THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS

THE following candidates have been elected members of the Society:

James Norman Davidson, B.Sc., M.D. (Edin.), University Lecturer on Biochemistry. (*Through the Scottish Section.*)

James Ross Fraser, B.Sc. (Lond.), F.I.C., Chemist in Government Department.

Granville Nicholas Gee, Chemist and Dyeing Technologist.

Daniel Evans Jones, M.Sc. (Wales), F.I.C., Chemist in Public Health Laboratory.

Francis Laurence Kinsella, Chemist with Feeding Stuffs Manufacturers. (*Through the North of England Section.*)

Robert Karran Matthews, F.I.C., Chief Assistant to Public Analyst and University Lecturer on Chemistry. (*Through the North of England Section.*)

William John Puregger, Branch Manager and Chief Chemist to Public Analysts in Australia.

Robert Edwin Stuckey, B.Sc. (Lond.), A.I.C., Ph.C., Staff Chemist at Forensic Science Laboratory. (*Through the North of England Section.*)

John Adam Cunningham Watson, A.I.C., Chemist with Feeding Stuffs Manufacturers. (*Through the North of England Section.*)

STATUTORY RULES AND ORDERS, 1940, No. 606

The Dripping (Maximum Prices) Order*

THE Society having been asked to suggest an appropriate fee for the analysis of samples of dripping submitted to Public Analysts by the Food Controller under this Order, the Emergency Committee of the Council has considered the matter and suggests that a suitable fee would be that recommended for the determination of meat in sausages in the circular letter sent to Public Analysts in May, 1940.

* cf. p. 567

The Composition of some Jam Fruits and the Determination of the Fruit Content of Jams

By C. L. HINTON, F.I.C., and T. MACARA, F.I.C.

(*Read at the Meeting, May* 1, 1940)

In an earlier paper[1] analytical data for the proximate composition of samples of a number of jam fruits were summarised. It was pointed out that there could be no finality in the figures, since the districts of supply, the varieties in cultivation, and the effects of seasonal conditions would all be liable to change over a period and thereby affect the average figures or the range of extremes. With a view to keeping in touch with any such trends, and to extending the data in some directions, further samples of fruits have been examined in the laboratories of the Research Association as opportunity permitted, and the results are given in the present paper.

Many of the samples were purchased locally, some were obtained direct from Covent Garden Market and others were samples from deliveries of jam fruit used by members of the Research Association. Included are a number of samples of fruit grown on the Continent.

The present data include figures for certain analytical characteristics not investigated in the earlier work, *viz.* combined acid (and hence also total acid), lead numbers and *p*H. These additional figures are the result of the working out of the lead precipitation method,[2] and the fact that experience over a number of years has shown this method to have considerable value in the examination of jams. Some of these figures were summarised in the paper mentioned,[2] but they are now given in detail.

Analytical Methods

A description is given of the methods used where these have not already been described,[1,2] or where alterations in the procedure have been made.

Preliminary Preparation of Sample.—Portions of the well-mixed minced material were quickly weighed out in duplicate for: (*a*) Determination of insoluble solids, seeds and pectin; (*b*) Preparation of an aqueous extract on which the determinations of soluble solids, acidity, etc., could be made.

In preparing stone fruits the stones from a weighed quantity of fruit were scraped free from any adherent tissue, allowed to dry in the air and weighed. Only the fleshy part of the fruit was analysed, and the analytical data for these fruits refer to the flesh only.

Insoluble Solids.—The residue of fibre, etc., after the first boiling and filtration, was boiled with two further quantities of water for periods of 15 minutes each and finally washed thoroughly on the filter. Filter and contents were dried overnight at about 105° C. All the filtrates and washings were reserved for the determination of pectin (see later).

Seeds.—The dried insoluble matter was detached from the filter, and boiled for a short time in weak sodium hydroxide solution (about $N/50$), to open the material and clean the seeds. These were allowed to subside, the alkaline liquor was poured off, and the seeds and fibre were boiled with water and then transferred to a filter and washed with hot water. The residue was turned out on to a white tile, and the seeds were teased out with a spatula, placed in a weighed box, dried at 105° C., cooled and weighed.

Pectin.—Experimental work in these laboratories has shown that pectin-containing fruit tissues tend to give up their pectin in varying degree according to the nature of the liquid by which they are extracted. It has, of course, been known

for some time that the *p*H of the extracting liquid is important. It now appears that the nature and concentration of salts in fruit juices can also have a marked effect. When a fruit is boiled with water, the pectin is being extracted not by water only, but by a diluted solution of the juice of the fruit. The composition of this solution will vary according to the composition and dilution of the juice. It is thus clear that the conditions of extraction of the pectin when the fruit is simply "boiled with water" may vary considerably.

In particular, it may be noted that in the preparation of an aqueous extract of the fruit, the pectin is being extracted by a fairly concentrated solution of the juice. When, however, the fruit is boiled with a fair amount of water, and the fibre, etc., is then washed copiously with boiling water, the pectin is virtually being extracted by an excess of distilled water only. It appears that the amount of pectin extracted by the latter procedure may be appreciably greater than by the former. Table I shows some results obtained by the two methods of procedure. Smaller differences of the same kind have been observed repeatedly also in the analysis of jams. It appears that they may partly, but cannot entirely, be accounted for by adsorption affects.

TABLE I—AMOUNTS OF PECTIN EXTRACTED FROM FRUITS BY DIFFERENT METHODS

Mode of extraction, etc.	Pectin extracted from: Blackcurrants (canned) Per Cent.	Raspberries (pulp) Per Cent.	Raspberries (pulp) Per Cent.
Boiled with an equal weight of water for 1 hour, and filtered, but not washed through. Pectin determined in an aliquot part of filtrate	(1) 0·39 (2) 0·35	0·13	0·08
Boiled with successive portions of water and washed through well	(1) 0·57 (2) 0·57	(1) 0·33 (2) 0·27	(1) 0·22 (2) 0·18

In view of this, it was thought desirable to modify the method for determining pectin in fruits, by applying it to the filtrate and washings from the insoluble matter. In some of the determinations the whole of the filtrate and washings was used, in others an aliquot portion. The subsequent procedure was that of the Carré-Haynes determination, without preliminary separation of the pectin with acetone; thus the results must be regarded as "crude calcium pectate."

Figures for pectin in fruits given in the earlier paper[1] are probably somewhat low in comparison with the present results, the pectin having been determined on aliquot parts of a 20 per cent. extract.

Soluble Solids.—For this and other determinations of soluble constituents, 250 g. of the minced sample were gently boiled with 250 ml. of distilled water for an hour, with frequent stirring. After cooling, the mixture was made up to 500 g., well mixed and filtered through paper or a fine sieve. The soluble solids content of the extract was then determined, usually by means of the specific gravity,[3] but in some instances by both specific gravity and dipping refractometer, the results being averaged.

The figures so obtained were in g. of soluble solids per 100 ml. of the extract. In preparing the extract, 100 g. of fruit yield only 200—I g. of total liquid (where I = percentage of insoluble solids in the fruit). Moreover, this (200—I) g. of liquid occupy only $\frac{(200—I)}{D}$ ml. in volume (D being the density or, sufficiently closely, the sp.gr. of the extract). Hence, to convert the results to the percentage by weight in the original fruit, they were multiplied by $\frac{(200—I)}{100\ D}$.

The same factor was applied, of course, to the results of other determinations carried out on the aqueous extract, *i.e.* acid and lead numbers.

Free and Combined Acid.—Previously to 1932 these determinations were made directly on the aqueous extract. Afterwards they were usually made on the pectin-free filtrate, by the procedure described earlier.[2] A few determinations were made on both the aqueous extract and the pectin-free filtrate; the differences, if any, were negligible.

All acidities, even those of malic acid fruits, are expressed for convenience as citric acid.

Lead Numbers.—These were determined as described in the earlier paper[2]. In the analyses of the last few years, use has been made of an improved method for finding the end-point of the lead titrations, especially for the aqueous lead number. This is as follows:

A first rough titration is made, with the molybdate solution added in 0·5 ml. portions. A fresh portion of lead filtrate is then diluted, and molybdate solution is added to within about 0·5 ml. of the expected end-point (as shown by the rough titration). The mixture is boiled, the precipitate is allowed to settle, and the partly clear liquid is decanted through a fluted filter. The precipitate is then transferred to the filter and washed thoroughly two or three times with a jet of almost boiling water. Finally, the filtrate and washings are heated to boiling and the titration is completed, the molybdate now being added 0·1 or 0·2 ml. at a time, and the mixture spotted out with indicator after each addition.

It will be noticed that the figures shown for "lead number per 1 per cent. of total acid" correspond numerically to what was before expressed as "lead number per 0·1 g. of total acid."[2] This is so, of course, because the first expression refers to a unit total acid content in the sample of 1 per cent. and the second refers to a unit total acid of 0·1 g. in 10 g. of sample on which the lead number is based, *i.e.* again 1 per cent. of the sample. The former mode of expression is rather more convenient, and in the present paper replaces the earlier one.

*p*H *Value.*—This was determined on the aqueous extract by means of the quinhydrone electrode in conjunction with a saturated calomel electrode. The formula for calculation of the *p*H from the reading in millivolts was: *p*H = (454—mv.)/ 0·0001984T, where mv. is the reading in millivolts, and T the absolute temperature as ° C. As a control for the accuracy of the system, a standard buffer solution (0·4 per cent. solution of cream of tartar) was used, having a *p*H of 3·57.

TABLE II—*p*H VALUES OF GOOSEBERRY EXTRACTS DETERMINED WITH THE HYDROGEN AND QUINHYDRONE ELECTRODES

No.	*p*H with hydrogen electrode	*p*H with quinhydrone electrode	
		Initial reading*	Subsequent readings
1	3·00	3·04	3·13 (18 mins.)
2	3·32	3·33	3·38 (7 ,,)
3	3·04	3·09	3·13 (9 ,,)
4	2·95	2·90	2·93 (5 ,,)
5	2·90	2·89	2·91 (5 ,,)
6	3·22	3·21	3·24 (6 ,,)
7	3·21	3·19	3·24 (10 ,,)
			3·40 (15 hours)

* About 1 minute after mixing in the quinhydrone, except for No. 3 (3 minutes).

With some extracts, especially of gooseberries and currants, a marked drift of the apparent *p*H occurred. This drift sometimes amounted to as much as 0·1 unit in a few minutes, but varied considerably from one sample to another.

Some comparative determinations on gooseberry extracts showed that the initial value obtained with quinhydrone was usually quite close to that obtained with the hydrogen electrode. (Table II)

In determining the *p*H of the fruit extracts, therefore, the initial reading was taken to give as nearly as possible the correct value.

It seems possible that the drift is due to some action of reducing substances in the fruit juice on the equimolecular mixture of hydroquinone and quinone which is formed when quinhydrone dissolves. The principal substance likely to have a sufficiently powerful reducing action is ascorbic acid; but without further work it would not be safe to ascribe the effect definitely to this substance.

Detailed Analytical Data for the Fruits

It has been thought desirable to give the detailed analyses of each individual sample (Tables III to XV). This is the most satisfactory way of making them available for use in the interpretation of the analysis of jams, etc.; extreme and average figures have only limited value, since a sample which is extreme in one respect is usually more normal in other ways. An asterisk is placed against figures which are more extreme than any of the corresponding figures of the earlier paper.[1]

Gaps in the tables occur because many of the samples were obtained primarily for other work, and time permitted the determination only of the analytical figures required for that work.

Notes.—*Gooseberries.*—The averages, where comparison is possible, are not very different from those reported earlier,[1] but rather low minima have been found for soluble solids and free acid.

The aqueous lead number (per 1 per cent. of acid) is a measure of the approximate proportions of citric and malic acids in the fruit. If the fruit acids were entirely citric acid, the figure would be 13·55; if they were entirely malic acid, it would be approximately 5·8.[2] The average value of 10·36 found, corresponds to a mixture of 41 per cent. of malic and 59 per cent. of citric acid. The extremes, however (11·7 and 9·0) correspond to 24 per cent. and 59 per cent. of malic acid respectively.

Strawberries.—When the "plugs" had not been removed in picking, they were taken out as far as possible prior to analysis. The averages were close to the earlier ones,[1] and none of the extremes lay outside the ranges previously established.

The weight of seeds is a fairly constant proportion of the total insoluble matter, averaging 54 per cent., the extremes being 44 per cent. and 61 per cent.

The lead number (aqueous) per 1 per cent. of acid indicates an average proportion of about 11 per cent. of malic acid in the acids of this fruit. A range of variation from 0 to 40 per cent. was shown by individual samples. Only one sample approached the latter figure, however, and this was in the period before the introduction of the improvement in the titration mentioned above. No subsequent sample has shown a larger proportion of malic acid than 18 per cent.

There is a fairly definite relation between the *p*H and the proportion of combined to total acid. This can be seen from Fig. 1. The dotted line is the neutralisation curve for citric acid in $N/10$ concentration, and the points for the strawberry samples lie fairly near this line. The deviations from the citric acid curve are doubtless due to various causes, *e.g.* the fact that the neutralising bases are not entirely alkalies, but to some extent alkaline earths; the presence of small amounts of malic acid; variations in concentration from the decinormal; lack of accuracy in the various determinations involved. On the whole, in spite of these sources of deviation, the correspondence is quite good. It is obvious that the *p*H of a fruit extract is not an independent property, but is governed by the proportions present of the organic acids and their salts.

Raspberries.—For the purpose of arriving at the average and extreme figures of Table V the data for 24 samples, mostly of Scottish fruits, specially examined in 1934 and recorded previously (T. Macara),[4] have been brought in.

In comparison with earlier figures,[1] the averages for soluble solids and free acid are about the same, but the average for insoluble solids is 1 per cent. lower. This must probably be attributed to the large proportion of samples of the Lloyd George

variety; this has come into considerable favour for jam-making, and it tends to have a smaller proportion of insoluble matter than other varieties. Its content of soluble solids and acid would appear to be more normal.

The weight of seeds averages 77 per cent. of the total insoluble solids, with a range from 70 to 82 per cent.

From the lead number per 1 per cent. of acid the average proportion of malic acid in the total acids is not more than about 3 per cent. In an extreme instance it was 19 per cent., but this sample was analysed in the early period of the lead precipitation method, and the result may be open to some doubt. Later figures, though few, indicate practically no malic acid in this fruit.

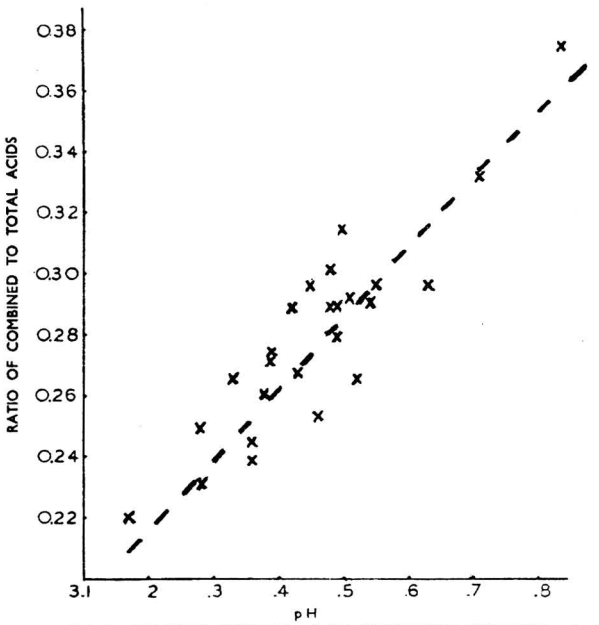

FIG. 1. RELATION BETWEEN pH OF STRAWBERRY EXTRACTS AND THE RATIO OF COMBINED TO TOTAL ACIDS.
[The broken line shown is the neutralization curve for citric acid.]

The relation between the *p*H and the proportion of combined acid in the total acid, as for strawberries, corresponds approximately to a partly neutralised solution of citric acid. Figures for pectin are not given, as it rapidly disappears from this fruit.

Redcurrants.—The averages on the whole are very close to the earlier ones.

The lead numbers, both aqueous and acetone, tend to be higher than can be accounted for by assuming the total acid present to be citric acid. (This would require values of 13·55 and 15·0 respectively for the lead numbers per 1 per cent. of total acid.) This seems to be due to precipitation of a certain amount of lead by the colouring matter of the fruit which, however, does not contribute to the acidity as determined by titration. The extracts from two samples were submitted to a decolorising treatment with vegetable carbon, and the lead numbers and total acid were determined on the decolorised solutions. It was found that the lead numbers per 1 per cent. of acid had fallen slightly, the acetone lead numbers now being close to the figure of 15·0 which would be expected from citric acid, or a mixture of citric and malic acids. (Table XVI)

TABLE III—GOOSEBERRIES

Date	Description	Insol. solids Per Cent.	Sol. solids Per Cent.	Acid as citric (cryst.) Free Per Cent.	Acid as citric (cryst.) Combined Per Cent.	Acid as citric (cryst.) Total Per Cent.	Lead numbers Aqueous	Lead numbers Acetone	Lead number per 1% total acid Aqueous	Lead number per 1% total acid Acetone	Pectin Per Cent.	pH (50% extract)
1930, June 2	Small, green	—	—	1·50	0·33	1·83	17·3	—	9·5	—	—	3·20
„ „ 4	Green, hard	—	—	1·99	0·32	2·31	23·8	—	10·3	—	0·99	3·14
„ „ 11	Some partly ripe, not fresh	—	7·5	1·91	0·36	2·27	24·3	—	10·7	—	0·76	3·30
„ „ 12	Very small, partly ripe, not fresh	—	6·2	1·70	0·39	2·09	24·5	—	11·7	—	0·59	3·37
„ „ 13	Green	—	7·5	2·06	0·37	2·43	23·4	—	9·6	—	0·86	3·07
„ „ 16	Green, fairly fresh	—	7·7	1·99	0·40	2·39	25·9	—	10·8	—	0·87	3·23
„ „ 25	Firm, some beginning to ripen	—	7·5	2·14	0·33	2·47	26·1	—	10·6	—	0·69	3·01
„ „ 26	Large, green, beginning to soften	—	10·2	2·49	0·33	2·82	29·9	—	10·6	—	0·79	2·94
„ „ 27	Large, green, firm	—	9·5	2·16	0·39	2·55	24·6	—	9·6	—	1·50	3·26
„ „ 30	Large, green, some beginning to ripen, not very fresh	—	7·7	2·26	0·35	2·61	27·3	—	10·5	—	0·82	2·90
„ July 3	Green, but beginning to ripen, not very fresh	—	7·7	2·04	0·34	2·38	24·1	—	10·1	—	0·64	3·02
„ „ 3	Green and firm	—	8·2	2·19	0·32	2·51	25·8	—	10·3	—	0·70	2·92
„ „ 17	Green, but softening	—	—	1·94	0·32	2·26	22·2	—	9·8	—	—	2·92
1931, June 15	Medium to small, firm, moderately ripe	2·48	7·8	2·24	0·36	2·60	28·6	—	11·0	—	—	2·96
„ July 1	Medium to small, green, firm	3·49	7·4	2·30	0·36	2·66	28·7	—	10·8	—	—	2·92
1932, June 1	Mostly very small, green, firm	2·25	6·5	1·27	0·38	1·65	—	—	—	—	—	3·40
„ „ 21	Rather small, a few turning red	2·24	8·2	2·26	0·39	2·65	28·1	40·9	10·6	15·4	—	3·01
„ July 11	Green and hard	2·84	7·6	2·22	0·36	2·58	28·6	39·8	11·1	15·4	—	2·95
1934, June 13	Rather small, green, very firm	—	—	1·72	0·44	2·16	—	—	—	—	—	—
1936, „ 8	Large, green	—	—	2·08	0·45	2·53	25·8	—	10·2	—	—	—
„ „ 17	Mixed sizes	—	—	1·54	0·26	1·80	16·2	—	9·0	—	—	—
	Highest	3·49	10·2	2·49	0·45	2·82	29·9	40·9	11·7	15·4	1·50*	3·40
	Lowest	2·24	6·2*	1·27*	0·26	1·65	16·2	39·8	9·0		0·59	2·90
	Average	2·66	7·81	2·00	0·37	2·37	25·0	40·4	10·36	15·4	0·84	3·08
	No. of samples	5	15	21	21	21	19	2	19	2	11	18

TABLE IV—STRAWBERRIES

Date	Description	Insol. solids Per Cent.	Seeds Per Cent.	Sol. solids Per Cent.	Acid as citric (cryst.) Free Per Cent.	Acid as citric (cryst.) Combined Per Cent.	Acid as citric (cryst.) Total Per Cent.	Lead numbers Aqueous	Lead numbers Acetone	Lead number per 1% total acid Aqueous	Lead number per 1% total acid Acetone	Pectin Per Cent.	pH (50% extract)
1930, June 17	Large, fully ripe	—	—	8·2	0·99	0·41	1·40	17·9	—	12·8	—	0·51	3·51
" " 18	Partly ripe	—	—	7·7	1·04	0·38	1·42	18·1	—	12·7	—	0·53	3·43
" " 20	Mostly fully ripe, but firm	—	—	9·3	1·08	0·35	1·43	17·9	—	12·5	—	0·68	3·36
" " 23	Ripe, not fresh, slight mould	—	—	7·6	1·05	0·33	1·38	18·3	—	13·3	—	0·43	3·36
" July 1	Full ripe, *ex* Kent	—	—	9·2	0·75	0·28	1·03	13·0	—	12·6	—	0·52	3·39
" " 2	Large, full ripe	—	—	—	0·95	0·37	1·32	15·5	—	11·7	—	0·50	3·49
" " 2	Small, full ripe, not fresh	—	—	—	0·74	0·37	1·11	12·5	—	11·3	—	0·39	3·71
" " 16	Poor specimens, just ripe (end of season pickings)	—	—	—	0·48	0·29	0·77	9·8	—	12·7	—	—	3·83
" " 21	Medium size, just ripe	—	—	—	0·93	0·38	1·31	16·7	—	12·7	—	—	3·49
1931, June 22	Good size, not fully ripe, *ex* Kent	2·05	0·91	9·3	1·24	0·35	1·59	16·7	—	10·5*	—	—	3·17
" " 30	Rather small, ripe and dry, but some mould	2·09	1·02	8·0	0·95	0·36	1·31	17·0	—	13·0	—	—	3·39
" July 3	Medium to small, some mould	2·14	1·22	8·0	1·05	0·38	1·43	17·5	—	12·2	—	—	3·33
1932, June 28	Rather small, fairly ripe, some crushed	2·63	1·40	9·0	1·30	0·42	1·76	22·7	27·0	12·9	15·3	—	3·38
" July 8	Mixed sizes, good condition	1·89	1·15	8·2	1·02	0·37	1·39	18·4	21·5	13·2	15·5	—	3·52
" " 5	Moderate size, mainly soft and ripe	1·64	0·76	7·6	0·71	0·30	1·01	13·0	15·8	12·9	15·6	—	3·63
1933, June 15	Medium size, mostly ripe	2·27	1·27	—	—	—	—	—	—	—	—	—	—
" " 27	Small, uniform, ripe	1·96	—	10·1	—	—	—	—	—	—	—	—	—
" " 30	Mixed sizes, mainly ripe, fresh	1·92	—	9·4	—	—	—	—	—	—	—	—	—
1936 " 30	Medium to small, dry	1·94	1·11	—	0·89	0·41	1·30	16·1	19·0	12·4	14·6	—	—
" July 2	Medium to small, dry	2·02	1·19	—	1·00	0·42	1·42	18·0	21·3	12·7	15·0	—	3·45
" " 7	Medium to small, some under-ripe	2·32	—	—	0·78	0·32	1·10	14·4	—	13·1	—	—	3·54
" " 20	—	—	—	—	1·17	0·39	1·56	19·7	24·3	12·6	15·6	—	3·28
1937, June 21	Dutch Jucunda, rather under-ripe	1·57	—	6·0	0·70	0·21	0·91	12·4	14·3	13·5	15·5	0·43	3·28
" " 25	Dutch Everns, rather over-ripe, crushed and mouldy	2·60	—	10·0	1·00	0·34	1·34	17·2	20·6	12·8	15·2	0·47	3·46
" " 30	Dutch Jucunda, ripe, slight mould, rather sandy	2·45	—	9·7	1·08	0·44	1·52	20·2	23·0	13·1	15·0	0·62	3·42
1938, " 23	Dutch Jucunda, medium size, ripe, rather wet and not fresh	2·11	—	8·8	0·93	0·38	1·31	18·2	20·6	13·8	15·6	0·63	3·48
" " 24	Dutch Everns, small, ripe, some crushed, fairly fresh	1·94	—	9·3	0·98	0·45	1·43	19·6	22·3	13·8	15·6	0·68	3·50
" July 2	Dutch, small, ripe, bruised, slight mould, plugs taken out	2·01	—	8·1	0·90	0·38	1·28	16·2	19·2	12·7	15·0	0·52	3·55
" " 22	Dutch, small, very much crushed, many mouldy, plugs taken out	2·59	—	7·8	0·88	0·38	1·26	16·8	19·7	13·2	15·5	0·53	3·48
	Highest	2·63	1·40	10·1	1·30	0·45	1·72	22·7	27·0	13·8	15·6	0·68	3·83
	Lowest	1·57	0·76	6·0	0·48	0·21	0·77	9·8	14·3	11·3	14·6	0·39	3·17
	Average	2·11	1·11	8·56	0·95	0·36	1·31	16·67	20·66	12·77	15·31	0·53	3·46
	No. of samples	19	9	20	26	26	26	25	13	25	13	14	25

* These results are doubtful and are not included in extremes and average.

TABLE V—RASPBERRIES

Date	Description	Insol. solids Per Cent.	Seeds Per Cent.	Sol. solids Per Cent.	Acid as citric (cryst.) Free Per Cent.	Acid as citric (cryst.) Combined Per Cent.	Acid as citric (cryst.) Total Per Cent.	Lead numbers Aqueous	Lead numbers Acetone	Lead number per 1% total acid Aqueous	Lead number per 1% total acid Acetone	pH (50% extract)
1930, July 10	Large, ripe, *ex* Kent	—	—	—	1·45	0·38	1·83	24·5	—	13·4	—	3·27
» » 10	Moderate size, ripe, *ex* Hants	—	—	—	1·96	0·37	2·33	33·1	—	14·2	—	3·04
» » 15	Fairly large, ripe, *ex* Kent	—	—	—	1·37	0·37	1·74	23·7	—	13·6	—	3·26
» » 15	Fairly large, ripe, *ex* Kent	—	—	—	1·57	0·40	1·97	25·9	—	13·1	—	3·28
» » 22	Medium size, ripe, fairly fresh	—	4·37	—	1·39	0·38	1·77	21·4	—	12·1	—	3·28
» » 23	Medium size, some under-ripe	—	4·09	—	1·24	0·31	1·55	19·7	—	12·7	—	3·20
1931, » 17	Mixed sizes, mostly soft and rather mouldy, poor quality	9·04	7·23	6·4	1·02	0·42	1·44	19·0	—	13·2	—	3·46
» » 29	Large, some bruised, generally good	5·00	3·71	8·2	2·02	0·34	2·36	29·9	—	12·7	—	2·87
1932, » 14	Large, crushed in lower layers, rather over-ripe	5·33	—	7·8	1·94	0·45	2·39	34·2	37·4	14·3	15·6	3·22
» » 16	—	5·25	4·19	7·5	2·01	0·47	2·48	33·8	37·3	13·6	15·0	3·17
» » 20	—	6·12	4·60	6·0	1·80	0·48	2·28	28·9	35·0	12·7	15·4	3·10
1933, » 25	Scottish	4·90	—	10·0	2·04	—	—	—	—	—	—	—
1935, Aug. 9	—	—	—	—	1·69	0·43	2·12	28·9	32·2	13·6	15·2	—
1936, July 28	Medium size, rather over-ripe	6·24	—	—	2·14	0·45	2·59	35·7	40·2	13·8	15·5	3·20
	Highest	9·04	7·23	12·2*	2·50	0·48	2·59	35·7	40·2	14·3	15·6	3·46
	Lowest	3·29*	2·38	6·0	1·02*	0·31	1·44	19·0	32·2	12·1	15·0	2·87
	Average	5·19	4·00	8·37	1·71	0·41	2·07	27·7	36·4	13·36	15·34	3·20
	No. of samples	**31**	**30**	**28**	**36**	**13**	**13**	**13**	**5**	**13**	**5**	**12**

Embracing also analyses of 1934 season, already published (T. Macara[4]).

TABLE VI—REDCURRANTS

Date	Description	Insol. solids Per Cent.	Seeds Per Cent.	Sol. solids Per Cent.	Acid as citric (cryst.) Free Per Cent.	Acid as citric (cryst.) Combined Per Cent.	Acid as citric (cryst.) Total Per Cent.	Lead numbers Aqueous	Lead numbers Acetone	Lead number per 1% total acid Aqueous	Lead number per 1% total acid Acetone	Pectin Per Cent.	pH (50% extract)
1930, July 16	Medium to large, ripe, good condition	—	—	—	2·12	0·35	2·47	35·4	—	14·3	—	—	3·05
» » 20	Medium to large, ripe, rather moist but otherwise good	—	—	—	2·70	0·39	3·09	43·1	—	14·0	—	—	3·02
» » 23	Slightly dirty	—	5·84	—	2·54	0·42	2·96	35·4	—	12·0*	—	—	3·03
» » 29	Large, fully ripe, wet with juice	—	3·10	—	2·40	0·45	2·85	37·7	—	13·2	—	—	3·07
» Aug. 5	Ripe, but not fresh, wet condition	—	3·83	—	2·40	0·42	2·82	34·9	—	12·4	—	—	2·95
1931, June 26	Fairly large, ripe, rather crushed	6·42	4·69	10·8	2·06	0·41	2·47	34·8	—	14·1	—	—	3·10
1932, » 7	Moderate size, rather under-ripe, dry	6·23	4·74	10·9	2·48	0·49	2·97	41·7	50·7	14·0	17·1	0·73	3·02
» July 15	Kentish	5·41	4·80	9·0	2·67	0·43	3·10	—	51·8	—	16·6	—	3·05
1934, » 17	Large, ripe, good condition	—	—	—	2·73	—	—	—	—	—	—	—	3·22
» » 18	Large, fresh condition, very few damaged	—	—	—	—	—	—	—	—	—	—	0·47	3·15
1938, » 29	—	—	—	—	2·60	0·50	3·10	41·8	49·7	13·5	16·0	—	—
» Aug. 9	Norfolk, medium size, ripe, good condition	—	—	—	—	—	—	—	—	13·5	15·3	—	—
	Highest	6·42	5·84	10·9	2·73	0·50	3·10	43·1	51·8	14·3	17·1	0·73*	3·22
	Lowest	5·41	3·10	9·0*	2·06*	0·35	2·47	34·8	49·7	12·4	15·3	0·47	2·95
	Average	6·02	4·50	10·23	2·46	0·43	2·87	38·5	50·7	13·64	16·25	0·60	3·07
	No. of samples	**3**	**6**	**3**	**10**	**9**	**9**	**7**	**3**	**8**	**4**	**2**	**10**

* These results are doubtful and are not included in extremes and average.

TABLE VII—BLACKCURRANTS

Date	Description	Insol. solids Per Cent.	Seeds Per Cent.	Sol. solids Per Cent.	Acid as citric (cryst.) Free Per Cent.	Combined Per Cent.	Total Per Cent.	Lead numbers Aqueous	Lead numbers Acetone	Lead number per 1% total acid Aqueous	Lead number per 1% total acid Acetone	Pectin Per Cent.	*p*H (50% extract)
1930, July 17	Medium size, ripe	—	—	—	3·66	0·67	4·33	58·9	—	13·6	—	—	3·10
" " 21	Mixed sizes, rather dirty	—	2·89	—	2·67	0·58	3·25	39·5	—	12·2	—	—	3·18
" " 24	Fairly large, ripe, not fresh	—	4·35	—	3·38	0·52	3·90	51·3	—	13·1	—	—	3·02
" Aug. 2	Mixed sizes, slightly moist with juice	—	3·94	—	2·73	0·45	3·18	42·6	—	13·4	—	—	3·06
1931, June 16	Belgian, very mixed size, poor condition	6·34	3·01	10·6	3·11	0·70	3·81	50·7	—	13·3	—	—	3·20
" " 18	Very small to medium size, fairly ripe, not fresh	5·98	2·33	10·9	3·18	0·67	3·85	50·8	—	13·2	—	—	2·98
" " 25	Medium to small, fairly ripe and fresh	7·87	4·00	11·3	2·85	0·67	3·52	46·0	—	13·1	—	—	3·22
" July 14	Medium size, very soft, some crushed	6·79	3·12	12·8	3·57	0·72	4·29	57·8	—	13·5	—	—	3·07
1932, " 13	Size varied, fair proportion under-ripe	5·98	—	8·9	3·69	0·80	4·49	64·5	74·1	14·2	16·3	—	3·17
" " 20	—	5·77	3·54	9·2	3·80	0·74	4·54	64·8	78·2	14·4	17·3	—	2·94
1933, " 5	—	5·53	—	13·6	3·40	—	—	—	—	—	—	—	—
" " 20	—	5·33	—	13·6	3·85	—	—	—	—	—	—	—	—
1934, Aug. 2	Large, fresh condition, dry	—	—	—	3·40	0·61	4·01	—	—	—	—	0·94	3·17
1936, July 27	Large, ripe	4·11	—	—	2·93	0·65	3·58	46·5	57·3	13·0	16·0	—	3·18
" " 28	Large, ripe	4·74	—	—	2·99	0·53	3·52	46·1	54·6	13·1	15·5	—	2·94
" Aug. 8	Medium size, over-ripe	4·94	—	—	2·66	0·52	3·18	39·7	49·9	12·5	15·7	—	3·08
" " 11	—	5·44	—	—	3·71	0·72	4·43	58·3	67·7	13·2	15·3	1·19	3·10
1937, July 16	Dutch, many very large, firm but ripe	5·24	—	13·6	3·03	0·66	3·69	48·7	60·5	13·2	16·4	1·47	3·18
" " 23	Dutch, very large, not over-ripe, but rather soft and moist with juice	4·26	—	11·5	2·42	0·58	3·00	40·9	48·1	13·7	16·0	1·04	3·09
1938, June 21	French, rather small, some unripe, not very fresh, heavily sprayed (lime-sulphur)	5·13	—	13·3	3·98	0·71	4·69	63·3	75·3	13·5	16·1	1·61	3·18
" " 23	French, moderate size, fairly ripe, some mouldy and crushed	5·68	—	11·3	3·86	0·69	4·55	60·7	73·0	13·4	16·0	1·66	3·14
" July 22	Dutch, medium size, ripe, good condition	5·46	—	15·4	2·67	0·66	3·33	48·9	55·7	14·7	16·8	1·41	3·29
" —	Latvian, canned	—	—	—	—	—	—	—	—	13·9	16·5	—	—
" —	Esthonian, canned	—	—	—	—	—	—	—	—	13·8	16·5	—	—
" —	Canadian, canned	—	—	—	—	—	—	—	—	14·4	17·1	—	—
	Highest	7·87	4·35	15·4	3·98	0·80	4·69	64·8	78·2	14·7	17·3	1·66	3·29
	Lowest	4·11*	2·33	8·9*	2·42*	0·45	3·00	39·5	48·1	12·2	15·3	0·94	2·94
	Average	5·57	3·40	11·99	3·25	0·64	3·86	51·6	63·1	13·47	16·25	1·33	3·11
	No. of samples	17	8	13	22	20	20	19	11	22	14	7	20

TABLE VIII—PLUMS

Date	Description	Insol. solids Per Cent.	† Stones Per Cent.	Sol. solids Per Cent.	Acid as citric (cryst.) Free Per Cent.	Acid as citric (cryst.) Combined Per Cent.	Acid as citric (cryst.) Total Per Cent.	Lead numbers Aqueous	Lead numbers Acetone	Lead number per 1% total acid Aqueous	Lead number per 1% total acid Acetone	ρH (50% extract)
1930, July 28	Italian, blue, medium size, moderately ripe	1·16	4·5	—	2·11	0·40	2·51	—	—	—	—	2·94
» Aug. 5	Fully ripe, some slightly crushed	1·19	4·5	—	1·82	0·37	2·19	—	—	—	—	2·93
» » 6	Blue type, small, fully ripe	1·57	4·3	—	1·72	0·52	2·24	—	—	—	—	3·06
» » 18	Blue-green, small, ripe	2·62	4·3	—	1·21	0·36	1·57	—	—	—	—	3·14
» » 25	Victoria, mixed sizes, unequally ripe, rather crushed	1·32	4·2	—	1·32	0·30	1·62	—	—	—	—	3·01
» » 29	Medium size, fully ripe	0·98	2·9	—	1·32	0·29	1·61	—	—	—	—	2·94
» Sept. 1	Red, large, ripe, but condition poor	1·23	4·0	—	1·78	0·32	2·10	—	—	—	—	2·90
» » 4	Golden, large, ripe to over-ripe, some slightly bruised	0·85	4·0	—	1·53	0·35	1·88	—	—	—	—	3·02
» » 10	Red, over-ripe, bruised and wet with juice	1·29	5·4	—	1·16	0·34	1·50	—	—	—	—	3·13
1931, Aug. 5	Blue, medium size, fully ripe	1·36	5·5	12·1	1·94	0·47	2·41	—	—	—	—	2·91
» » 31	Victoria, fairly large, mixed ripe, over-ripe and green	1·31	2·8	11·2	1·61	0·35	1·96	—	—	—	—	2·92
» Sept. 2	Victoria, mixed sizes, ripe, bruised	1·31	4·6	10·4	1·65	0·36	2·00	—	—	—	—	2·88
» » 4	Golden, very uneven in size and ripeness, fair condition	1·25	3·9	12·1	1·68	0·37	2·05	6·9*	—	3·4*	—	2·97
» » 14	Red, small to medium size, ripe	1·75	3·3	16·6	2·62	0·43	3·05	7·6*	—	2·5*	—	2·80
1932, » 1	Blue type, hard and greenish	1·76	3·8	12·0	1·37	0·38	1·75	9·7	24·9	5·5	14·2	3·08
» » 14	Victoria, Kentish, fairly large, ripe and sound	1·15	3·7	11·7	1·31	0·23	1·54	8·6	19·0	5·6	12·3	2·82
» » 22	Blue type, mainly ripe	1·39	4·0	15·6	1·84	0·51	2·35	18·0	34·4	7·6	14·6	3·15
	Highest	2·62*	5·5	16·6	2·62	0·52	3·05	18·0	34·4	7·6	14·6	3·15
	Lowest	0·85	2·8	10·4	1·16	0·23	1·50	8·6	19·0	5·5*	12·3	2·80
	Average	1·38	4·11	12·72	1·65	0·37	2·02	12·1	26·1	6·2	13·7	2·97
	No. of samples	17	17	8	17	17	17	3	3	3	3	17

* These results are doubtful and are not included in extremes and average.

TABLE IX—GREENGAGES

Date	Description	Insol. solids Per Cent.	† Stones Per Cent.	Sol. solids Per Cent.	Acid as citric (cryst.) Free Per Cent.	Acid as citric (cryst.) Combined Per Cent.	Acid as citric (cryst.) Total Per Cent.	Lead numbers Aqueous	Lead numbers Acetone	Lead number per 1% total acid Aqueous	Lead number per 1% total acid Acetone	ρH (50% extract)
1930, July 25	Small, hard, unripe	2·93	5·0	—	1·49	0·54	2·03	—	—	—	—	3·08
» Aug. 7	Rather small, ripe	1·96	5·2	—	1·12	0·50	1·62	—	—	—	—	3·13
» » 13	Mixed in size, ripe, bruised	2·33	5·1	—	1·25	0·44	1·69	—	—	—	—	3·12
» Sept. 4	English, small, firm, ripe, not fresh	1·41	7·1	—	1·17	0·41	1·58	—	—	—	—	3·16
1931, July 27	Italian, mostly sound and ripe	1·14	6·5	9·8	1·41	0·41	1·82	—	—	—	—	3·06
» Aug. 7	Medium size, full ripe	1·98	5·3	17·2	1·16	0·64	1·80	—	—	—	—	3·21
1932, Sept. 5	English, ripe, soft, a little bruised and some mould	1·38	2·9	15·5	1·73	0·44	2·17	11·9	28·2	5·5	13·0	3·00
» » 12	English, large, ripe and soft	1·14	3·5	13·2	1·01	0·36	1·37	8·1	17·1	5·9	12·5	3·19
» » 21	—	—	—	—	0·96	0·49	1·45	—	15·0	—	10·3	3·46
1936, » 18	English, medium size, rather unripe	—	—	—	1·97	0·43	2·40	16·6	34·1	6·9	14·2	—
» » 25	—	—	—	—	2·20	0·56	2·76	17·1	38·1	6·2	13·8	—
	Highest	2·93*	7·1	17·2	2·20*	0·64	2·76	17·1	38·1	6·9	14·2	3·46
	Lowest	1·14	2·9*	9·8*	0·96*	0·36	1·37	8·1	15·0	5·5	10·3	3·00
	Average	1·79	5·1	13·91	1·41	0·48	1·89	13·4	26·6	6·12	12·74	3·16
	No. of samples	8	8	4	11	11	11	4	5	4	5	9

† On entire fruit; other data on stone-free portion only.

TABLE X—DAMSONS

Date	Description	Insol. solids Per Cent.	† Stones Per Cent.	Sol. solids Per Cent.	Acid as citric (cryst.) Free Per Cent.	Acid as citric (cryst.) Combined Per Cent.	Acid as citric (cryst.) Total Per Cent.	Lead numbers Aqueous	Lead numbers Acetone	Lead number per 1% total acid Aqueous	Lead number per 1% total acid Acetone	pH (50% extract)
1930, Sept. 1	Rather small, ripe and sound	1·92	8·4	—	2·53	0·58	3·11	—	—	—	—	3·01
" " 5	Small, ripe	1·74	8·9	—	2·39	0·65	3·04	—	—	—	—	3·01
" " 8	Mostly small, ripe, not fresh	2·26	9·9	—	2·21	0·63	2·84	—	—	—	—	3·11
" " 12	Medium size, ripe, not quite fresh	2·15	10·8	—	2·05	0·60	2·65	—	—	—	—	3·10
" " 23	Large, fully ripe or over-ripe, and wet with juice	2·18	7·3	—	2·01	0·63	2·64	—	—	—	—	3·12
1931, " 6	Medium to small, firm and fresh, sour flavour	2·50	6·8	13·6	2·26	0·53	2·79	9·8*	—	3·5*	—	2·96
" " 16	Mostly small, ripe, not fresh	2·39	7·7	13·0	2·49	0·64	3·13	9·4*	—	3·0*	—	3·00
1932, " 20	Rather over-ripe	2·43	5·8	15·6	2·14	0·64	2·78	19·4	43·3	7·0	15·6	3·19
" Oct. 3	Californian, rather large, some soft and over-ripe	1·44	4·6	14·8	2·01	0·38	2·39	13·3	34·9	5·6	14·6	2·98
	Highest	2·50	10·8	15·6	2·53	0·65	3·13	19·4	43·3	7·0	15·6	3·19
	Lowest	1·44	4·6	13·0	2·01	0·38	2·39	13·3	34·9	5·6	14·6	2·96
	Average	2·11	7·8	14·25	2·23	0·59	2·82	16·4	39·1	6·3	15·1	3·05
	No. of samples	9	9	4	9	9	9	2	2	2	2	9

† On entire fruit; other data on stone-free portion only.
* These results are doubtful and are not included in extremes and average.

TABLE XI—APPLES

Date	Description	Insol. solids Per Cent.	Sol. solids Per Cent.	Acid as citric (cryst.) Free Per Cent.	Acid as citric (cryst.) Combined Per Cent.	Acid as citric (cryst.) Total Per Cent.	Lead numbers Aqueous	Lead numbers Acetone	Lead numbers per 1% total acid Aqueous	Lead numbers per 1% total acid Acetone	pH (50% extract)
1930, July 25	Hard, green	—	—	1·61	0·24	1·85	—	—	—	—	2·86
" " 30	Hard, green, sour	—	—	1·37	0·19	1·56	—	—	—	—	2·86
" Aug. 6	Small, green	—	—	1·27	0·28	1·55	—	—	—	—	3·00
" " 7	Large, green, sour	—	—	1·61	0·22	1·83	—	—	—	—	2·80
" " 15	Large, green, bruised	—	—	1·34	0·24	1·58	—	—	—	—	2·92
" " 27	Small, green, bruised	—	—	1·24	0·20	1·44	—	—	—	—	2·89
" Sept. 3	Medium size, beginning to ripen, bruised	—	—	0·96	0·25	1·21	—	—	—	—	3·11
" " 8	Medium size, dark green, hard, bruised, sweet taste	—	—	0·82	0·22	1·04	—	—	—	—	3·16
" " 19	Very large, beginning to redden and ripen, slightly bruised	—	—	0·94	0·22	1·16	—	—	—	—	3·10
" " 29	Medium size, fairly ripe, slightly bruised	—	—	0·82	0·30	1·12	—	—	—	—	3·38
" Oct. 3	Medium size, green, but fairly ripe	—	—	0·61	0·28	0·89	—	—	—	—	3·45
1932, Aug. 30	Kentish	2·21	9·8	1·21	0·24	1·45	10·9	22·2	7·4	15·1	2·96
" Sept. 9	Hard, yellow-green	2·09	11·2	1·15	0·28	1·43	10·1	21·2	7·1	14·9	3·09
" " 16	Bramleys, rather large, green, some rather sweet	2·61	11·1	1·08	0·24	1·32	9·7	19·6	7·4	15·0	3·13
" " 29	Rather large, green, fairly sweet	3·20	11·7	0·82	0·28	1·10	6·9	16·5	6·3	15·0	3·22
	Highest	3·20	11·7	1·61	0·30	1·85	10·9	22·2	7·4	15·1	3·45
	Lowest	2·09	9·8	0·61	0·19	0·89	6·9	16·5	6·3	14·9	2·80
	Average	2·53	11·0	1·12	0·25	1·37	9·4	19·9	7·05	15·0	3·06
	No. of samples	4	4	15	15	15	4	4	4	4	15

TABLE XII—BILBERRIES

Date	Description	Insol. solids Per Cent.	Seeds Per Cent.	Sol. solids Per Cent.	Acid as citric (cryst.) Free Per Cent.	Combined Per Cent.	Total Per Cent.	Lead number (Aqueous)	Lead number per 1% acid (Aqueous)	Pectin Per Cent.	pH (50% extract)
1929, Sept. 29	Cumberland (a small loss of juice occurred in transit)	3·87	—	9·4	1·10	—	—	—	—	0·37	—
1930, Aug. 26	Norwegian, fully ripe, good condition	3·96	2·31	—	1·21	0·24	1·45	11·6	8·0	0·51	2·95

TABLE XIII—BLACKBERRIES

Date	Description	Insol. solids Per Cent.	Seeds Per Cent.	Sol. solids Per Cent.	Acid as citric (cryst.) Free Per Cent.	Combined Per Cent.	Total Per Cent.	Lead numbers Aqueous	Acetone	Lead number per 1% total acid Aqueous	Acetone	Pectin Per Cent.	pH (50%) extract
1930, Aug. 25	Large, firm and black, but not fully ripe	—	4·34	—	2·26	0·34	2·60	22·5	—	8·7	—	—	2·64
» Sept. 5	Large, hedgerow fruit, uneven ripeness	7·44	5·56	—	1·20	0·37	1·57	14·5	—	9·2	—	—	3·12
» » 11	Hedgerow (Middlesex), medium size, firm, uneven ripeness	9·72	7·30	—	1·75	0·47	2·22	21·6	—	9·7	—	—	2·95
» » 19	Small, fairly ripe, wet with rain ..	9·55	8·01	—	0·48	0·45	0·93	9·5	—	10·2	—	—	3·83
» » 22	Medium size, fully ripe	9·15	7·65	—	0·72	0·45	1·17	11·9	—	10·2	—	—	3·60
» » 23	Small, not very ripe, rather wet ..	9·47	7·63	—	0·79	0·52	1·31	11·6	—	8·9	—	—	3·62
» » 30	Large, uneven ripeness, wet with rain	8·44	—	—	1·09	0·56	1·65	13·3	—	8·1	—	—	3·39
» Oct. 7	Medium size, full ripe, slightly wet with juice	10·74	8·88	—	0·60	0·47	1·07	10·0	—	9·3	—	—	3·79
1931, Sept. 22	Rather small, fairly ripe and fresh ..	9·21	6·93	8·1	0·36	0·47	0·83	7·2	—	8·6	—	—	4·21
» Oct. 1	Medium size, barely ripe, some mould	9·64	7·26	7·9	0·64	0·49	1·13	10·1	—	8·9	—	—	3·86
1932, Sept. 24	Large, fair condition, but some mouldy	5·05	3·79	7·7	1·82	0·40	2·22	19·4	34·4	8·8	15·6	—	3·16
» » 28	Rather large, slightly under-ripe, fresh condition	7·86	—	8·2	1·03	0·43	1·46	14·6	22·2	10·0	15·2	—	3·29
	Highest	10·74	8·88	8·2	2·26*	0·56	2·60	22·5	34·4	10·2	15·6	—	4·21
	Lowest	5·05*	3·79	7·7*	0·36*	0·34	0·83	7·2	22·2	8·1	15·2	—	2·64
	Average	8·75	6·73	8·0	1·06	0·45	1·51	13·9	28·3	9·22	15·4	—	3·46
	No. of samples	11	10	4	12	12	12	12	2	12	2	—	12
	CANNED BLACKBERRIES.												
1934 —	Solid Pack, U.S.A.	8·56	—	10·35	0·69	0·31	1·00	10·0	—	10·0	—	0·56	—

TABLE XIV—APRICOTS

Date	Description	Insol. solids Per Cent.	† Stones Per Cent.	Sol. solids Per Cent.	Acid as citric (cryst.) Free Per Cent.	Acid as citric (cryst.) Combined Per Cent.	Acid as citric (cryst.) Total Per Cent.	Lead numbers Aqueous	Lead numbers Acetone	Lead number per 1% tota lacid Aqueous	Lead number per 1% tota lacid Acetone	Pectin Per Cent.	*p*H (50% extract)
1932, July 1	—	1·16	5·5	9·9	0·92	0·62	1·54	14·7	23·5	9·6	15·3	—	3·90
1938, " 2	Italian, ripe	1·58	—	14·0	2·02	0·67	2·69	27·0	40·1	10·0	15·2	0·88	3·46
" " 2	Italian, unripe	1·57	—	12·4	2·15	0·63	2·78	—	—	—	—	1·04	3·42
1939, Jan. 6	South African	1·78	6·5	15·7	1·19	0·59	1·78	—	—	—	—	0·90	3·86
	Highest	1·78	6·5	15·7	2·15	0·67	2·78	27·0	40·1	10·0	15·3	1·04	3·90
	Lowest	1·16	5·5	9·9	0·92	0·59	1·54	14·7	23·5	9·6	15·2	0·88	3·42
	Average	1·52	6·0	13·0	1·57	0·63	2·20	20·8	31·8	9·8	15·25	0·94	3·66
	No. of samples	4	2	4	4	4	4	2	2	2	2	3	4

† On entire fruit; other data on stone-free portion only.

TABLE XV—APRICOT PULPS

Date	Description	Insol. solids Per Cent.	† Stones Per Cent.	Sol. solids Per Cent.	Acid as citric (cryst.) Free Per Cent.	Acid as citric (cryst.) Combined Per Cent.	Acid as citric (cryst.) Total Per Cent.	Lead numbers Aqueous	Lead numbers Acetone	Lead number per 1% total acid Aqueous	Lead number per 1% total acid Acetone	Pectin Per Cent.	*p*H (50% extract)
1932, Nov. 8	—	1·24	—	10·7	1·36	0·54	1·90	20·8	25·9	11·0	13·6	0·58	3·45
1937, Oct. 2	Average pasty consistency	1·00	—	14·6	1·80	0·38	2·18	24·0	—	11·0	—	—	—
" " 2	Average pasty consistency	1·20	—	13·0	2·05	0·63	2·68	27·0	—	10·1	—	—	—
" " 2	Very thick and fruity	1·66	—	11·8	2·64	0·55	3·19	33·0	—	10·4	—	—	—
" " 2	Average pasty consistency	1·15	—	16·2	1·33	0·63	1·96	22·4	—	11·4	—	—	—
1938, Feb. 22	South African	1·41	—	16·4	1·70	0·56	2·26	20·3	32·8	9·0	14·7	0·81	—
" Aug. 23	Spanish (casks)	0·92	—	12·0	2·17	0·30	2·47	28·2	37·5	11·4	15·2	0·59	2·86
" " 23	Spanish (casks)	0·89	—	12·5	2·17	0·33	2·50	28·3	38·3	11·3	15·3	0·68	2·89
" " 23	Syrian (casks)	1·73	—	13·0	1·83	0·68	2·51	27·2	36·1	10·8	14·4	0·69	3·41
" " 23	Syrian (casks)	1·54	—	13·0	1·69	0·73	2·42	27·4	37·1	11·3	15·3	0·78	3·44
	Highest	1·73	—	16·4	2·64	0·73	3·19	33·0	38·3	11·4	15·3	0·81	3·45
	Lowest	0·89	—	10·7	1·33	0·30	1·90	20·3	25·9	9·0	13·6	0·58	2·86
	Average	1·27	—	13·32	1·87	0·53	2·40	25·86	34·62	10·77	14·75	0·69	3·20
	No. of samples	10	—	10	10	10	10	10	6	10	6	6	5

Not determined when present; other data on stone-free portion only.

TABLE XVI—EFFECT OF REMOVAL OF COLOUR ON THE LEAD NUMBERS OF REDCURRANT EXTRACTS

No.		Lead No. per 1 per cent. of total acid	
		Aqueous	Acetone
1	Before decolorising	13·5	16·0
	After ,, ...	13·2	15·2
2	Before ,, ...	13·5	15·3
	After ,, ...	13·3	14·8

The aqueous lead numbers (per 1 per cent. of acid) indicate that the average proportion of malic acid in this fruit is practically nil. In one sample it appeared to be as high as 20 per cent., but this again was one of the early analyses by the lead method, and is open to some doubt.

Blackcurrants.—The minimum figures for insoluble and soluble solids and free acid are lower than before[1] and the averages for soluble solids and free acid are also appreciably lower.

The proportion of seeds in the insoluble matter varies from 39 per cent. to 61 per cent., the average being 49 per cent.

As with red currants, the lead numbers per 1 per cent. of acid exceed the figures to be expected from citric acid. This again appears to be due to lead-precipitating effects of the colouring matter. The lead numbers and acidities were determined on the extracts of the last three samples after treatment with decolorising carbon. Appreciably lower figures for the lead numbers per 1 per cent. of acid were found, and the acetone lead numbers were now much nearer the figure for citric acid or citric and malic acids. (Table XVII)

TABLE XVII—EFFECT OF REMOVAL OF COLOUR ON THE LEAD NUMBERS OF BLACKCURRANT EXTRACTS

No.		Lead No. per 1 per cent. of total acid	
		Aqueous	Acetone
1	Before decolorising	13·9	16·5
	After ,, ...	12·4	15·0
2	Before ,, ...	13·8	16·5
	After ,, ...	13·0	15·3
3	Before ,, ...	14·4	17·1
	After ,, ...	12·6	15·4

Allowing for the average effect of the colouring matter, as thus indicated, the average lead number per 1 per cent. of acid shows that about 19 per cent. of the total acid is malic acid. The extreme figure found might indicate up to 35 per cent., but this was obtained in the early days of the lead method. In only one later sample does the proportion seem to be at all high—about 32 per cent.

The *p*H values are on the whole rather higher (about 0·1) than would correspond to the proportion of combined to total acid, assuming the latter to be citric acid or a mixture of citric and malic acids. Possibly somewhat high results may have been due to the upward drift during the determination, mentioned earlier. This was often rather marked with blackcurrants.

Plums.—The average figure for insoluble solids is higher than previously recorded,[1] but there is little difference in the averages for soluble solids and free acid. An appreciably higher maximum value for the insoluble solids is to be noted.

Few samples were examined by the lead process, as, until the use of the pectin-free solution was introduced in 1932, the titrations with stone fruits were difficult and very probably in error. This may account for the very low figures (per 1 per cent. of acid) obtained for the two samples examined in 1931.

The *p*H values, when compared with the proportion of combined to total acid, were on the whole rather lower (about 0·1) than would correspond to the neutralisation curves of either malic or citric acid.

Greengages.—The average and maximum soluble solids are much higher than before[1]; the same applies to a less extent to the free acid.

Damsons.—A rather lower average than previously was found for the free acid.[1] Extreme figures for insoluble and soluble solids and free acid all lie within the previous limits.

The aqueous lead numbers indicate very little citric acid. The two results of this determination for 1931 samples are open to some doubt for the reason mentioned in discussing the results for plums.

Apples.—No new extreme limits were established for insoluble and soluble solids and free acid, and the averages for these are close to the previous ones.[1]

There is some evidence, on comparing the *p*H values with the notes on flavour and appearance, that the *p*H followed the degree of ripeness, the riper fruit having higher *p*H. A correspondence of this kind was not observed with the other fruits.

Bilberries.—The Cumberland sample had lost a little juice in transit, and the figure for insoluble solids may therefore be slightly high. The percentage of the soluble constituents would not have been appreciably altered, however.

Blackberries.—Lower minimum figures than before have been found for the insoluble solids and free acid; also a much higher maximum. The very wide range of free acid content shown by this fruit may be noted. Since the combined acid does not vary correspondingly, the *p*H tends to follow the free acid and therefore also shows a wide range.

Apricots and Apricot Pulps.—No figures for apricots were included in the earlier paper.[1]

Although there is no absolute certainty, a general consideration of the figures for each of the samples of pulp, in comparison with the averages and with the data for the fresh fruit, makes it reasonably likely that but little water had been used in their preparation. It may be mentioned that several other samples were met with in which the analysis pointed fairly definitely to the addition of an appreciable proportion of water. These, of course, have not been included.

The lead numbers per 1 per cent. of acid would, of course, not be affected by any added water. They indicate about 40 to 50 per cent. of the acids to be malic.

The *p*H values, when compared with the proportion of combined to total acid, lie fairly close to the neutralisation curve of a mixture of equal parts of citric and malic acids.

Use of the Data in Determining the Fruit Content of Jams

Of the analytical characteristics included in the Tables, the insoluble solids content has perhaps been the most frequently used in assessing the fruit content of jams, but suffers from the disadvantage of the possible uneven distribution of the fruit from jar to jar. This difficulty does not apply to the soluble constituents of the fruit, since these, as a result of the boiling, are fairly evenly distributed throughout the liquid or jelly portion of the jam, including that part which permeates the fruit itself, and the effect of the uneven distribution of the fibrous portions of the fruit is negligible.

Unfortunately, the total soluble constituents derived from the fruit cannot be determined in a jam, because of the merging of these constituents, part of which are sugars, in the added sugar in the jam. Hence the data for soluble solids content,

although affording a useful and independent figure in the examination of fruit pulps. are useless for jams.

The acid and salt constituents (free and combined acid) are, however, available, but here again certain difficulties arise. In the first place, when a jam is made from sulphited fruit pulp, any oxidised SO_2 in the pulp remains as sulphate in the finished jam, an equivalent amount of free fruit acid being liberated from the salt constituents. Hence the free acid will be higher and the combined acid lower than in the original fruit. The sum of the two, however, will be unaltered. Under such conditions, therefore, only the total fruit acid in the jam can be accepted as being directly related to that in the original fruit.

A second difficulty arises from the possible presence in the jam of added liquid pectin containing a certain amount of acid derived from the apple material used as a source, or added for the purpose of extracting the pectin. The lead precipitation method[2] was introduced to overcome this difficulty, and gives useful results with the citric acid fruits.

Another source of uncertainty is the possible addition, during the boiling of the jam, of some acid or salt such as citric acid or a citrate. The magnitude of this difficulty, however, should not be exaggerated. It is very unlikely that any such additions would amount to more than the equivalent of a few per cent. of fruit in the jam; otherwise the effects on flavour, colour, or setting properties would be likely to be objectionable.

INTERPRETING RESULTS.—The discussion of these points indicates that the determination of the fruit content of a jam is beset with difficulties, and, in general, the results are only approximations unless the composition of the fruit used in making the jam is known. It should be noted, however, that the lower the fruit content the closer the results will be to the truth (in terms of percentage of fruit in the jam).

As stated in a previous communication,[1] it is unwise to rely on the insoluble solids figure alone, especially when this is obtained from only one sample. With the best will in the world it is not possible for the manufacturer to ensure that the fibre of the fruit is uniformly distributed throughout all the jars from a single batch. Public Analysts meet with a serious difficulty in this respect, for, should it be found desirable to initiate a prosecution under the Sale of Food and Drugs Act, they must confine their report to the official sample. On the other hand, it has been a growing practice of many authorities of late years to take informal samples, and while the results of these cannot be referred to in a prosecution, the knowledge obtained from their analyses should strengthen the analyst's conclusions. If the analyses of two or more samples of the same make of jam are compared, it will generally be found that, while the insoluble solids may vary considerably, the total acidity and other constituents are reasonably uniform. When this happens it would obviously be unfair to base an opinion on the insoluble solids figure obtained in the analysis of the official sample, if this happened to be lower than the results obtained from the informal samples.

If all the determinations suggested below are carried out, a much sounder conclusion can be arrived at from a consideration of all the figures for fruits containing mainly citric acid. It is true that the lead numbers are much less helpful with fruits containing mainly malic acid, *e.g.* plums, apples, etc., but generally these fruits are so cheap that there is little inducement to lower the quality of the jam seriously.

In calculating the fruit content of a jam from all the analytical data, both the minimum and average figures for the particular fruit should be used. Should the results calculated from the lead numbers and acid figures on the minimal basis indicate a deficiency in fruit, the analyst would be justified in initiating proceedings. It would not, however, be safe to accept the results based on a similar calculation from the insoluble solids figure, unless they were confirmed by results obtained from

other samples or by those calculated from the acid figures and lead numbers on the basis of average data.

When a low fruit content is indicated by the calculation of all constituents on the basis of the average data for these constituents, it is suggested that no prosecutions should be initiated, but that the manufacturers should be given an opportunity of proving that the full quantity of fruit was used. These instances occur as the result of more than the normal amount of condensed steam entering the pulp. Steam pulps must always contain condensate. It should be noted in this connection that fruit preserved with sulphur dioxide sometimes contains excess of water, and the manufacturer may be unaware of this fact. This occurs not infrequently with blackcurrant pulp which is steamed before preservation, and arises from an excessive condensation of steam. Without a full analysis of the contents of each cask, it is difficult to detect occasional casks of such pulp. Manufacturers have been warned to buy pulp made by the hot process on the basis of weight of fresh fruit per cask, and to check this by determinations of the acidity and specific gravity of the juice.

It should be noted that, so far, no single sample of fruit has been found to contain the minimum percentages of both the insoluble solids and acids. With the raspberries containing the lowest percentage of insoluble solids, the acids were up to the average. Blackcurrants having a low insoluble solids have been found to contain less acid than the average, but always more than the minimum. Therefore when considering the results it should not be necessary to base a decision on the results calculated from all constituents on the minimum basis.

A microscopical examination of the jam should always be made, particularly of jams containing seeds, *e.g.* raspberry jam. Cases have been known in which seeds had been added in substantial amount to a raspberry and gooseberry jam, but the very low proportion of raspberry fibre, other than seed, revealed the sophistication, which was afterwards admitted. The proportion of seeds to fibre is also a valuable indication of this form of adulteration. Some results are given in the tables indicating the proportions generally occurring in the original fruit.

Improvements in the Lead Precipitation Method as Applied to Jams

In applying the lead method to jams, as described in the original paper,[2] experience has revealed some minor difficulties and sources of uncertainty. These and the means of dealing with them are described below.

Filtration of Jam Solution.—Filtration of the 50 per cent. extract of the jam through paper is often slow. This can be avoided by using coarse muslin or a 30-mesh sieve.

Possible Error in Determination of Combined Acid.—Errors in the determination of the combined acid have been found to arise owing to traces of sulphates or chlorides in the jam. These substances, when ignited at even a moderate temperature in presence of an excess of sugar, decompose and form carbonates, thus increasing the alkalinity of the ash and causing the combined acid to appear high. If sugar is absent, or is present only in relatively small proportion as in the fruits themselves, this decomposition does not occur, at least with small amounts of these salts.

It was found that the effect could be prevented by adding a sufficient amount of alkaline material, such as potassium or calcium carbonate, to the solution before ashing. For 50 ml. of pectin-free filtrate, 30 ml. of $N/10$ potassium carbonate solution is a suitable addition. In dissolving the ash subsequently, of course, more acid is required, and in calculating the alkalinity of the ash, that of the added carbonate must be deducted.

Avoidance of Errors in the Precipitation of the Lead Malate.—It was shown in the original paper[2] that the proportion of lead malate which is precipitated varies somewhat with the amount present, and with the amount of citrate also present.

The proportions of the two acids must be kept within certain rough limits in order to secure precipitation of a constant proportion of the malate.

This was provided for in analysing different fruits by arranging for the amount of fruit extract taken for the analysis to be varied, and for suitable amounts of either malic acid or citric acid to be added, according to the kind of fruit.

These arrangements may sometimes be interfered with in determining the aqueous lead number of jams containing much added pectin. The presence of the latter entails that a larger proportion of malic acid is present than in the fruit from which the jam is named. Some of the acid is also perhaps lactic acid. This means that the jam from a fruit which is naturally a "citric acid fruit" (Group ii) comes to resemble, in its acid content, a "mixed acid fruit" (Group i); and a jam from a "mixed acid fruit" (Group i) approaches a "malic acid fruit" (Group iii). There will be a tendency for the lead malate to be precipitated to a smaller extent than under the standard conditions. This is most likely to occur with gooseberry, apricot or blackberry jams containing much liquid pectin.

The difficulty can be overcome by treating the jam in question according to the group it tends to resemble. Thus for the aqueous lead number of an apricot jam of this type, the amount of pectin-free filtrate to be taken will be that containing about 0·65 g. of total acid; and instead of 3 ml. of 10 per cent. malic acid solution, 3 ml. of 5 per cent. citric acid solution will be added. Usually it is known beforehand that the sample is one likely to require this modification. If this is only discovered after the analysis has been carried out in the usual way, the determination of the aqueous lead number should be repeated as modified.

The acetone lead number does not appear to be liable to this source of error.

Insufficiency of Solution for Titrations.—It sometimes happens in analysing jams very low in acid content, that the amount of pectin-free filtrate available is insufficient to permit of using the proper amount for the aqueous lead number. In that event the deficiency may be made up with a definite amount of citric or malic acid, as required. An allowance for this can be made, though the accuracy of the final lead number is reduced to some extent.

An alternative method is to take two-fifths quantities for the lead number determinations, the amounts of reagents, of course, being reduced in the same proportion, and to make up the mixture to 100 ml. before filtration. For the first rough titration, 25 ml. of the filtrate should be taken, the full 50 ml. being used for the final titration.

Addition of Malic or Citric Acid to Control the Precipitation of Lead Malate.—It has been pointed out that there is a lack of clarity in the requirement that the strength of the solutions of these acids should be correct to within 1 per cent. of the total.[2] This statement means that the strength of the solutions should be within the range 9·9 to 10·1 per cent. or 4·95 to 5·05 per cent. respectively, and does not refer to the total acid in the solution taken for the test.

Improvement in End-point of the Lead Titration.—This has already been described in connection with the analysis of the fruits (see p. 542).

Correction of Lead Numbers for Sulphate present in Jams.—Sulphate may be present in jams as a natural constituent of the fruit or as a result of oxidation of sulphur dioxide either in pulp used in making the jam, or, to a negligible extent, in the jam itself. Lead sulphate is partly insoluble under the conditions of the aqueous lead precipitation and completely so under those of the acetone lead precipitation. In the former, neither the amount precipitated nor the amount retained in solution is constant; both vary with the amount of sulphate present in the mixture. This effect is seen in the following experiment, in which different amounts of sulphate were added to solutions prepared to represent jam solutions under examination by the aqueous lead test.

Mixtures as shown (Table XVIII) were dissolved in water, and each was treated with 20 ml. of 10 per cent. lead acetate solution, made up to 250 ml.,

mixed, and filtered. The titrations with molybdate were carried out as usual, on 50 ml. of filtrate, two individual workers each making two titrations. The averages of the four titrations of each filtrate are shown in the Table.

From these figures it is possible to derive corrections to be applied to the titration of the aqueous lead number. These are shown in Table XIX. The determination of the sulphate can be made on the ash if the precaution is taken of adding excess of alkali prior to ashing, as already described (p. 556).

TABLE XVIII—LEAD PRECIPITATED IN THE AQUEOUS LEAD TEST BY VARYING AMOUNTS OF SULPHATE

(7·5 ml. of 5 per cent. citric acid, 10 ml. of *N*/10 NaOH and 50 g. of sugar in each mixture)

Potassium sulphate added g.	Back titration with molybdate ml.	Difference due to extra lead precipitated by sulphate ml.
0·0	9·81	—
0·0125	9·66	0·15
0·025	9·60	0·21
0·050	9·41	0·40
0·075	9·30	0·51

The acetone lead number is also raised by any sulphate present. Here the lead sulphate is completely precipitated, and the correction is therefore equal to the lead equivalent of the sulphate present. Each 0·01 g. of K_2SO_4 in the amount (P) of pectin-free filtrate used for the test requires a deduction of 0·25 ml. from the titration difference before the acetone lead number is calculated.

TABLE XIX—CORRECTION FOR SULPHATE (AQUEOUS LEAD NUMBER)

Sulphate as K_2SO_4 in the amount (P) of pectin-free filtrate taken g.	Deduction from titration difference ml.
0·01	0·1
0·02	0·2
0·03	0·3
0·05	0·4
0·07	0·5
0·10	0·6

It may be mentioned that the amounts of sulphate in jam only become significant in this respect when fruit pulp containing much oxidised sulphur dioxide or sulphite has been used in manufacture.

MODIFIED CALCULATION OF THE FRUIT CONTENT FROM THE LEAD NUMBERS.—In the earlier paper[2] the acetone lead number (per 1 per cent. of total acid) in a jam containing no lactic acid was taken to be 15, the value for citric or malic acid. On this figure was based the calculation of, and the allowance for, any lactic acid which might be present.

The data now reported show that the value is usually slightly higher than 15, and appreciably higher with black- and redcurrants. For these fruits it was shown that the high figures appear to be due to the colouring matters. These colouring matters are not removed in the ordinary way, and, as in any event they are natural constituents of the fruit, it seems more correct in calculating the fruit content of jams to use the average value for the acetone lead number of the particular fruit in question.

For blackcurrants which, with redcurrants, differ most widely from the figure for the pure acids, a calculation shows that the fruit content indicated will be about 2 per cent. too low if the figure 15 is used in place of the average figure 16·25 found for that fruit. The percentage of lactic acid calculated from the acetone lead number is likely to be more nearly correct if a value appropriate to the kind of fruit present is used.

For convenience in reference, the average values for the lead numbers (per 1 per cent. of acid) of the fruits are collected in Table XX, and the average total acid contents are also shown. The values of L_1 and A_1 are those to be substituted in formula VI of the earlier paper[2]; and the values of L_1' are those now to be used in

TABLE XX—AVERAGE VALUES FOR THE TOTAL ACID CONTENT AND LEAD NUMBERS (PER 1 PER CENT. OF ACID) OF JAM FRUITS

	Total acid (A_1) Per Cent.	Aqueous lead number per 1 per cent. of total acid (L_1)	Acetone lead number per 1 per cent. of total acid (L_1')
Gooseberries	2·37	10·36	15·4
Strawberries	1·31	12·68	15·31
Raspberries	2·07	13·36	15·34
Redcurrants	2·87	13·46	16·25
Blackcurrants	3·86	13·47	16·25
Blackberries	1·51	9·22	15·4
Apricots	2·20	10·6	14·9

place of the citric or malic acid value of 15 in forming an opinion as to the presence of lactic acid and in calculating and correcting for it. It should be noted that if l', the acetone lead number per 1 per cent. of acid, corrected for sulphate as described above, lies above the value of L_1' for the fruit in question, there can be no allowance for lactic acid to be made in evaluating l (aqueous lead number per 1 per cent. of acid), since obviously there cannot be a negative amount of lactic acid. The fact that l' lies above the average value for the fruit simply means that the fruit used is slightly divergent from the normal. There may actually be a small amount of lactic acid present, but in such a case it is clearly impossible to allow for it. The error is not likely, however, to be significant.

For routine tests, the titration of the ash to arrive at the combined acid (and hence the total acid) may sometimes be omitted, reliance being placed simply on the acetone lead number for finding the total fruit acid (*i.e.* less lactic acid). The corrected value of a for substitution in formula VI[2] is then given directly by

$$a = \left(\frac{L'}{L_1'}\right)$$

In such a shortened procedure, in order to find the amount of pectin-free filtrate to be used for the lead number determinations, it is necessary to make a guess at the approximate total acid content from the free acid titration alone.

In conclusion, we wish to thank Messrs. Chivers & Sons Ltd., for help in obtaining samples of fresh fruits from Holland, and the Council of the Research Association for permission to publish this work with a view to assisting in the maintenance of the Jam Standards.

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The Gravimetric Determination of Phosphate and Vanadate

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The possibility of determining various acid radicals by precipitation as insoluble uranyl salts deserves consideration, as the high atomic weight of uranium makes the percentage weight of the radical in the precipitate very small and thus considerably increases the accuracy of the determination. Lewis and Davis[1] have described a method for determining the arsenate radical based on the precipitation of uranyl ammonium arsenate. The present paper deals with the conditions of precipitation, analysis and use of the corresponding salts of phosphoric and vanadic acids.

Determination of Phosphate Group.—Uranyl ammonium phosphate was first described by Arendt and Knop,[2] and L. Barthe[3] has shown that the alkyl amines yield similar derivatives. Priority in suggesting the use of this precipitate in quantitative analysis must be given to Kitchin,[4] who studied the precipitation of disodium hydrogen phosphate with uranyl salts. Although Kitchin employed the formula $(U_2O_3)_2P_2O_5$, then in vogue for uranyl phosphate, and accepted the rough atomic weight value U = 120, yet his results on recalculation in the light of modern data show that he achieved an accuracy lying within 0·1 per cent.

I have studied the application of this method to the phosphates of sodium, ammonium, calcium and barium, and have also used it for the determination of phosphorus in urine and organic compounds. The results indicated that it affords a rapid and accurate method for the determination of phosphate. The general procedure for organic compounds is briefly as follows:—

Determination of Phosphate.—The organic compound (0·1 to 0·4 g.) is decomposed in a Kjeldahl flask by the method of Lewis and Davis (*loc. cit.*)[1] or by means of a mixture in equal volumes of fuming nitric acid and sulphuric acid. A known volume of urine may be similarly decomposed. After complete oxidation, the colourless liquid is neutralised with ammonia (methyl red as indicator) and then slightly acidified with acetic acid, and treated with 2 g. of ammonium acetate. The solution is heated to boiling, approximately *N*/10 uranyl acetate solution is added dropwise until precipitation is complete, and, after (preferably) standing overnight, the precipitate is transferred to a No. 40 Whatman filter-paper, dried, and ignited in a platinum crucible to uranyl pyrophosphate:—$2(UO_2)NH_4PO_4 \rightarrow (UO_2)_2P_2O_7 + 2NH_3 + H_2O$. 1 g. of pyrophosphate $\equiv$ 0·1988 g. of P_2O_5. Overheating will make the residue green, owing to slight decomposition, but addition of a few drops of nitric acid and gentle re-ignition gives the stable yellow pyrophosphate.

The results with organic compounds were very satisfactory. In the inorganic work it was found that the alkali metal and barium salts gave practically theoretical results, but in presence of much calcium the results were generally from 0·1 to 0·5 per cent. too low.

Uranyl Ammonium Vanadate.—This salt does not appear to have been described (*cf.* Mellor, "*Treatise of Inorganic and Theoretical Chemistry*," 1929, Vols. **8**, **9** and **12**), although J. J. Berzelius obtained uranium metavanadate from alkali tetravanadate solutions. Lachatre[5] has shown that ammonium metavanadate in acetic acid solutions gives the tetra- and hexavanadates.

It has been found that, under definite experimental conditions, it is possible to precipitate the vanadate quantitatively as the uranyl ammonium salt. Analysis of the dried yellow precipitate gave NH_3, 3·90; U, 55·95; loss on ignition, 12·1 per cent.; this corresponds with the formula $NH_4(UO_2)VO_4,3/2H_2O$, which requires NH_3, 3·95, U, 55·35, and ignition loss, 12·32 per cent. Friedel and Cumenge[6] have carried

out an accurate investigation of the analogous mineral carnotite and suggest the similar formula, $K(UO_2)VO_4,3/2H_2O$.

Experimental Method.—The following method was applied to pure (AnalaR) ammonium metavanadate dried in a vacuum desiccator, and dissolved in dilute sulphuric acid:—To 25 ml. of the vanadate solution, containing about 0·05 g. of V_2O_5, ammonium hydroxide is added until the solution is just alkaline (neutral red, light filtered with methylene blue).* The solution is then made very slightly acid with acetic acid and 2 g. of ammonium acetate are added. The solution is heated to boiling and, on addition of excess uranyl acetate, a dense, yellow precipitate is thrown down. After standing for some hours to facilitate the formation of definite crystals, this precipitate is transferred to a No. 4 Jena glass Gooch crucible and dried in an air oven at 105° C. to constant weight. 1 g. of $NH_4(UO_2)VO_4,3/2H_2O \equiv 0{\cdot}2114$ g. of V_2O_5. Some typical analyses corroborating this factor are given below. It is important to adjust the *p*H of the solution fairly carefully, as the vanadate precipitate is far more soluble than the corresponding phosphate and arsenate.

	A.	B.	
NH_4VO_3 g.	V_2O_5 g.	$NH_4(UO_2)VO_4$, 3/2 H_2O g.	A/B Per Cent.
0·07382	0·05738	0·2736	20·97
0·08576	0·06667	0·3160	21·16
0·06536	0·05080	0·2409	21·09
0·1001	0·07784	0·3599	21·50
0·04802	0·03733	0·1782	20·96
0·0096	0·00746	0·0360	20·64
		Mean =	21·05

Concentrations of V_2O_5 exceeding 0·08 g. in 25 ml. are to be avoided, as the precipitate is liable to contamination by adsorbed impurities; with concentrations in the above range, however, the results are very satisfactory, and, owing to the heavy nature of the precipitate formed, the method can be employed for very dilute vanadate solutions. Attempts were made to ignite the precipitate to uranyl pyrovanadate $(UO_2)_2V_2O_7$, after filtration, but, although the results were fairly good (within 0·5 per cent.), they were not sufficiently exact for accurate work, and the method should be employed only when an approximate result is required. It is probable that the decomposition by heat is attended by some slight by-reaction which makes the ignition procedure rather inexact; the Gooch method has been found to be far superior.

References

1. D. T. Lewis and V. E. Davis, *J. Chem. Soc.*, 1939, 285.
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3. L. Barthe, *Compt. rend.*, 1911, **152**, 1396.
4. A. Kitchin, *Chem. News*, 1873, **27**, 199.
5. M. Lachartre, "*Mellor's Treatise of Inorganic and Theoretical Chemistry*," 1929, Vol. 9, 759.
6. C. Friedel and E. Cumenge, *Compt. rend.*, 1899, **128**, 532.

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June, 1940

* A 0·1 per cent. alcoholic solution of neutral red mixed with an equal volume of a 0·1 per cent. solution of methylene blue. For details of light filtering, see Vogel, "*Textbook of Quantitative Analysis*," p. 69; Fowles, "*Volumetric Analysis*," p. 71.

Studies in Internal Electrolysis. V

The Determination of Small Quantities of Copper in Presence of Cadmium

By JAMES G. FIFE, M.Sc., F.I.C.

The separation and determination of copper and cadmium has recently aroused considerable interest, as is shown by the work of Rây and Bose,[1] Shennan,[2] Majumdar[3] and Smith.[4] The object of the present investigation was to provide a rapid electrolytic method for the determination of small quantities of copper in presence of cadmium. It was found that this could be effected by the method of internal electrolysis.

Table I

Experiment	Copper taken g.	Conc. sulphuric acid added ml.	Copper found g.	Time of electrolysis, minutes
1	0·0020	nil	0·0020	20
2	nil	nil	nil	17
3	0·0040	nil	0·0040	30
4	0·0010	nil	0·0011	14
5	0·0005	nil	0·0006	21
6	0·0080	nil	0·0080	24
7	0·0100	2	0·0102	34
8	0·0025	2	0·0025	25
9	0·0022	1	0·0022	18
10	0·0007	2	0·0007	18
11	0·0150	3	0·0152	36
12	0·0030	3	0·0030	30
13	0·0200	3	0·0201	50
14	0·0004	4	0·0004	19

The apparatus employed was that described by Sand[5] with the modification used in the determination of small proportions of cadmium and of nickel in zinc.[6] The anodes consisted of cadmium tape prepared by rolling out pure stick cadmium and soldering copper connecting wires to the top end of the cadmium tape. Satisfactory results have been obtained by using a platinum cathode, an anolyte containing cadmium sulphate equivalent to 5 g. of cadmium and also 0·2 g. of hydroxylamine sulphate per 100 ml., and a catholyte of approximately 300 ml. containing the copper to be determined (added as sulphate), 0·2 g. of hydroxylamine sulphate, cadmium sulphate equivalent to 5 g. of metal and in some experiments small quantities of sulphuric acid. The electrolysis was carried out between 70 and 80° C., for about 15 to 60 minutes, according to the amount of copper present; when a new apparatus is set up, however, the time required for solutions of known composition should be determined.

The results are shown in Table I.

Table II

Experiment	Copper taken g.	Cadmium added g.	Copper found g.	Time of electrolysis, minutes
15	0·0096	5·17	0·0095	51
16	0·0117	5·35	0·0117	58

In further experiments, known amounts of pure cadmium and electrolytic copper were dissolved in 10 ml. of conc. sulphuric acid and 2 ml. of conc. nitric acid, the solution was neutralised with ammonia and then acidified with 3 ml. of conc.

sulphuric acid, and 0·2 g. of hydroxylamine sulphate was added. The solution was electrolysed as described above. The results are shown in Table II.

In Expt. 16 the determination was carried to constant weight, showing that prolongation of the electrolysis has no harmful effects.

It is known that copper and cadmium form a eutectic mixture containing approximately 1·2 per cent. of copper.[7] A copper-cadmium alloy was prepared containing 1·03 per cent. of copper, and weighed portions of this were dissolved in 10 ml. of conc. sulphuric acid and 2 ml. of conc. nitric acid. The solution was filtered, neutralised with ammonia and acidified with 3 ml. of conc. sulphuric acid, and 0·2 g. of hydroxylamine sulphate was added.

The results are shown in Table III.

TABLE III

Wt. of alloy taken g.	Copper found g.	Copper Per Cent.
0·436	0·0044	1·01
1·518	0·0158	1·04

I wish to thank Dr. A. J. Lindsey for his interest in this work.

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2. R. J. Shennan, ANALYST, 1939, **64**, 14.
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4. G. S. Smith, *id.*, 1940, **65**, 87.
5. H. J. S. Sand, *id.*, 1930, **55**, 309.
6. J. G. Fife, *id.*, 1936, **61**, 681.
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July 16th, 1940

Notes

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

A LABORATORY TEXTILE SCOURING APPARATUS

METHODS of removing, by scouring, oils used for textile fibre lubrication, have been studied, and detergent baths used for scouring have been compared. Many of these methods proved unsatisfactory, but the simple apparatus shown in the diagram has given results which are as consistent as those obtained with the Society of Dyers and Colourists' Wash Wheel.[1]

It consists of a flask F containing the detergent solution, in which the material to be scoured is placed. The exit tube E has a Gooch crucible G fixed to the end to act as a filter. The inlet tube I has a Bunsen non-return valve at the end.

The detergent solution is circulated through the reciprocating pump R. This consists of a cork collar C hollowed out to form a seating for a steel ball B, thus forming a non-return valve. The piston of the pump has a 1-inch stroke and consists of a cork P, bound round with thin string to form "piston rings." The piston rod is a glass tube connected by means of a connecting rod to the eccentric EX, which makes 8 revolutions per minute.

When the pump is in operation the scouring liquor is forced intermittently in one direction only (shown by the arrows) and in the flask F a swirling motion is set up, which agitates the material being scoured.

The whole apparatus is contained in a water-bath controlled by the thermostat T.

An example is given in Fig. 2, to illustrate the reproducibility obtainable. Wool was oiled under standard conditions with three oils adjusted to have the same viscosity, *viz.* S = sperm oil,

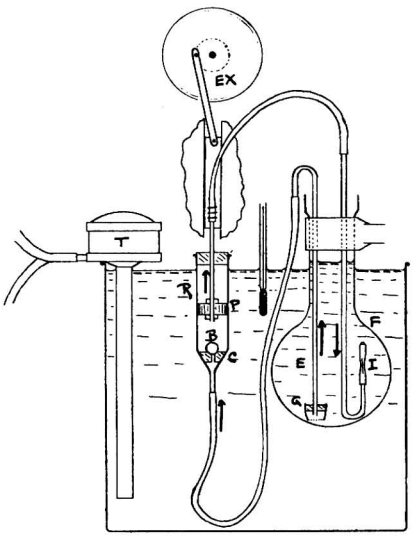

Fig. 1

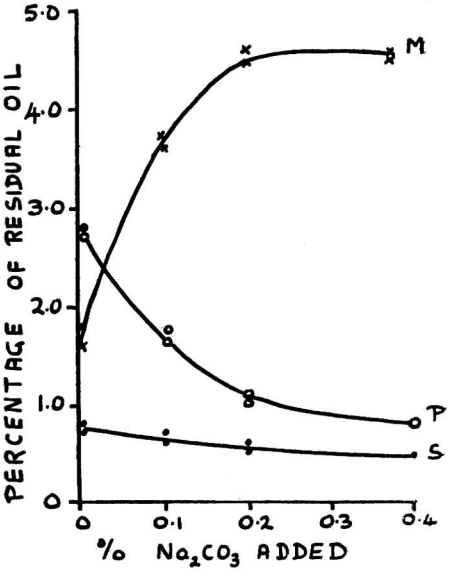

Fig. 2

M = refined mineral oil, and P = refined mineral oil containing a polar compound (6·3 per cent. of oleyl alcohol, as first proposed by J. B. Speakman). Scouring was carried out in every instance in a 0·2 per cent. soap solution for 20 minutes at 37° C., the amount of soda being varied.

REFERENCE

1. *Society of Dyers & Colorists: Report of Fastness Committee.*

LISTER & CO., LTD.
MANNINGHAM MILLS
BRADFORD

W. GARNER

April 18th, 1940

DETECTION OF SMALL AMOUNTS OF PROTEINS ON THE SURFACE OF METAL PLATES

DURING the past two years considerable use has been made in these laboratories of the chloramine test for proteins[1] for the detection of small amounts of protein material—usually albumen or casein—on the surface of zinc or aluminium lithographic printing plates. When investigating the causes of various defects in lithographic plates it has often been important to ascertain whether black specks and patches on the plate are, or are not, printing images formed by albumen or casein.

The chloramine test was originally devised by Cross and Bevan for the detection of small amounts of gelatin sizing in paper and for showing localised spots of gelatin. The test has been modified slightly for use with metal plates and is now carried out in these laboratories in the following manner:

A small portion of the plate to be tested is cut out and all printing ink is cleaned off by thorough rubbing with cotton wool and benzene. The cleaned plate is then placed in a test-tube and moist chlorine gas is passed in for half-an-hour to chlorinate the protein. (This is more convenient than Cross and Bevan's original method of chlorination by immersion in hypochlorous acid solution, which is liable to attack the metal plate.) The strip is removed, rinsed with cold water and placed in a 2 per cent. solution of sodium dihydrogen phosphate at 45° to 50° C. for exactly five minutes, to destroy free chlorine and hypochlorous acid, after which it is transferred to a solution containing 1 per cent. of potassium iodide and 0·1 per cent. of starch, or the solution may be poured on to the plate; specks of protein matter on the surface are stained with the dark blue starch-iodine colour.

The test has been found to work well with specks of light-hardened bichromated albumen and with specks of casein on a plate. It has also been found possible in several instances to determine whether black specks ("scumming") on a litho plate were, or were not, due to specks of

the albumen used in the preparation of the plate not having been washed off properly. Another application of the test has been to ascertain whether scumming has arisen from casein transferred to the plate from the coating of the paper being printed.

I wish to thank the Council of the Printing and Allied Trades Research Association for permission to publish this note.

REFERENCE

1. C. F. Cross, E. J. Bevan and J. F. Briggs, *J. Soc. Chem. Ind.*, 1908, **27**, 260. C. F. Cross and E. J. Bevan, "*A Text Book of Paper-Making*," London, 1936, p. 420.

J. H. YOUNG

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August 8th, 1940

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 special interest to the Society. Notes made from such Reports are submitted to the Publication Committee.

CITY OF BIRMINGHAM

REPORT OF THE CITY ANALYST FOR THE SECOND QUARTER, 1940

OF the 1388 samples submitted, 23 were taken formally.

MILK CHEESE.—A sample sold as "milk cheese" was found to have been made from partly skimmed milk instead of whole milk. The manufacturers agreed to state the facts on the label.

"VINEGAR ESSENCE."—A sample consisted of a colourless, dilute solution of acetic acid of 19·4 per cent. strength, and was labelled "Vinegar Essence (Kosher) 20 per cent. Five parts of water to 1 part of Essence." If these instructions had been followed, the strength of the resultant mixture would have been only 3·25 per cent. It was found that the firm selling this product had formerly been conducted by two refugees, and that since their internment the business had been disorganised. The sale was discontinued.

SACCHARATED IRON TABLETS.—A sample contained only 2·3 g. of saccharated iron carbonate per tablet instead of 4·9 g., the remainder being in the oxidised condition. The deficiency may have been due to deterioration during storage. The firm was cautioned.

COLOURLESS TINCTURE OF IODINE.—A sample contained 7·1 per cent. of free ammonia instead of 1·4 per cent. and 4·6 per cent. of ammonium iodide instead of 3·25 per cent. A formal sample from the same shop contained 2·3 per cent. of free ammonia and only 1·45 per cent. of ammonium iodide. The vendor attributed the incorrect composition to an error in calculation. He was cautioned.

H. H. BAGNALL

Legal Note

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

FALSE TRADE DESCRIPTION OF BLEACHING POWDER

ON August 21st, at Newport Pagnell Police Court, a Cheshire chemical manufacturer was summoned under Section 2 of the Merchandise Marks Act, 1887, for selling bleaching powder under a false trade description—"Chloride of lime." A packet of his preparation, purchased from the Newport Pagnell branch of a chain stores, bore on its label a statement that it was suitable for A.R.P. work. Analysis showed the powder to contain only 13·76 per cent. of available chlorine and to be adulterated with 32 per cent. of sand. It was contended for the defence that the powder could be correctly described as "chlorinated lime" and that that term was synonymous with "chloride of lime." The Bench considered, however, that the description "chloride of lime" implied that the substance was bleaching powder and should therefore comply with the B.P. requirements as to chlorine-content and freedom from "an unusual impurity." According to the B.P. Codex bleaching powder is sometimes known as "chloride of lime."

The defendant was fined £20 with 10 guineas costs.

Ministry of Health

A COMMUNICATION from the Ministry of Health dated September 9th, 1940, notifies the following amendments to the list of Public Analysts appointed by Local Authorities with the approval of the Minister.

Authority	Analyst
Swinton and Pendlebury, Borough	T. R. HODGSON.
West Suffolk, County	W. F. GREEVES (Deputy).
Norfolk, County	,, ,,
Lowestoft, Borough	,, ,,
East Suffolk, County	,, ,,
Great Yarmouth, Borough	,, ,,
Oxford, County Borough	J. H. WEBER (Deputy).
Wakefield, County Borough	F. W. JAFFE (Deputy).
Kingston upon Hull, County Borough ..	D. J. T. BAGNALL.

Ministry of Food

Emergency Powers (Defence)

Food (Jam)

The Jam (Maximum Prices) Order, 1940. Dated August 20, 1940*

THIS Order comprises 8 Articles and 3 Schedules. Article 1 prohibits the sale or exposure for sale of jam of a description mentioned in the first column of par. 5 of the First Schedule unless,

(*a*) such jam conforms to one or other of the quality standards mentioned in paragraphs 2, 3 and 4 of that Schedule, and

(*b*) is labelled in accordance with the provisions of the Second Schedule.

Article 2 prohibits the sale by retail of any jam of a description and quality standard mentioned in the Third Schedule at a price exceeding that specified. This provision does not apply to jam sold by retail by the manufacturer thereof, where the total quantity of jam manufactured in the year ended June 30, 1939, did not exceed 5 tons.

Article 3 provides

(1) That in any prosecution for the sale of jam of a quality standard other than that indicated on the label, or of weight less than was represented to the purchaser, it shall be a sufficient defence for the person charged to prove:

(*a*) that the jam was purchased in the container in which he sold it and with a written warranty as to its quality standard or as to its weight;

(*b*) that he had no reason to believe that the quality standard or weight was otherwise than as warranted.

(2) A warranty shall only be a defence if the defendant has within seven days of the service of the summons sent to the prosecutor a copy of the warranty with a notice stating that he intends to rely on it, and specifying the name and address of the person from whom he received it, and has also sent a like notice of his intention to that person.

(3) A servant of the person who purchased the jam shall be entitled to rely upon the warranty.

(4) The person by whom the warranty is alleged to have been given shall be entitled to appear at the hearing and to give evidence.

(5) Any statement relating to quality standard or weight on any label affixed to the wrapper or container of the jam or in an invoice or similar document relating to any jam mentioned in the invoice or document shall be deemed to be a written warranty.

Article 4 provides that the Order shall not apply to (*a*) jam manufactured outside the United Kingdom; (*b*) the sale of jam sold as part of a meal by a caterer in the ordinary course of his catering business.

Article 5 prohibits any fictitious or artificial transactions or unreasonable charges in connection with the sale or disposition of any jam.

Article 6 authorises the Minister to give or grant general directions, authorisations or licences.

Article 7 provides that infringements of the Order are offences against the Defence (General) Regulations, 1939.

Article 8 provides that the Order shall come into force on September 1st, 1940, and may be cited as the Jam (Maximum Prices) Order, 1940.

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* H.M. Stationery Office, York House, Kingsway, London, W.C.2. 1940. Price 2d. net.

THE FIRST SCHEDULE

Provisions Relating to Quality Standards

1. In this Schedule:
   (*a*) "Fruit Content" means the quantity of fresh fruit or fruit pulp of the variety or varieties of fruit named in the description of the jam (excluding added fruit juice or fruit pectin) used in the manufacture of the jam, expressed as a percentage based on the number of pounds of such fresh fruit or fruit pulp required to be used in making 100 lbs. of finished jam.
   (*b*) "Minimum percentage of soluble solids" means the minimum percentage by weight of soluble solids ascertained by means of a refractometer when the jam is cold, no correction being made for insoluble solids.
2. "Fresh Fruit Standard Jam" means jam,
   (i) of which the percentage of soluble solids is not less than 68·5 per cent.;
   (ii) of which the fruit content complies with the provisions of the second column of the scale in par. 5 below;
   (iii) of which the fruit content consists of fresh fruit only, and
   (iv) which does not contain any added colouring matter or any added preservative other than sugar.
3. "Full Fruit Standard Jam" means jam,
   (i) of which the percentage of soluble solids is not less than 68·5 per cent., and
   (ii) of which the fruit content complies with the provisions of the second column of the scale in par. 5 below.
4. "Lower Fruit Standard Jam" means jam,
   (i) of which the percentage of soluble solids is not less than 68·5 per cent., and
   (ii) of which the fruit content complies with the provisions of the third column of the scale in par. 5 below.
5. MINIMUM FRUIT CONTENT

First column	Second column	Third column
Description of jam	Fresh Fruit Standard Jam and Full Fruit Standard Jam Per Cent.	Lower Fruit Standard Jam Per Cent.
Strawberry	42	20
Blackcurrant	30	
Raspberry	38	
Green gooseberry	35	
Red gooseberry	40	
Victoria plum	40	
Green or golden plum	35	
Red plum	40	
Strawberry and gooseberry	40(20/20)	20(10/10)
Raspberry and gooseberry	40(20/20)	20(10/10)
Gooseberry and strawberry	40(30/10)	20(15/5)
Gooseberry and raspberry	40(30/10)	20(15/5)

NOTE.—The figures in brackets in the second and third column above denote the respective contents of the fruits in the order given.

The SECOND SCHEDULE deals with provisions relating to labelling and the THIRD SCHEDULE gives the maximum retail prices of the three qualities of jam.

STATUTORY RULES AND ORDERS. 1940. No. 606

The Dripping (Maximum Prices) Order, 1940. Dated April 25, 1940*

THIS Order contains eight articles:

(1) "Dripping" means the clear unbleached and unadulterated fat, untreated by any chemical process, of sweet smell, and produced from or by the rendering of Fat and Bones of Sheep, Oxen or Pigs, the finished product of such Rendering or Processing to contain a minimum of 99 per cent. of Saponifiable Matter and a maximum of 2 per cent. of Free Fatty Acids.

"Sale by Wholesale" means any sale other than a sale by retail and includes a sale to a person carrying on business as a fishfryer.

Articles 2 to 5 fix the maximum prices that may be charged and the charge that may be made for a returnable package.

Article 6 provides that the provisions of the Animal Oils and Fats (Provisional Control) (No. 2) Order, 1939(*a*), as amended(*b*), relating to Home Melt Technical Tallow shall apply to any Home Melt Edible Tallow which is not dripping as defined in Article 1 of this Order.

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* H.M. Stationery Office. 1940. Price 1d. net.

(*a*) S.R. & O., 1939 (No. 1480), I, p. 1032. (*b*) S.R. & O., 1940, No. 162.

International Society of Leather Trades Chemists

DETERMINATION OF THE COLOUR OF VEGETABLE TANNIN SOLUTIONS*

THE Committee has studied the application of the Bolton and Williams Colorimeter (ANALYST, 1935, **60**, 447) to the measurement of the colour of vegetable tannin solutions and the calculation of the readings into Lovibond figures. In the earlier experiments the calculations (based on a chart by K. A. Williams) were made by means of the formulae:

$$R = 13{\cdot}32D_3 - 11{\cdot}98D_6$$
$$Y = 251{\cdot}0D_6 - 20{\cdot}30D_3$$

where R and Y are the required Lovibond units, and D_3 and D_6 are "absorption densities" derived from the Bolton and Williams readings at 530 and 640$m\mu$, respectively, by means of equations of the type $D = 2 - \log$ (100—per cent. light absorbed) $= K \times$ depth of layer $\times$ concentration.

A "brightness" figure was obtained by dividing the sum of the red, green and blue (460$m\mu$) transmissions by 3, and the transmissions by subtracting the absorptions from 100.

It was found that with highly coloured liquors there was a pronounced divergence between the two sets of results, and further work showed that the main errors were due to the use of neutral tint Lovibond glasses and to not renewing the copper sulphate solution of the Bolton and Williams instrument often enough. It is recommended that it should be changed at least daily.

In May, 1940, the preparation of a new chart was begun by K. A. Williams, based on more accurate readings than the original ones. A Bolton and Williams colorimeter was adapted to hold the modern Lovibond racks of colour glasses, and it was found that a figure for a combination of two glasses could be obtained, agreeing within the range of the experimental error with the determined figure, by calculation from the separate figures for each glass. From this chart it is anticipated that it will be possible to work out two tables showing the B. & W. figures corresponding respectively with the Lovibond Red and Yellow readings.

The Committee formed the following conclusions:

1. The agreement between the results obtained by different observers with the Bolton and Williams Colorimeter is very good, in view of the fact that most of the observers had never used the instrument before.
2. The concordance between the figures obtained with the colorimeter is very much better than those given by the tintometer.
3. It is agreed that the Bolton and Williams Colorimeter is very simple and easy to work, is very sensitive to differences in colour, and gives definite and reproducible results. The method of reporting results so that they will be easy to visualise is still under consideration, but the new table is very promising.

The Committee records its indebtedness to Mr. Williams for his help and mentions that he has no financial interest in the colorimeter.

International Union of Chemistry

FIFTH REPORT OF THE COMMITTEE ON ATOMS†

THE revised International Table of Stable Isotopes for 1940 is published in English, German, Italian and French, rough or indirect measurements being given in brackets. The following changes in the Table are recommended:

The symbol (M) for Mass Number is replaced by the symbol (A) in conformity with general usage.

LITHIUM.—The figure 7·9 for the percentage abundance of ^{6}Li is probably too high owing to the uncertain correction for the isotope effect in free evaporation. A lower value (7·5) is now recommended in accordance with the measurements of Hoff Lu (*Phys. Rev.*, 1938, **35**, 845).

CARBON.—The very complete investigations of Nier and Gulbransen (*J. Amer. Chem. Soc.*, 1939, **61**, 697) show that the percentage abundance of ^{13}C varies appreciably in nature. The mean value 1·1, now recommended, agrees excellently with the earlier work of Brosi and Harkins (*Phys. Rev.*, 1937, **52**, 472).

CHROMIUM AND IRON.—Percentage abundances calculated from the work of Nier are recommended (*Phys. Rev.*, 1939, **55**, 1143).

MOLYBDENUM.—The more accurate results of Mattauch and Lichtblau (*Z. phys. Chem.*, 1939, B**42**, 288) are incorporated.

EUROPIUM.—The work of Lichtblau (*Naturwiss.*, 1939, **27**, 260) has indicated that the heavier of the twin isotopes of this element is slightly the more abundant.

HAFNIUM.—A new rare isotope has been discovered by Dempster and its abundance estimated to be 0·3 per cent. (*Phys. Rev.*, 1939, **55**, 794).

URANIUM.—The measurements of Nier (*Phys. Rev.*, 1939, **55**, 150) indicate the presence of the third rare isotope 234 (UII) and provide accurate figures for the abundances of the other two.

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* Report No. 1 of the British Section Committee. *J. Int. Soc. Leather Trades Chem.*, 1940, **24**, 257–264.

† *Chairman:* F. W. Aston. *Members:* N. Bohr, O. Hahn, W. D. Harkins, F. Joliot, R. S. Mullikin, M. L. Oliphant. 28 Rue St. Dominique, Paris. 1940.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS

Food and Drugs

Oxidation Turbidities [in Beer] and their Causes. E. Singruen. (*Modern Brewer*, 1940, **23**, 31; *J. Inst. Brewing*, 1940, **46**, 285.)—Chill-haze and oxidation-haze are both due to the precipitation of protein-tannin compounds, but the changes which result in the former only are reversible. The latter contains 50 per cent. of protein and a little mineral matter (including silica and iron oxide), and is coloured by pigments and resins. Its formation is controlled by the sensitiveness of the beer to oxidation, which in turn depends on the nature and proportions of the constituents of the beer, and on the *p*H, being a minimum for the normal *p*H range of beer. Iron and copper, and probably also phosphate, calcium, ammonium and hydroxyl ions and yeast co-zymase, are active oxygen-carriers in these oxidation processes; and it is believed that organic compounds containing the grouping $-C(OH)=C(OH)$ are also involved in autoxidation. Indicator—time tests for beer stability suggest that the stability decreases if the wort is oxidised before fermentation (*e.g.* as in the hot aeration process), and that storage at a high temperature or slow cooling after pasteurisation, accelerates oxidation; agitation also increases haze-formation. The stability is increased as the result of the boiling operation, and the simultaneous increase in colour suggests that the melanoidins may contribute to the reducing properties, which, however, are little influenced by the fermentation process. Beer which contains 5 to 7 ml. of air per bottle will not keep for more than half as long as beer in air-free bottles. J. G.

Determination of Iron in Bread and Bread Ingredients. C. Hoffman, T. R. Schweitzer and G. Dalby (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 454–455.)—Loss of iron during ashing is entirely prevented by addition of 5 ml. of *N* sodium hydroxide solution per 1 g. of sample. The weighed substance is thoroughly mixed with the lye, dried at 100° C., and ashed at a low red heat in an electric muffle. The iron in the ash is determined colorimetrically as usual. W. R. S.

Body Fats of the Pig. V. Component Glycerides of Perinephritic and Outer Back Fats from the same Animal. T. P. Hilditch and W. H. Pedelty. (*Biochem. J.*, 1940, **34**, 971–979.)—The pig fats were examined by a method similar to that previously used (Hilditch and Paul, *Biochem. J.*, 1938, **32**, 1775) in the examination of ox depot fat. The fats were systematically crystallised from acetone and grouped into three or four fractions according to their iodine values, and the respective component acids were determined by ester fractionation. The amounts of fully-saturated glycerides in the two fractions most sparingly soluble in acetone were then estimated by oxidation with potassium permanganate in acetone solution, and, when possible, their component acids were estimated by ester fractionation. The fractions most soluble in acetone (in which the di- and tri-unsaturated glycerides are concentrated) were completely hydrogenated, and the products were fractionated with ether. The component acids in each sub-fraction so obtained were estimated by ester-fractionation, and the component glycerides in each were calculated as binary mixtures of tristearin and palmitodistearin, or of palmitodistearin and dipalmitostearin, according to the proportions of palmitic and stearic acid found. From these results, the amount of tristearin in the hydrogenated product was calculated, and thence the amount of tri-C_{18} glycerides in the acetone-soluble fractions of the original fat. The data thus obtained permit the component glycerides of each fraction of the fat to be grouped into (*a*) dipalmitomono-C_{18}, palmitodi-C_{18} and tri-C_{18} glycerides, and (*b*) fully-saturated, mono-unsaturated-disaturated, di-unsaturated-monosaturated and tri-unsaturated glycerides. From the proportions of these classes of glycerides, the individual components in each fraction of the original fat were deduced. The minor fatty acids present were included with the appropriate major acid. The method now introduced is believed to be much more accurate than those used in the past, and it is claimed that a practically complete statement of the component glycerides in animal depot fats can be given with a fair chance of certainty. The molar percentages of the main components of the two pig fats were found to be:

	Outer back fat	Perine-phritic fat
Palmitodi-"olein" ..	46–53	35–40
"Oleo"-palmitostearin ..	34–27	39–34
"Oleo"-dipalmitin ..	5	9
Stearodi-"olein" ..	0–7	0–5
Tri-"oleins"	10–3	8–3
Fully saturated glycerides	5	9

The results now obtained, together with those on ox depot fat previously reported, support the hypothesis that stearo-glycerides are formed in the animal body by the saturation of pre-formed oleo-glycerides. In addition, a gradual increase in the proportions of dipalmito-glycerides is observed as the general degree of saturation increases. F. A. R.

Detection of Saccharin in Foodstuffs. W. F. Reindollar. (*J. Assoc. Off. Agr. Chem.*, 1940, **23**, 288–290.)—The two tests for saccharin given in the *Methods of Analysis* of the A.O.A.C. depend upon the sweet taste of

the ethereal extract and conversion of the saccharin into salicylic acid. Neither test is specific, since other extracted matter may have a sweet taste, and the procedure in the second test does not destroy naturally occurring or added salicylic acid. The following test, depending upon the conversion of saccharin into phenolsulphonephthalein, is recommended: The sample (25 g.) is mixed with sufficient boiling water to produce 75 ml. and, after standing for 1 hour with occasional shaking, the mixture is acidified with 3 ml. of glacial acetic acid, and 5 ml. of 20 per cent. neutral lead acetate solution are added. The mixture is diluted to 100 ml., allowed to stand for 20 minutes and filtered, and an aliquot portion (60 ml.) of the filtrate is acidified with 5 ml. of hydrochloric acid and extracted with a mixture of equal parts of ether and petroleum spirit (b.p. 30° to 60° C.). The ethereal solution is washed with 5 ml. of water and, after preliminary concentration, the solvent is allowed to evaporate at room temperature. The residue is heated for 2 hours at 135° to 140° C. with 5 ml. of a solution of crystalline phenol in an equal weight of sulphuric acid, and the product is dissolved in a little hot water and poured into about 250 ml. of cold water. After 3 hours the liquid is filtered, made alkaline with 10 per cent. sodium hydroxide solution, and diluted to 500 ml. A magenta or reddish purple colour indicates the presence of saccharin in the original sample. A yellow, buff, or pale salmon colour is not significant, and will be given by a blank experiment made with similar material. Vanillin, if present, must be removed by repeated extraction of the ethereal extract with carbon tetrachloride. With some foods (*e.g.* ginger ale) the preliminary treatment with lead acetate is unnecessary. The method gives satisfactory results with foodstuffs containing 80 p.p.m. of saccharin (equivalent in sweetening power to 4 per cent. of sugar), and will probably detect smaller amounts. A. O. J.

Determination of Vanillin in Vanilla Extracts. H. J. Lynch and N. Deahl. (*J. Assoc. Off. Agr. Chem.*, 1940, **23**, 429–431.)—It was found that in the determination of vanillin in concentrated vanilla extracts containing added vanillin by the official colorimetric method (*Methods of Analysis* of the A.O.A.C., 1935, 307) a considerable portion of the vanillin was not recovered, and the method was examined to discover the cause of its failure. In a modification of the official method the extract containing 8 to 12 mg. of vanillin (usually about 5 ml.) was diluted with water so that a further addition of 4 ml. of the solution of neutral and basic lead acetates produced 50 ml. The mixture was filtered and the precipitate was washed with water until the filtrate measured 100 ml. A 5-ml. portion of this filtrate was assayed by the official method, but, although higher results were obtained, the whole of the vanillin was not found. Subsequent investigation showed that the vanillin could be removed quantitatively from the lead precipitate only by the use of an inconveniently large amount of washing water, and the results indicated that precipitation should be made in a much more dilute solution. The following method is proposed: The sample, containing 8 to 12 mg. of vanillin, is diluted to 100 ml. with water, and a 5-ml. portion of the dilute solution is treated with 0·2 ml. of a solution containing 50 g. each of the neutral and basic acetates of lead per litre. A standard vanillin solution (5 ml.) containing 0·1 mg. of vanillin per ml. is used as control. To each of the solutions 5 ml. of the vanillin reagent (Folin and Denis, *J. Ind. Eng. Chem.*, 1912, **4**, 670; Abst., ANALYST, 1912, **37**, 501) are added, and after 5 minutes the mixtures are diluted to 50 ml. with saturated sodium carbonate solution. Finally the liquids are allowed to stand for 10 minutes and filtered, and the blue colours are compared in a colorimeter. By this method 99·5 per cent. of the known vanillin-content of a sample was found, compared with 79·46 per cent. by the official method. It would appear that in the official method originally developed by Folin and Denis (*loc. cit.*) vanillin is lost either by precipitation as a lead compound or by adsorption on the lead resinate. It is generally accepted that a good vanilla extract should contain about 0·2 g. of vanillin per 100 ml. when assayed by the official method. Such extracts yield over 0·3 per cent. of vanillin by the modified method. A. O. J.

Biochemical

Estimation of Manganese in Organic Material containing Large Amounts of Calcium and Chlorides. T. W. Ray. (*J. Biol. Chem.*, 1940, **134**, 677–681.)—Studies on the metabolism of manganese in the mouse necessitated the estimation of manganese in the ash of the whole animal. The dead mice were dried in an electric oven at 105° C. and ashed in silica beakers in a silica-lined muffle-furnace maintained thermostatically at a temperature not exceeding 700° C. After 8 hours' heating, the ash was allowed to cool and dissolved in 10 ml. of conc. nitric acid. Four drops of conc. sulphuric acid were added (not more, to avoid the formation of more calcium sulphate than could be dissolved), and the solution was evaporated to dryness over a free flame to remove the chlorides. The residue was dissolved in 12 ml. of 25 per cent. nitric acid, and the solution was diluted to 20 ml. (or more, if the mice had been fed on a manganese-rich diet). To an aliquot portion of the solution were added 0·2 ml. of 0·25 per cent. silver nitrate solution and 0·25 g. of potassium persulphate. The mixture was then heated in a water-bath for a few minutes to develop the full permanganic acid colour; it was compared with a standard similarly treated. The most accurate readings were obtained with not more than 0·02 mg. of manganese in 10 ml. of solution. The recovery of added manganese varied between

96 and 103 per cent. of the theoretical. The method was also applied successfully to the estimation of manganese in milk. F. A. R.

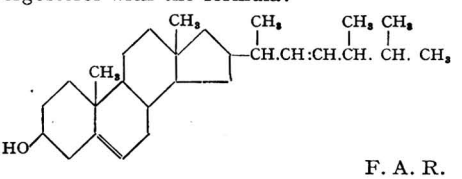
Determination of Fumarate and Malate in Animal Tissues. H. A. Krebs, D. H. Smyth and E. A. Evans. (*Biochem. J.*, 1940, **34**, 1041–1045.)—The polarimetric method for the determination of *l*(-) malate is not specific. The following method, based on reduction of fumarate to succinate, which is then estimated manometrically with succinic dehydrogenase, can be applied to quantities of fumarate from 0·05 mg. upwards; the concentration of malate is calculated from the equilibrium constant. The tissue suspension is deproteinised at 40° C., by adding one-fifth of its volume of 5 per cent. metaphosphoric acid, and filtered. An aliquot portion of the filtrate is transferred to a measuring cylinder, and 0·5 g. of zinc filings, 2·3 ml. of 10 *M* phosphoric acid and 0·25 ml. of copper sulphate solution (20 per cent. solution of $CuSO_4,5H_2O$) are added to each 10 ml. of solution. After one hour the succinic acid is extracted with ether in a continuous extractor. The extract is treated with 1 ml. of 0·1 *M* disodium phosphate solution, and the ether is removed by distillation. The residue is transferred to a measuring cylinder, adjusted to *p*H 7 and made up to a known volume (1 to 3 ml.). The succinate in the solution is estimated manometrically with succinic dehydrogenase (Krebs, *Biochem. J.*, 1937, **31**, 2095). The concentration of *l*(-) malate is calculated from the equilibrium constant. At *p*H 7·4 the ratio *l*(-)malate/fumarate was found to be 2·65 at 50° C., 3·17 at 40° C., 3·54 at 30° C., and 4·57 at 20° C. Malate, tartrate, oxaloacetate, glutamate, citrate and aconitate do not yield succinate under these conditions, but maleäte (not present in biological material) behaves like fumarate. F. A. R.

Estimation of Nicotinic Acid in Biological Materials by means of Photoelectric Colorimetry. D. Melnick and H. Field. (*J. Biol. Chem.*, 1940, **134**, 1–16.)—Several workers have used the colour produced by treatment with cyanogen bromide and an aromatic amine for estimating nicotinic acid in foodstuffs (*cf.* ANALYST, 1939, **64**, 441, 755; 1940, **65**, 182, 183), and this method has been adopted in the present instance. The preliminary treatment has been modified, however, direct acid hydrolysis of the test substance being used, followed by preferential charcoal adsorption for the decolorisation of the hydrolysate. The test material, containing 10 to 400γ of nicotinic acid, is treated in a test-tube, graduated at the 10- and 15-ml. marks, with 5 ml. of conc. hydrochloric acid and water to make the volume up to 15 ml. The test-tube is immersed in boiling water for 30 to 40 minutes, then cooled, and the volume is adjusted to the original 15 ml. Ten ml. of absolute ethyl alcohol are added, and the solution is transferred to a 150-ml. conical flask. Exactly 200 mg. of adsorbent charcoal are added, and the mixture is shaken and filtered at room temperature. An aliquot part of the filtrate (8·33 ml.) is pipetted into the graduated test-tube, a drop of phenolphthalein solution is added, and the solution is neutralised in the cold to *p*H 7 with conc. sodium hydroxide solution, and its volume is adjusted to 10 ml. A 3-ml. aliquot portion is mixed with 7 ml. of an alcoholic buffer solution of *p*H 2·4 (1960 ml. of water, 10 ml. of 85 per cent. phosphoric acid, 30 ml. of 15 per cent. sodium hydroxide solution and 333 ml. of absolute ethyl alcohol), and the colour of the solution is measured in an Evelyn photoelectric colorimeter, filter 420 being used. A second 3-ml. aliquot portion is treated with 6 ml. of cyanogen bromide reagent (water saturated with bromine at 5 to 10° C. is just decolorised in the cold by means of 10 per cent. potassium cyanide solution) from a burette, followed immediately by 1 ml. of a 4 per cent. solution of aniline in absolute ethyl alcohol. After mixing, the colour of the solution is measured in the colorimeter with the same filter as before. The reading given by the extract alone is subtracted from that obtained after addition of the reagents, and the difference gives the photometric density produced by the nicotinic acid and nicotinamide present. The amount of nicotinic acid cannot, however, be calculated from a standard curve, owing to the presence of different amounts of inhibitors in the extract. A third 3-ml. aliquot portion of the extract is therefore treated with 10γ of nicotinic acid, and the procedure is repeated. The difference between this and the second reading gives the photometric density produced by 10γ of nicotinic acid, and from this value the amount of nicotinic acid present in the extract is calculated. In contrast to those described in previous reports, the reagents thus prepared were stable for at least 5 months. The recovery of known amounts of nicotinamide added to biological materials varied from 97 to 108 per cent. in 14 experiments and was usually between 99 and 102 per cent. F. A. R.

Coupled Oxidation of Carotene and Fat by Carotene Oxidase. J. B. Sumner and R. J. Sumner. (*J. Biol. Chem.*, 1940, **134**, 531–533.)—The name "carotene oxidase" was given to an enzyme which peroxidises unsaturated fats and bleaches carotene, bixin, xanthophyll and other carotenoids; it is present in legumes, especially in soya beans. In earlier experiments the carotene was dissolved in an unsaturated oil, and under these conditions bleaching was rapid. When, however, a solution of carotene in a mixture of acetone and alcohol was added to the enzyme extract, bleaching of the resulting carotene suspension was very slow; thus 0·79 mg. of carotene required 18 to 20 hours without oil, and only 32 seconds when mixed with 4·6 mg. of hempseed oil. The presence of additional fat reduced the rate of bleaching. Unsaturated fats were much more effective, weight for weight, than saturated fats. Fat previously peroxidised by carotene oxidase had no

increased action on the bleaching of carotene, nor did it cause immediate bleaching when carotene was dissolved in it; to exert its effect the fat must be mixed with the carotene before being suspended in water. Thus the bleaching action of carotene oxidase appears to be a coupled reaction. F. A. R.

Brassicasterol, the Characteristic Sterol of Rapeseed Oil. E. Fernholz and H. E. Stavely. (*J. Amer. Chem. Soc.*, 1940, **62**, 1875–1877.)—Brassicasterol, m.p. 148° C., was first isolated by Windaus and Welsch from the sterol fraction of rapeseed oil *via* the sparingly soluble acetate tetrabromide. This was difficult to distinguish from the acetate tetrabromide of stigmasterol, but on debromination it yielded brassicasterol acetate, m.p. 158° C. (stigmasterol acetate melts at 142° C.). The constitution of brassicasterol has now been elucidated. Ozonolysis of a carefully purified specimen yielded methyl isopropyl acetaldehyde, identical with the aldehyde formed under similar circumstances from ergosterol. On catalytic hydrogenation, ergosterol and brassicasterol give the same saturated sterol, ergostanol. Thus brassicasterol is 7 : 8-dihydroergosterol with the formula:



F. A. R.

Ascorbic Acid Oxidase from Summer Crook-neck Squash (*C. pepo condensa*). P. L. Lovett-Janison and J. M. Nelson. (*J. Amer. Chem. Soc.*, 1940, **62**, 1409–1412.)—The existence of the so-called ascorbic acid oxidase has been doubted by some workers, who attribute the oxidation of ascorbic acid to the catalytic effect of traces of copper or a copper-protein complex. Evidence is now put forward suggesting that ascorbic acid oxidase is a true enzyme, consisting of a copper-protein, similar in type to tyrosinase. After several possible sources had been examined, summer crook-neck squash was chosen as the best source of the active substance. The juice, obtained by expression of the minced rinds, was freed from inert material by treatment with barium acetate solution, and the filtrate, freed from excess of barium, was treated with ammonium sulphate until 0·6 saturated. The precipitate, which was active, was treated with disodium hydrogen phosphate solution, and the filtrate was made 0·9 saturated with magnesium sulphate. This treatment precipitated about two-thirds of the enzyme, and the remainder was precipitated by fully saturating the filtrate with magnesium sulphate. It was not possible to effect further concentration of the first precipitate, but the second precipitate was treated as follows:—It was dialysed and adsorbed on alumina, from which it was eluted with secondary sodium phosphate solution. The eluate was dialysed and treated with 0·1 saturated lead acetate solution and acetone cooled in solid carbon dioxide. The precipitate formed was removed in the centrifuge and the supernatant liquid was re-treated, the procedure being repeated three times in all. The combined precipitates were dissolved in secondary sodium phosphate solution and the solution was subjected to further adsorption, elution, dialysis and fractionation until no further concentration resulted. The copper content of the enzyme was estimated by the method of Warburg (*Biochem. Z.*, 1927, **187**, 255) at various stages in the purification. A mixture of 2 ml. of 0·2*M* sodium pyrophosphate solution (*p*H 7·75), 0·1 ml. of 0·2*N* hydrochloric acid and 0·2 ml. of a solution containing 6 mg. of cysteine hydrochloride was introduced into each of the side-arms of six Warburg respirometers. To the first was added 0·1 ml. of a solution containing 0·1γ of copper, to the second 0·2 ml. of a similar solution, and to the fourth, fifth and sixth, 0·1, 0·2 and 0·3 ml. respectively of the enzyme solution. The third was a blank with reagents but no enzyme. Each mixture was then diluted to 2·6 ml. with water. When the temperature of the bath was constant at 25° C., the contents of each side-arm were transferred to the flasks of the apparatus containing a buffer solution of *p*H 7·6. Manometer readings, taken at 5-minute intervals, were converted into c.mm. of oxygen. By subtracting the value for flask 4 from those for flasks 1 and 2, the uptakes due to 0·1 and 0·2γ of copper were obtained; and by subtracting the value for flask 4 from those for flasks 5 and 6, the uptakes due to 0·1 and 0·2 ml. of enzyme were obtained. The uptake due to 0·1 ml. of enzyme solution divided by the uptake due to 0·1 ml. of copper solution gave the concentration of copper in the enzyme solution. It was found that the percentage of copper in the preparation increased as the enzyme activity increased until it reached a value of 0·15 per cent. This value was not exceeded in spite of further attempts at purification. The correspondence between copper content and enzyme activity is strong evidence for the conclusion that the active group of ascorbic acid oxidase contains copper. The purified enzyme has 1100 times the activity of copper alone, 13,000 times the activity of a copper-albumin mixture and 4100 times the activity of a copper-gelatine mixture, suggesting that the purified material is, indeed, a true enzyme. In view of Theorell's observation that dihydroxymaleic acid oxidase contains manganese as activator, the presence of manganese was sought in the ascorbic acid oxidase, but the amount was found to decrease as the enzyme activity increased. Similarly, the final product was almost completely free from peroxidase activity. The enzyme had little or no action toward *p*-cresol, catechol or hydroquinone. F. A. R.

Effect of Zinc on Alkaline Phosphatases. E. Hove, C. A. Elvehjem and E. B. Hart. (*J. Biol. Chem.*, 1940, **134**, 425–442.)—Bone

phosphatase preparations were made by grinding cleaned fresh bone (rat femur) and allowing the suspension to autolyse for 24 hours; the filtered solution contained about 4 mg. of dry matter per ml. Preparations of intestinal phosphatase were similarly prepared from minced rat intestine; after dialysis the solution contained 2 mg. of dry matter per ml. The activity of the phosphatase preparations was measured with sodium β-glycerophosphate as substrate, but disodium phenyl phosphate, sodium hexose diphosphate and sodium pyrophosphate were also used. The phosphate esters were made up as a $4{\cdot}8mM$ solution (1 ml. = 0·15 mg. of phosphorus) in a $50mM$ veronal buffer solution, the final pH being adjusted to 9·2. Ten ml. of the substrate solution and 0·5 ml. of enzyme solution were made up to a total volume of 11·5 ml. and incubated at 37° C. The reaction was stopped by adding 2 ml. of 10 per cent. trichloroacetic acid solution, and the inorganic phosphorus produced in the reaction was estimated by the method of Fiske and Subbarow (ANALYST, 1926, **51**, 205). It was found that crude intestinal phosphatase activity was increased 40 to 100 per cent. by addition of zinc ions, whereas crude kidney and bone phosphatase activities were progressively inhibited by concentrations of zinc of 4×10^{-3} to $70 \times 10^{-3} mM$; this effect was independent of the nature of the substrate. The effect of zinc and magnesium together was greater than that produced by either alone. Dialysis of the crude intestinal phosphatase resulted in a preparation that was not activated by zinc ions, but which, like the other two phosphatase preparations, showed progressive inhibition. The dialysate, when added to the dialysed intestinal enzyme, restored the original antivatability with zinc, but the dialysate alone had no effect on the substrate. The zinc coactivator present in the dialysate was a product of mucosal tissue autolysis, and its effect was simulated by a casein hydrolysate. Subsequently it was shown that all α-amino acids behave as zinc co-activators, though β-amino-, keto- and hydroxy-acids and aliphatic acids or organic amines have little or no effect. The optimal concentration of the amino-acids was studied in detail for glycine; it was found that the optimal zinc and optimal glycine concentrations were interdependent, optimal stimulation being obtained with 1 mole of zinc for every 75 to 100 moles of glycine. At high glycine concentrations the zinc optimum was broad and flat, whereas at lower glycine concentrations it was sharp. The phosphatases of the bone and intestines of zinc-deficient rats were compared with similar preparations from normal animals. Whereas the activities of the bone phosphatases differed little, the intestinal phosphatase activity of zinc-deficient rats was considerably lower than that of normal controls. These results probably explain why zinc is essential to the life and growth of rats; the lowered blood non-protein nitrogen in zinc deficiency would also be explained. Moreover, if it is true that amino-acid absorption involves phosphorylation, the slower amino-acid absorption observed with zinc-deficient rats would be explained by the lowered intestinal phosphatase activity of these rats. F. A. R.

Identification of the Rice Factor. H. J. Almquist, E. L. R. Stokstad, E. Mecchi and P. D. V. Manning. (*J. Biol. Chem.*, 1940, **134**, 213–216.)—Chicks reared on a diet consisting of casein, water-washed sardine-meal, yeast, wheat-germ oil or soya bean oil, minerals, vitamins A and D, glucose or starch and sucrose grew more rapidly when the pure carbohydrates were replaced by polished rice, or when cartilage was added to the basal diet. It was subsequently found that gelatin or synthetic glycine in combination with chondroitin resulted in a rate of growth equal to that produced by the cartilage supplement. Glycine or chondroitin, given separately, did not effect the same growth increase as the two substances administered together. Thus the "rice factor" is identical with, or at least is replaceable by, a mixture of glycine and chondroitin. F. A. R.

Oxidation of Vitamin E. C. Golumbic and H. A. Mattill. (*J. Biol. Chem.*, 1940, **134**, 535–541.)—It has been suggested that the irreversible oxidation of a phenolic substance may occur in two steps, the initial step being reversible and consisting in the production of a phenoxyl radicle; this forms an unstable oxidation-reduction system with the phenol. When such a reversible step governs the rate of the irreversible oxidation of a phenol, it is possible to measure an apparent oxidation potential by potentiometric indicators. Such an apparent oxidation potential (the potential at which 20 to 30 per cent. was oxidised in 30 minutes) of α-tocopherol was measured by two different methods and was found to lie between the normal oxidation potentials of mono- and dimethylhydroquinones. α-Tocopheryl quinone, the oxidation product of α-tocopherol, was found to be biologically inactive when pure. Previous statements to the contrary are explained by the presence of unchanged α-tocopherol due to incomplete oxidation; with gold chloride, but not with ferric chloride, oxidation is complete at room temperature. Pure α-tocopheryl quinone is also without antioxygenic action on lard. Various substituted hydroquinones with oxidation potentials of the same order as α-tocopherol were found to be biologically inactive, alone or with phytol. These observations indicate that the reaction: α-tocopherol $\rightarrow$ α-tocopheryl quinone is irreversible in the organism, but it may take place in two steps, of which the first is reversible. This may be of biological importance. F. A. R.

Vitamin-free Diets for Animal Experiments. A. L. Bacharach. (*Nature*, 1940, **146**, 28–29.)—The difficulty of obtaining supplies of rice starch for use as the sole or predominant source of carbohydrate for basal

diets required in work on vitamin A, vitamin B_1 or vitamin E may be overcome by the use of wheat starch. Experiments illustrating this conclusion are described. Wheat starch cannot, however, satisfactorily replace rice starch for diets used in work on the rat "filtrate factor," discrimination between negative controls and animals receiving filtrate factor concentrate made from fresh liver being seldom satisfactory. It is believed that the wheat starch used had adsorbed or otherwise retained appreciable quantities of filtrate factor from the wheat berry. No corresponding information is available concerning the effect of wheat starch on diets used for work on vitamin B_6 (adermin, "eluate factor"). J. G.

Bacteriological

Molecular Constitutions of Catenarin and Erythroglaucin. W. K. Anslow and H. Raistrick. (*Biochem. J.*, 1940, **34**, 1124–1133.)—Catenarin, $C_{15}H_{10}O_6$, which constitutes 15 per cent. of the dry weight of the mycelium of *Helminthosporium catenarium* Drechsler, is 1 : 4 : 5 : 7 - tetra - hydroxy - 2 - methylanthraquinone. Erythroglaucin, $C_{16}H_{12}O_6$, which is a metabolic product of species in the *Aspergillus glaucus* series, is the 7-methyl ether of catenarin, *i.e.* 1 : 4 : 5-trihydroxy-7-methoxy-2-methylanthraquinone. F. A. R.

Agricultural

Plant Insecticide Materials from Empire Sources. (*Bull. Imp. Inst.*, 1940, **38**, 150.)—Derris: Wide variations in the composition of *D. elliptica* and *D. malaccensis* roots from different countries have been found as follows:

No. of samples	Country of origin	Moisture	Total extract (ethereal)	Rotenone calculated from carbon tetrachloride samples	Rotenone purified by alcohol	
					Range	Mean
8	Tanganyika	4·5 to 10·2	3·3 to 22·6	1·0 to 11·0	0·95 to 9·3	6·3
3	Sarawak ..	10·0 ,, 10·6	10·3 ,, 13·1	1·7 ,, 3·6	1·3 ,, 3·3	2·3
6	Seychelles ..	5·9 ,, 7·9	8·1 ,, 18·4	1·2 ,, 7·0	1·0 ,, 6·5	3·4*
1	,, ..	6·1	14·9	3·1	2·6	
5	Trinidad ..	4·9 ,, 7·6	9·0 ,, 22·0	1·1 ,, 8·6	0·8 ,, 7·2	3·8
3	Dominica ..	6·0 ,, 8·3	3·8 ,, 13·3	0·8 ,, 4·6	0·6 ,, 3·6	2·3
2	Fiji ..	9·8 ,, 9·9	6·9 ,, 9·3 (Chloroform)	1·5 ,, 1·8	1·3 ,, 1·6	1·45
1	Tanganyika	7·2	18·0	—	7·6*	
1	Mauritius ..	7·1	12·6	—	4·85*	

* Purified by trituration with cold alcohol (other samples crystallised from hot).

Mundulea sericea (or *M. suberosa*).—Two South African Union samples from root and bark respectively contained no rotenone or only traces. Owing to the reported toxicity of this plant, which was formerly attributed to rotenone, biological tests were made at Rothamsted with *Aphis rumicus*. An alcoholic root extract was completely toxic at 1 per cent. concentration (in terms of root used). Other results were:—Concentrations (as percentages) 0·5, 0·25, 0·1: moribund and dead insects (per cent.), 50, 20, 10. Stems and bark of the same plant gave negative biological tests, but stems from an Indian sample were toxic, and *M. suberosa* leaves from India were completely toxic at 1 per cent. One sample of Tanganyika bark, rotenone-free, contained saponins and alkaloids; a second contained 0·7 per cent. of rotenone (0·8 per cent. on moisture-free material) and ethereal extract, 4·6 per cent.

Tephrosia vogelii (Uganda): Leaves from a Toro estate (1*a*), from white-flowered Kampala Plantation plants (1*b*) and from purple-flowered Kampala Plantation plants (1*c*) gave alcoholic extracts which, on dilution with aqueous solutions of saponin (0·5 per cent.) to 5 per cent. alcoholic solutions, were toxic to *Aphis rumicus* as follows:—Concentration of leaf, g./100 ml.: 1·0, 0·5, 0·25, 0·1, 0·05; percentage of insects paralysed: (1*a*) 100, 100, 25, 5, 5; (1*b*) 100, 100, 25, 10, 0; (1*c*) 100, 100, 85, 5, 5 (control spray toxicity 3 per cent.). *Tephrosia toxicaria* (Natal) roots contained 1 per cent. of ethereal extract and only a trace of rotenone. Insecticidal properties are due to tephrosin.

Pyrethrum: Samples of flowers harvested at different stages, containing various amounts of stalk, and obtained from different countries, were analysed. Two of the St. Helena samples —12*a*, dried flowers; 12*b*, dried stalks—were grown experimentally from seed supplied by Kew Gardens in 1934 and examined in 1937. Pyrethrin I (0·46 per cent.) and pyrethrin II (0·56 per cent.) of the flowers were satisfactory (moisture, 8·6 per cent.); the stalks contained only 0·05 per cent. of each. Other results (as percentages) were: *Tanganyika* (14 samples): moisture, 7·0 to 10·7; pyrethrin I, 0·38 to 0·64; pyrethrin II, 0·33 to 0·80; total pyrethrins, 0·73 to 1·40. A sample of pyrethrum powder contained: moisture, 10·7; pyrethrin I, 0·30; pyrethrin II, 0·32; total pyrethrins, 0·62 per cent. *St. Helena* (1 sample): moisture, 10·0; pyrethrin I, 0·45; pyrethrin II, 0·59; total pyrethrins, 1·04 per cent. *Ceylon* (1 sample): moisture, 7·3; pyrethrin I, 0·47; pyrethrin II, 0·57; total pyrethrins, 1·04. There is an optimum time for harvesting.

Chrysanthemum frutescens (Tanganyika, 1

sample): moisture, 9·1; pyrethrin I, 0·05; pyrethrin II, 0·07; total pyrethrins, 0·12 per cent. Biological tests on this were negative. In appearance the plant resembled normal pyrethrum. E. B. D.

Toxicity to Sheep of Lead Arsenate and Lead Arsenate Spray Residues. J. L. St. John, E. C. McCulloch, J. Sotola and E. N. Todhunter. (*J. Agr. Res.*, 1940, **60**, 317–329.)—An investigation of outbreaks of illness or death of cattle and sheep fed in autumn on pasture in sprayed orchards showed that they were due to chemical poisoning; approximately 15 per cent. of the poisoned animals recovered. Orchard grasses from an area where approximately 1000 sheep died contained 0·44 per cent. of arsenic and 1·44 per cent. of lead. Experiments with healthy lambs (average wt., 80 lbs., or 36·29 kg.) showed that the total lethal dose of arsenic, as lead arsenate, was 1·5 g. (about 41·0 mg. per kg.) when given daily in small amounts (0·25 to 2 g.) in gelatin capsules. Lead was determined by the 1935 tentative methods of the Association of Official Agricultural Chemists, the Gerritz preliminary treatment being applied (*cf. Ind. Eng. Chem., Anal. Ed.*, 1935, **7**, 167) and colorimetric comparisons being made. Arsenic was determined by the bromate or the Gutzeit method. The lead and arsenic recovered from the total urine and faeces, from the stomach contents, and from a number of organs amounted to less than the quantity consumed. Maximum recovery was from the stomach contents, urine and faeces, the proportion of lead eliminated being larger than that of arsenic, elimination of arsenic per day being greater in urine than in faeces with larger consumption and *vice versa* with smaller amounts. Lead, which was eliminated mainly in the faeces, was always proportionally higher than the arsenic in these. The experimental animals were fed with a ration of rolled oats, rolled barley and alfalfa hay, in proportions 3 : 4 : 14 (salt *ad lib.*). Addition of apples to the ration apparently did not alter toxicity of lead and arsenic. Lead arsenate on sprayed foliage became less toxic after several weeks than the experimental capsules, and investigation of possible change in form of the lead and arsenic is required. E. B. D.

Gas Analysis

Elimination of Traces of Carbon Monoxide from Hydrogen. K. Kawakita and B. Ichiyanagi. (*J. Soc. Chem. Ind. Japan*, 1940, **43**, 121B.)—The gas is passed at ordinary pressure over activated reduced iron at 350° to 400° C., the carbon monoxide being decomposed into carbon and carbon dioxide, which latter is chemically absorbed. For the detection of traces of carbon monoxide the hydrogen was passed through blood solutions, the absorption spectrum of which was examined before and after treatment. The quantitative determination of the carbon monoxide was carried out by oxidation with iodine pentoxide (temperature not given), which gave satisfactory results with carbon monoxide contents lower than 0·04 per cent. With a catalyst obtained by reduction of 8 g. of ferric oxide, the carbon monoxide was reduced to about 0·001 per cent. at 360° C. at a velocity of 4 litres per hour; it could no longer be detected with a catalyst obtained from 16 g. of ferric oxide and a gas velocity of 5 litres per hour at 360° C. W. R. S.

Organic

Quantitative Determination of Certain Polyalcohols in Presence of Each Other. N. Allen, H. Y. Charbonnier and R. M. Coleman. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 384–387.)—The reaction of periodic acid with polyalcohols (*cf.* Malaprade, ANALYST, 1928, **53**, 299) has been applied to the determination of, and to distinguish between, certain polyalcohols, notably glycerol and ethylene glycol. These two polyalcohols may also be determined in presence of one another by an acidimetric-iodimetric titration, or potentiometrically with the glass electrode, and a combination of the Malaprade reaction and an acid dichromate oxidation enables solutions containing three glycols to be analysed. A quantity of sample containing oxidisable matter corresponding with the equivalent of not more than 0·09 g. of glycerol is diluted to 50 ml. and allowed to stand for 40 to 80 minutes in a glass-stoppered flask at 20° C. with 50 ml. of a filtered 0·05 *N* solution of periodic acid (calculated as a monobasic acid, *i.e.* 11 g. per litre). When oxidation is complete, 100 ml. of water are added, followed by 20 ml. of 0·1 *N* sodium hydroxide solution, 2 drops (not more) of methyl red indicator, and then sufficient of the 0·1 *N* alkali to destroy the pink colour. To the titrated solution are added 150 ml. of water, 30 ml. of 20 per cent. potassium iodide solution and 25 ml. of 6 *N* sulphuric acid, and the mixture is titrated with 0·2 *N* sodium thiosulphate solution with starch as indicator; the end-point is sharp, but the final solution has a pink shade owing to the methyl red. Allowance is made for the blank obtained with 50 ml. of water, and since 1 ml. of *N* sodium hydroxide ≡ 0·09206 g. of glycerol (acidimetric titration), the glycerol content (*G* per cent.) may be calculated. Since, further, 1 ml. of *N* sodium thiosulphate solution ≡ 0·023015 g. of glycerol (iodimetric titration), this titration enables the joint percentage of glycerol and ethylene glycol (*T*) to be determined in terms of glycerol; then the ethylene glycol content is 1·348 (*T*–*G*) per cent. If a third glycol, which is not oxidised by periodic acid (*e.g.* diethylene glycol) is present, it is determined by oxidation with dichromate. Thus the sample (half the quantity previously taken) is diluted to 25 ml. and boiled under a reflux condenser for 20 minutes with 25 ml. of a 2·4 per cent. solution of potassium dichromate and 40 ml. of conc. sulphuric acid. The cooled solution is then diluted to 300 ml., 10 ml. of 20 per cent.

potassium iodide solution are added, and the iodine is titrated with 0·2 *N* sodium thiosulphate solution in the usual way, allowance being made for a blank. Then, since 1 ml. of *N* sodium thiosulphate solution ≡ 0·006576 g. of glycerol, the combined glycerol, ethylene glycol, and diethylene glycol contents may be calculated in terms of glycerol (*X* per cent.); then the diethylene glycol-content is 0·807 (*X*–*T*) per cent. For the potentiometric method 50 ml. of the 0·05 *N* periodic acid solution are diluted with 150 ml. of water and titrated with 0·1 *N* sodium hydroxide solution; the first equivalent-point (at about *p*H 5·5) is determined (*B* ml.), an equal volume of reagent is added, and the resulting *p*H (about 10·0) is determined. The sample (*w* g.) is then oxidised with the periodic acid as described above, and after dilution with 100 ml. of water the solution is titrated with the 0·1 *N* alkali to the first equivalent-point (*A* ml.). Titration is then continued to the *p*H value found for the second equivalent-point of the blank (*X* ml.). Then 0·9206 (*A*–*B*)/*w* = percentage of glycerol; and 0·6205 (3*B*–*X*–*A*)/*w* = percentage of ethylene glycol. Results tabulated for a number of mixtures of the above polyalcohols in known proportions record errors ranging from −4·68 to +12·94 per cent.; the error for the determination of the ethylene glycol is always greater than for glycerol, since the former is not determined directly. The acidimetric method for glycerol may be used in presence of a variety of organic compounds, so long as these do not react with periodic acid to form an acid. Unknown solutions of polyalcohols should first be oxidised with periodic acid, and the results of the acidimetric iodimetric titrations calculated in terms of glycerol. If these figures agree with that obtained from the dichromate oxidation and calculated in the same way, glycerol is the only polyalcohol present; if the values are different a mixture of polyalcohols is indicated. The fact that glycerol reacts with periodic acid to produce formic acid, whereas ethylene glycol does not, is the basis of the following qualitative test to distinguish between them when they are the only two constituents present. The solution (2 ml.), containing a drop of methyl red, is adjusted with acid or alkali until it is faintly acid to the indicator; 2 ml. of a solution of periodic acid are treated similarly. If a pink colour develops when the solutions are mixed, glycerol is present. J. G.

Determination of Unsaturation in Aliphatic Hydrocarbon Mixtures by Bromine Absorption. J. B. Lewis and R. B. Bradstreet. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 387–390.)—The Francis method (ANALYST, 1926, **51**, 534) has been modified as follows (see also Thomas, Block and Hoekstra, *Ind. Eng. Chem., Anal. Ed.*, 1938, **10**, 153):—From 0·7 to 1·0 g. of the sample is added from a Lunge weighing-pipette to a mixture of 20 ml. of 10 per cent. (by vol.) sulphuric acid saturated with potassium bromide, and 15 ml. of *n*-heptane. This mixture is at once titrated with 0·5 *N* potassium bromide-bromate solution, until a faint yellow colour is obtained, and 1 ml. of the bromide-bromate solution in excess is then added. The solution is shaken for 2 minutes, and the iodine liberated on addition of 5 ml. of a saturated solution of potassium iodide is titrated with 0·1 *N* sodium thiosulphate solution, with starch as indicator. Then the bromine number = (0·0799 × normality of $KBrO_3$ × net vol. of $KBrO_3$ × 100)/wt. of sample. This modified procedure eliminates the preliminary trial titration and the necessity for cooling the sample prior to titration found necessary by other workers (*loc. cit.*), thereby halving the time required for control tests. It is applicable to straight-chain olefines, di*iso*butylene and other branched-chain olefines, of low mol. wt., and is unaffected by the presence of pure aromatic hydrocarbons, although erratic results are obtained with highly-branched polymers and in presence of certain sulphur compounds; thus low concentrations of *iso*amyl mercaptan and higher concentrations of benzyl mercaptan, amyl mercaptan and *iso*amyl disulphide lower the bromine number of di*iso*butylene. The effects of these sulphur compounds can, however, be overcome by the use as a catalyst of 1 ml. of a 10 per cent. solution in water of mercuric chloride, zinc sulphate or uranium acetate, or of 0·1 g. of solid silver bromide. Data tabulated for a number of pure unsaturated aliphatic hydrocarbons, synthetic blends made from naphtha (A.S.T.M.), *n*-heptane, di*iso*butylene and heptene-1, and also for a number of saturated compounds, show satisfactory agreement with the theoretical values. (*Abstractor's Note.*—The solubility of mercuric chloride in cold water is less than 10 per cent.)

J. G.

Isolation of Arachidonic Acid. G. Y. Shinowara and J. B. Brown. (*J. Biol. Chem.*, 1940, **134**, 331–340.)—The phosphatides of the lipids of beef suprarenals were converted into the corresponding methyl esters by direct alcoholysis. The esters were dissolved in acetone, and the solution was cooled to −20° C., causing the saturated esters to crystallise out. These were filtered off, and the filtrate was cooled to −70° C.; the resulting crystals, containing most of the methyl oleate, were filtered off, and the filtrate, containing esters that were 50 to 60 per cent. methyl arachidonate was concentrated and cooled again to −70° C. The final filtrate contained 70 to 75 per cent. methyl arachidonate. By fractional distillation, the purity was raised to 90 to 95 per cent. An alternative method of purification was also used in which the impure material was brominated in ethereal solution at 0° C. and the resulting octabromides, m.p. 228·5 to 229·5°, were debrominated with zinc and boiling methyl alcohol. The two methods gave essentially the same substance, except that the second method yielded purer material with an appreciably lower polybromide value than the first method. The following con-

stants were found for methyl arachidonate prepared by the crystallisation-distillation and bromination-debromination methods respectively: n_D, 1·4800–1·4845, 1·4823; iodine value, 289·3–306·2, 312·8–319·0; polybromide value, 87·0–97·7, 86·3–86·6; thiocyanogen value, 159·0, 161·1. Arachidonic acid is known to be a straight chain eicosatetraenoic acid, $C_{20}H_{32}O_2$; the straight chain structure was confirmed by analytical data on its hydrogenation product. Ozonolysis experiments, designed to determine the position of the double bonds, indicated that two of these are probably in the 6 and 18 positions; the evidence is against the presence of double bonds in the 9 and 12 positions as in linolic and linolenic acid. There is some evidence from the diene number that the methyl arachidonate prepared by the debromination method contains 5 per cent. of conjugated double bonds, supporting the belief that this ester is a mixture of at least two compounds, one of which contains a conjugated double bond system. F. A. R.

Analyses of Certain South African Woods, with Special Reference to their use as Producer-Gas Generator Fuels. P. E. Hall. (*J. Chem. Met. Mining Soc. S. Africa*, 1940, **40**, 350–352.)—The woods tested were (1) *Eucalyptus cladocalyx*, *E. saligna* and *E. paniculata*; (2) *Acacia karroo*, *A. saligna*, *A. cyclopis* and *A. mollissima*; (3) *Pinus insignis*, *P. pinaster*, *P. patula*, *P. longifolia* and *P. caribaea*. The most important properties in the evaluation of woods for producer-gas operation were found to be the following:—*Sp.gr.*—Segments cut from "rounds" of wood, 0·25 inch thick, were weighed in air before and after waxing, and then in water, the sp.gr. being calculated on the assumption that the volumes of the waxed and unwaxed woods were equal. The values for the above 3 groups of woods were 0·57 to 1·03, 0·68 to 0·86 and 0·38 to 0·65, respectively. A high sp.gr. is an advantage, because a vehicle must provide carrying capacity for its own fuel. *Ash content.*—Whilst none of the woods had a value exceeding 1 per cent., the light powdery nature of the ash is such that it will contaminate the gas made and so necessitate a relatively large area of filtration, a matter of importance with down-draught producers. *Charcoal yield.*—Values obtained (Gray–King low temperature assay) were 20·7 to 25·8, 20·9 to 25·3, and 22·6 to 28·7 per cent., respectively. Since most of the fuel gas is derived from the charcoal, a high value is an advantage. *Tar yields* (Gray–King) were 10·6 to 28·0, 13·5 to 18·7, and 15·9 to 19·8 per cent., respectively. Unless the producer is of the down-draught type (in which the tar is cracked in the fuel-bed) a high tar-content is a disadvantage, since it tends to gum up the fuel gas-line and to interfere with the filter, so that frequent stoppages for cleaning are required. The calorific values were 7,410 to 7,680, 7,360 to 7,740 and 7,530 to 8,470 B.Th.U. per lb., respectively. On the whole, there are no marked differences in suitability between the woods, but the above considerations suggest that the *Eucalyptus* group should be satisfactory fuels by reason of their high sp.gr. and charcoal yield, low ash content, and reasonable tar-yield; *E. saligna* has a rather low sp.gr. and *E. paniculata* a high tar-yield and a low charcoal yield. Woods of the *Acacia* group should prove fairly satisfactory, since the charcoal, tar-yield and sp.gr. are themselves fairly satisfactory. The low sp.gr. of the *Pinus* woods is a disadvantage (especially with portable generators); this is offset to some extent by their high charcoal yields as compared with the other woods. J. G.

Species Identification of Wood and Wood Fibres. (*Tech. Assoc. Pulp and Paper Ind. Amer., Standards*, Suggested Method, T.8 sm-37, Dec. 1937, pp. 1–12.)—Paper samples are torn up, and a small piece is boiled with 1 per cent. sodium hydroxide solution for a few minutes to remove the sizing. The alkali is washed out, the residue is shaken well with water, a drop of the resulting suspension is placed on a microscope slide, and a cover-slip is applied. The fibres may be stained with a 1 per cent. solution of methylene blue, a drop of which is placed by the side of the cover-slip and drawn under it by means of absorbent paper. After 3 minutes the excess of dye is removed by irrigation with water in the same way. Wood samples are broken into pieces about 2 cm. long and 3 mm. wide, which are boiled well in water until all air is expelled. The water is then replaced by conc. nitric acid, to which is added powdered potassium chlorate, and boiling is resumed until bubbles are evolved. When the fragments are bleached the chips are washed and disintegrated into the fibrous state by vigorous shaking. Reference standards should be prepared in this way from authentic specimens of wood. An illustrated list of the diagnostic features of 30 American pulpwoods is provided, and this, together with 3 keys, are used to identify the wood (*cf.* Maby, ANALYST, 1932, **57**, 2). Diagnostic features not referred to by Maby include the following:—Tyloses, which are white, colourless or sometimes dark-coloured cell-like intrusions, which partly or completely fill the vessels of many hardwoods. They usually have thin walls, which are rarely pitted, although thick profusely-pitted walls sometimes occur. Ray-flecks are the characteristic broad faces of the wood ray as seen on a radial section; they may not, however, always be conspicuous, by reason of their lack of height, colour contrast or lustre. Two types of ray cells are distinguished, *viz.* those with bordered pits (ray tracheid cells) and those with none (ray parenchyma cells); unlike the tracheids, which are dead cells, the latter remain alive so long as they are part of the sapwood. The ray cells are to a great extent removed from chemical pulps by screening, but they are useful for the identification of mechanical pulps. The spiral thickening of the cell wall also is characteristic of certain woods, and it may occur at the tips of the vessel segments, where vessels come into

contact, or throughout each vessel segment. The following principal diagnostic features are described for each tree listed in the key, and serve for its identification:—*Gross features.*—Colour, hardness, texture and density of sapwood and heartwood; odour; width and uniformity of distribution of rings; nature of transition from spring-wood to summer-wood; texture and size of rays; number, appearance and size of longitudinal and transverse resin canals and ray flecks. *Minute anatomy.*—Number and average diameter of tracheids; position, number, grouping and nature of bordered pits; appearance of ray tracheids, and nature of their walls; presence of thin-walled epithelial cells associated with resin canals; dimensions of longitudinal and transverse canals; and degree of homogeneity of rays. J. G.

Inorganic

Colorimetric Determination of Copper with Triethanolamine. J. H. Yoe and C. J. Barton. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 456–459.)—The colorimetric determination of copper was made spectrophotometrically. With triethanolamine, the sensitiveness is substantially the same as with ammonia, though slightly greater at low copper concentrations. Triethanolamine has no odour and is not subject to losses by volatilisation. Ammoniacal copper solutions obey Beer's law, giving a linear relation between concentration and transmission at $625m\mu$ over the range 0 to 1000 p.p.m. of copper in $0{\cdot}3M$ and in $2{\cdot}5M$ ammonia; solutions in triethanolamine do not entirely conform to Beer's law at various concentrations from 1 to 5 per cent., as the curves show a slightly greater slope with low copper concentrations (up to about 60 p.p.m.). A series of curves are given showing the results obtained with the two reagents. W. R. S.

Colorimetric Determination of Free Chlorine in Effluents. F. J. Hallinan. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 452–453.)—The method, which is applicable to coloured effluents, is based upon the formation of an insoluble adsorption compound of α-naphthoflavone and iodine which is filtered off and subsequently dissolved in alcoholic potassium iodide solution. The effluent (50 ml.) is treated with 1 g. of solid potassium iodide and a buffer (1 g. of potassium sodium tartrate for *p*H 6·7, ammonium acetate for *p*H 8·9, sodium diethylbarbiturate for *p*H 10·0), which are dissolved by stirring, and after 2 minutes with 0·5 ml. of 0·2 per cent. α-naphthoflavone solution in 95 per cent. alcohol. After 3 minutes' standing the liquid is vigorously stirred to promote flocculation and filtered by suction through hard paper in a Gooch crucible. The clear filtrate is tested for iodine with more of the reagent, and if a positive reaction is obtained a fresh test must be carried out on a smaller volume of effluent. The precipitate is washed with chlorine-free water and extracted with about 5 ml. of a 0·1 per cent. alcoholic solution of potassium iodide, and the yellow filtrate is matched against permanent colour standards corresponding with known concentrations of chlorine. The method is very delicate, 0·05 p.p.m. of chlorine giving a perceptible yellow tinge in the small volume of alcoholic filtrate. W. R. S.

Colorimetric Determination of Iron with Salicylaldoxime. D. E. Howe and M. G. Mellon. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 448–450.)—A weight of sample containing 0·01 to 1 mg. of iron is dissolved in 10 ml. of nitric acid (1 : 3), and the red fumes are boiled off. After cooling, 10 ml. of 3 per cent. hydrogen peroxide, a slight excess of ammonia, and dilute hydrochloric acid to faint acid reaction (litmus), are added. Any insoluble matter is filtered off and well washed. The filtrate is treated with 10 ml. of 0·1 per cent. salicylaldoxime solution and, after agitation, with 1 g. of ammonium acetate. The solution is diluted to 100 ml., and an aliquot part is transferred to a Nessler tube and compared with a scale of standards prepared in the same manner less than 24 hours before use. A Duboscq colorimeter may also be used, the solutions obeying Beer's law. The reagent is prepared from a weighed amount of salicylaldoxime dissolved in 5 ml. of alcohol and diluted with the required volume of water. The interfering ions are copper, nickel, cobalt, molybdenum, uranium, chromium, phosphate, fluoride, and borate. They should be absent or removed from the solution. W. R. S.

Phosphate Separation in Qualitative Analysis. J. N. Friend, R. H. Vallance and H. J. G. Challis. (*Nature*, 1940, **146**, 63.)—Since zirconium phosphate is insoluble in acid solution, a soluble zirconium salt can be used in place of ferric chloride for the removal of phosphates after the separation of the Group II metals as sulphides in qualitative analysis. Hydrogen sulphide is removed from the Group II filtrate by boiling, in the usual way, and a few drops of nitric acid are added, followed by an acid solution of zirconium nitrate, a drop at a time, until precipitation is complete. The solution is warmed, and after separation of the precipitated zirconium phosphate the filtrate is tested with a drop of reagent to ensure that no phosphate remains. Ammonium chloride and ammonium hydroxide are then added, and the solution is boiled well (important) and filtered. The filtrate may contain the members of Groups IV, V and VI, and is dealt with in the usual way, whilst the precipitate contains any iron, chromium or aluminium and the excess of zirconium salt. Aluminium is separated by extraction with boiling sodium hydroxide solution, the extract is neutralised with acid, and the aluminium is precipitated with ammonia. The residue (which is brown if iron is present) is oxidised by addition of water and sodium peroxide, when any chromium passes into solution as chromate. A solution of any insoluble residue in acid is tested for the presence of iron, and a confirmatory

test for chromates should be applied to the filtrate because zirconium salts sometimes contain titanium, and this will have formed the coloured pertitanate, which will mask or simulate the colour of the chromate. The advantage of the method is that it is more straightforward than that usually adopted, whilst the disadvantage is the cost of pure zirconium nitrate. Zirconia, however, is relatively cheap, and the presence of hafnium in it is immaterial as it behaves like zirconium under the above conditions. The reagent is best prepared by boiling a solution of the nitrate with an excess of sodium hydroxide (to extract any aluminium), washing the precipitate repeatedly by decantation, and allowing it to settle overnight; it is then suspended in water and dissolved by the addition of half the volume of warm 50 per cent. nitric acid.

J. G.

Determination of Sulphides in Depilatories. E. M. Hoshall. (*J. Assoc. Off. Agr. Chem.*, 1940, **23**, 437–444.)—Liquid depilatories contain from 2 to 10 per cent. of alkali sulphides calculated as the anhydrous salts. Powder depilatories contain 20 to 30 per cent. of barium or strontium sulphides, and mixtures of zinc sulphide and lime have also been used. Paste preparations form the largest class, and contain 5 to 20 per cent. of barium or strontium sulphide and occasionally calcium hydrosulphide and saccharosulphide. Methods for the determination of the sulphide-content of these preparations were examined, and many were found too tedious and cumbersome for routine purposes, but the following modification of Mohr's method gave satisfactory results:—An amount of depilatory equivalent to less than 0·12 g. of hydrogen sulphide is added to 50 ml. of 0·1*N* arsenious oxide solution, and the mixture is acidified with 20 ml. of dilute sulphuric acid (1 + 1) and shaken vigorously in a closed flask. If the depilatory contains much carbonate the flask is closed by means of a stopper carrying a separating funnel with a stem reaching nearly to the surface of the liquid, and the acid, mixed with 10 ml. of the arsenious oxide solution, is introduced gradually from the separating funnel. Traces of hydrogen sulphide and the liberated carbon dioxide bubble up through the acid, and the carbon dioxide can be removed by gentle shaking. When the reaction has stopped the contents of the funnel are rinsed into the flask. When cold the mixture is diluted to 250 ml. and filtered, the first 20 ml. of the filtrate being rejected. A portion (100 ml.) of the filtrate is treated with starch solution, sufficient 0·1*N* iodine solution to produce a blue colour, and 1 to 2 g. of sodium bicarbonate in excess of the amount required for neutralisation and titrated with 0·1*N* iodine solution. One ml. of 0·1 *N* arsenious oxide solution is equivalent to 0·002556 g. of hydrogen sulphide. The method was found to be sufficiently accurate for routine purposes, and no substances interfering with the reaction were encountered. Discrepancies in the results, when they occurred, were found to be due to instability of the sulphides (particularly sodium sulphide) in presence of other constituents of the depilatory. With preparations that carbonise or dissolve only with difficulty, 15 ml. of hydrochloric acid may be used instead of dilute sulphuric acid.

A. O. J.

Microchemical

Permanganate Acid Ashing Micromethod for Iodine Determinations. I. Values in Blood of Normal Subjects. D. S. Riggs and E. B. Man. (*J. Biol. Chem.*, 1940, **134**, 193–211.)—The various methods hitherto used for the estimation of iodine in blood are subject to certain errors inherent in each method, and an entirely new method of oxidising the organic matter has been introduced, and is claimed to avoid these errors. Ten ml. of the oxalated blood are oxidised with potassium permanganate and dilute sulphuric acid, ceric sulphate and copper being used as catalysts. The iodine is removed by distillation with oxalic acid, and the iodine in the distillate is oxidised to iodate by potassium permanganate and then determined by titration with sodium thiosulphate. *Apparatus.*—The distillation apparatus consists of (1) a 900-ml. Kjeldahl flask fitted with a ground-glass joint; (2) a distilling arm, made of wide glass tubing bent twice at right-angles, with a ground-glass joint at each end, one of which fits the Kjeldahl flask. Sealed through one bend of the distilling-arm is a dropping funnel, the stem of which is made of capillary tubing; the capillary extends to within 8 mm. of the bottom of the flask; (3) a spiral condenser, 17 cm. long, with a ground-glass joint into which the other end of the distilling-arm fits; (4) a 250-ml. wide-mouthed Erlenmeyer flask with a short horizontal side-arm sealed on at the base; this tube has a capacity of 3 ml. and is calibrated at 2 ml.; (5) a thermometer (120 to 200° C.) attached to the capillary tube of the distilling arm by strong rubber bands. *Digestion.*—Into the digestion flask are introduced 14 g. of potassium permanganate (recrystallised from water), a small piece of copper, 10 mg. of ceric sulphate (washed twice with 5 times its weight of boiling 95 per cent. alcohol) and 15 to 25 ml. of redistilled water. While the mixture is shaken, 10 ml. of oxalated blood are added, and then 10 ml. of 18 *N* sulphuric acid, and the flask is again shaken. A violent reaction takes place, and after about 5 minutes, when it has subsided, a further 200 ml. of 18 *N* sulphuric acid are added. The flask is heated cautiously over a wire gauze until the contents are boiling steadily, digestion being continued until a temperature of 195° C. is reached; the solution is then allowed to cool to 100° C., 150 ml. of 1 per cent. potassium permanganate solution are added, the flask is heated to 145° C. for about 15 minutes and again allowed to cool to 100° C., and the thermometer is washed with 25 ml. of water and removed. *Distillation.*—The digestion

flask is connected to the distillation apparatus, and the receiving flask, containing 1 ml. of *M* potassium carbonate solution and 1 ml. of 0·1 *M* sodium bisulphite solution, is tilted so that the tip of the condenser dips below the surface of the liquid. The digestion flask is heated to 138 to 140° C. and oxalic acid solution (saturated at 30° C.) is added slowly through the dropping-funnel. Two or 3 ml. are added beyond the point at which the solution is decolorised, followed by water so long as the distillation is continued. About 170 ml. of distillate are collected in 30 minutes. The distillate in the receiving flask is evaporated cautiously on an electric hot-plate to a volume of 6 or 7 ml. *Oxidation.*—The flask is placed in a water-bath maintained at 70 to 80° C., and 8 drops of 0·2 *M* potassium permanganate solution are added. If these are decolorised, more permanganate is added until a purple colour persists. After 4 minutes 10 drops of 8 *N* sulphuric acid are added, and after a further 4 minutes the flask is removed from the water-bath and 0·75 *N* sodium nitrite solution is added dropwise, with constant shaking, until the solution is quite clear. One drop of nitrite solution is added in excess, followed immediately by 2 drops of 5 *M* urea solution. The flask is replaced in the water-bath until the volume is about 2 ml. (measured with the aid of the side arm). *Titration.*—The flask is cooled in ice-water and a drop of 1 per cent. stabilised starch solution and then 0·06 ml. of freshly prepared 0·2 per cent. potassium iodide solution are added. The solution is titrated with 0·001 *N* sodium thiosulphate solution delivered from a micro-burette, the tip of which dips just under the surface of the liquid being titrated. *Blank.*—A blank is prepared by the method used in the estimation itself, with the following modifications: 15·5 g. of permanganate are used for the oxidation, anticoagulant equal to the amount present in the blood is introduced into the digestion flask, 85 ml. of water are used to wash the thermometer, and, before oxidation of the 7 ml. of distillate with permanganate, a small, accurately measured quantity (0·5 ml. of 0·00005 *N*) of biiodate solution is added. A control titration is carried out on the same amount of iodate added to a mixture of 1 ml. of 0·1 *M* sodium bisulphite solution and 1 ml. of *M* potassium carbonate solution and carried through the final oxidation and titration processes. The value of the blank is the difference between the titres of the control iodate samples and of the distillate to which iodate was added. *Results.*—In 59 experiments inorganic and organic iodine was recovered, with an average error of 6·5 per cent.; the average recovery for all experiments was 95 per cent. F. A. R.

Micro-determination of Succinic Acid. G. J. Goepfert. (*Biochem. J.*, 1940, **34**, 1012–1014.)—The following method for the estimation of minute amounts of succinic acid in nutrient media containing nitrate, sulphate and phosphate ions was devised. The solution, containing volatile acids and 2 ml. of 0·1 *N* succinic acid solution, was acidified with conc. sulphuric acid until acid to Congo red, heated on the water-bath, and treated with 0·1 *N* potassium permanganate solution until a brown precipitate was formed. This was dissolved by addition of sodium sulphite, and the solution was evaporated to dryness. The residue was dissolved in 15 ml. of chloride-free water, 2 ml. of conc. sulphuric acid were added, and the solution was saturated with potassium sulphate and extracted with ether for 3 or 4 hours in a continuous extractor. To the extract were added 5 ml. of chloride-free water, and the ether was removed by distillation. The aqueous residue was boiled over a flame for 30 seconds and cooled under the tap, and one drop of *m*-nitrophenol indicator (0·3 per cent. in water) was added, followed by 0·05 *N* sodium hydroxide solution until one drop caused the solution to turn yellow. It was decolorised with one drop of 0·1 *N* nitric acid, and a measured excess of 0·02 *M* silver nitrate solution was added immediately. The solution was made neutral by adding one drop of 0·05 *N* ammonia and allowed to stand in the dark for 2 hours. The precipitate was collected on a Gooch or sintered-glass crucible and washed successively with 3-, 3- and 2-ml. portions of 1 per cent. ammonium nitrate solution. The filtrate was treated with 2 drops of dichlorofluorescein indicator (0·1 per cent. in 70 per cent. ethyl alcohol) and about 8 drops of 1 per cent. soluble starch solution (chloride-free). The excess silver was titrated with 0·02 *M* potassium bromide solution until the pink colour disappeared. (One ml. of 0·02 *M* silver nitrate solution ≡ 1·18 mg. of succinic acid.) In 25 samples the recovery was 98 ± 2 per cent. F. A. R.

Micro-estimation of Uronic Acids. E. M. Kapp. (*J. Biol. Chem.*, 1940, **134**, 143–150.)—When glucuronic acid is boiled in hydrochloric acid in presence of naphthoresorcinol, a purple ether-soluble pigment is formed (Tollens' reaction). The usual conditions employed for this reaction are not optimal, however, and an improved method, suitable for quantitative purposes, has therefore been devised: A sample containing 10 to 40 γ of free glucuronic acid (or 7 to 30 γ of galacturonic acid) in 3·5 ml. of water is treated in a 10- or 15-ml. centrifuge tube with exactly 0·1 ml. of a 1 per cent. solution of naphthoresorcinol in 95 per cent. ethyl alcohol and then with 1·5 ml. of conc. hydrochloric acid. After mixing, the tube is immersed in a boiling-water bath for 4·5 hours with occasional shaking (2 hours is sufficient for galacturonic acid), cooled and centrifuged. Most of the supernatant liquid is withdrawn by means of a pipette and 10 volumes of water are added to the remainder. The resulting aqueous suspension is extracted several times with 1-ml. portions of ether, and the combined extracts, measuring not more than 6 ml., are dried with sodium sulphate and filtered into a 10-ml. flask. The solution is made up to the mark, and the colour is measured as soon as possible in a step-photometer with filter S 57 and

20 mm. cells, against an ether blank. The colour produced by the reagents alone is measured, and the value so found is subtracted from the value given by the sample. The amount of glucuronic or galacturonic acid is calculated from a standard curve obtained with pure specimens, or from the two equations: Mg. of glucuronic acid = (D — 0·08)/16·7, and Mg. of galacturonic acid = (D — 0·08)/24·5, where D is the observed photometric density.

The two substances are readily distinguished from one another by the rate of colour development, the maxima for galacturonic and glucuronic acid being reached in 2 and 4·5 hours respectively. A number of substances, including pentoses, interfere with the colour, and must be removed before carrying out the reaction. The agreement between the amount of glucuronic acid added to previously analysed specimens of urine and the amount found to be present was good, the largest discrepancy being 3 γ in 39 γ. F. A. R.

Physical Methods, Apparatus etc.

Estimation of *o*-Nitrophenol in *p*-Nitrophenol, and *o*-Aminophenol in *p*-Aminophenol by Fluorescence Analysis. W. Seaman, A. R. Norton and O. E. Sundberg. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 403–405.)—The reaction depends on the fact that *o*-aminophenol (*e.g.* produced by reduction of *o*-nitrophenol) reacts with carboxylic acids or their anhydrides to form benzoxazole and associated fluorescent by-products, whereas *m*- and *p*-aminophenols do not. A quantity of sample equivalent to 1·3 g. of nitrophenol is boiled under a reflux condenser with 125 ml. of water, 30 ml. of conc. hydrochloric acid and 5 g. of zinc dust for 15 minutes. The mixture is cooled and filtered, the residue is washed with three 10-ml. portions of water, and the total filtrate is treated with 15 ml. of ammonia, followed by sufficient 2 per cent. ammonia to bring the *p*H to about 5·1 (*i.e.* alkaline to Congo red paper and faintly alkaline to red litmus paper); this ensures that no zinc hydroxide is precipitated. Most, or all, of the *o*-aminophenol but little of the *p*-aminophenol may then be removed from the cold solution by extraction with three 25-ml. portions of ether; these are united, washed with two 5-ml. portions of water, and evaporated rapidly just to dryness, the formation of an adherent skin being avoided so far as possible. The residue is mixed with 5 g. of sublimed benzoic acid without delay (to avoid oxidation), and the mixture is heated in an oil-bath at 155° to 160° C. for 15 minutes after the contents have melted; carbonisation should be avoided. The cold residue is rubbed alternately in ammonia and in water until no more will dissolve, and the resulting blue solution is extracted with two 25-ml. portions of benzene, which are then mixed and extracted, first with 10-ml. portions of 1 per cent. sodium hydroxide solution until no more blue colour is removed, and then with water until the washings are neutral to litmus. The fluorescence of the benzene solution is matched in a test-tube (150 × 25 mm.) against a series of standards prepared from known mixtures of pure *p*-nitrophenol with 0 to 0·5 per cent. of *o*-nitrophenol. A fairly good comparison may be made with the aid of an ordinary desk-lamp, if the eyes are shielded against direct light and the tubes are placed vertically on a dark background and illuminated from the side and viewed from above. A more sensitive comparison may be made in filtered ultra-violet light in a dark room, the tubes being held in a rack at arm's length, slightly above eye-level over the lamp. The minimum detectable quantity of *o*-nitrophenol in *p*-nitrophenol is 0·05 per cent., and although no mixtures containing *m*-nitrophenol were used, it was shown that *m*-aminophenol gives no fluorescence. Any red colour obtained with commercial samples of *p*-nitrophenol is eliminated in this way, although the effect of the red colour obtained when more than 1·3 g. of sample is used, or when the method is used to estimate commercial *o*-aminophenol (the reduction process being omitted) cannot then be avoided. The following modified technique should then be used. A solution of 1 g. of sample in a mixture of 100 ml. of water and 3 ml. of conc. hydrochloric acid is filtered, and the above procedure followed from the stage of the adjustment of the *p*H value. The final benzene extract is shaken first with 50 per cent. ammonia (twice), and then with 10-ml. portions of 33 per cent. hydrochloric acid until no further colour is removed. If the solution is then filtered through a dry paper, it may be matched in the usual way without any appreciable loss of fluorescence; if desired, the standards may be treated similarly. A solution in benzene of phenyl benzoxazole prepared by the reaction of *o*-aminophenol with benzoyl chloride had a red-brown fluorescence, but successive recrystallisations from acetone and a petroleum hexane cut converted this into a green fluorescence, which ultimately disappeared once the correct m.p. had been attained and had not reappeared after 16 months. It is concluded that the fluorescence matched is due to a by-product of the benzoxazole condensation. J. G.

Quantitative Analysis of Plant Tissues for Lithium by the Ramage Flame Spectrographic Method. N. L. Kent. (*J. Soc. Chem. Ind.*, 1940, **59**, 148–153T.)—The weighed sample is dried at 100° C., and ashed, and about 20 mg. of the ash are weighed into a small specimen tube and dissolved in a volume of dilute nitric acid (1:10) such that 0·1 ml. of solution contains exactly 0·25, 0·5, 1 or 2 mg. of ash, according to the amount of lithium believed to be present. Then 0·1-ml. portions of the solution are dropped on to quarter-circles of filter paper (No. 00, 9 cm.), which are dried on a glass plate over a 60-watt lamp and rolled into tight spirals about 1·75 in. long. The spiral is then "fed" horizontally into the

hottest part of the flame of a (Hilger) Ramage oxy-coal gas burner (*cf.* Steward and Harrison, *Ann. Bot.*, 1939, NS3, 427) at the rate of about 2 in. per 10 seconds, and the resulting spectrum is photographed by means of a Hilger Medium Quartz Spectrograph, E3, panchromatic plates (Ilford process, 10 × 4 in.) being used, since the most sensitive lithium lines are in the red at 670·8 $m\mu$. A novel feature of the present apparatus is the inexpensive feeder. This consists of a horizontal piston, into which the paper "spiral" can be fixed in such a way that when a large hand flywheel is turned the piston moves forward, thereby enabling the tip of the spiral to be advanced rapidly through the relatively cool outer cone of flame, until it begins to burn brightly at the edge of the inner cone formed by the junction of the oxygen and coal gas. Manipulation of the flywheel enables the spiral to be maintained in this position, and a hinge serves to swing the piston in and out of position for loading and removal of the ash; 50 exposures can be made in 2 hours, 25 spectrograms being recorded at each end of the plate, which is developed in metol-quinol at 20° C. for 3·5 minutes and fixed in acid "hypo" for 15 minutes. The standard spectrograms used for comparison purposes are prepared in a similar way from solutions of lithium sulphate equivalent to 1 mg. to 0·05μ g. of lithium per 0·1 ml., the maximum sensitiveness of the method being obtained with quantities between 0·4 and 4·0μ g. It is therefore sometimes necessary to make a preliminary trial analysis in order to ensure that this condition will be satisfied in the final analysis. The maximum probable error, as estimated from the errors of the individual operations, is 9 per cent. The objects of the analyses were to ascertain how much lithium is taken up by plants from normal soils and from soils treated with lithium chloride or nitrate, and any resulting differences in the distribution, concentration and uptake of the lithium in the plant. With each species of plant examined (*viz.* healthy wheat and celery plants, tomato crown galls dissected from the stems of plants that had been innoculated with *B. tumefaciens* and treated with various amounts of lithium nitrate, and the dead leaves which fell from these plants), there was a high degree of correlation between the concentrations of lithium in the soil and in the plant. The greatest accumulations in wheat were found in the oldest leaves, and in celery in the margin of the largest leaves. The tolerance for lithium was in the decreasing order, wheat, celery and tomato. Plants appear to have little power to prevent the absorption of lithium, and it is suggested that the accumulation of lithium in the leaves depends more on their growth-rates than on transpiration; lithium performs no useful function, and merely accelerates leaf-fall. The following are the lithium-contents (in mg. per 100 g. of fresh material) obtained for plants grown on untreated soil:—Wheat leaves, 2·81, roots, 1·73; celery leaves, 1·5, petioles, 0·09, roots, 0·28; tomato petioles, 0·26. The corresponding figures for the lithiated plants having the maximum individual lithium-contents were, 33·8, 36·5, 29·8, 1·66, 4·08, and 1·49, respectively. J. G.

Standard Method for Testing the Fastness to Light of Coloured Textile Materials. L. A. Lantz. (*J. Tex. Inst.*, 1940, **31**, S13–14.)—The results obtained by the method proposed in the Report of the Society of Dyers and Colourists (ANALYST, 1934, **59**, 783) were compared with those obtained by the methods suggested by other organisations, with the object of agreeing on a common range of standards which might be adopted internationally. An improved set of Tentative Standards dyed on wool* are, therefore, now submitted for publication in accordance with the agreements made by the Textile Institute with the Society and with the British Standards Institution; these are:—(1) 0·8 per cent. Brilliant Wool Blue FFR extra (I.G.); (2) 1·0 per cent. Brilliant Wool Blue FFB extra (I.G.); (3) 1·2 per cent. Brilliant Indocyanine 6B (I.G.); (4) 1·1 per cent. Polar Blue G conc. (Gy.); (5) 0·8 per cent. Solway Blue RS (I.C.I.); (6) 2·5 per cent. Alizarine Light Blue 4GL (S.); (7) 2·5 per cent. Soledon Blue 4BC pdr. (I.C.I.); (8) 3·0 per cent. Indigosol Blue AGG (I.G.). The parallel red series formerly suggested has been abandoned as superfluous. Appropriate adjacent members of this series are exposed together with the material to be tested until the latter shows definite signs of fading; its fastness number is then that of the Standard which has faded to the same extent. The standards are little affected by variations in atmospheric humidity and temperature, and they allow reliable figures to be obtained under all conditions of daylight and in different climates. If an artificial source of light is used it should approximate to daylight as closely as possible; when it is an enclosed carbon arc, provision should be made for the control of the humidity and temperature of the atmosphere surrounding the samples, which should be disposed in a circle around the arc on a rotating device, so that the distribution of the light falling on them is equalised. When fastness measurements are based on times of exposure to a constant source of light (as distinct from comparisons with a series of standards) the suggested Standards may be used to control the exposures and to calibrate the lamp used. J. G.

Microscopical Investigation of True Woollen Cloths. Chemical and Microscopical Determination of Additions of Casein Wool. J. Straub and G. J. Van Amerongen. (*Chem. Weekblad*, 1940, **37**, 236–238.)—For the determination of the proportions of damaged wool in cloth used for military uniforms, a thread from the cloth is cut into a piece 0·5 to 1 mm. long and mounted in a drop of 50 per cent. glycerin or a mixture of glycerin and gelatin on a microscope slide

* Obtainable from the Society of Dyers and Colourists, in sets, 4½ × 3″; price, 5s. per set.

and covered with a cover-glass on which is etched a scale which enables the widths of the fibres to be estimated. A table shows the relationship between the thicknesses and the colours of undamaged and damaged wool as seen under the microscope. Most of the fibres of 0 to 15μ in thickness were white; those of 15 to 30μ were white and either blue-green or yellow-green, respectively; those over 30μ were black and blue-green or yellow-green, respectively. The number of fibres falling in these three thickness groups were, respectively, 7, 28 and 0 for the undamaged wool and 5, 25 and 7 for the damaged wool. Estimations of the proportions of undamaged fibres based on the frequency of distribution of the thicknesses and colours observed gave closely concordant results. The magnifications used for these counts were · 60 ×, but the details of the differences between damaged and undamaged wool were shown better at magnifications of 130 ×. Casein wool is recognisable by its comparatively thin uniform fibres, with their narrow central canal, and the absence of scalariform markings (magnification, 130×). Photomicrographs illustrate the differences between the three types of fibre. The average weights per cm. of length of true wool and casein wool from a sample of cloth were (after correction for the effects of mechanical treatment) 9·1 to 5·1γ, respectively, and these figures were used to calculate the weights of each type of wool present in the cloth from the microscopical counts obtained with 3 threads each from the warp and weft. In the chemical method described (*cf.* Herzog, *Technologie der Wolle*, 1938) 0·4 g. of the sample is dissolved in a 40-ml. porcelain crucible in 10 ml. of 20 per cent. hydrochloric acid, or in a mixture of sodium hydroxide solution and bromine water (*cf.* Trotman and Bell, *J. Soc. Chem. Ind.*, 1926, **45**, 10; ANALYST, 1934, **59**, 715). After 30 to 60 minutes 1 ml. of a solution containing 25 g. each of cupric nitrate and sodium chloride and 10 g. of sodium nitrate in 100 ml. of water (Benedict-Denis reagent) are added, and the mixture is evaporated to dryness on the water-bath. Evaporation is repeated after a further addition of 4 ml. of reagent, and the residue is ignited in a muffle at a glowing heat for 3 minutes with the crucible covered, and then for 10 minutes with it open. The sulphate-content of a solution of the residue in 10 ml. of 10 per cent. hydrochloric acid and 10 ml. of water is then determined by precipitation as barium sulphate, and the sulphur-content of the original sample may be calculated from this. The average sulphur-contents of the true wool and casein wool in the cloth under examination were found to be 2·81 and 0·43 per cent., respectively, and these data enabled the proportions of the two types of fibre in the cloth to be calculated from its sulphur-content. The ratios of casein wool to true wool in the cloth, as found by the chemical and microscopical methods, were 29 : 71 and 28 : 72 respectively, and as certified by the manufacturers 30 : 70. J. G.

Reviews

THORPE'S DICTIONARY OF APPLIED CHEMISTRY. Fourth Edition. By Sir J. F. THORPE, C.B.E., D.Sc., F.I.C., and M. A. WHITELEY, O.B.E., D.Sc., F.I.C. Vol. IV. Pp. 603. London: Longmans, Green & Co. Price 70s.

This volume was published on June 10th of the present year. On the same day, with tragic suddenness, Prof. Sir Jocelyn Thorpe peacefully passed away in his home in Sussex. Appreciations of Thorpe and tributes to his genius and geniality have appeared in many places; to these one must add that, since the mantle of Sir Edward Thorpe fell upon him, he has by this Dictionary made a memorial for himself which will cause his name to be remembered (with that of Sir Edward) with gratitude by the rising generation who could not know him so well as those who are older.

It may perhaps be impossible to make Thorpe's Dictionary quite the same without "Thorpe," but the framework exists and Dr. Whiteley has the co-operation of many well-known experts as contributors.

The present volume ranges from Digallic Acid to Feeding Stuffs; it differs a little from the preceding three in that it contains perhaps a smaller number of headings, but certainly a greater number of what may be called major articles. Even these are too numerous to mention in a brief review; some of them amount almost to small text-books on their subjects. For example, the articles on Explosives, including Gaseous Explosion and Explosives used in Coal Mines, occupy no less than 146 pages and vie with certain well-known textbooks in their comprehensiveness of survey in this field. Although it is probably true that no

new major explosives have been introduced lately, the many modifications—both of production and of product—are of great importance; these are here clearly but concisely described. There are, too, some major articles of particular interest to the laboratory worker, such as those on laboratory distillation, laboratory extraction apparatus and on disinfectants. The articles on azo dyestuffs and on dyeing (48 and 68 pp. respectively) are full of interest to the general chemist; including the analyst. The classification and exposition of the constitution of azo dyes in terms of function is interesting and, so far as the Dictionary is concerned, novel. This method conveys a picture of the structure of these interesting compounds more readily than conventional though fuller formulae. There is some overlapping between the articles on dyestuffs and dyeing, but this is perhaps inevitable, and we think that out of a 68-page article on dyeing, one-quarter page is not a wholly adequate allowance for the complexities of modern fur dyeing.

As in previous volumes, there is a nice balance between matters industrial and matters theoretical, and it is very satisfactory that the authors maintain the principle of setting out clearly the scientific bases of the subjects treated as well as the practical industrial applications.

In perusing the list of contributors it is as pleasant to welcome new names as it is gratifying to see some old and much-honoured contributors, among whom may be mentioned Dr. Bernard Dyer who has revised and brought up-to-date his contribution on feeding stuffs. The general usefulness of the volume is such that it becomes indispensable to the consulting chemist, who naturally turns to it in all his problems. Even the expert in a particular field finds much in it that is helpful. We are sorry to note the increase in price, as the purchase of these volumes is already a quite considerable item of expenditure, but we suppose in these days it is unavoidable. These are volumes which people who use such books must acquire, more or less regardless of cost. H. E. Cox

INORGANIC CHEMISTRY. FRITZ EPHRAIM. Third English Edition. By P. C. L. THORNE, M.A., M.Sc., Ph.D., F.I.C., and A. M. WARD, Ph.D., D.Sc., F.I.C. Pp. xii + 911. London: Gurney & Jackson. 1939. Price 28s.

An organic chemist, surveying the achievements of inorganic chemistry up to date in, say, the year 1895, might well ask (in fact often did ask) the question put to Ezekiel in the valley of dry bones—"Can these bones live?" The inorganic chemist of that day, less cautious than the prophet, was very apt to reply, "No they can't; you will have to take them as they are." The coming together of the bones, the clothing them with flesh and skin, in some instances even the breathing into them of the breath of life, has been very largely a miracle of this century. The process is very far from complete; in some regions the bones seem hardly to have stirred, but in a surprisingly large number they are giving every indication of being alive. It makes a fascinating story—this achievement—and nowhere has it been better presented than in the book under review; it is more, not less, fascinating by very reason of the fact that it is incomplete.

The distinctly unconventional method of approach of former editions has been preserved; in the words of the preface, "The materials of inorganic chemistry are dealt with collectively rather than individually." To describe this method adequately is impossible; perhaps the nearest approach one can make is that the basis of classification tends to be anionic rather than cationic as in ordinary textbooks. Covering, as it does, however, the whole wide sweep of a subject so liable to change, not only in detail but even in fairly basic conception, it is necessarily, to a large extent, a new book. The afore-mentioned method of approach is undeniably stimulating; it has many and obvious virtues, among them being the cross-sections it gives of information to be found in other textbooks; it has also drawbacks which, perhaps, are not quite so obvious. The book is such a mine of valuable information that one needs must want not only to read and study it, but also to

use it as a book of reference; here, however, one comes up against the unconventional arrangement. The unfamiliar setting has removed those landmarks which, though their use may be subconscious, serve as pointers in most textbooks, and one is left with a singularly lost feeling if what is wanted does not appear in the index. It is difficult to think of a remedy for this; the index contains some 4100 references, and to provide one that would meet all requirements would be a labour of Hercules.

It is a thankless task looking for flaws in such a really outstanding work, but there are a few statements scattered through its pages which cannot be allowed to go unchallenged. For instance, on p. 453, "Solutions of . . . ferric salts to which tartaric acid has been added, are not precipitated by . . . ammonium sulphide." This statement, if true, would make invalid one of the major separations of mineral analysis. Again (p. 752), "Secondary alkali monofluophosphates are neutral to phenolphthalein but acid to methyl orange," and (p. 844), "Tempered steel may contain *more than* 90 *per cent.* of this carbide" (Fe_3C).

These, and a few others of the same sort, must surely be proof-reading slips, although it is hard sometimes to see what can have been intended. As has been said, these lapses are very few, but their occurrence is most regrettable, inasmuch as they shake confidence in the many statements which cannot be so readily checked. Nevertheless, when one considers the immense labour that must have gone to the mere compiling of material for this wonderful book, criticisms such as these shrink into insignificance. The volume is well bound and well printed on good paper; misprints exist but are very few indeed; proof-reading slips are more numerous but still few. An unusual and attractive feature is a brief appendix giving aid to the younger chemist in looking up literature before embarking on a line of research. It is both impossible and unnecessary, however, to enumerate and comment on the various excellent sections that this work contains; the fundamental thing about it is its unity. Through it, more than through any book I know, one sees the picture, dim as yet and blurred, of that which, grasped by the alchemists by an act of faith but denied until recently by science for lack of sufficient evidence, is now emerging into the light of day and growing steadily clearer as the years go by—the unity underlying the bewildering phenomena of inorganic chemistry.

B. S. EVANS

PHYSICAL CONSTANTS OF HYDROCARBONS. Volume II. G. EGLOFF. Pp. 605. New York: Reinhold Publishing Co.; London: Chapman & Hall. 1940. Price 72s. net.

It will not be necessary to introduce Volume II of Dr. Egloff's well-known work to those who are familiar with Volume I. The second of this four-volume series is the result of a survey of data available on the physical constants of the non-aromatic cyclic hydrocarbons. The aliphatics were covered by the first volume.

For each hydrocarbon the carbon skeleton and the available data on melting and boiling points, specific gravity and refractive index are given. The text is conveniently arranged so that new experimental results can be added if desired. Every figure shown bears its own bibliographical reference. There is a short introduction in which the author discusses the structure of the alicyclic hydrocarbons and defines his nomenclature.

The cyclanes (cycloparaffins or naphthenes may be more familiar titles for some) have perhaps lagged behind other hydrocarbons in their application to chemical industry. Most people will agree with Egloff's opinion that a new chemical industry could well be developed based on cyclane chemistry. The almost unbelievable progress made recently in petroleum technology, of which no small amount is due to Egloff, allows hydrocarbons to be broken down and built up differently almost according to plan. These developments, coupled with the vast amount of cyclanes in nature (500,000,000 barrels from crude petroleum alone

during 1939 according to Egloff's estimate), must increase the importance of these hydrocarbons.

Although the volumes in this series find their most obvious application in oil research laboratories, workers in many other fields will find them invaluable—in fact, it is not difficult to predict that generations of chemists yet unborn will one day find themselves indebted to Egloff for the service he is now performing.

P. N. FULLER

THE MANUFACTURE OF COMPRESSED YEAST. By F. G. WALTER, A.A.C.I. Pp. viii + 254, with 29 figures, including 3 plates. London: Chapman & Hall, Ltd. 1940. Price 15s. net.

This volume is intended to provide a comprehensive account of the manufacture of yeast on an industrial scale, from the underlying scientific principles involved to the examination and packing of the finished product. The contents include a chapter entitled "The Yeasts, etc.," in which descriptions are given of alcoholic fermentation, the common varieties of yeasts, moulds, bacteria, enzymes, various cereals with other sources of starch, and the carbohydrates. This is followed by sections devoted to the preparation of nutrient media, the isolation of pure yeast cultures and the production of seed yeast, whilst the four succeeding chapters describe in detail the manufacture of yeast on an industrial scale from cereals, molasses, glucose and spirit fermentations by the differential and continuous processes. A chapter on the drying of yeast and yeast nutrients in panary fermentations is followed by the final one providing details of the design, construction and operation of the yeast factory, laboratory equipment, the determination of *p*H values, and methods of analysis of the raw materials and the finished yeast.

It is evident that the author is far more familiar with operations of the yeast factory than with the more scientific part of the subject, for the few errors which occur are confined almost entirely to the first two chapters. Thus the yeast vacuole does not consist of glycogen, as stated on p. 1; cassava juice is not mainly hydrocyanic acid, nor are the granules of cassava starch far smaller than those of any other starch-producing plant (p. 29); the term "inversion" is not appropriate to the conversion of starch into dextrin, maltose, etc., in the mash tun (p. 65), and ammonia cannot be directly titrated with *N* sodium hydroxide solution even by using methyl orange indicator (p. 90). In addition, several typographical errors occur throughout the text, although these are not serious.

The larger portion of the book, dealing with the design, construction and operation of the yeast manufacturing plant is excellent and thoroughly comprehensive, its value being enhanced by the numerous tables and charts of operation given together with frequent references to the necessity for biological cleanliness throughout. In this section the only item that invites criticism is the undue number of buffer solutions, ranging from *p*H 2·0 to 5·4, for use with methyl orange as the sole indicator.

The binding, printing and illustrations are excellent, but the index, although fairly satisfactory, requires revision. Not only are some items in the text omitted, but numerous entries are included under their adjective, verb, and so forth instead of under their principal noun. This work contains much valuable information, and, with the elimination of the errors referred to above, would be an admirable textbook and reference manual for all interested in the industrial manufacture of yeast, which is being used to an ever-increasing extent in animal and human dietary and in therapeutic practice.

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