Chem.*, 1922, 14, 324-325.)—One hundred c.c. of 50 per cent. alcohol and 10 c.c. of the extract are placed in a 500 c.c. flask which is connected with a

vertical condenser; the lower end of the condenser is fitted with a rubber stopper carrying a mercury valve, and a receiving flask is attached to the stopper. The receiving flask is surrounded by a bath containing ice-water. The alcoholic mixture is distilled until 100 c.c. of distillate have been collected, the distillate then transferred to a 500 c.c. flask, the receiver rinsed out with 50 per cent. alcohol, phenolphthalein is added, and the solution neutralised with 0.1 *N* sodium hydroxide solution; a definite excess of this solution is then added, the flask is closed, placed aside for about eighteen hours at ordinary temperature, heated on a water-bath under a reflux condenser for thirty minutes, cooled, and the excess of alkali titrated with 0.1 *N* acid. The quantity of ester present may be expressed in terms of ethyl acetate.

W. P. S.

**Reports on Lavender Oil. Établissements Antoine Chiris.** (*Perfumery Essent. Oil Record*, 1921, 12, 404-406; *Chem. Abstr.*, 1922, 16, 988-989.)—The rotatory power of lavender oils obtained by open-fire distillation increases with the ester content, rising from  $-5^\circ$  for oils poor in esters to  $-8^\circ$  for superior oils. The ester content of oils thus distilled rarely exceeds 48.5 per cent. On the other hand, the rotatory power of steam-distilled oils containing more than 40 per cent. of esters varies between  $-6^\circ$  and  $-9^\circ 30'$ . Other conditions being similar, lavender oil distilled with steam contains from 7 to 8 per cent. more esters than that obtained by fire-distillation. A specialist can classify fire-distilled oils, but not the steam-distilled products, by their odour. Steam-distilled oils are less soluble, have higher densities, and no longer satisfy the requirements of the U.S.P. Hence, it is suggested that the U.S.P. figures for solubility and density require revision.

**Oil of Cade. R. Huerre.** (*Bull. Sci. Pharmacol.*, 1921, 28, 508-12; *Chem. Abstr.*, 1922, 16, 788.)—Twenty-five c.c. of the sample are shaken with a solution of 10 grms. of sodium hydroxide in 26 c.c. of water, and the mixture left for six hours and then extracted with four portions (25 c.c. each) of water, or until the washings are free from alkali. The aqueous solution is treated with ether, the ethereal extract dehydrated with anhydrous sodium sulphate, filtered and distilled, and the residue weighed. Five grms. of this residue are treated with 15 grms. of glacial acetic acid saturated with hydrogen chloride, care being taken to prevent the temperature rising, and, after standing for 12 hours, the mixture is transferred to a crystallising dish. Any crystals (cadinene dihydrochloride) forming within 24 hours are filtered off, dried at the ordinary temperature and weighed.

Positive results were obtained with all the samples of oil of cade examined, whilst no crystals were found in the case of thick oil of pine, oil of *Juniperus virginiana*, of *Cedrus libani* or of *Cedrus atlantica*. The conclusion is drawn that when the crystals are found in the case of a pyrogenic oil it has been derived from dry distillation of *Juniperus oxycedrus*.

D. G. H.

**Chemical Composition of Belladonna Leaves. A. Goris and A. Larsonneau.** (*Bull. Sci. Pharmacol.*, 1921, 28, 499-503; *Chem. Abstr.*, 1922, 16, 787.)—On extracting belladonna leaves so as to obtain the volatile bases as

sulphates, adding sodium carbonate, and extracting the mixture with ether, the extract contained not more than 2 per cent. of pyridine, the greater part of the volatile bases consisting of methylpyrroline and pyrrolidine. The aqueous residue from the ether extract was found to give indications of the presence of a 1·4-diamine, possibly tetramethyldiaminobutane. It is a point of practical interest that such volatile bases are included by the usual methods employed for estimating the fixed alkaloids, and are weighed as atropine. D. G. H.

**Adrenaline Tests.** L. Zechner and F. Wischo. (*Pharm. Monatsh.*, 1921, 2, 141-5; *Chem. Abstr.*, 1922, 16, 787.)—The authors give results obtained with various adrenaline tests in which the following oxidising agents which develop characteristic colorations are used: *Ferric chloride*: The optimum concentrations and quantities are: One drop of a 50 per cent. solution for adrenaline solutions of 1:100, 1 drop of a 5 per cent. solution for solutions of 1:1000, 1 drop of a 0·5 per cent. solution for lower concentrations, with, in each case, an optimum temperature of 10 to 15° C. *Potassium dichromate*: One drop of a 5 per cent. solution added to 1 c.c. of 1:1000 adrenaline solution gives colorations ranging from yellow to orange and red, and finally brown, with turbidity and eventually flakes, whereas no turbidity is produced on adding 1 drop of a 0·5 per cent. solution of potassium permanganate. *Potassium permanganate*: One drop of a 0·1 per cent. solution gives a red coloration and is sensitive in dilutions of 1 in 100,000. D. G. H.

**Commercial Hydrogen Peroxide containing Preservatives.** P. Poetschke. (*J. Ind. Eng. Chem.*, 1922, 14, 181-185.)—A sample of commercial hydrogen peroxide examined contained 11·75 grains of benzoic acid, 0·93 grains of salicylic acid and 1·28 grains of acetanilide per gallon. Results of tests to determine the efficiency of these substances, separately or together, showed that a mixture of benzoic acid and salicylic acid in the proportions mentioned had a decided preservative effect, and that this was not enhanced by the addition of acetanilide. A mixture of quinine sulphate (2·3 grains) and saccharin (1 grain per gallon) also has a preservative action; the saccharin masks the bitter taste of pure hydrogen peroxide, but does not appear to increase the keeping qualities of the peroxide. Without the addition of quinine sulphate, hydrogen peroxide lost 37 per cent. of its available oxygen in four weeks; when quinine sulphate was present the loss was only 14 per cent. in the same period. W. P. S.

## Bacteriological, Physiological, etc.

**Toxicology of Arsenic.** N. Tarugdi. (*Boll. Chim. Farm.*, 1921, 60, 569-576; *Chem. Abstr.*, 1920, 16, 539.)—Excised organs of guinea pigs which had been poisoned with disodium hydrogen arsenite were left to putrefy for four weeks or longer. The garlic-like odour characteristic of arsine or alkylarsines had then developed, and when the air of the container was passed through silver nitrate solution and the latter tested in the Marsh apparatus, arsenic was found. Animal tissues, such as muscle and liver, taken from normal animals and treated with



disodium hydrogen arsenite in an amount insufficient to stop putrefaction, also produced volatile arsenic derivatives. When, however, the mixture was sterilised by heating, or was treated with an excess of arsenic, no volatile arsenic could be detected.

**Toxicity of Neo-Arsphenamine.** M. C. Hart and W. B. Payne. (*J. Amer. Chem. Soc.*, 1922, **44**, 1150-1160.)—Experiments with rats show that the toxicity of commercial samples of neo-arsphenamine ranges from 200 to 360 mgrms. per kilo. weight of animal. Variability in the test rats may introduce considerable discrepancies in the results, and a method is suggested for the standardisation of the rats. The toxicity of arsphenamine is shown to have a negligible influence on that of the neo-arsphenamine derived from it and the effects on this conversion, of variations in the conditions are indicated. A curve is given showing the acute lethal activity of a freshly-prepared solution of neo-arsphenamine.

T. H. P.

**Destructive Action of the Amylase of Cows' Milk on Various Kinds of Starch.** F. Welzmüller. (*Biochem. Zeitsch.*, 1921, **125**, 179-86; *Chem. Abstr.*, 1922, **16**, 937.)—The optimum temperature for amylase from milk is about 37° C., whereas that of malt or pancreas diastase is about 54° C. The activity of milk amylase begins to be impaired at 42° C. It hydrolyses the starches of peas and beans almost as readily as it does potato starch, whilst the "easily digested" grains are much less easily attacked.

R. F. I.

**Estimation of Peroxidase in Milk.** F. E. Rice and T. Hanzawa. (*J. Ind. Eng. Chem.*, 1922, **14**, 201-202.)—A method proposed originally by Bach and Chodat (*Ber.*, 1904, **37**, 1342) for the estimation of peroxidase in plant extracts is recommended; it depends on the oxidation of pyrogallol to insoluble purpurogallin by hydrogen peroxide, peroxidase acting as a catalyst in the reaction. The details of the method as applied to milk are as follows: Fat is first removed by submitting the slightly warmed milk to centrifugal action; 10 c.c. of the separated milk are then placed in a stoppered bottle, and 50 c.c. of water, 10 c.c. of 5 per cent. pyrogallol solution and 10 c.c. of 1 per cent. hydrogen peroxide solution are added. The mixture is shaken thoroughly, the bottle then filled completely by adding a mixture consisting of 60 c.c. of water, 10 c.c. of 5 per cent. pyrogallol solution and 10 c.c. of 1 per cent. hydrogen peroxide solution, the stopper is inserted tightly, and the mixture kept at ordinary temperature for seven days. At the end of this time the mixture is filtered through a weighed Gooch filter, the precipitate washed first with the pyrogallol-hydrogen peroxide mixture mentioned, and then with as small a quantity of water as possible; the washing is complete when the washings cease to give a dark blue coloration with ferric chloride solution. The filter and its contents are dried, washed with a small quantity of petroleum spirit to remove traces of fat, again dried, and weighed. The weight of the purpurogallin precipitate (in mgrms.) yielded by 10 c.c. of milk is termed the peroxidase number. This value is about 28 for unboiled milk, and in mixtures of boiled and

unboiled milk it varies regularly with the peroxidase concentration. The value is reduced when milk is kept on ice for two days or when it is heated at 63° to 66° C.; the critical temperature for the destruction of the peroxidase appears to lie between 69° and 74° C. Mercuric chloride or formaldehyde cannot be used for preserving samples of milk in which the peroxidase is to be estimated; the former yields a precipitate with the casein, and the latter reduces the whole of the peroxidase.

W. P. S.

**Nutritive Value of Edible Oils and Fats. Oil-bearing Seeds and Crude Vegetable Oils and Fats.** J. C. Drummond and S. S. Zilva. (*J. Soc. Chem. Ind.*, 1922, 41, 125–127T.)—An exhaustive study of the vegetable oils and their raw materials as a source of vitamin *A* shows that, with the sole exception of linseed oil, they are of very low vitamin *A* value as compared with the chief animal oils. Extraction with petroleum spirit under such conditions as to exclude oxidation of the vitamin shows that most of the vitamin passes into the oil, giving oils of very low potency; but with linseed this is not so, the oil does not appear to contain the whole of the vitamin of the seed. Crude palm oil contains a relatively high proportion of vitamin *A*, but it has not been found possible to prepare from this a fraction consisting of the unsaponifiable constituents (in which the vitamin has been shown to be principally contained) suitable for raising the vitamin content of margarine.

H. E. C.

**Limits of the Agglutination Test for Ricin.** H. Waites. (*J. Soc. Chem. Ind.*, 1922, 41, 113–114T.)—For the purpose of examining castor meal residues which have been treated to destroy toxic properties the agglutination test (*cf.* Lander and Geake, *ANALYST*, 1914, 39, 262) has been compared with the method of subcutaneous injection into guinea pigs. For the test 100 grms. of castor-seed meal are mixed with 400 c.c. of Ringer's solution, allowed to stand 24 hours and filtered with the aid of the pump through three thicknesses of paper. (Ringer's solution consists of sodium chloride 9.00 grms.; potassium chloride, 0.25 gm.; sodium bicarbonate, 0.15 gm.; calcium chloride, 0.20 gm.; and water, 1000 c.c.) One c.c. of fresh defibrinated blood, diluted to 1:20 with Ringer's solution and filtered, is used for the test; the requisite quantity of extract is added, and the volume made up to 3 c.c. with Ringer's solution. The limit of sensitiveness is such that 2 c.c. of undiluted extract give less agglutination in 30 minutes than 0.5 c.c. of 1:500 dilution of standard extract, prepared, as described above, from castor seed which has been pressed but not otherwise treated. This corresponds to a limit of agglutination of 1:2000 under these conditions, or about 1:800,000 of ricin (assuming 0.5 per cent. ricin to be present in castor seed). Injection into guinea pigs gives an agglutination limit of about 1:40,000.

H. E. C.

**Preservation of Fish Frozen in Chilled Brine.** L. H. Almy and E. Field. (*J. Ind. Eng. Chem.*, 1922, 14, 203–206.)—Fish were frozen in air at –10°C. and in 10 per cent. brine at its freezing point; generally, the fish frozen in air lost slightly in weight, whilst there was a gain when they were frozen in brine. After

cold storage for some months the quantities of ammonia and amine nitrogen in the fish increased, and were slightly higher in the air-frozen than in the brine-frozen specimens, but the difference was not sufficient to indicate any difference in keeping quality. The flesh of the brine-frozen fish seemed to be the firmer, and it was possible to squeeze more juice from the air-frozen than from the brine-frozen fish. There was no great difference in the character of the bacterial flora of the two sets of fish, and from the standpoint of bacterial decomposition during storage there appears to be little choice between the two methods of freezing.

W. P. S.

**Relation between Chemical Constitution and Antiseptic Action in the Coal Tar Dyestuffs.** T. H. Fairbrother and A. Renshaw. (*J. Soc. Chem. Ind.*, 1922, **41**, 134-144T.)—Standard solutions of dyes were added to broth prepared from animal tissues and the mixtures sterilised for 30 minutes in plugged tubes by means of steam at 30 lbs. pressure. The tubes were then inoculated with recent broth cultures of the organisms, incubated at 37° C. for two days, then subcultured into broth or other media and again incubated for two days. A large number of results are tabulated. Many, but not all, of the dyes containing one or more amino groups in the molecule have an antiseptic action, but no definite antiseptic power is exhibited by those from which this group are missing. The effect of the amino groups may be modified or neutralised by the presence of other groups in the molecule. The observed antiseptic action may possibly be due to a combination between the dye base and the cell molecules of the organism, since all dyes showing this action are basic. Antiseptic action does not depend upon molecular weight, nor is it explained by the quinonoid theory of Armstrong. It is shown that the relationship between chemical constitution and the absence of antiseptic power is much more definite.

T. J. W.

**Attack of Minerals by Bacteria. Oxidation of Zinc Blende.** A. Helbronner and W. Rudolfs. (*Comptes rend.*, 1922, **174**, 1378-1380.)—Bacteria have been isolated which are able to convert zinc blende into zinc sulphate, the zinc thus rendered soluble having no deterrent influence on the further action of the organisms; the presence of sulphur favours the oxidation. The bacteria employed in the process recently described by Lipmann for the preparation of soluble phosphate from a mixture of tricalcium phosphate with sulphur are found capable of producing sufficient sulphuric acid to render soluble the natural silicates and carbonate of zinc. With minerals containing both zinc and lead, the zinc, but not the lead, is converted into the soluble form by these bacteria, which hence serve for the separation of the two metals.

T. H. P.

**The "Cotton-wool Plug Test" for Indole.** S. N. Gore. (*Indian J. Med. Research*, 1921, **8**, 505-7; *Chem. Abstr.*, 1922, **16**, 939.)—The following solutions are required:—*Böhme's Solution, A*: One grm. of *p*-dimethylaminobenzaldehyde and 20 c.c. hydrochloric acid in 95 c.c. absolute alcohol; and *Böhme's Solution, B*: One grm. of potassium persulphate in 100 c.c. of water. The under-surface of

the plug is moistened evenly first with a few drops of solution *A*, and then with a few drops of solution *B*, and is then replaced and pushed down the culture tube until it is about 1 inch above the surface of the broth. The prepared tube, after incubation, is placed upright in a gently boiling water-bath for about 15 minutes. This allows time for any indole to volatilise and give a positive reaction, even when only present to the extent of 0.0005 mgrm. indole per 1 c.c. of the broth culture. In the presence of indole a rose coloration develops on the under surface of the plug.

R. F. I.

**Estimation of Ammoniacal Nitrogen in Nitrogenous Organic Substances, especially in Protein Materials and their Decomposition Products.**

**J. Froidevaux.** (*Comptes rend.*, 1922, 174, 1238-1240.)—At the ordinary temperature concentrated sodium carbonate solution liberates ammonia from ammonium salts or amines with moderate rapidity, whereas protein nitrogen is set free extremely slowly. When ammonia-free air is passed first through 15 c.c. of a protein solution containing free or combined ammonia mixed with 35 c.c. of a solution containing 600 grms. of sodium carbonate per litre and then through a known volume of 0.1 *N*-sulphuric acid, the ammoniacal nitrogen passes over completely in about 8 hours. The curve connecting amount of nitrogen thus liberated with the time consists of two straight branches with a curved joining line; the point of intersection of the two straight lines gives the quantity of ammoniacal nitrogen.

T. H. P.

**Alkalimetric Estimation of Amino-Acids and Peptides.**

**R. Willstätter and E. Waldschmidt-Leitz.** (*Ber.*, 1921, 54B, 2988-93; *Chem. Abstr.*, 1922, 16, 944.)—The acid ion in ammonium salts may be estimated in alcoholic solution (97 per cent.) by direct titration with an alcoholic solution of caustic alkali, one c.c. of a 1 per cent. alcoholic solution of phenolphthalein being added as indicator for each 100 c.c. of the solution of the ammonium salt. The carboxyl groups in aliphatic amino acids may also be directly titrated in 97 per cent. alcoholic solution of tyrosine in 80 per cent. alcohol, phenylalanine in 70 per cent., and polypeptides in 40 per cent. alcohol; peptones and proteins also show a satisfactory end-point in dilute alcohol. When 50 per cent. alcohol is used, the amino-acids, which predominate as products of proteolysis, require for neutralisation only 28 per cent. of the theoretical amount of fixed alkali. Therefore in the case of a mixture of amino-acids and polypeptides the proportions of these two groups may be estimated by titration in 50 per cent. alcohol (*A*), and in 97 per cent. alcohol (*B*). The amount of alkali ( $x$ ) required to neutralise the amino-acid is  $100(B - A) \div 72$ ; and the amount of alkali required for the polypeptides is  $B - x$ .

R. F. I.

**Separation of Aliphatic Amines from each other and from Ammonia.**

**H. Franzeri and A. Schneider.** (*Biochem. Zeitsch.*, 1921, 116, 195-207; *Chem. Abstr.*, 1922, 16, 902.)—Plant bases can be classified into two groups on the basis of the solubility of their hydrochlorides in chloroform. Ammonium chloride and

methylamine hydrochloride are insoluble, whilst di- and tri-methylamine hydrochlorides, ethylamine hydrochloride, and probably those of the higher amines, are soluble in that solvent. Ammonia can be separated from mono-methylamine by means of yellow mercuric oxide, unless the amount of ammonia is large; in that case the methylamine is purified by extraction of its hydrochloride with absolute alcohol. Dimethylamine can be separated from trimethylamine by treatment with a potassium iodide solution of iodine, but this method cannot be used for the separation of diethylamine from triethylamine, or for separating the higher amines.

**Colorimetric Estimation of Amino-Acid Nitrogen in Blood.** O. Folin. (*J. Biol. Chem.*, 1922, 51, 377-391.)—Blood is treated with tungstic acid (*J. Biol. Chem.*, 1919, 38, 106), the precipitate filtered off, and 10 c.c. of the filtrate transferred to a test-tube. One c.c. of a solution of glycine, leucine, phenylalanine or tyrosine (containing 0.07 mgrm. of nitrogen and 0.365 per cent. of hydrochloric acid), 8 c.c. of water, one drop of phenolphthalein solution and 1 c.c. of 1 per cent. sodium carbonate solution are placed in a similar tube. To the blood filtrate one drop of phenolphthalein solution is added and sufficient sodium carbonate solution to yield a colour equal to that of the standard. Two c.c. of a fresh 0.5 per cent. solution of  $\beta$ -naphthoquinone solution are added to each tube, and the solutions are left to stand overnight in darkness. On the following day 2 c.c. of a solution containing 25 per cent. of acetic acid and 2.5 per cent. of sodium acetate are added, followed by 2 c.c. of 4 per cent. sodium thiosulphate solution, the liquids then diluted to 25 c.c., and the colours compared. The results obtained were satisfactory with glycine, aspartic acid, glutamic acid, cystine, valine, phenylalanine, tyrosine, lysine and histidine, but divergent figures were given by alanine, proline, tryptophane and arginine. Elaborate details of the preparation and purification of  $\beta$ -naphthoquinone are given.

T. J. W.

**Colorimetric Estimation of Amino Acid Nitrogen in Normal Urine.** O. Folin. (*J. Biol. Chem.*, 1922, 51, 393-394.)—From 2-3 grms. of permutit are added to 5 c.c. or a larger volume of the urine diluted to 25 c.c., and the flask is gently shaken for five minutes, after which the urine is decanted and again shaken with a similar weight of permutit, thus removing all traces of ammonia. Since the amino-acids nitrogen of urine is very variable in amount, it is advisable to prepare several standards with different amounts of a glycooll solution containing 0.1 per cent. of nitrogen, 0.365 per cent. of hydrochloric acid, and 0.2 per cent. of sodium benzoate. The process of estimation adopted is the same as that described for the estimation of amino-acid nitrogen in blood (see previous Abstract).

T. J. W.

**Colour Reaction for Urea.** V. Arreguine, jun., and E. D. Garcia. (*Anales Assoc. Quim. Argentina*, 1921, 9, 183-191; *Chem. Abstr.*, 1922, 16, 939.)—One c.c. of the solution to be tested is boiled for one minute with 2 c.c. of concentrated hydrochloric acid and 0.02 to 5 grms. of resorcinol, and the liquid then cooled and shaken with an equal volume of ether. If urea is present, the

etheral layer becomes pink, and, on then adding ammonia to alkalinity, the ether is decolorised, and the aqueous layer becomes violet with a maroon fluorescence. The reaction is also obtained with guanidine, saliva, and other substances yielding urea on hydrolysis, but the results are uncertain with thio-urea. The reaction is not suitable for the quantitative colorimetric estimation of urea. Resorcinol and hydrochloric acid give a pink coloration when heated together, but the colouring matter is insoluble in ether.

**Colorimetric Estimation of Homogentisic Acid in Urine.** A. P. Briggs. (*J. Biol. Chem.*, 1922, **51**, 453-454.)—Two c.c. of a 5 per cent. solution of ammonium molybdate in 5 *N* sulphuric acid and 2 c.c. of a 1 per cent. potassium dihydrogen phosphate solution are added to 1 or 2 c.c. of the urine diluted to about 15 c.c., and the mixture diluted to 25 c.c. Similar additions are made to an appropriate amount of standard hydroquinone solution containing 1 mgrm. per c.c., the contents of the flasks well mixed, and the green coloration produced compared in a colorimeter. It has been found experimentally that 1 mgrm. of hydroquinone is equivalent to 0.79 mgrm. of homogentisic acid by this method. An error of 1 per cent. is produced by the presence of phenol, cresol, resorcinol and uric acid, and one of 3 per cent. by the addition of pyrocatechol. Sulphides give rise to an intense blue-green coloration, but this may be removed by the addition of silver sulphate. Albumin interferes with the colour comparison by forming an opalescence, but may be precipitated by trichloroacetic acid. The results obtained showed an error of within 10 per cent. when compared with those given by the Wolkow and Baumann method. T. J. W.

**Estimation of Guanidine.** A. H. Dodd. (*J. Soc. Chem. Ind.*, 1922, **41**, 145-147T.)—To estimate the yield of guanidine obtained by heating dicyanodiamide and dilute acids in an autoclave (in which process the intermediate product formed is guanylurea) the following method was worked out. The solution containing less than 1 per cent. of guanidine was added to a solution, heated to 90° C., containing 20 grms. of picric acid dissolved in 100 c.c. of *N* sodium hydroxide solution, and the mixture allowed to stand six hours. The precipitate was transferred to a Gooch crucible and completely rinsed in with a portion of the filtrate, the filter then being washed with exactly 50 c.c. of water at 10° C. The results obtained varied with different volumes of washing water, with the concentration of the precipitant and with the salt of guanidine present. Both compounds may be estimated by precipitating the guanylurea with neutral sodium picrate, followed by alkaline sodium picrate (the above solution to which was added 20 c.c. of *N* sodium hydroxide solution) to precipitate the guanidine. By the above method the author has shown that two equivalents of an ammonium salt fused with one equivalent of dicyanodiamide yield 93 per cent. of guanidine nitrate, and that the method devised by Ulpiani of oxidising dicyanodiamide gives bad results. T. J. W.

## Water. Analysis.

### Activation of Water by Means of Metallic Copper and its Oxides.

**R. Wernicke and A. Sordelli.** (*Anales Assoc. Quim. Argentina*, 1921, **9**, 145-182; *Chem. Abstr.*, 1922, **16**, 979.)—Water was distilled in a closed apparatus in a current of hydrogen, and treated with metallic copper which had been ignited in a current of hydrogen. In like manner, copper oxide was ignited in pure oxygen and added to water which had been distilled in a current of oxygen. No copper was dissolved in either case, and the water had no effect upon a culture of *B. paratyphosus* A. On adding air or small amounts of carbon dioxide, however, a certain proportion of copper was rapidly dissolved, and the water then had a sterilising action on the bacterial culture. The reagent of Röhman and Spitzer, which can detect 0.00000005 per cent. of copper was found to be most sensitive for the test. It consists of 1.5 grm. of  $\alpha$ -naphthol, 1.5 grm. of *p*-phenylenediamine, and 1.5 grm. of sodium hydroxide in 100 c.c. of redistilled water, and is diluted to 1:100 before use. On heating 3 c.c. of the solution under examination with 0.5 c.c. of the dilute reagent a red or violet coloration is produced when copper is present. The method is not suitable for quantitative colorimetric estimations.

**Active Carbon Dioxide and Hydrogen-ion Concentration in the Examination of Water.** **J. M. Kolthoff.** (*Zeitsch. Unters. Nahr. Genussm.*, 1922, **43**, 184-192.)—The author replies to Tillmans's criticisms (*ibid.*, 1921, **42**, 98) on his previous papers (*ANALYST*, 1921, **46**, 249). The calculated value of the concentration of the free carbon dioxide which is in equilibrium with solid calcium bicarbonate and carbonate agrees with the experimental value of Tillmans and Heublein until the bicarbonate reaches the concentration of 5 milli-equivalents per litre, if the solubility product is taken as  $1.2 \times 10^{-8}$ . When the concentration of the bicarbonate increases, concordant values are obtained with the value  $1.6 \times 10^{-10}$  for the solubility product. These facts are taken into account in the preparation of a table and of curves giving the amounts of free carbon dioxide in equilibrium with different bicarbonate and calcium concentrations. For saturated calcium carbonate solution, the value of  $P_H$  at 14° C. is 10.2, the solubility at this temperature being 16 mgrms. per litre. For the determination of  $P_H$  between the values 6.8 and 6.0, the use of dibrom-*o*-cresolsulphophthalein (bromcresol purple) is recommended.  
T. H. P.

### Estimation of Nitric Acid in Drinking Water by Mayrhofer's Method.

**A. Reuss.** (*Zeitsch. Unters. Nahr. Genussm.*, 1922, **43**, 174-183.)—When nitric acid is estimated by the indigo method, in which 5 c.c. of the water are mixed with 5 c.c. of concentrated sulphuric acid, and the hot liquid titrated with indigo solution, it is often found that the titration proceeds more readily and uniformly with a natural water than with pure nitrate solution. This difference is traced to the presence in the former of sodium chloride, which considerably facilitates the reaction, especially with small amounts of nitric acid. The table given by Mayrhofer for calculating the proportion of nitric acid appears to correspond with

a content of 1 grm. of sodium chloride per litre, the titre of the indigo solution being determined by means of a solution containing 0.0962 grm. of potassium nitrate (0.060 grm.  $\text{HNO}_3$ ) and 1 grm. of sodium chloride per litre. The titration is conveniently carried out in a 25 c.c. flask and exactly 5 c.c. of sulphuric acid are run down the wall of the flask containing the water, and then mixed by rapid rotation. The indigo solution is added at the rate of 2-3 drops per second, except for the last few drops, which are added more slowly; with very small amounts of nitric acid the addition of the indigo solution is regulated according to the disappearance of the blue colour.

T. H. P.

## Agricultural Analysis.

**Soil Solutions.** J. E. Greaves and C. T. Hirst. (*J. Ind. Eng. Chem.*, 1922, 14, 224-226.)—Clear soil extracts may be obtained by adding 2 grms. of potassium alum to the soil-water mixture (soil, 100 grms., water 500 c.c.), by filtering<sup>1</sup> the soil-water mixture through a Pasteur-Chamberland filter, or by subjecting it to centrifugal action. A considerable loss of nitrate nitrogen occurs when lime, ferric sulphate or ferric alum is used as clarifying agent. Of course, when sulphates are to be estimated the use of alum is not permissible. Five minutes' agitation of the soil-water mixture is sufficient to obtain all chlorides and nitrates in solution, but a longer time is required for sulphates, depending on the quantity and kind present; a larger proportion of water than that mentioned may also be required in the case of sulphates. When alum is used as the clarifying agent in the estimation of nitrates the addition of another antiseptic is unnecessary, but when alum is not used and the solution has to be kept for some time it is well to add 0.5 c.c. of chloroform to each sample mixture.

W. P. S.

## Organic Analysis.

**Use of Phthalate Solutions for Hydrogen Electrode Standards.** E. T. Oakes and H. M. Salisbury. (*J. Amer. Chem. Soc.*, 1922, 44, 948-951.)—In view of the suggestion recently made to replace the calomel electrode by phthalate solutions as standard hydrogen electrodes the authors have investigated the reliability of such solutions. It is found that, even when carefully purified, phthalate solutions undergo a progressive decrease in hydrogen-ion concentration when in continued contact with the hydrogen electrode, this decrease being the result of a change, probably a reduction, of the phthalate itself. If this phenomenon is due to the presence of impurities, the latter must act as catalysts in the reduction of the phthalate, as they cannot exist in sufficient quantity to account otherwise for the whole change taking place.

T. H. P.

**Detection and Estimation of Free Sulphuric Acid and Sulphoacetates in Cellulose Acetates.** M. Entat and E. Vulquin. (*Ann. Chim. anal.*, 1922, 4, 131-135.)—It is shown that cellulose acetates do not usually contain any free sulphuric acid; such free acid has frequently been reported present owing to the



fact that sulphoacetates of cellulose are partially hydrolysed by water and so give a precipitate with barium chloride. The only method of testing for free sulphuric acid and estimating it is by electrometric titration with barium hydroxide as previously described by the authors (*cf.* ANALYST, 1917, 42, 221). Ten grms. of the sample are digested for half an hour with 200 c.c. of water at a temperature below 15° C., filtered, and titrated electrometrically with barium hydroxide; on plotting the E.M.F. against the volume of barium hydroxide solution the break in the curve indicates neutrality. For the estimation of sulphoacetates, 10 grms. are heated in a sealed tube with 50 c.c. of water for 5 hours in an autoclave at 125–130° C., the solution is filtered, the residue washed, and the filtrate titrated as before. Below 125° C. hydrolysis is not complete. Cellulose acetates of good quality contain less than 0.03 per cent. of sulphoacetates reckoned as sulphuric acid.

H. E. C.

**Estimation of Free Sulphur and the Coefficient of Vulcanisation in Vulcanised Rubber.** W. J. Kelly. (*J. Ind. Eng. Chem.*, 1922, 14, 196–198.)—

In addition to the classes of sulphur mentioned by the author in a previous paper (ANALYST, 1920, 45, 424), vulcanised rubber may contain sulphur combined with organic accelerators; a portion of this class of compounds may be soluble in acetone, whilst another portion, together with metallic sulphides, may be insoluble in acetone. Methods for the estimation of all the different classes of sulphur are as follows:—**TOTAL SULPHUR.**—0.5 gm. of the sample is dissolved in a solution prepared by dissolving 16 grms. of zinc in 199 c.c. of concentrated nitric acid, 10 c.c. of fuming nitric acid saturated with bromine are then added, and the mixture is heated until excess of bromine has been expelled; about 0.5 gm. of potassium chlorate is now added and the solution is evaporated to dryness. The flask containing the residue is heated over a flame until the zinc nitrate has been decomposed and all organic matter oxidised, the residue then dissolved in hydrochloric acid and the sulphuric acid precipitated as barium sulphate. **ACETONE-SOLUBLE SULPHUR.** *Total.*—One gm. of the sample is extracted with acetone, the extract is evaporated, the residue oxidised with nitric acid and bromine solution and potassium chlorate, and the sulphuric acid precipitated as barium sulphate. *Free sulphur.*—One gm. of the sample is extracted with acetone, and the extract is evaporated to dryness at a temperature not exceeding 60° C.; the free sulphur is then separated from organic matter by means of alcohol saturated with sulphur as described in the previous paper (*loc. cit.*). **ACETONE-INSOLUBLE SULPHUR.** *Total.*—This is estimated, as described above, on a sample of the rubber extracted previously with acetone. *Sulphur combined with resins, etc., soluble in alcoholic potassium hydroxide solution.* The rubber is extracted with acetone, then boiled for eight hours with alcoholic potassium hydroxide solution, washed with alcohol, and extracted for many hours with alcohol to remove all the alkali solution. The united alcoholic solutions are evaporated, the residue is oxidised, and the sulphuric acid precipitated as barium sulphate. *Metallic sulphides.*—After the rubber has been extracted with acetone and alcoholic potassium hydroxide solution it is

treated in a flask with 50 c.c. of ether saturated with hydrogen chloride, and the hydrogen sulphide evolved is collected in potassium bromide-bromine solution; the last traces of hydrogen sulphide are expelled by distilling a portion of the ether, and the bromide-bromine solution is treated with nitric acid and the resulting sulphuric acid estimated. *Sulphur combined with accelerators, etc., soluble in ether-hydrogen chloride solution.*—The residual rubber from the preceding estimation is washed with ether, the united ethereal solutions are evaporated, and the residue obtained is oxidised, etc. *Sulphur combined with rubber.*—The rubber remaining after extraction with acetone, alcoholic potassium hydroxide solution and ether-hydrogen chloride, is treated as described for the estimation of total sulphur.

W. P. S.

**Essential Oil of Naal Grass (Sudan.) A. F. Joseph and B. W. Whitfield.**

(*J. Soc. Chem. Ind.*, 1922, **41**, 144–145T.) Three samples of oil obtained from Naal grass gave the following analytical results:

	Sp. gr.	Refractive index $n_D^{20}$	Optical rotation	Acid value	Ester value	Ester after acetylation	Solubility in alcohol.
(1)	0.954	1.495	-31°	—	29.6	198.1	1 vol. in
(2)	0.955	1.492	-35°	3.6	21.8	189.2	1.2 vols of 70 per cent.
(3)	0.953	1.495	-50°	4.4	26.5	189.4	1.6 „ „ 75 „
A.	0.9405	1.4965	+26°	4.6	9.3	99.1	0.5 „ „ 80 „

The fourth set of results was obtained by Schimmel with a sample of oil stated to be prepared from Naal grass (*Cymbopogon nervatus* Chiov.), but from the discordant figures given above it is doubtful if this sample was genuine. The oils examined by the authors were obtained by distilling the grass flowers with water or steam, a yield of 0.8 to 1.5 per cent. being obtained. The colour was light yellow and did not darken on keeping for two years, whilst the odour resembled that of ginger grass oil. On distillation, even at a pressure of 10 to 15 mm. of mercury, a viscous condensation product was formed, and with difficulty the oil was separated into *l*-limonene and perilla alcohol.

T. J. W.

**Plant Incrustations. E. Schmidt and Duysen.** *Ber.* 1921, **54B**, 3241–4; *Chem. Abstr.*, 1922, **16**, 949.)—Incrustations can be removed from plant tissues by immersing the latter for 24 hours in a saturated solution of chlorine peroxide in acetic acid, which solution is prepared by passing the gas into 50 per cent. acetic acid contained in a brown flask cooled by a mixture of ice and salt. This solution is stable, even in diffused light. After immersion, the tissues are removed and washed. The cell walls are somewhat swollen, but are not dissolved by the reagent. A solution of iodine in zinc chloride solution will give a blue reaction with polysaccharides in the tissues from which the incrustations have been removed. On the other hand, tissues which give a red coloration with hydrochloric acid no longer do so after treatment with the reagent. The so-called phytomelane, which occurs in the fruit of many *Compositae*, is dissolved by the chlorine peroxide solution.

R. F. I,

**Zinc Chloride as Wood Preservative. Report of Committee No. 4--Preservatives.** (*Proc. Amer. Wood-Preservers' Assoc.*, 1921, 50-80; *Chem. Abstr.*, 1922, 16, 625.)—Analytical data, given in detail, indicate that none of the impurities in commercial zinc chloride is present in sufficient amount to influence its action as a preservative. Detailed directions for sampling zinc chloride, and standard specific gravity tables for zinc chloride solution are given, and modified methods of analysis are suggested. *Visual Method for Determining the Penetration of Zinc Chloride.*—A mixture of potassium ferricyanide, starch and potassium iodide is sprayed on to discs of treated wood, a blue coloration (due to the liberation of iodine by the action of zinc ferricyanide) only being produced in those portions originally reached by the zinc chloride. The treatment has little effect upon the strength of wood, provided that it is not afterwards exposed to abnormal temperatures.

**Detection of Benzene in Petroleum Spirit. P. Schwarz.** (*Chem. Zeit.*, 53, 401).—A mixture of equal parts by vol. of aniline and 94 to 96 per cent. alcohol can be used for the rapid detection of 5 per cent. of benzene in petroleum spirit. If 2 c.c. of the mixed solvent are mixed with 5 c.c. of pure petroleum spirit in a cylinder, the aniline, and sometimes part of the alcohol, will separate after a short time. But if 5 per cent., or more, of benzene were present a clear solution is at once obtained. As a confirmation the fraction of the sample distilling between 80° and 110° C. should be collected, and the usual qualitative tests applied to it.

**Iodine and Bromine Values of Petroleum Products. E. M. Johansen.** (*J. Ind. Eng. Chem.*, 1922, 14, 288-292.)—The iodine and bromine addition and substitution values of a considerable number of petroleum products are recorded. A modified Hanus solution was used, the iodine and bromine being dissolved in carbon tetrachloride, whilst the bromine values were estimated by means of a solution of bromine in the same solvent. The following are selected from the results given:—

	Iodine Value		Bromine Value	
	Addition	Substitution	Addition	Substitution
Straight run naphtha	0·53	1·58	0·65	1·55
Cracked naphtha	14·70	8·16	9·39	4·62
Lamp oil distillate	1·32	5·04	0·69	3·13
Cracked lamp oil	23·23	9·52	21·81	7·57
Lubricating oil	0·83	12·86	2·09	5·83
Paraffin wax	1·10	—	1·16	2·25
White medicinal oil	0·05	—	0·40	1·90
Motor gasoline	5·91	2·73	3·71	1·15

There appears to be no fixed relation between the bromine and iodine values; increase of time of contact, higher temperature, and lower proportion of oil to halogen usually increased the values, the increase being mainly due to substitution, particularly in the case of the bromine values.

W. P. S.

**Estimation of Volatile Combustible Matter in Pitch Coke.** H. E. Lloyd and F. W. Yeager. (*J. Ind. Eng. Chem.*, 1922, **14**, 220–222.)—The most concordant results are obtained by heating 2 grms. of the sample (ground to pass a 60-mesh sieve) in a 10 c.c. platinum capsule provided with a tightly fitting lid; the capsule and its contents are placed in an electric furnace at 950°C. for exactly seven minutes, cooled, weighed, and again heated for seven minutes. The loss occurring during this second heating is deducted from the first loss, and is a correction representing the extent of the oxidation of the non-volatile portion of the coke. W. P. S.

## Inorganic Analysis.

**Preferential Catalytic Combustion of Carbon Monoxide in Hydrogen.** A. B. Lamb, C. C. Scalione and G. Edgar. (*J. Amer. Chem. Soc.*, 1922, **44**, 738–757.)—Carbon monoxide in amounts under 1 per cent. is preferentially oxidised at room temperature by “hopcalite” (a specially prepared mixture of 60 per cent.  $\text{MnO}_2$  and 40 per cent.  $\text{CuO}$ ) in presence of hydrogen and oxygen; when the carbon monoxide content is as much as 1 per cent. the catalyst becomes heated and hydrogen is oxidised, heating the hopcalite to incandescence and destroying its properties. If there is moisture present, the oxidation is only complete at temperatures of about 90° C. A mixture of carbon monoxide (under 1 per cent.) and oxygen, hydrogen, and nitrogen, may therefore be analysed by passing it through potassium hydroxide, and then through sulphuric acid (to dry it), then passing it over hopcalite, and finally through absorption bulbs to absorb the carbon dioxide formed. As the catalyst adsorbs some of the carbon dioxide it is afterwards heated to 140° C., by means of a bath of boiling xylene, in a current of purified air, the adsorbed carbon dioxide is driven off into the absorption bulbs, and the total carbon dioxide titrated. The effects of temperature, impurities, rate of flow of the gases, and concentration are described, particularly in relation to the technical removal of carbon dioxide from hydrogen. H. E. C.

**Analysis of Liquid Nitrogen Peroxide.** A. Sanfourche. (*Bull. Soc. Chim.*, 1922, **31**, 316–319.)—The impurities occurring in liquid nitrogen peroxide are usually nitrous oxide and nitric acid, which normally do not exceed 10 per cent. and 1 per cent. respectively; a larger quantity of nitric acid would be due to excess of moisture. Samples should be aspirated into an ice-chilled and perfectly dry sample bulb which should be sealed and kept in ice. For the estimation of the nitric acid 10 c.c. are run into an ice-chilled Dewar vacuum flask, and the volatile constituents are evaporated by drawing dry air through the liquid at the rate of 3 or 4 bubbles per second; about four hours are required for the evaporation. The nitric acid, which remains in little drops which, when warmed by holding the flask in the hand, should not evolve any nitrous fumes, is taken up with a little water and titrated with  $N$  sodium hydroxide solution. The density of liquid nitrogen peroxide is 1.5 at 0°C., so that if  $n$  c.c. of sodium hydroxide are required,  $0.42 n$  gives the percentage of nitric acid. If greater accuracy is desired, the

sample should be weighed. The nitrous oxide and the peroxide are estimated simultaneously by means of the nitrometer and by titration with 0.1 *N* potassium permanganate solution. One c.c. of ice-chilled nitrogen peroxide is added to 20 c.c. of ice-cold sulphuric acid, and dissolved by shaking; at this low temperature there is no loss by volatilisation. When the normal temperature has been regained, aliquot parts by volume, in a burette, or by weight in a Lunge pipette, are taken for the estimation in the nitrometer and titration with 0.1 *N* permanganate solution. If *p* and *m* are the respective weights of NO and N<sub>2</sub>O<sub>3</sub> found in the same quantity of the sulphuric acid solution, the corresponding quantities of NO<sub>2</sub>, N<sub>2</sub>O<sub>3</sub>, and HNO<sub>3</sub>, are:  $\text{NO}_2 = \frac{100p - 79m - a(p - 0.315m)}{32.6 - 0.46a}$ ,  $\text{N}_2\text{O}_3 = m - 0.413\text{NO}_2$ ,  $\text{HNO}_3 = \frac{a}{100 - a} (\text{NO}_2 + \text{N}_2\text{O}_3)$ ; where *a* is the percentage of nitric acid already found.

H. E. C.

### Colorimetric Estimation of Arsenic by means of Quinine Molybdate.

**D. Chouchak.** (*Ann. Chim. anal.*, 1922, 4, 131-135.)—Minute quantities of arsenic form a colloidal precipitate with quinine molybdate, which is compared colorimetrically with standards. The method is more sensitive and more convenient than the Marsh or other test. The following solutions are required:—*A*: 9.5 grms. of molybdic acid and 3.5 grms. of anhydrous sodium carbonate dissolved in 100 c.c. of water. *B*: nitric acid solution containing 25 c.c. of nitric acid (sp. gr. 1.34) in 100 c.c. *C*: a solution of arsenic oxide containing 0.01 mgrm. per c.c. made by oxidising 1.3207 grms. of arsenious anhydride with nitric acid, evaporating it nearly to dryness and dissolving it in 1 litre of water, and diluting 10 c.c. of this solution to 1000 c.c. The reagent is prepared by dissolving 0.5 gm. of neutral quinine hydrochloride in 10 c.c. of water, adding to it 5 c.c. of solution *C*, 10 c.c. of *B*, and then, drop by drop, 1 c.c. of *A*. The precipitate so formed redissolves and the mixture is diluted to 120 c.c., mixed and filtered through a paper previously washed with nitric acid and then with water. (This reagent keeps for several months.) When the residue from the evaporation of a solution of arsenic is treated with a few drops of this reagent a turbidity appears with as little as 0.00002 mgrm. The most convenient standard is prepared by adding 0.5 c.c. of solution *B*, then 2 c.c. of water, and 20 c.c. of the reagent, to 2.5 c.c. of the arsenic solution *C*. The solution to be examined is evaporated on the water bath and 0.5 c.c. of solution *B*, 4.5 c.c. of water, and 20 c.c. of the reagent are added to the dry residue; the comparison with the standard is made in a colorimeter after 15 minutes. For the application of the method to the estimation of the arsenic in organic mixtures, the organic matter must be removed; this may be done by any of the standard methods; arsenious compounds must be oxidised to the arsenic condition, and phosphoric acid, free acids, and salts of the group II. metals must be removed, if present. In the presence of metals precipitated by hydrogen sulphide it is best to remove the arsenic by distillation, to evaporate the distillate to dryness after oxidation with bromine and nitric acid, and then to estimate the arsenic as described above. When organic matter is present and is destroyed by

means of mixed sulphuric and nitric acids, the bulk of the acid should be driven off by evaporation; then to the remainder should be added water, a little potassium bromide and chloride, and hydrazine sulphate, this mixture being then evaporated to low bulk in a distillation flask and the residue distilled three or four times with 1 c.c. of hydrochloric acid; the distillate is oxidised and treated as before. Distillation is also recommended if the organic matter has been removed by ignition with lime or magnesia. Excellent quantitative results were obtained with organic mixtures and wines containing arsenic.

H. E. C.

**Errors Caused by Nitrates and Nitrites in the Estimation of Arsenic by the Distillation Method, and a Means for their Prevention.** J. J. T. Graham and C. M. Smith. (*J. Ind. Eng. Chem.*, 1922, 14, 207-209.)—The presence of nitrates or nitrites causes low results to be obtained in the ordinary distillation method for the estimation of arsenic, in which cuprous chloride or ferrous sulphate is used as the reducing agent; this is due to some volatile substance, possibly nitrosyl chloride, which passes into the distillate and re-oxidises the arsenic, the extent of the oxidation depending largely on the length of time which elapses before the distillate is titrated. The error may be avoided by using a mixture of hydrazine sulphate and sodium bromide as the reducing agent; in this case the nitrates and nitrites are probably reduced to nitrogen. The method is as follows:—A quantity of the sample, containing the equivalent of not more than 0.6 gm. of arsenic pentoxide, is placed in a distillation flask connected with a condenser and a series of absorption vessels or receivers; 50 c.c. of the reducing solution (20 grms. of hydrazine sulphate and 20 grms. of sodium bromide dissolved in 1 litre of dilute hydrochloric acid) are added, the mixture is boiled for three minutes, 100 c.c. of concentrated hydrochloric acid then introduced by means of a tapped funnel, and the mixture distilled until its volume is reduced to about 40 c.c. A further quantity of 50 c.c. of hydrochloric acid is added and the distillation continued until the volume is again reduced to 40 c.c. The contents of the receivers are mixed, diluted to 1 litre; 200 c.c. of this solution are neutralised with sodium bicarbonate, an excess of 5 grms. of the latter is added, and the solution is titrated with standardised iodine solution, starch solution being used as indicator. An alternative method of titrating the arsenic in the distillate consists in treating 200 c.c. of the latter with 10 c.c. of concentrated hydrochloric acid and then titrating the mixture with standardised potassium bromate solution, methyl-orange being used as indicator. The end-point is sharp and is denoted by the discharge of the red colour.

W. P. S.

**Physical Properties of Commercial Lead Arsenates.** R. H. Robinson. (*J. Ind. Eng. Chem.*, 1922, 14, 313-317.)—The specific gravity of different samples of lead arsenate varied from 5.7 to 6.0, but there appeared to be no relation between the specific gravity and the dry volume of the substance, the volume occupied by 1 gm. ranging from 1.8 to 4.3 c.c. The arsenates were of very uniform composition, and contained only traces of water-soluble arsenic, but the size of the particles varied considerably, and this affected the manner in which the samples

could be suspended in water and their "spreading" quality when applied as a spray to foliage. The best indication of efficiency is the amount of lead arsenate which will remain in suspension in water for a specified length of time; substances such as calcium caseinate, glue, soap, sugars, gums, etc., are sometimes added to increase the suspension and "spreading" qualities of the arsenate. W. P. S.

**Detection of Traces of Osmium.** M. Hirsch. (*Chem. Zeit.*, 1922, 46, 390.)—The substance to be tested is dissolved in fuming nitric acid, or fused with alkali hydroxide and nitrate, the fused mass dissolved in water, and the liquid acidified with strong nitric acid. The acid liquid is distilled, and the distillate collected in cold water. The acid liquid thus obtained is treated with strong potassium thiocyanate solution and well shaken with ether or amyl alcohol. A blue layer is obtained in presence of osmium, the colour of which becomes deeper on standing. The reaction is easily obtained with concentrations of 1:100,000, and is still perceptible at 1:1,000,000. W. P. S.

**Alleged Adsorption of Alumina from Aluminium Sulphate Solutions by Cellulose.** A. Tingle. (*J. Ind. Eng. Chem.* 1922, 14, 198–199.)—The withdrawal of alumina from aluminium sulphate solutions by cellulose, as stated by many writers, is in all probability due to precipitation by non-cellulose material present as an impurity. Adsorption of alumina by pure cellulose could not be detected by the author when either neutral or basic aluminium sulphate solutions were employed. W. P. S.

**Detection and Estimation of Nickel and Cobalt in Silicate Rocks.** O. Hackl. (*Chem. Zeit.*, 1922, 46, 385–386.)—The powdered rock is disintegrated by means of hydrofluoric and sulphuric acids, or by fusion with sodium carbonate followed by removal of silica. For the minute quantities of nickel and cobalt usually present, Macintosh's method is sufficiently accurate. The solution is treated with hydrogen sulphide until the iron is reduced and the precipitate coagulates; the filtrate is made ammoniacal, again treated with hydrogen sulphide, and left to stand in a warm place. A large excess of 5 per cent. hydrochloric acid is then added, the solution allowed to stand and stirred from time to time, and the precipitate (containing nickel, cobalt, and a little iron) filtered off and washed with acidulated hydrogen sulphide water; it is ignited in a porcelain crucible and dissolved in *aqua regia*. The solution is diluted, and treated with tartaric acid, and then with dimethylglyoxime and ammonia. The small precipitate may be ignited to nickel oxide, after wrapping a second paper round the wet filter and incinerating it at very low temperature to avoid sublimation. For the estimation of nickel and cobalt, the *aqua regia* solution of the sulphides is freed from iron by the succinate method or by two or three successive precipitations with ammonia; the filtrate is boiled with sodium hydroxide and bromine, and the precipitate ignited and reduced in hydrogen. This gives the sum of nickel and cobalt. The metals are dissolved in *aqua regia* and the cobalt precipitated with nitroso- $\beta$ -naphthol, nickel being found by difference. Eleven samples analysed gave Ni 0.016 to 0.14 and Co 0.006 to 0.032 per cent. W. R. S.

**Trivalent Titanium. II. Estimation of Copper and Iron in Presence of Each Other. W. M. Thornton.** (*J. Amer. Chem. Soc.*, 1922, **44**, 998–1001.)—

The principle of the suggested method is as follows: The copper and iron (ferric) as sulphates are titrated in presence of a soluble thiocyanate with standard titanous sulphate, the sum of the two metals being thus indicated. The coagulated cuprous thiocyanate is removed by filtration, the small amount of ferric sulphate formed in the solution by atmospheric oxidation reduced by addition of further titanous sulphate, the thiocyanate rendered inactive towards permanganate by introduction of silver nitrate in excess, and the iron titrated with standard permanganate solution.

The solution of cupric and ferric sulphates, mixed with 10 c.c. of aqueous sulphuric acid (1:1), is cooled to 15° C. and, after addition of 2.5 to 5 c.c. of 10 per cent. ammonium thiocyanate solution according to the quantity of copper expected, the liquid is titrated with the standard titanium trisulphate solution until the pink colour just disappears. The solution is then heated to incipient boiling to coagulate the precipitate, cooled somewhat and filtered, and the precipitate washed with cold water. The filtrate is cooled to 15° C., the slight red colour removed by very careful addition of the titanium solution, and the solution cooled still further, treated with sufficient silver nitrate to precipitate the whole of the thiocyanate, and, without being filtered, titrated with permanganate solution. Allowance must be made for any iron present in the titanium reagent. When the proportion of copper is very high and that of iron very low, it is advisable to add an exact volume of standardised ferric ammonium sulphate solution, and to correct the results of both titrations accordingly; in this way the delicacy of the first colour indication is enhanced without appreciable reduction of accuracy. For sharpness of the second end-point, small volume, low temperature and good daylight are desirable. The titrations may be performed in air without appreciable inaccuracy.

T. H. P.

**Volumetric Estimation of Titanium in Bauxium. H. J. Winch and V. L. Chaudratreya.** (*Chem. News*, 1922, **124**, 231–232.)—

The bauxite (0.3 gm.) is fused with bisulphate, and the cake dissolved in a conical flask by boiling with dilute hydrochloric acid. The solution is treated with strong hydrochloric acid, 5 grms. of ammonium chloride, and 0.15 gm. of pure tin powder free from iron. A small funnel covered with a watchglass is inserted, and the solution gently boiled; when all the tin is dissolved, cold air-free water and 50 c.c. of 4 per cent. mercuric chloride solution are added, followed by 5 c.c. of ferric (free from ferrous) chloride solution (10 grms. Fe per litre). The solution is titrated with dichromate, ferricyanide being used as external indicator. This gives the sum  $\text{Fe}_2\text{O}_3 + \text{TiO}_2$ , the factor for ferric oxide being the same as that for titanium dioxide. A second portion of 0.3 gm. is treated like the first, but the ferric chloride is reduced in the boiling solution by stannous chloride solution, added drop by drop until the yellow colour just disappears; cold boiled water is added, and the assay titrated as before. The difference between the first and second titrations gives the quantity of titanium dioxide.

W. R. S.



**Colorimetric Estimation of Vanadium in Steel.** G. Misson. (*Bull. Soc. Chim. Belge*, 1922, 31, 123–126.)—The following solutions are required:—Nitric acid (sp. gr. 1·2) free from chlorine. Potassium permanganate (8 grms. per litre). Hydrogen peroxide, prepared from 100 c.c. nitric acid diluted with 900 c.c. distilled water, into which is projected sodium peroxide (40 grms.) in small quantities at a time whilst stirring and cooling. Ammonium metavanadate (2·294 grms.) is dissolved in 500 c.c. of boiling distilled water acidified with 20 c.c. of pure nitric acid, then cooled and diluted to 1 litre (1 c.c.=0·001 gm. V). *Carbon steel*.—One gm. is dissolved in 20 c.c. of nitric acid; the solution is boiled a few minutes with 10 c.c. of the permanganate solution and then with 10 c.c. of hydrogen peroxide to dissolve the precipitated manganese peroxide. After cooling, 5 c.c. of hydrogen peroxide are added, the solution made up to 80 c.c., and the colour compared with those of solutions prepared by adding 0·5, 1·0, 1·5 c.c., etc., of ammonium vanadate to 1 gm. charges of vanadium-free steel, which are treated like the assay. *Alloy steels*.—One gm. is dissolved in *aqua regia*, the solution evaporated to dryness, the residue once more evaporated with *aqua regia* to a small bulk, and the tungstic acid filtered off after dilution with hot water. The filtrate is evaporated twice with strong nitric acid to expel as much chlorine as possible. The residue is taken up in dilute nitric acid, and the liquor boiled successively with permanganate solution and hydrogen peroxide, as in the preceding case. The remainder of the chlorine is then precipitated with a slight excess of silver nitrate. An aliquot part of the filtrate is treated with 5 c.c. of hydrogen peroxide, and the colour compared with standards prepared as before; if chromium, nickel, etc., are also present, they must be determined beforehand, and suitable additions of potassium chromate, nickel nitrate, etc., made to the standards. The comparison must be made without delay, as the colour produced is not very stable.

W. R. S.

**Preservation of Volumetric Solutions of Uranium.** (*Union. Pharm.*, Jan. 15, 1922; *Ann. Chim. anal.*, 1922, 4, 144.)—Solutions of uranium acetate preserved in nearly-full stoppered bottles undergo decomposition which is due to the action of light on the traces of formic acid present in the uranium acetate and in the acetic acid. The action is catalytic and, once started, is continuous in the dark; but it only takes place in the absence of oxygen, or when all the oxygen in the bottle has been absorbed by the oxidation of uranous salts. The solution may therefore be preserved unchanged, if kept in an amber-coloured bottle which is not completely full, so that there may be plenty of oxygen in excess. H. E. C.

## Physical Methods, Apparatus, etc.

**Change of Properties of Substances on Drying.** H. B. Baker. (*J. Chem. Soc.*, 1922, 121, 568–574.)—The boiling points of certain liquids and the melting points of sulphur and iodine have been found to undergo a considerable rise after the substances have been dried over phosphoric oxide for a number of years. Surface tension determinations support the hypothesis that this is due

to increased molecular complexity in the dried liquids. No changes in density were observed. The following table shows the results obtained:—

	Period of drying Years	Original boiling point °C.	New boiling point °C.	Rise °C.
Bromine	8	63	118	55
Mercury	9	358	420–425	62
Hexane	8½	68.4	82	14
Benzene	8½	80	106	26
Carbon disulphide	1+27	49.5	80	30
Carbon tetrachloride	9	78	above 112	34
Ethyl ether	9	35	83	48
Methyl alcohol	9	66	above 120	54
Ethyl alcohol	9	78.5	138	60
Propyl alcohol	9	95	134	39

The carbon disulphide had originally been purified in 1894.

H. E. C.

### Simple Formula for the Calculation of the Specific Heats of Solids.

**H. J. Krase.** (*J. Amer. Chem. Soc.*, 1922, **44**, 784–786.)—The well known theories of specific heats of solids agree in that, for a large class of solids, the specific heat,  $C_v$ , is a function of the temperature; *i.e.*  $C_v = f(T/\theta)$ ,  $T$  being the absolute temperature and  $\theta$  the characteristic temperature for each substance. The following is a simple form of the function which is found to give numerically accurate values

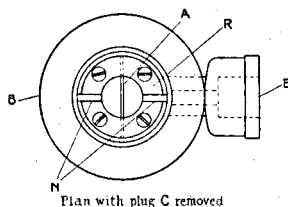
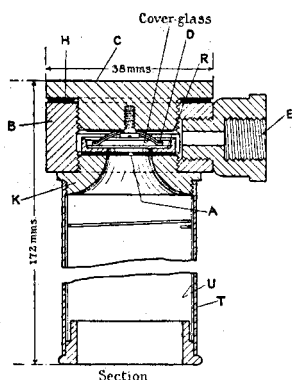
of  $C_v$  in calories per grm.-atom.  $C_v = 2.91 + 2.89 \tanh 2.95 \log T/\theta$ . Similarly the entropy can be calculated from the integration of the same formula  $S_v = 6.70 \log T/\theta + 5.18 \log \cosh 2.95 \log T/\theta + 1.62$ . The values of  $\tanh$  and  $\log \cosh$  are readily obtained from tables of hyperbolic functions.

H. E. C.

### Suspended Impurity in the Air. J. S. Owens.

(*Proc. Roy. Soc.*, 1922, [A], **101**, 18–37.)—Methods of estimating the amount of dust in air depending on filtration followed by solution in water or on spraying, are unsatisfactory because of the disintegration of aggregate particles, the partial solubility of the dust, and, in the case of spraying methods, the difficulty of wetting the particles. A method giving satisfactory results with black particles consists in filtering the air through paper and comparing the shades of the black marks left on the paper. In an excellent method, devised by the author, the air is made to impinge from a small jet with high velocity on to a glass cover-slip, with the result that the dust adheres to

the glass and can then be examined microscopically. The apparatus (see figure) consists of a brass sleeve B, with a plug C, made airtight by the leather



washer H. K is a piece of brass perforated by a small funnel-shaped hole varying in diameter from 2 to 10 mm.; A, the slot forming the jet, is of hard brass or copper, and is held in place by a brass ring suitable for receiving a  $\frac{3}{4}$  in. coverglass in a recess on its upper surface; the coverglass is held in place by the spring D. The connection E is for an air pump of about 50 c.c. capacity. To the outer part of K is fixed the thin tube T, which is lined with blotting paper, and has a capacity about twice that of the air pump; before making a test the blotting paper is saturated with water. The slot is varied according to the air to be examined, about 1 cm. in length being suitable for town air and 2 cm. for cleaner air. The air is drawn through the apparatus at a high velocity, in the limit equal to that of sound, a few pumpfuls being drawn before the coverglass is inserted. The particles, which adhere to the cover-glass, possibly owing to condensation of moisture, are afterwards counted microscopically. The highest velocity possible should be employed, as at low velocities there is a tendency for the particles to form aggregates in the jet.

Results obtained show that visibility is closely related to the number of dust particles; and the apparatus is useful for testing this relationship, for examining the dust in expired air and in mine air, and for the investigation of the vertical distribution of dust particles. As a result of investigating the haze overlying this country in periods of gentle easterly winds, the author considers that this is due to the smoke from continental fires, a fair proportion of the suspended impurity in the air of Britain being due to German industrial activity. Records made during westerly winds contain numbers of denser spherical particles, probably consisting of fused ash from furnaces fired with powdered coal.

Permanent records may be set up by mounting the cover-slips in Canada balsam, but this makes the count appear lower, and the results should be multiplied by  $\frac{7}{3}$  to make them equal to the unmounted count. Using a  $\frac{1}{12}$  in. oil immersion objective of n.a. 1.28, the limit of resolution of the particles observed is about  $0.216\mu$ . (Cf. also *Meteorological Office Rep.*, 249; *ANALYST*, 1922, 256.)

H. E. C.

**Method of Colour Measurement for Oils.** L. W. Parsons and R. E. Wilson. (*J. Ind. Eng. Chem.*, 1922, 14, 269-278.)—The method described is intended chiefly for use with petroleum oils, but may be modified to apply to many other liquids. The Duboscq colorimeter is employed and the depth of standard colour solution necessary to match a given layer of unknown oil is determined; the thickness of the layer of the unknown oil varies from a very thin film for dark oils to a 10 cm. layer for the lightest coloured oils. The standard solution consists of a solution of a dark oil in kerosene; this solution is diluted to approximately 35 on the Lovibond colour scale, and then standardised exactly in the colorimeter with a 50 Lovibond colour glass. The latter is selected because up to this point there is no deviation between the Lovibond and true colour curves. A single standard enables the true colour of petroleum oils from 1 to 5000 colour to be determined.

W. P. S.

**Continuous-Flow Calorimeter.** F. G. Keyes, L. J. Gillespie and S. Mitsukuri. (*J. Amer. Chem. Soc.*, 1922, 44, 707-717.)—In determining the heat of neutralisation of a solution of hydrochloric acid by one of sodium hydroxide the two solutions are led down from reservoirs arranged to give a constant hydrostatic pressure, through coiled silver tubes in a constant temperature bath, and from these into a special form of silver mixing calorimeter, with a vacuum jacket, immersed in a very accurate thermostat. The temperatures are recorded by platinum resistance thermometers, one in the thermostat and the other in the calorimeter. Corrections are applied for the heat lost at the particular rate of flow, heat due to friction, and, in the case of sodium hydroxide, for the carbonate present in the solution. Constant temperature in the calorimeter is secured by an arrangement of silver baffle plates, and the neutrality is indicated by bromothymol-sulphonephthalein. The rise of temperature is observed for ten rates of flow and the mean error of the observations is probably less than 0.1 per cent. of the heat. The value for the formation of 0.107 *N* NaCl at 32.3° C. is 13280 calories per mol.

H. E. C.

**Maintenance of the Adiabatic Condition in Calorimetry.** F. Barry. (*J. Amer. Chem. Soc.*, 1922, 44, 899-937.)—The results of the numerous measurements referring to the calorimetry of slow processes show that the loss or gain of heat may be limited to 0.5 grm.-calorie in 10 hours if certain precautions are taken. Use must be made of a closed calorimetric system allowing no measurable evaporation into the insulating air-gaps, which must be dry. If any conducting parts pass directly from the calorimeter into the surrounding air, the outer environment must be controlled thermostatically with a precision characteristic of the system employed. With the usual calorimetric system having a heat-capacity of about 1000 grm.-calorie units, the air-gaps surrounding the vessel should be not less, and need not be more, than 40 mm. wide. With such gaps the system is practically adiabatic, if the temperature of the bath fluctuates within  $\pm 0.01^\circ$  C., and that of the environment within  $\pm 0.2^\circ$  C. of the temperature of the calorimeter, the total leakage of heat being little more than three times that of a perfect vacuum gap. Stirring of the closed system is advantageous and may be continuous, although intermittent stirring is preferable; the loss of heat thus involved is negligible.

The open calorimeter may be safely used for the adiabatic measurement of rapid reactions, either with dry surfaces in a gap initially dry or with surfaces not visibly wetted in a saturated gap containing no water, provided always that correction for aggregate thermal disturbances due to leakage by evaporation be made by interpolation from the results of observations made before and after the period of the reaction. An experimental method for the determination of leakage by conduction in any closed calorimetric system is described, and a simple empirical rule is given for estimating its approximate magnitude in closed cylindrical systems of convenient dimensions.

T. H. P.

## Reviews.

A COMPREHENSIVE TREATISE ON INORGANIC AND THEORETICAL CHEMISTRY. Vol. I., pp. 1065, and Vol. II., pp. 880. By J. W. MELLOR, D.Sc. London: Longmans, Green & Co. 1922. Price 63s. net per volume.

The task of compiling a survey of the whole of inorganic and theoretical chemistry is an exceptionally onerous one. Furthermore, it requires the services of a well-read chemist who is capable of assimilating the vast store of information and presenting it logically and systematically. Such a work has been undertaken by Dr. Mellor, with whose remarkable ability we are already familiar.

At the present time there is no complete treatise on inorganic and theoretical chemistry in the English language, the student having to rely largely on foreign compilations. The importance of Dr. Mellor's proposed work, the first two volumes of which have appeared, is therefore considerable. There is no doubt that it will be heartily welcomed by teachers and students.

The two volumes now under review are very clearly and carefully written. The magnitude of the work does not permit of a detailed discussion of each point raised. It may be said confidently, however, that every effort has been made to substantiate each point with the fullest bibliographical references, which are given at the end of each section.

The separate treatment of physical and inorganic chemistry, now so often adopted in text books and lecture rooms, although solely one of convenience, is a source of danger to the student. There is a tendency for him to regard them as distinct, thereby failing to realise the connection between them and consequently the importance of physical chemistry for the proper understanding and development of inorganic chemistry. The method employed by Dr. Mellor in his *Modern Inorganic Chemistry*, of correlating the facts and theories of inorganic and physical chemistry, was certainly the main reason for the general adoption of that book. Encouraged by its success, the larger work is being compiled on similar lines.

The general arrangement of Volumes I. and II. is good. In the preface the author refers to the Periodic Classification as being "justified solely by expediency and convention," and that the appearance of order imparted by it is "superficial and illusory." Further, that "it has a tendency to make teachers over-emphasise unimportant and remote analogies, and to under-estimate important and crucial differences." In the circumstances, he is discussing the elements mainly in accordance with the system, and apparently with these reservations.

Volume I. is chiefly historical and introductory. Chapter I.—73 pages—on "The Evolution and Methodology of Chemistry," furnishes a very interesting beginning. The remaining chapters deal with the laws of combination, hydrogen, oxygen and their compounds, and such subjects as the theory of solutions, crystals and crystallisation, equilibria, phase rule, thermo-chemistry, thermo-dynamics, etc. The latter subjects are discussed somewhat fully in order to simplify explanations of a physico-chemical character required in subsequent volumes. As the subject of atomic weight determinations is one of fundamental importance,

one would have thought that an adequate general discussion would have been found in this volume instead of the brief account on pages 180 and 181. In the case of the atomic weights of hydrogen and oxygen, the subject is dismissed after a treatment of about a page in length. It must be mentioned, however, that a complete bibliography is appended. The equation on page 283, representing the action of warm dilute sodium hydroxide on aluminium to form  $\text{Al}(\text{ONa})_3$ , is not in accordance with fact.

In Volume II. the halogens and alkali metals are discussed. As to the arrangement of the matter, the following point should be noted. Compounds, other than hydrides, oxides, halides, sulphides, sulphates, carbonates, nitrates and phosphates, are described with the appropriate acids or acidic elements only. The treatment of these elements and their compounds appears exhaustive. On the contrary, no reference could be found to either of the following considerations:—(a) Hulett and Bonner's useful application of solutions of hydrochloric acid of constant boiling point to the making of standard volumetric solutions of that acid (*J. Amer. Chem. Soc.*, 1909, **31**, 390); or (b) the preparation of solutions of potassium hydroxide free from carbonate by decomposing water with electrolytically prepared potassium amalgam (Ming Chow, *J. Amer. Chem. Soc.*, 1920, **42**, 488). Here again, the author has given a full bibliography, but has barely indicated the general lines along which the determinations of the atomic weights of the nine elements discussed in this volume have been carried out.

The historical treatment of the subjects covered in these two volumes is remarkably extensive and interesting. Numerous quotations, which have either a philosophical bearing, or are, for instance, illustrative of the controversies which arose owing to the enunciation of new theories, help to make the volumes pleasant reading.

Both the general reader and the research student will find plenty of inspiration in the first two volumes, which will command a foremost place among works of reference. Reference, however, might have been facilitated if a fuller index had been given to each volume. This applies more especially to the theoretical subjects which have been discussed, yet only obtain little or no mention in the index. No author-index is given.

The two volumes are well produced, and appear surprisingly free from typographical errors.

In conclusion, Dr. Mellor is to be heartily congratulated on the production of these two volumes, the undoubtedly high standard of which enables us to anticipate the forthcoming volumes of this valuable treatise with keen interest.

HUBERT T. S. BRITTON.

ORGANIC CHEMISTRY, OR THE CHEMISTRY OF THE CARBON COMPOUNDS. By VICTOR VON RICHTER. Edited by Professor R. ANSCHÜTZ and Dr. H. MEERWEIN. Volume II., THE CHEMISTRY OF THE CARBOCYCLIC COMPOUNDS. Translated by E. E. FOURNIER D'ALBE, D.Sc., A.R.C.Sc. Pp. xvi. + 760. London: Kegan Paul, Trench, Trubner & Co., Ltd. 1922. Price 35s. net.

"Small Richter" is too well known to every student of organic chemistry to

need any general description, but during the war it has increased in size enormously, and has also developed the quality of unconscious humour. Many quaint wordings, such as the "progeny" of certain organic compounds, have resulted from the translation, but, although these are sometimes quite incorrect, even in the case of words of an essentially technical nature (for example the mistranslation of "Kohlenwasserstoff" by "carbohydrate"), yet they do not seem to be in danger of confusing the meaning, at least in the mind of the modern type of reader, since with each of its many successive editions Richter's book of organic chemistry has been steadily losing all value as a text book for the uninitiated, and becoming more and more merely a convenient work, in the hands of an organic chemist, for rapid or preliminary reference of a less thorough and drastic variety than that offered by its encyclopædic namesake, M. M. Richter's *Lexikon*.

Two serious defects appear in the new edition, the one permanent, and the other temporary. The book is already rather badly out of date, owing to its being now nine years since the production of the corresponding publication in Germany; it is also temporarily much less useful, as a work for rapid or preliminary reference, than the old American edition, because all heterocyclic compounds have been deleted, in preparation for a separate Volume III. on these substances, and enquiry at the publishers has failed to elicit a date for the appearance of this very necessary companion volume.

English references are made indirectly through German abstracts, and, sometimes, are not made at all. Nevertheless, in spite of these defects, much space could be occupied in detailing the advantages and improvements which have been incorporated in the various portions of this edition, among which may be mentioned those portions dealing with the polymethylenes, the oxidation of benzene nuclei, the halogenation and the nitrogenous derivatives of the hydrocarbons condensed nuclei, quinones, and terpenes.

The book is undoubtedly still the best of its kind that we have in the English language.

ARTHUR FAIRBOURNE.

**POWER ALCOHOL: ITS PRODUCTION AND UTILISATION.** By G. W. MONIER WILLIAMS, O.B.E., M.C., M.A., Ph.D., F.I.C. Oxford Technical Publications. Pp. xii. + 323. London: Henry Frowde and Hodder & Stoughton. Price 21s. net.

The author of this excellent treatise cannot be too highly commended for his work. He has ransacked the literature and presented the result in a book which is most interesting to read, clearly expressed, well arranged, and will be found of the greatest value by all who desire to obtain a digest of existing knowledge on this subject.

The author first discusses and summarises in an introductory chapter the relative merits of the various types of fuel available for power purposes and the conditions under which alcohol is likely to become a motor fuel of importance. Shale oil is not mentioned in this discussion. The theory of the production of alcohol by yeast fermentation from the carbohydrates is next considered, and an account is given of the latest work of Neuberg and others. The methods employed

in the commercial production of alcohol from starch and sugar by fermentation are then described in detail, with special reference to the methods best adapted for use where the greatest yield of alcohol and its production by the most economical methods are the main considerations. An interesting account is given of the "amylo" process, in which the saccharification of starch is effected by certain diastase-producing moulds under aseptic conditions. The methods of distillation employed for separating the alcohol from the fermented wash, and the physical principles involved, are also clearly explained.

Chapter IV. deals with the economics of alcohol production from crops, and this is one of the most important in the book. Although alcohol can be produced from a great variety of vegetable products, the economical value of any particular product depends upon the price at which the material required for one gallon of alcohol can be delivered at the distillery. With this fundamental fact in view, the author compares all the most important cereals, tubers, roots and other natural raw materials containing starch and sugar, including by-products such as molasses, showing in the case of each the yield of alcohol per ton and per acre, together with the cost of cultivation and the quantity of waste material which has to be disposed of. The conclusion is reached that it is not possible at present to produce alcohol at a price which would enable it to compete with petrol, that its cost must be considered as that of a supplementary and not a competitive fuel, and that its rôle is not to drive petrol off the market, but to provide an alternative fuel in the event of an actual and permanent shortage of supplies. The low cost at which alcohol was produced in Germany prior to the War and the extent to which it was used in industry are shown to have been mainly the effect of State subsidies, resulting from a policy originally directed towards the development of agriculture. Under present conditions, the use of alcohol is more likely to be successful as a local industry, but if some biological or other process were to be discovered by which the cellulose of the plant could be converted directly into fermentible carbohydrates an immense step forward would be taken.

The efforts which have been made to produce alcohol from cellulose materials by fermentation of the sugar produced by acid hydrolysis of the cellulose itself, or which are found in the waste liquors of the sulphite cellulose process, are detailed in the next chapter, and the author concludes that much further research is needed to establish the conditions necessary for complete success. Alcohol can be obtained synthetically from ethylene and from acetylene, but the author rightly questions whether the carbon of coal, which is the source of these hydrocarbons, cannot be better utilised in the form of cyanamide which, as a manure for crops, can be made to yield far more alcohol indirectly from the carbon of the atmosphere. In the chapter on Excise Supervision and Denaturation the author remarks that the selection of a suitable denaturant is one of the most important and most difficult of the problems connected with the use of power alcohol, and that although substantial prizes have been offered for its solution these have never been won. The discovery of one or more simple chemical denaturants, capable of being accurately estimated in the spirit, would remove a great difficulty by



rendering possible the importation of denatured spirit, and so avoiding the costly business of denaturing imported spirit in bond.

In Chapter VIII. the principles of the internal combustion engine are outlined and the physical principles involved in determining the efficiency of the engine are explained. The chemical and physical properties of alcohol are then considered in detail from the motor fuel standpoint, and an account is given of all the most important researches, including the latest work of Ricardo and others, by which the relationship of alcohol to the purely hydrocarbon fuels has been established by experiments made under carefully controlled conditions on specially designed machines. In the succeeding chapter an account is given of some of the most important tests which have been carried out on commercial engines. Fuel mixtures containing alcohol form the subject of a brief concluding chapter.

The book is copiously supplied with references and has a good index. I have noticed only two small errors. On page 233 the composition given for commercial petrol is 3 per cent. short of 100, and on page 271, line 23 from top of page, the words "alcohol" and "petrol" have been reversed.

I have felt it a pleasure to be able to add my testimony to the value of this work to all chemists who have to act in a consultative capacity.

L. ARCHBUTT.

#### SOAPS AND PROTEINS: THEIR COLLOID CHEMISTRY IN THEORY AND PRACTICE.

By MARTIN H. FISCHER, M.D., and others. Pp. ix.+272. London: Chapman & Hall. 1921. Price 24s. net.

Readers who may wonder why a distinguished physiologist should write a book on soaps will find the reason for this enterprise stated in a very candid preface. The author looks to the colloid chemistry of the proteins for the explanation of many physiological and pathological processes, but finds the proteins too complex for direct analysis. He has therefore turned to the soaps, as being simpler, and yet sufficiently like the proteins in their colloidal behaviour to enable him to explain the many peculiarities of the latter bodies by analogy. This expectation is probably over-sanguine *a priori*, and is certainly not realised by the chapters on proteins and tissues, which consist largely of ingenious, but unconvincing, parallels between soap-water and protein systems. A particularly striking example of the shortcomings of the method is the elaborate comparison between the heat-coagulation of albumin and the hydrolysis of boiled sodium palmitate solution (p. 231), which certainly does nothing to elucidate this fundamental and much-investigated property of albumin.

These chapters occupy about a fifth only of the book, the rest being devoted to the soaps. The experimental work consists in the preparation of a very large number of pure salts of acids of the acetic, oleic and linolic series, and the investigation of their power to form gels with water and various organic solvents. The results form a valuable collection of material, and an interesting generalisation is established: that practically all the acids whose sodium salts and potassium salts tend to form gels with large water content have an even number of carbon

atoms in the empirical formula. The author is, however, chiefly concerned with applying the results to his theory of the sol-gel transformation, which he states, in its simplest form, thus:—"A change from what is, at the higher temperature, essentially a solution of *soap in water*, to that which is, at the lower temperature, a solution of *water in soap*." The term "solution" is used here, and throughout the book, in a highly elastic sense, but the author insists repeatedly and emphatically, that the methods of, *e.g.* electrochemistry are not applicable to such "solutions" of water-in-soap or water-in-protein. This fundamental thesis is in direct contradiction with the observations of McBain and his co-workers, published some time ago, that the *conductivities of soap sols and gels, at the same temperature and concentration, are identical*. A similar result for gelatin sols and gels containing salts was obtained by S. Arrhenius nearly forty years ago. The author, however, persistently ignores the results and theories of other investigators, or refers to them only to dismiss them summarily. Few readers familiar with the large amount of very careful work devoted to the soaps and proteins within the last decade will be inclined to admit that the author's positive contributions to these subjects justify his superior attitude.

The book is well printed and copiously illustrated, principally by half-tone blocks, showing well over a thousand test tubes and bottles containing soap solutions, gels and froths. The majority of these could have been omitted without detriment, more especially those (like Figs. 76, 96, 97, etc.) in which the elementary precaution of preventing the high lights on the glass has been neglected.

EMIL HATSCHKEK.

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## Publications Received.

METALLOGRAPHY. By C. H. DESCH, D.Sc. (Text-Books of Physical Chemistry.) Third Edition. Pp. 400. London: Longmans, Green & Co. 1922. Price 16s. net.

COAL TAR COLOURS IN THE DECORATIVE INDUSTRIES. By A. CLARKE. London: Constable. 1922. Price 6s.

NOTES ON QUALITATIVE ANALYSIS [Supplement]. By H. J. H. FENTON, D.Sc., F.R.S. Cambridge University Press. 1922. Price 3s. 6d. net.

JOURNAL OF SCIENTIFIC INSTRUMENTS (Preliminary number, May, 1922). Produced by the Institute of Physics, with the co-operation of the National Physical Laboratory. Pp. 35. Proposed subscription 30s. per annum. The *Journal* will not be issued unless sufficient promises of support are received.

CONTENTS.—Foreword (Sir J. J. Thomson); Scheme for a Journal of Scientific Instruments (E. H. Rayner); Instruments and Apparatus in Relation to Progress in Physiology (A. V. Hill); Use of a Reference Plate for the Micrometric Measurement of Astronomical Photographs (Sir F. Dyson); New Fundamental Bench Mark of the Ordnance Survey (C. Close); The Optical Sonometer (Twyman and J. H. Dowell); Two Machines for Rapidly Weighing Loads of a few Milligrammes (Research Staff, Gen. Electric Co.); Reid Control for Aeroplanes (J. Robinson); Measurement of the Internal Diameters of Transparent Tubes (J. S. Anderson); Laboratory Arts: Production of Uniform Cylinders of Small Diameter (G. A. Tomlinson); The Physical and Optical Societies' Exhibition. Review, Notes, Abstracts of Patents.