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The Routine Examination of Dairy Products with Special Reference to the Mojonnier Tester.

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(*Read at the Meeting, April* 2, 1924.)

In any creamery or factory where dairy products are in process of manufacture, it often becomes necessary to make estimations of fat and solids-not-fat in short spaces of time if the results of the control are to be of any practical use. Such a case is to be found in the manufacture of ice-cream on a large scale, where every "mix" of, say, 300 gallons, has to be standardised before it can pass to the freezing machines.

We have found the Mojonnier tester to be most useful in such a case, and the following work is presented, not only to demonstrate the ease and speed of manipulation, but also to show the reliability of the results obtained by this method.

It is not proposed to describe the Tester, as a full description will be found in Mojonnier and Troy's book on the technical control of dairy products,* but it may be said that, in a comparatively small space it combines the means of making rapid and accurate estimations of the total solids and the fat in milk and its products, the total solids being estimated by drying in vacuo and the fat by a modified Röse-Gottlieb method. The economy in time effected by using this method will be appreciated when it is stated that it is possible by means of the Total Solids apparatus, to estimate the solids in milk in 21 minutes, whilst the

* Mojonnier and Troy, *Technical Control of Dairy Products*, 1st ed., pp. 62–69. (Published by Mojonnier Bros. Co., Milk Engineers, Chicago, 1922.)

time required for estimation in duplicate is only 30 minutes. For other dairy products varying times are required, as is shown in the following table:

TABLE I.

Quantities of materials used and the time required in the vacuum oven for estimation of total solids.

Product.	Milk.	Cream and Butter.	Dried Milk Products.	Ice Cream.	Cheese.	Evaporated Unsweetened Milks.	Sweetened Condensed Milks.
Wt. of material (grms.)	2	0·5–1·0	0·5	2	0·5	1·0–2·0	0·5
Time in oven (mins.)	10	10	15	10	20	15	20
Total time required for the estimation	21	21	26	21	31	26	31

With sweetened condensed milk there is always a certain amount of moisture remaining, and accordingly, when working under the conditions specified above, 0·3 per cent. is subtracted from the solids content, as it has been found that this is the additional loss after 90 minutes in the vacuum oven.

That the results obtained are consistent is shown in Table II., which gives typical results of duplicate estimations obtained in our laboratories with various products.

TABLE II.

Typical results of duplicate estimations of Total Solids.

	Sample I.		Sample II.		Sample III.		Sample IV.	
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Milk	12·22	12·24	11·36	11·35	12·21	12·23	12·30	12·33
Cream	55·65	55·61	39·52	39·60	54·45	54·45	54·84	54·79
Butter	84·46	84·47	84·31	84·28	85·78	85·80	84·46	84·51
Milk powder	97·82	97·80	97·75	97·70	98·22	98·24	98·02	97·99
Ice cream	38·06	38·03	34·59	34·63	34·51	34·53	34·95	34·95
Cheese	65·77	65·70	48·26	48·22	72·65	72·71	76·80	76·72
Unsweetened condensed milk	25·64	25·69	37·73	37·65	30·94	31·00	39·81	39·80
Sweetened condensed milk	76·11	76·21	73·50	73·33	78·47	78·36	72·90	72·96

It may also be stated that the apparatus is being used with success in the estimating of total solids in sauces, syrups, confectionery, tomato purée, fruit pulps, etc.

The Mojonnier modification of the Röse-Gottlieb method for the estimation of fat has been made particularly simple and rapid, the time occupied for a complete estimation being 30 minutes.

As with the total solids estimation, varying quantities of materials are required according to their type, as is shown in the following table:

TABLE III.

Quantities of Materials required for Fat Estimations.

	Grms.
Milk	10
Ice cream	5
Evaporated milk	5
Sweetened condensed milk	5
Thin cream	2
Rich cream	1
Butter, cheese, and other solid products such as milk powder, etc.	1

In nearly every case it is found that all the fat is removed in the two extractions specified in the Mojonnier method, and that a third extraction gives an additional weight of only 0·2 to 0·3 mgrm., which can be accounted for by the residue from the added solvents. We have found that the only case in which the whole of the fat is not obtained in 2 extractions is when working with spray process milk powder, which requires three extractions, no further increase in weight being obtained with additional extractions. Certain investigations were carried out to see if the ammonia used in the process had any saponifying action on the butter fat. For example, the residues after extraction were acidified and the mixture re-extracted with ether and petroleum spirit. It was found that in no case was the increased weight thus obtained more than could be accounted for by the traces of impurities in the solvents used, showing that no saponification had taken place. On re-extracting the fat with petroleum spirit in the dishes, the loss in weight of each dish was exactly the weight of the fat previously found.

This is also borne out by the figures given in the following table, which shows the results of complete estimations carried out on weighed quantities of butter fat:

TABLE IV.

Butter Fat Extractions.

Butter fat taken. Grms.	Butter fat found. Grms.
0·1362	0·1362
0·2100	0·2097
0·3463	0·3465
0·5192	0·5188
0·5638	0·5641

A point which we have found to be of major importance is the amount of shaking given to the contents of the extraction flasks, and unless this is carried out vigorously and for the space of time specified, abnormal results will be obtained, as is shown in the following table, where comparative sets of figures are

given for thorough mixing and also for continually inverting the flask for the same period. (Table V.)

TABLE V.

Effect of Shaking on the Estimation of Fat.

Material.	Vigorous shaking.		Gentle shaking.	
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Milk powder	26·07	26·06	24·55	25·32
Milk powder	26·34	26·31	24·62	24·80
Cream	49·54	49·50	48·81	49·05
Cream	50·62	50·69	49·62	49·93

In order to make the conditions of the extraction more comparable with those obtaining during the analysis of a milk product, estimations were made on a mixture of butter-fat and skim milk powder (fat content 1·51 per cent.), the weights of each substance used being approximately those of fat and solids-not-fat in 1 grm. of cream containing 50 per cent. of fat. The results obtained are shown in the following table, together with those obtained by other methods, the comparative tests being made to show relative results which would be obtained in the ordinary course of analysis, and not as a detailed investigation on the accuracy of the other methods. (Table VI.)

TABLE VI.

Estimations of Fat in mixtures of butter-fat and skim-milk powder.

(Skim-milk powder used contains 1·5 per cent. butter-fat.)

Method.	Total weight of Fat. Grms.	Weight obtained. Grms.	Difference as per cent. of fat taken.
Babcock. (Reading to bottom of meniscus × 0·18)	4·933	4·986	+1·07
	4·198	4·284	+2·03
Gerber. (One c.c. amyl alcohol used. Reading to bottom of meniscus × 0·111 and 0·0054 added for meniscus	0·495	0·504	+1·82
	0·502	0·509	+1·40
Gerber. (Previous reading × 0·110 and 0·0054 added for meniscus.)	0·495	0·499	+0·81
	0·502	0·505	+0·60
Gerber. (0·25 c.c. amyl alcohol used. Reading × 0·111 and 0·0054 added for meniscus.)	0·519	0·519	0·00
	0·496	0·494	−0·04
Mojonnier. (Two extractions.)	0·5297	0·5298	+0·01
	0·5080	0·5075	−0·10
Röse-Gottlieb. (Three extractions.)	0·2577	0·2580	+0·12
	0·2835	0·2835	0·00
Werner-Schmid. (Three extractions.)	0·5106	0·5133	+0·53
	0·6097	0·6127	+0·49

Details of the estimations recorded in Table VI. were as follows:

(*a*) The Babcock method was carried out with the constituents of 9 grms. of cream; the reading from the base of the fat column to the bottom of the upper meniscus being multiplied by 0·18 to obtain the weight of fat represented by the reading.

(*b*) In the Röse-Gottlieb estimation the equivalent of 0·5 grm. of cream was used.

(*c*) The Gerber results were arrived at in three different ways, depending on various quantities of amyl alcohol used, and also on two types of calculation.

(i.) Using 1 c.c. of amyl alcohol. Reading from base of the fat column to the bottom of the upper meniscus multiplied by 0·111, and 0·0054 added. (Based on Day and Grimes' results.*)

(ii.) Using 1 c.c. of amyl alcohol. Reading multiplied by 0·110 and 0·0054 added.

(iii.) Using 0·25 c.c. of amyl alcohol. Calculation as (i.).

From these results it appears that the recovery of fat is most nearly the theoretical amount in the modified Gerber, the Mojonnier, and the Röse-Gottlieb methods.

The whole series of methods was extended to different milk products, with the results shown in Table VII.

TABLE VII.

Typical estimations of fat.

	Babcock. Per Cent.	Gerber I.† 1 c.c. amyl alcohol. Per Cent.	Gerber II.† 0·25 c.c. amyl alcohol. Per Cent.	Röse-Gottlieb. Per Cent.	Werner Schmid. Per Cent.	Mojonnier. Per Cent.
Milk	3·35	3·40	—	3·37	3·45	3·40
	3·35	3·40	—	3·37	3·45	3·40
Cream	48·8	48·3	48·2	48·16	48·36	48·23
	48·8	48·3	48·2	48·27	48·23	48·27
Cheese	34·8	38·8	35·3	35·45§	35·63	35·50
	34·8	36·8	35·3	35·45	35·69	35·54
Milk powder (full cream), roller process	24·8	25·4	—	25·48	25·50	25·50
	24·2	25·4	—	25·40	25·51	25·54
Milk powder (full cream), spray process	25·0	27·5	—	27·26	27·01	27·35
	25·4	27·5	—	27·30	27·12	27·37

In connection with these figures the following points should be noted:

(*a*) In the Gerber, Mojonnier and Werner-Schmid methods, 1 grm. of material was weighed out in all cases except for milk.

* ANALYST, 1893, **18**, 123.

† Gerber I. Factor: Reading × 0·110 + 0·0054; Gerber II. Factor: Reading × 0·111 + 0·0054.

§ Double the strength of ammonia.

(*b*) In the Babcock method 2 grms. were weighed in the milk-bottle; for cream 9 grms. were weighed out, and a cream bottle used.

(*c*) In the ordinary Röse-Gottlieb estimation 0·5 grm. of material was used.

(*d*) In the case of spray process powder, the material was weighed into a small beaker, dissolved in water, and poured into the vessel in which the estimation was to be made, the beakers being subsequently rinsed with the reagents used.

These results bear out the conclusion drawn from the results given in the previous table.

A point of great importance, and one which we do not think has been sufficiently recognised, is the effect of the quantity of amyl alcohol on the reading of fat in the case of estimations by the Gerber method in cream.

If only 0·25 c.c. of alcohol is used, a correct result is obtained by using the factor 0·111 and adding 0·0054 for the meniscus, but if 1 c.c. of alcohol is used, abnormally high figures result, as the fat column contains an appreciable quantity of amyl alcohol and amyl esters. Actual analysis of such fat showed the presence of 2·28 per cent. of volatile matter, of which 1·44 per cent. was amyl alcohol and 0·78 per cent. amyl esters, calculated as amyl butyrate.

Results show that the Babcock method is not very accurate, and this has been pointed out by American workers.*

The great difficulties which have been noted by various workers in the estimation of fat in spray process milk powder, and which we ourselves have experienced in the course of extended studies on such powders, have led us to test many methods. In general, as a routine method we have adopted the Gerber method, using a weighed quantity of powder as described above, and whirling two or three times for 3 minutes at about 1300 revs. per min. till the reading is constant. We find that the value of 0·110 grm. of fat for each division and an allowance of 0·0054 grm. for the meniscus gives satisfactory results, readings being made at 65 °C.

That the Mojonnier modified Röse-Gottlieb method gives consistently reliable results is shown by the following typical estimations. (Table VIII.)

TABLE VIII.

Typical duplicate estimations by means of the Mojonnier Tester.

Product.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Milk	3·10	3·10	3·40	3·40	3·72	3·73	3·52	3·52
Cream	49·58	49·60	51·30	51·24	50·72	50·74	48·97	49·00
Cheese	35·50	35·54	27·18	27·18	34·96	34·89	31·51	31·55
Unsweetened condensed milk	8·99	8·98	9·54	9·54	10·36	10·34	9·12	9·13
Milk powder	27·00	27·02	26·78	26·75	26·96	26·97	27·10	27·11
Ice cream	13·94	13·95	14·22	14·23	14·26	14·26	14·32	14·31
Sweetened condensed milk	8·53	8·53	9·02	9·01	8·74	8·76	9·38	9·35

* Mojonnier, *Technical Control of Dairy Products*, p. 51; *Dairy Science*, VI., p. 549; and *J. Ass. Off. Agr. Chem.*, 7, 159 *et seq.*

Our results may be summarised as follows:

(1) The total solids of milk products can be estimated rapidly and with consistent results by means of the Mojonnier tester. A single estimation can be made in 21 minutes; in duplicate in 30 minutes.

(2) The Mojonnier modification of the Röse-Gottlieb method for the estimation of fat has been shown to give accurate results. In the case of spray process milk powder three extractions are recommended.

(3) The time required for one estimation is 30 minutes. Both fat and total solids can be estimated in 35 minutes.

(4) A modified Gerber method is recommended for the routine estimation of fat in milk powders.

(5) It has been shown that where the proportion of fat to the milk solids is high, as in cream, the amount of amyl alcohol used in the Gerber method affects the result to a considerable extent.

We wish to acknowledge with thanks the permission of Messrs. J. Lyons and Co., Ltd., to publish these results.

Discussion.

Dr. L. H. Lampitt said that, originally, their reason for taking up the Mojonnier apparatus was a purely commercial one. When in America he had met and been rather impressed by Dr. Timothy Mojonnier—who was a chemist first and a laboratory and dairy-equipment expert second—and had inspected many dairies and creameries in which the Mojonnier equipment was used. At that time his firm had been unable to compete in their laboratory with the ice-cream question. They had a plant with a capacity of 25,000 gallons of ice-cream per day; this was divided over 600 gallon mixes during 20 hours, which gave them 45 minutes to analyse samples. The Mojonnier Tester afforded an excellent method of obtaining the necessary control, and, in his opinion, it was as good as any other routine method of determination, its great advantages being accuracy and speed. It was certainly an expensive apparatus, but it had proved itself to be well worth its cost. Each test cost 8½d. in materials, but the expense was balanced by the speed with which the tests could be carried out. His firm proposed to instal another Mojonnier tester to be used entirely for moisture estimations in the Baking Section. It was anticipated that this apparatus would turn out to be as efficient as that used for ice-cream.

Mr. Hinks enquired whether the percentage of total solids of milk obtained in the Mojonnier tester was the same as that obtained by drying the milk at 100° C. for three hours in a water oven in the usual way.

Mr. F. E. Day said that in his experience he had found the Mojonnier apparatus useful for estimations of fat, but had always felt dubious about evaporating in open pans with ether. For moisture they had had good results with new milks, but with condensed milks they had obtained very curious results—after 10 to 20 minutes' drying they had obtained a figure which corresponded to the water of hydration of milk sugar.

Mr. E. R. Bolton, remarking upon the great necessity for speed which had mainly prompted the authors to instal the Mojonnier apparatus, made the suggestion that the time might be still further curtailed by a modification of the procedure on the lines of the process suggested by Revis (Analyst, 1907, **32**, 284), in which a small quantity of acetone was added before evaporation of the milk in estimating total solids. He always used this method, not only for milk, but also

for condensed milk, and its rapidity and accuracy were astonishing. In one case he had carried out a series of tests which were duplicated with the Mojonnier apparatus in America, and the results agreed as closely as if they had been carried out by the same observer in one laboratory.

Mr. G. RUDD THOMPSON said that he could see no reason why results obtained in this way should not be equal to those obtained by the ordinary laboratory methods; they ought to be better. In his opinion, the evaporation of an ethereal solution of fat-solids under glass entailed a great waste of material (he referred more especially to the ether carried through a fan to the open air) it was not only a great waste, but there was a danger from fire.

The Freezing Point of Sudan Milk.

BY A. F. JOSEPH, D.Sc., F.I.C., AND F. J. MARTIN, M.A., PH.D., F.I.C.

THE recent improvements in apparatus for determining the freezing point of milk have led us to use the method for obtaining additional information on which to base a comparison between the properties of milk of tropical and temperate climates. In a previous communication[1] we drew attention to the fact that cows' milk in the tropics is usually richer both in fat and solids-not-fat than that in temperate climates, and we are now able to supplement this by showing that in the Sudan cows' milk has the same freezing point as has been reported elsewhere. Some data have also been collected relating to the milk of other animals.

In carrying out this work we have used the cryoscope described by Dr. Hortvet in 1921[2]; the method was critically examined by the Association of Official Agricultural Chemists and reports were published in 1922 and 1923.[3, 4] A few years before Hortvet's work appeared, the question was investigated by Monier-Williams,[5] who, however, considered that the experimental difficulties involved in obtaining reliable results were such that the method was not likely to be adopted. Using Hortvet's cryoscope and following his instructions, we have obtained very concordant results and are satisfied that no special skill is required in its use.

THE LOWERING OF THE FREEZING POINT OF COWS' MILK.—The first point investigated was the effect of the breed of animal on this property. For this purpose samples were taken from 51 animals of three different breeds, with the following results:

Breed.		Demietta.	Shorthorn crosses.	Native Khartoum district.
Number examined		16	9	26
Freezing-point depression, °C.	Average	0·558	0·553	0·556
	Highest	0·579	0·578	0·573
	Lowest	0·538	0·538	0·546
Fat, per cent.	Average	3·8	4·4	4·1
	Highest	5·8	5·7	5·6
	Lowest	2·5	3·6	3·3
Solids-not-fat, per cent.	Average	9·0	9·3	9·1
	Highest	9·6	10·0	10·0
	Lowest	8·7	8·7	8·6

The average lowering of freezing point for all the samples is 0·556, that for the different breeds being substantially the same. The mean value for 129 results quoted by Bailey[3] is 0·548, the limits being 0·530 to 0·566. None of our samples has given a lower value than 0·530, and only four were higher than 0·566. As a difference of ·01 °C. in the freezing point corresponds to only 2 per cent. of added water, we should be justified in using the standard recommended for America.

VARIATION IN DEPRESSION OF FREEZING POINT IN DIFFERENT PORTIONS OF THE SAME MILKING.—A common difficulty in dealing with the milk of individual animals is caused by the great difference in fat content of the first and later portions of the milking. The following results show that the freezing point is practically the same throughout.

PERCENTAGE OF FAT.

Cow No.	1st portion.	2nd portion.	3rd portion.	4th portion.
70	2·5	3·7	3·0	5·1
73	1·3	1·8	2·9	6·9
18	2·2	3·5	4·7	6·2
75	1·8	2·4	1·7	5·7
Mean	1·95	2·85	3·07	5·97

FREEZING POINT DEPRESSION.

	°C.	°C.	°C.	°C.
70	0·561	0·558	0·559	0·559
73	0·561	0·559	0·562	0·559
18	0·570	0·569	0·568	0·568
75	0·556	0·556	0·558	0·553
Mean	0·562	0·560	0·562	0·560

VARIATION OF FREEZING POINT DEPRESSION WITH THE PERIOD OF LACTATION.—The samples from which the results were given in the first table were taken from animals at various stages of their lactation period, and, when grouped according to this, give the following results:

Period of Lactation.	No. of Animals.	Average Depression of Freezing Point. °C.
1st Month	8	0·552
2nd ,,	12	0·560
3rd ,,	8	0·556
4–6th ,,	10	0·553
7–10th ,,	14	0·554

These differences are unimportant.

DEPRESSION OF FREEZING POINT OF THE MILK OF ANIMALS OTHER THAN COWS.—A number of samples from goats, sheep, and asses, and one of camel's milk, gave the following results:

Animal.		Goats.	Sheep.	Asses.	Camel.
Number examined		12	8	6	1
Freezing-point depression, °C.	Average	0·575	0·564	0·554	0·616
	Highest	0·591	0·591	0·563	—
	Lowest	0·564	0·547	0·546	—
Fat, per cent.	Average	4·1	6·1	0·7	4·7
	Highest	7·5	8·2	1·9	—
	Lowest	2·8	3·0	0·1	—
Solids-not-fat, per cent.	Average	9·2	11·7	7·6	8·6
	Highest	10·3	12·5	8·5	—
	Lowest	8·8	10·9	5·4	—

In the case of 4 goats, the milk was drawn in two portions which were examined separately.

Goat No.	1st Portion. Depression F. Point. °C.	1st Portion. Fat. Per cent.	1st Portion. Solids-not-fat. Per Cent.	2nd Portion. Depression F. point. °C.	2nd Portion. Fat. Per Cent.	2nd Portion. Solids-not-fat. Per Cent.
1	0·570	2·8	8·5	0·570	3·6	8·5
2	0·591	3·3	9·7	0·593	5·3	9·3
3	0·570	4·0	9·3	0·570	5·2	9·3
4	0·570	3·2	9·3	0·571	3·9	8·8
Mean	0·575	3·3	9·2	0·576	4·5	9·0

FACTORS WHICH INFLUENCE THE FREEZING POINT DEPRESSION.—In the reference already quoted[3, 4] attention is drawn to certain conditions which may cause abnormal freezing points in genuine samples. Bailey finds that the morning milk has a larger depression than the evening, but the differences seldom exceed 0·02° C., and are frequently less than ·01° C. We have not, up to the present, collected any local data on this point, but the differences are not great enough to impair seriously the usefulness of the method.

The case of diseased animals always presents difficulty, but, for practical purposes, they might well be grouped as "non-genuine."

A common cause of an increased depression of freezing point is the development of acidity. Bailey has devoted much attention to this point and confirms the opinion of Monier-Williams, that where the sample does not smell and taste distinctly sour, the freezing point is unlikely to be depressed by as much as 0·002° C.

In hot weather here the acidity increases so rapidly that a preservative must be added if the examination is delayed even for only a few hours. We have found mercuric chloride, added in the proportion of 1 in 2000, to be very satisfactory, and this is illustrated by the following results for a sample tested immediately on receipt and after standing 24 hours, with and without the addition of mercuric chloride:

	Depression of freezing point.	Acidity (i.e. c.c. of 0·02 *N* alkali required per litre of milk).
On receipt	0·578	18
After 24 hours	0·806	95
Do., but $HgCl_2$ added	0·578	20

SUMMARY.—1. The freezing point of cows' milk in the Sudan is practically the same as in the United States.

2. Different portions of the same milking give the same freezing point.

3. The period of lactation only exerts a very small influence on freezing point.

4. The milk of goats and sheep gives a greater depression of freezing point than that of cows; that of asses gives the same.

5. Abnormal results due to the rapid development of acidity in a hot climate are prevented by the use of mercuric chloride (one part in 2000).

Our thanks are due to the Director of the Veterinary Department and to the staff of the Sanitary Service, Khartoum, for furnishing us with the samples.

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Alcoholysis and the Composition of Oils and Fats.

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

IT is indeed refreshing to read the remarks by Channon, Drummond and Golding in a recent paper in the ANALYST (1924, **49**, 311) in connection with the determination of the composition of the mixed fatty acids of fats by the method of fractional distillation of the methyl or ethyl esters. Already, in 1913, the present writer (ANALYST, 1913, **38**, 8), as a result of a certain amount of work with this process, stated:—"From the experiments carried out it has been concluded that the process of alcoholysis is a suitable one for determining the qualitative composition of an oil, and that it also gives considerable information in regard to the quantities of the constituents present; the process, however, is much too lengthy for use as an ordinary laboratory test, and would seem to be chiefly of theoretical interest. The quantitative results have not been encouraging, but the process probably gives results within about 5 to 10 per cent. of the true value—that is to say, it should decide between 35 and 45 per cent. for the content of lauric acid in coconut oil," and this opinion was repeated in a later paper (ANALYST, 1914, **39**, 78), although in the latter paper, unfortunately, there is a fairly obvious misprint in the last line but one, which possibly somewhat obscures the meaning.

Of late years, this method has been very largely used both in this country and in America for the elucidation of the composition of oils and fats, and some extraordinary claims have been made for the process. These claims are, in the opinion of the writer, not justified by the facts and a short time ago the following sentence was written by him (*Chemical Age*, 1924, **10**, 188): "It is perhaps a little difficult to understand why the only one of the acids of high boiling point which appears in the lower fractions should be oleic, and also, to take one example, why in one of the lower fractions there should be 0·608 grm. of caproate, 0·320 grm. of caprylate and 0·160 grm. of oleate to the total exclusion of all other esters. The experience of the writer in the fractional distillation of large quantities of methyl esters obtained from coconut oil would, moreover, scarcely support this assumption. . . ." With all these ideas Channon, Drummond and Goulding are evidently in perfect agreement, and they give a most excellent account of their objections in their recent paper quoted above, the extraordinary and inexplicable excellence of the results obtained by Crowther and Hind on their artificial mixture of fatty acids notwithstanding. They do not, however, draw particular attention to the enormous differences in composition found by Crowther and Hynd and by Holland and Buckley, respectively, in their examination of butter fat. The largest amount of stearic acid found by the former was 5·9 per cent., whilst the figures obtained by the latter varied from 7·8 to 20·4 per cent.

The different results which may be found by various workers with the method of alcoholysis, and other methods, are well shown by the variation in the figures which have been obtained on the composition of the mixed fatty acids of coconut oil.

It has been shown by Jensen (ANALYST, 1905, **30**, 397) that coconut oil contains no butyric acid and but little caproic, the volatile acids being mainly capric and caprylic. Paulmyer (*J. Soc. Chem. Ind.*, 1907, **26**, 881) found that the fatty acids of coconut oil consisted of capric, 20 per cent.; lauric, 40 per cent.; myristic, 25 per cent.; palmitic, 11 per cent.; and oleic, 5 per cent.; together with capric and caprylic acids; while Caldwell and Hartley (ANALYST, 1909, **34**, 274) found by a different method at least 30 per cent. of lauric acid. Haller and Youssoufian found, by the method of alcoholysis, caproic, caprylic, capric, lauric, myristic, palmitic, stearic, and oleic acids. They did not give any quantitative results, but they stated that lauric and myristic acids predominated, whilst palmitic, stearic, and oleic were present in relatively small quantities. The writer, also, by the method of alcoholysis, deduced the following composition:

Caproic acid	2 per cent.
Caprylic acid	9 ,, ,,
Capric acid	10 ,, ,,
Lauric acid	45 ,, ,,
Myristic acid	20 ,, ,,
Palmitic acid	7 ,, ,,
Stearic acid	5 ,, ,,
Oleic acid	2 ,, ,,
	100 per cent.

but in this case the quantities of capric acid and stearic acid are possibly too high, whilst that of oleic acid is probably too low. Walker (*J. Chem. Soc.*, 1923, **123**, 2837) has shown that, in a sample of coconut oil examined by him by a method of fractional precipitation, it was unlikely that more than 2 per cent. of capric acid was present. In the most recently quoted examination of coconut oil, referred to by E. F. Armstrong and John Allen in the recent Presidential address to the Society of Chemical Industry (*J. Soc. Chem. Ind.*, 1924, **43**, 207 T), the following figures are given, which figures are stated to have been obtained by the method of alcoholysis:

Caproic acid	0·25 per cent.
Caprylic acid	0·25 „ „
Capric acid	19·5 „ „
Lauric acid	40 „ „
Myristic acid	24 „ „
Palmitic acid	10·6 „ „
Stearic acid	0·0 „ „
Oleic acid	5·4 „ „
	100·0 per cent.

The extraordinary differences between these figures and other figures obtained both by the same and other processes, all of which differ among themselves, can, presumably, mean only one of two things, or possibly a mixture of both—either that there are enormous differences in the composition of coconut oils from different sources, or that, as is alleged by Channon, Drummond and Golding, the fractionation of the esters as an exact quantitative method is of little value.

In regard to this first point, there are undoubtedly differences in the composition of different samples of coconut oil, but the wide fluctuations in the composition, as indicated by the so-called quantitative analyses, are by no means upheld by the somewhat narrow variations in the analytical figures usually obtained. In the opinion of the writer, as has been previously expressed, the second is by far the more important of the two reasons, and, although the method of alcoholysis is undoubtedly valuable as a means of detecting the constituents of a mixture of fatty acids, it is only valuable in so far that better methods are, in general, not available.

The following remark, taken from Myddleton and Barry's recent book "Fats; Natural and Synthetic," shows the kind of confidence this method inspires, even in those who make use of it, "The figures indicated by the letter *a* in Tables VI.–IX. (*i.e.* tables which give the results of analyses of oils obtained in part by alcoholysis) have been adjusted by not more than 1·5 per cent.* of the observed figure to indicate clearly that no change takes place in the amount of this constituent, the 1·5 per cent. being within the limits of experimental error."

* Whether this means 1·5 per cent. of the oil or 1·5 per cent. of the actual observed figure for the fatty acid is not too clearly stated—if the latter, then it may mean in some cases possibly ten per cent. or more of the oil.

The process may have some quantitative value, but for exact work it is not only useless but worse than useless, as it seems to be leading very seriously astray workers whose time might be better spent. The work of Channon, Drummond, and Golding in exposing the pitfalls into which some of the later workers seem to have fallen will be thankfully received by all those who have any desire to see the examination of fats placed on a sure footing. The present writer would take the liberty of emphasising everything that these authors have said, and would like particularly to draw attention to the remarks made by Dr. Drummond at the close of the discussion.

Note.

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.

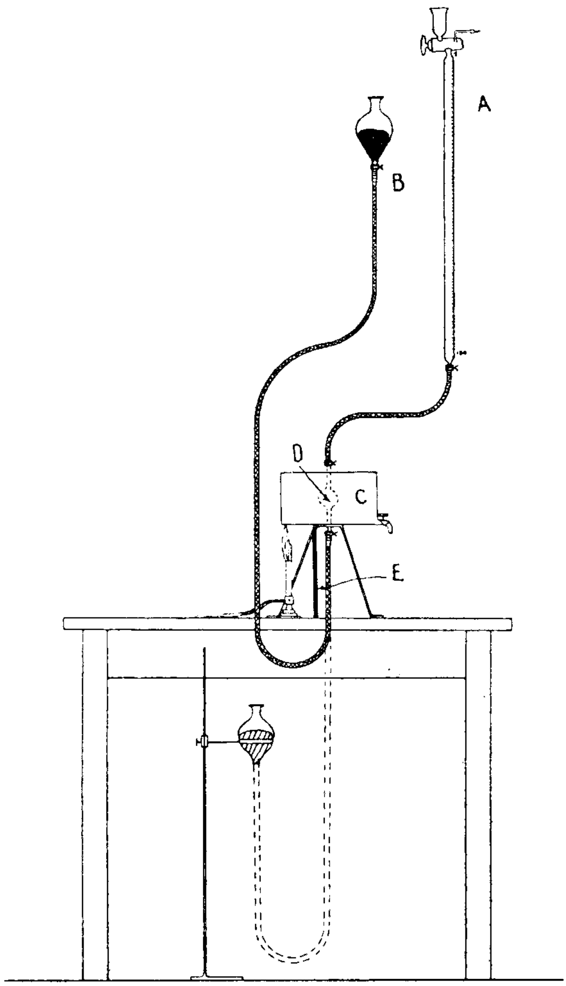
THE ESTIMATION OF CARBONATES IN HIGHLY COLOURED LIQUIDS.

In the apparatus shown in the diagram A is a nitrometer connected at the lower end with a glass bulb, D, having a capacity of about 100 c.c. This bulb is also connected by pressure tubing with the mercury reservoir, B, which can be placed either above the working bench or below it in the position B′. The apparatus is filled with clean mercury. The bulb is set in an oval-shaped bath which can be heated at one side and emptied when necessary. The rubber tubing is protected from the flame by a screen, E.

The estimation is made as follows:—Twenty-five c.c. of the liquor are poured into the cup of the nitrometer. The liquor is drawn into the nitrometer by lowering B and opening the threeway cock. No air must be allowed to enter. The cup is washed with two separate quantities of about 10 c.c. of distilled water and both lots of wash water are drawn into the nitrometer. Twenty c.c. of 16 per cent. sulphuric acid are added and then 2 c.c. of "paroleine." The latter is to prevent the carbon dioxide which is evolved from dissolving in the residual liquor when the apparatus is cooling down. The mercury reservoir is lowered to the position B′, and the height adjusted so that all the liquor is in the bulb. The rubber connection between D and A is 16 to 18 inches long and, when an estimation is in progress, should be in a continuous upward sweep. When all the liquor is in the bulb, D, water at 65 to 70° C. is poured into the bath C, and the temperature maintained at that point. It is not advisable to overheat the bath. The liquor in D boils gently and is allowed to do so for 15 minutes. The liquor is then transferred to A by raising the reservoir B, and the whole is left to cool. The level of the mercury in B is maintained somewhat below that in A. The bath, C, is emptied.

The volume of the carbon dioxide evolved is measured, after cooling, by adjusting the height of the reservoir B in the usual manner. The observed volume

should be reduced by 0·1 c.c. for every 10 c.c. of liquor, washings and acid. This represents the dissolved air and gives a sufficiently accurate correction for most purposes. (The actual value may be found after reading off the volume of the



gas by running strong sodium hydroxide solution into the nitrometer, shaking well and then levelling up.)

In order to calculate the weight of carbonate as grms. of Na_2CO_3 per 100 c.c. of liquor, the formula (a) is used.

$$\frac{K\,(V-v)\,(P-B-c)}{(273+T)} \text{ grms.} \quad \ldots\ldots\ldots\ldots\ldots\ldots (a).$$

Here V represents the volume in c.c. of gas after levelling; v, the correction in c.c. for dissolved air; P, observed barometric pressure; B, pressure of aqueous vapour at T° C.; c, correction for temperature of mercury column; T, temperature of the laboratory in °C.; and K, = 0·006846; Log K = $\bar{3}$·8355.

The full formula is:

$$\frac{(V-v)\,(P-B-c)}{(273+T)}\left[\frac{273}{760}\times\frac{1{\cdot}977}{1000}\times\frac{106}{44}\times\frac{100}{25}\right] \quad \ldots\ldots\ldots(b).$$

1·977 = weight in grms. of one litre of carbon dioxide at N.T.P.

106 = weight in grms. of sodium carbonate equivalent to 44 grms. of carbon dioxide.

The portion of formula (b) in square brackets = K.

The actual time required for this estimation (excluding the time occupied in boiling and cooling) is about half an hour.

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Notes from the Reports of Public Analysts.

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

METROPOLITAN BOROUGH OF HAMMERSMITH.

ANNUAL REPORT OF THE PUBLIC ANALYST FOR 1923.

DURING the twelve months 626 samples were submitted for examination, of which 600 were purchased under the provisions of the Sale of Food and Drugs Acts, and 12 under the Public Health (Milk and Cream) Regulations. Of the total number examined, 509 were reported as genuine, 37 as adulterated, and 54 as of inferior quality. The percentage of adulteration was 6·1, as compared with 6·6 in 1922.

MILK.—Of the 435 samples examined, 54 were reported as inferior and 22 as adulterated. Four milks were found to be artificially coloured with annatto.

COCOA AND CHOCOLATE.—Four samples of cocoa, out of 25 examined, contained arsenic in quantities ranging from 1/130th to 1/80th of a grain of arsenious oxide per lb. Cautionary letters were sent to the vendors.

MEAT AND FISH PREPARATIONS.—Seven samples were examined, and, of these, 3 contained boron preservatives, the amounts found being 18·2 grains, 17·5 grains, and 13·3 grains (in terms of crystallised boric acid) per lb.

DRUGS.—Thirty-two samples were examined, and two of these (borax) were found to contain arsenic in the proportion of 10 parts of arsenious oxide per million. Cautionary letters were sent.

P. A. ELLIS RICHARDS.

Legal Notes.

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

RICE HUSKS IN SHARPS.

ON May 17th, a firm of millers was summoned before the Norton (Malton) Bench of Magistrates for selling sharps which contained rice husks.

Mr. Hodge, prosecuting on behalf of the Ministry of Agriculture, stated that, on December 15th, 1923, the official sampler to the East Riding County Council had taken a sample of the sharps, but the defendants, who had been notified, did not attend. The sample was sent to Dr. Voelcker for analysis, and he reported that it contained some finely-ground rice husks, which were of no value as food, and action was accordingly taken under Section 6 of the Fertilisers and Feeding Stuffs Act.

On the back of the invoice was a printed notice to the effect that "all goods herein invoiced are, or may be, mixtures." This statement, counsel contended, was contrary to the spirit of the Act, and rendered void the special purpose for which that section was passed, i.e. to prevent a purchaser receiving any ingredient worthless for feeding purposes.

Dr. J. A. Voelcker, giving evidence in support of his certificate, said that a casual examination of the sample did not show anything wrong, but the microscope indicated the presence of rice husks. He had accordingly reported that the sample was impure, and that it contained a material worthless for feeding purposes.

Counsel for the defence urged that the question at issue was one of law rather than of fact. Defendants from the first admitted that, owing to the difficulty of obtaining wheaten sharps. it was necessary to add some ingredient. It was not suggested by the prosecution that rice husks were deleterious to poultry or stock, but only that they were worthless for feeding purposes. The question was whether they were worthless altogether. Rice husks had a value, and cost £5 9s. 8d. per ton at Hull, so that it would appear they had a value and were not, therefore, a worthless ingredient. On that ground he submitted that the summons would have to be dismissed.

A member of the defendant firm gave evidence to the effect that it was the custom in the milling trade to mix sharps, as in this case, but these were always sold as "mixed," and not pure wheaten sharps. They believed that rice husks had a food value, but when they were mixed with the sharps they charged less for the mixture.

The Chairman said the Bench had come to the conclusion that there could be no doubt that there had been adulteration, and agriculturists ought to be protected against that sort of thing. If they bought sharps they should receive sharps. As this was the first case of the kind to come before the Bench, they had decided to deal leniently with the four defendants, and a fine of £5 each, together with costs—£3 each—would be imposed.

APPEAL CASE UNDER THE RAG FLOCK ACT.

CHADWICK *v.* KERSHAW.

ON June 30th an appeal was made to the Bucks Quarter Sessions at Aylesbury by Messrs. H. E. Kershaw, Ltd., who, on May 29th had been fined £10 by the Wycombe Borough Magistrates for selling flock which did not conform to the standard of cleanliness prescribed by the Rag Flock Regulation of 1912. The sample taken contained 243 parts of soluble chlorine per 100,000, as compared with the permissible amount of 30 parts per 100,000.

Mr. Fox Davies, for the Borough Council, said that what the Court below had found was that the sample taken was not up to the Government standard of cleanliness, and that there were in it jute, coconut fibre, and pieces of woven material. If the sample had been all coconut fibre, no matter what its state of cleanliness, it did not come under the Act, but what the respondent maintained was that as woven material was present, it came under the Act, and it was on that ground that the Court below had convicted. The defence, he understood, was that the jute fibre was fed into the machine on pieces of Hessian cloth, and their contention was that it was not rag. He would contend, however, that if only a small portion of woven material were found, then it became rag flock and came under the Act.

Mr. A. P. Davson, F.I.C., said that he had analysed a sample of the material and found it to contain 243·0 parts per 100,000 of soluble chlorine. In that sample he had found about 8 per cent. of pieces of material which had evidently been woven. He had analysed coconut fibre, and his average results showed 70 parts of chlorine per 100,000. The composition of the sample might fairly be taken as: Coir fibre, 19 per cent.; pieces of spun and woven material, 8 per cent.; and jute fibre, 73 per cent. He came to the conclusion that the remainder of the jute material was probably woven, because the pointed ends which were common in raw jute could not be detected.

He was asked in cross-examination whether he was aware that coir fibre was steeped in tidal waters for a year or 18 months, and that this would account for the salt present in it, and that in this coir there was as much as 640 parts of chlorine per 100,000. Mr. Davson replied that neither his analysis nor that of others supported this assumption.

Several flock manufacturers gave evidence to the effect that they had no hesitation in saying that the material in question had been made out of old bagging, or sacking. It had been "pulled" in the ordinary way, but was unwashed. Commercially it was not possible to make flock out of fibre.

Mr. C. A. Mitchell said that he had analysed two samples of coconut fibre—one prepared at Mitcham and the other imported from Ceylon. These contained 14 and 19 parts of soluble chlorine per 100,000 respectively. He had also analysed the actual coir fibre in a sample of the mixture sold by the defendants, and had found it to contain 17 parts of chlorine per 100,000. Hence, it could not have been the source of the excessive chlorine in the material.

A manufacturer of coir fibre stated that the average annual amount of coir fibre imported from India was 300 tons, whilst that from Ceylon was 11,000 tons per annum. Ceylon fibre was not treated in salt water, but in mountain streams. Witness added that he was only giving evidence because an unmerited slur had been cast on coconut fibre, which, if not contradicted, would be injurious to the reputation of the fibre used for bedding and upholstery purposes.

Mr. Robertson, for the defence, stated that the only woven material present in this material was the long strip of washed Hessian cloth, which was used as a

carrier for introducing the said jute fibre into the machine, and he called the defendants to give evidence to this effect.

The foreman at the works, also called, said that the material consisted of 50 per cent. of coir fibre, 45 per cent. of flax and hemp waste, and 5 per cent. of jute.

Dr. J. F. Shaw, A.R.C.S., called for the defence, said that he was very familiar with jute and that the sample produced was composed largely of raw jute fibre and coir fibre, with a few small strings derived from the Hessian cloth. He had not detected any trace of flax. He was familiar with the methods of preparing coir fibre in Madras, and understood that a satisfactory condition could not be obtained except in tidal waters.

Witness was proceeding to give text book figures as to the amount of chlorine in Indian coir, when exception was taken to this evidence by Mr. Fox Davies.

Mr. Fox Davies maintained that he had proved beyond doubt the same arguments that were put forward in the case of *Cooper* v. *Swift*. The analysis showed that there was woven material in the sample, and by putting in even 8 per cent. of woven material the appellants had brought it within the definition of the Act.

After consultation, Lord Parmoor, the Chairman, said that they regarded the case as one of great importance. They accepted the evidence that the Hessian cloth was only used as a carrier to take the material through the machine. Although the proportion of woven material was found to be very small, they felt that it did come within the Act; therefore the conviction of the Court below must be upheld.

Mr. Robertson thereupon applied to the Court to state a case for the High Court, and Lord Parmoor said that they would agree to do this.

Government of Madras.

REPORT OF THE CHEMICAL EXAMINER FOR THE YEAR 1923.*

THE report of Dr. Clive Newcomb on the work of the Chemical Examiner's department shows that 3973 analyses and examinations were made, as against 3824 in 1922. The number of general analyses increased from 570 to 688, mainly owing to work for the Customs department, and a fresh record was established for the number of stain cases (1655 as against 1583 in 1922).

HUMAN POISONING CASES.—In the 204 cases of suspected human poisoning investigated, poison was detected in 92, giving a percentage of detections of 45·1 per cent., as compared with 49·1 in 1922.

The poison most commonly found was again mercury, with 27 cases, and arsenic next, with 20 cases. Opium was found in 12 cases, aconite in 11, oleander in 6, strychnine in 5, and atropine in 4. There was one case each of powdered glass, nitric acid, lysol, and kerosene oil.

Datura, which was found in 4 cases, is not a very common poison in Madras, and in most instances it is used as an aid to robbery. In one of the cases the drug was given in coffee, and the victim, feeling giddy some 20 minutes later, suspected foul play and reported the matter to the station police, who arrested the two accomplices. On them were found three packets of yellow seeds, and from each of these a poisonous mydriatic alkaloid was extracted. The victim recovered after 5 days in hospital.

* G.O. No. 693, P.H., May 3, 1924.

In another case, a woman took datura for "stomach ache," and died 12 hours later, in spite of medical treatment. A mydriatic alkaloid was found in the stomach and contents, but not in the urine, which was collected *post-mortem*.

In the case of aconite poisoning a man was given a sweetmeat made of sugar and coconut and shared this with an acquaintance. Both were taken very ill with typical symptoms of aconite poisoning, but recovered under treatment. The vomits of both victims yielded, on extraction, a substance giving the reactions of an alkaloid, fatal to a frog, and, when applied to the tongue, producing tingling, followed by numbness. This was reported as a poisonous alkaloid giving the physiological reactions of aconitine. In this case the question arose whether it was likely that anyone would go on eating a substance the poisonous nature of which was indicated by the tingling of his tongue. When one tastes aconite, however, the tingling does not come on for some ten minutes, and full numbness often does not develop for half an hour or longer.

In another a man died after eating a curry. The curry contained an alkaloid giving the physiological reactions of aconite, and the symptoms of the victim were those of aconite poisoning. No aconitine could be detected, however, in the stomach or intestines, and it is suggested that the case should be put on record as one where aconitine was not found.

CATTLE POISONING.—Forty cases were investigated, and poison was detected in 17. The active principle of yellow oleander (thevetin) was the poison most commonly found (8 cases), and arsenic was second (7 cases). Mercury was found in 2 cases.

STAIN CASES.—During the year 380 cases were investigated for blood or semen or both. Of these, 353 (with 1549 articles) were examined for blood alone, and it was detected in 306 cases and 1050 articles. Of the 1018 specimens of blood stains submitted to the Imperial Serologist, at Calcutta, 924 were reported to be human blood, 8 ruminant animal blood, 2 bird's blood, 5 non-mammalian blood, and 7 blood non-human. In 65 cases the origin of the blood could not be determined, and in 17 cases the amount of stain was insufficient for the tests.

In one of the cases two pieces of white-washed wall were submitted on which were, what at first sight, appeared to be two prints of blood-stained hands left behind by the murderer. The prints on examination, however, did not give any of the reactions for blood.

This raised the question of the possibility of blood stains on white-washed walls becoming altered in the course of a few weeks, so that they would no longer answer to the reactions for blood. Stains were, therefore, made on a white-washed wall protected from direct sunlight, and exposed for a year at 25° to 35° C.

The appearance of the stains did not alter after the first 48 hours, and up to two months no difficulty was experienced in getting any of the reactions for blood. After a year the stains still gave a blue coloration with guaiacum and turpentine, and when treated with 10 per cent. potassium cyanide solution and ammonium sulphide and examined under the microscope, still gave the spectrum of cyano-hæmoglobin, though the colour was not the bright rose-pink of fresh stains, but of a browner shade. The reduced hæmatin spectrum could not be obtained after this interval, even when the stain was soaked for some time in water before boiling with potassium hydroxide and reducing with ammonium sulphide. As the other test is conclusive, it seems a safe inference that a blood stain on white-wash, if kept dry, will be detectable up to one year, at least.

SEMINAL STAINS.—In all the seminal stain cases Florence's test in the form described in last year's report (ANALYST, 1923, **48**, 490) was used. Certain stains,

originally proved to be seminal, were kept for over a year in an envelope, and, although no spermatozoa could be found in them after they were a day or so old, they still gave a pronounced reaction in Florence's test after the whole period.

Florence's reagent has been found to be liable to lose some of its iodine if kept long, and should be made up fresh, or re-saturated with iodine, every few months. The reagent was also found useful in showing up the nature of paste stains, which may resemble seminal stains.

Ministry of Health.

INTERIM REPORT OF THE FOOD PRESERVATIVES COMMITTEE ON THE TREATMENT OF CHILLED BEEF AND OTHER FOODS WITH FORMALDEHYDE.*

THE Departmental Committee on the Use of Preservatives and Colouring Matters, appointed in July, 1923, was requested to make a special report on a process (named after its inventor the Linley Process) for the preservation of chilled beef by means of formaldehyde, but decided to issue an interim report dealing with the use of formaldehyde for food in general, and to devote special attention to its particular application as a preservative for chilled beef.

It is pointed out in the report, which is dated June 12th, that formaldehyde, when ingested, exerts an irritant action upon mucous membranes, and, after prolonged use, appears to cause inflammatory changes in the liver and also in the kidneys, where a portion of it is excreted. In the body it is partly oxidised to formic acid and partly excreted unchanged; in its passage through the body it combines with tissue proteins, and this combination renders its excretion slow, so that it is probably cumulative, and this feature makes its use as a preservative of food particularly objectionable. Formaldehyde may retard digestion, even when present in food in comparatively great dilution, and it combines with the protein constituents of foods, forming a compound which is less digestible than the original substance. Even if this formaldehyde-protein compound is ultimately digested and the products of digestion are absorbed, it is possible that these products are abnormal in character, and present knowledge does not justify us in asserting that they could be regarded as unobjectionable and without effect upon the normal metabolic processes of the body. On the contrary, recent advances in our knowledge of biological processes point to the far-reaching importance of the effects produced upon metabolism and nutrition by extremely minute amounts of substances ingested and absorbed, and, in view of this, a substance such as formaldehyde, which is known to produce gross changes in the composition of food materials and to be inimical to life, even in high dilution, must be regarded with grave suspicion in regard to its possible ultimate effects upon the normal processes of metabolism.

Formaldehyde may apparently be used for other purposes than those of merely preserving fresh food. Dr. (now Sir) G. S. Buchanan in a report to the Local Government Board, 1909, refers to its possible use for concealing incipient decomposition and for hiding any smell of staleness or putrefaction. Quite

* H.M. Stationery Office. 1924. Price 2d. net.

apart from the question whether formaldehyde is undesirable as a preservative for fresh food, its use for the purpose of foisting upon the consumer an article which has already begun to decompose is unquestionably reprehensible.

From the evidence given before the Committee, it would appear that the undesirability of using this substance as a preservative is generally recognised by traders. When formaldehyde is added to food nowadays, it is usually done by irresponsible persons, and its use as a food preservative appears to be comparatively rare and sporadic. Although butchers are being supplied, to some extent, with a solution of formaldehyde for treating the surface of meat, it is possible that they may be unaware of the material they are using. It may be added that, with scarcely an exception, every country which has made legal enactments or regulations in connection with the purity of its food supply has prohibited the addition of formaldehyde to food, either specifically, or implicitly in a general prohibition of preservatives.

Hence, having regard to the interests of the trader and of the consumer, the Committee have no hesitation in recommending that the addition of formaldehyde or any of its derivatives to food or drink should be absolutely and specifically prohibited.

The Linley Process.—The object of the Linley process is to enable beef to be carried chilled at a temperature of about 29° to 30° F. from distant countries, such as the Argentine and Australia, so as to arrive in this country in good condition and free from moulds which are apt to develop on the surface of lightly chilled beef after the lapse of a certain time. The process was in operation some years before the war in connection with the transport of chilled beef to this country from the Argentine, but by no means all of the Argentine chilled beef sent here was treated by the process. The Linley apparatus was installed in several abattoirs in the Argentine, but none of the abattoirs in that country controlled by United States firms ever adopted the process, and without its aid these firms were able to export chilled beef to England successfully. During 1909 and 1910 five trial consignments of chilled beef treated by the Linley process were sent from Australia, but after that date shipments were discontinued.

Early in 1923 the Argentine Government had under consideration the question as to whether the Linley process should be recognised, but, before making any pronouncement on the matter, consulted the British Government, and were informed that the revival of the process would not be in the interest of the consumer. In a decree dated February 20, 1923, the Argentine Government prohibited the treatment of meat by the Linley process.

The chief reason given for desiring the resumption of the process is that it will enable the establishment of a trade in chilled beef from Australia, since, under present conditions, the journey from Australia is too long to permit the successful transport of chilled beef to this country without the aid of some such process. For the information of the Committee, and in order to demonstrate the feasibility of carrying chilled beef treated by the Linley process from Australia, a firm of importers interested in the process offered to arrange for a trial consignment to be sent to London. It proved impossible to make the necessary arrangements in Australia, and the consignment was shipped from New Zealand instead. On April 1 the consignment arrived at the Port of London; the hold was opened on April 3, and the beef was inspected by the Committee as it was removed from the hold. The quarters were frozen hard, and there were copious growths of mould upon them. The whole consignment was subsequently condemned by the Port Medical Officer as unsound and unfit for food, and was surrendered by the importer for destruction.

Samples were taken from the exposed surface of muscular tissue of both hind and forequarters and were examined by Dr. G. W. Monier-Williams, for the presence of formaldehyde. The method used was that elaborated by Dr. Schryver and described in the Local Government Board's Food Reports, No. 9, 1909. The amount of formaldehyde found was relatively small, but it is noteworthy that traces were detected as deep as 30 millimetres from the surface of the meat, showing that formaldehyde penetrates into the meat with ease and does not confine itself merely to the surface. Analyses made on behalf of the Local Government Board of chilled beef treated by the Linley Process and imported into this country before the process was discontinued also demonstrated the readiness with which formaldehyde penetrates into the meat in this process.

The inventor ascribed the failure of the experiment to several causes, the chief of which were that the cattle had not been properly slaughtered and that sufficient care was not taken in the subsequent dressing and handling of the carcasses, that the meat had not been properly hung, and that the fan for circulating the air in the hold had not been working effectively.

The Committee think it fair to state that the conclusions at which they have arrived have not been influenced by the failure of this experiment.

If it were justifiable to assume that formaldehyde was sufficiently unobjectionable to enable it to be tolerated in small quantities in foods, it might be possible to permit its use in connection with chilled beef and to prescribe a limit for the amount which might be present, say, 2 to 3 parts per million in a surface layer of beef 7 to 10 millimetres thick. There would, however, be considerable practical difficulties in the equitable enforcement of such a prescription.

Moreover, declaration would be a necessary corollary to treatment with formaldehyde, and meat traders in this country would have to be allowed to treat their beef in similar fashion, if they so desired.

It appears possible that a section of the trade might benefit financially by the treatment of chilled beef with formaldehyde, but we think that the true interests of the trade as a whole would not be furthered by a revival of the practice. There appears to be much in the statement that the treatment of beef with formaldehyde may correct, in part at any rate, improper and defective methods of preparation, handling, storage, and transport, but it is undesirable that preservatives generally should be used to support the perpetuation of bad methods in dealing with foodstuffs. The Committee are satisfied that the consumer will not be prejudiced by their non-interference with existing conditions in the meat trade, and that no ultimate advantage to the consumer would accrue from a revival of the Linley Process.

Having regard to all the information which has been given to them, they are of opinion that no exception in favour of the Linley Process should be made from their general condemnation of the use of formaldehyde in any article of food or drink.

Deaths.

WE regret to have to record the deaths of two of our Members:

Sir George Beilby, August 1st.

Lionel Gowing-Scopes, August 16th.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Estimation of Moisture in Wheat and Flour. H. Snyder and B. Sullivan. (*Ind. Eng. Chem.*, 1924, **16**, 731, 744.)—In twenty-eight comparative estimations of moisture in flour, 1·43 per cent. more moisture was found by drying the samples for five hours at 105° C. than when they were dried for the same length of time in an ordinary water-oven. A second similar series of tests showed that drying at 100° C. under reduced pressure (600 to 750 mm. "vacuum") yielded 1·67 per cent. more moisture than was found by drying in the water-oven. W. P. S.

Estimation of Egg Solids in Alimentary Pastes (Noodles). R. Buchanan (*J. Assoc. Off. Agric. Chem.*, 1924, **7**, 407–424.)—Juckenack's method, consisting in estimation of the phosphoric acid in the hot alcoholic extract, was, until recently, regarded as the most reliable procedure for the estimation of eggs in egg products, but Rask, Hertwig (ANALYST, 1924, 37) and others find that this method results in incomplete extraction. More complete extraction is effected either by digestion, first with hot ammoniacal alcohol and afterwards with ether (Rask and Phelps), or by digestion with 70 per cent. alcohol, followed by extraction with a mixture of alcohol and ether (Hertwig). Hydrolysis by means of hot hydrochloric acid and extraction of the fat from the resulting solution has been suggested, but organic phosphoric acid is decomposed in this way.

Alimentary pastes have been prepared from semolina, flour, whole eggs or yolks, either dried or frozen, and dried albumin, mixed in various proportions, the resulting products being analysed for lipoids and lipoid P_2O_5 by the Rask and Phelps and the Hertwig methods, and for the moisture, total nitrogen, water, soluble nitrogen, and water-soluble protein nitrogen precipitable by 40 per cent. alcohol. Ratios between certain of these substances, mulplied by 100, as proposed by Hertwig, distinguish clearly between whole egg and yolk noodles, and also between these and flours and semolinas. The recovery in the whole egg and yolk noodles of the lipoid P_2O_5 of the ingredients ranged from 81·3 to 104·5 per cent. by the Rask and Phelps method, and from 80·1 to 105·5 per cent. by the Hertwig method. If use is made of the factor 1·1 to compensate for loss of lipoid P_2O_5 during manufacture of the noodles, a close approximation is attained to the actual whole egg and yolk solids content. The formulae and basic values employed in the calculations, which are those of Hertwig, all refer to dry matter and are as follows:—Average percentages of lipoid P_2O_5 in flours, whole eggs, and commercial yolks are respectively 0·055, 1·38, and 1·78; percentage of whole egg solids in any sample of noodles is thus given by $\frac{(A-0{\cdot}055)\,100}{1{\cdot}38-0{\cdot}055}=75{\cdot}5\,(A-0{\cdot}055)$, where A represents the percentage of lipoid P_2O_5 found, multiplied by 1·1. Similarly,

for samples containing commercial yolk, the percentage of commercial yolk solids will be 58·0 (A − 0·055).

Noodles stored for about a year in either sealed jars or paper boxes showed considerable and similar losses in lipoid P_2O_5, but only slight change in the proportions of water-soluble protein nitrogen precipitable by 40 per cent. alcohol.

T. H. P.

Separation of Trimyristin from Milk. M. Piettre and C. Roeland. (*Compt. rend.*, 1924, **278**, 2283–2285.)—When milk fat is dissolved in a mixture of ether and alcohol and allowed to evaporate slowly, well-defined needle-shaped crystals, melting at 52–54° C., are obtained in 24–48 hours; these have been identified as trimyristin. The crystals are insoluble in water, slightly soluble in alcohol, and readily soluble in ether; they may be purified by recrystallisation from alcohol-ether, about 2 grms. of crystals being obtainable from 1 litre of milk. The best procedure is to mix the cream from 500 c.c. of fresh milk with 200 c.c. of Adams' mixture (alcohol 75 per cent., 1000 c.c., ether 1100 c.c.) and, after separation, remove the upper layer (which consists of an alcohol-ether solution of the fat), filter it, and allow it to evaporate at 8 to 10° C. H. E. C.

Estimation of Raffinose in Sugars. Proportion of Raffinose in Molasses. E. Saillard. (*Comptes rend.*, 1924, **178**, 2189–2192.)—In test experiments with 7 to 8 per cent. solutions of pure raffinose inversion was found to be (by Clerget's method) 51·56 at 20° C. Taking the inversion of sucrose as 34 at 20° C., S the percentage of sucrose, γ that of raffinose, A the direct polarisation, B polarisation after inversion and C the sum of A and B, $A = S + \gamma$, $B = -0{\cdot}34S + 0{\cdot}5156\gamma$; whence, sucrose $= C - 0{\cdot}4844A/0{\cdot}8556$, and hydrated raffinose $= A - S/1{\cdot}57$. In the case of molasses sucrose is usually calculated from the formula $S = (A + B) \times 100/144 \times 1/2t$. As a rule A is greater than S, and the nitrogen and raffinose content influence this difference, raffinose being probably formed to a greater extent at the end of the season, or in the beet silos. D. G. H.

Estimation of Reducing Sugars and Sucrose. W. Thomas and R. A. Dutcher. (*J. Amer. Chem. Soc.*, 1924, **46**, 1662–1669.)—The method of Benedict and Osterberg (*J. Biol. Chem.*, 1918, **34**, 195) for the estimation of sugars in urine has been adapted, with modifications, to the estimation of reducing sugars and sucrose in plant extracts. This colorimetric method is superior to the gravimetric, volumetric and optical methods for the estimation of small amounts. The following reagents are necessary:—(1) The picrate-picric solution.—To 500 c.c. of 1 per cent. sodium hydroxide solution are added 36 grms. of picric acid (dried at 60° C. and purified) and 400 c.c. of hot water. The mixture is shaken, cooled and diluted to 1 litre. (2) The mercuric nitrate solution.—A solution of 110 grms. of mercuric oxide is added gradually to 80 c.c. of concentrated nitric acid, stirred, heated to boiling, cooled, and to it 30 c.c. of 5 per cent. sodium hydroxide solution are added, and the whole made up to 1 litre. A representative sample (about 50 grms.) of the plant parts is plunged into 95 per cent. boiling

alcohol free from acids and aldehydes, filtered, washed with 75 per cent. alcohol, dried in an oven at 70° C. and pulverised. The sugars are removed by extracting the powdered plant in a Soxhlet apparatus, using the alcoholic filtrate, until the extract is colourless. A 20 c.c. to 100 c.c. aliquot portion, containing from 0·025 to 0·150 grm. of sugars, is evaporated to remove the alcohol, and the residue dissolved in 100 c.c. of water. A slight excess, say 10 c.c., of mercuric nitrate solution is added; then small quantities of solid sodium bicarbonate are added gradually, with stirring, until all frothing ceases. The solution must be kept just alkaline. It is then filtered into a 250 c.c. flask, the precipitate washed with 5 per cent. sodium bicarbonate solution, and the solution and wash liquid made up to the mark and shaken. From 30 c.c. to 50 c.c. are treated with 0·3 to 0·5 grm. of zinc dust in a 75 c.c. test-tube, one drop of concentrated hydrochloric acid added, the tube stoppered and left for 15 minutes and the contents then filtered. From 5 c.c. to 10 c.c. of the filtrate, tested to ascertain the absence of mercury, are pipetted into a 50 c.c. pyrex test-tube, 10 c.c. of the picrate-picric solution added and 2 c.c. of a 25 per cent. sodium carbonate solution. Tubes containing this solution and a standard colour solution simultaneously prepared (10 c.c. of standard glucose solution, 10 c.c. of the picrate-picric solution and 2 c.c. of the sodium carbonate solution) are plugged and immersed for 20 minutes in a water-bath at 95°, removed, cooled to room temperature, and the liquids compared in any standard colorimeter. The products of hydrolysis of sucrose are equivalent amounts of glucose and fructose, and, since sucrose up to 0·1 *M* concentration has no picrate-picric colour, it is possible to estimate the quantity present in solution not exceeding this, by the increase in colour after inversion. Mercuric nitrate is better than lead acetate as a clarifying agent. P. H. P.

Estimation of Starch. W. Thomas. (*J. Amer. Chem. Soc.*, 1924, **46,** 1670–1675.)—A colorimetric method for the estimation of starch by means of conversion into glucose and maltose by the mixed enzyme taka-diastase—secreted by *Aspergillus oryzæ*—without a secondary hydrolysis is described. Under the conditions of conversion, the ratio of dextrose to maltose is remarkably constant, and the analytical error introduced by regarding this ratio as 2·0 is very small, since the picrate-picric reducing ratio of dextrose to maltose is relatively high. From 1 to 4 grms. of the vacuum-dried residue, freed from sugars as above, is gelatinised by heating to boiling for 40 minutes with 200 c.c. of water, with stirring, cooled to 38° C. and incubated at this temperature, with occasional stirring for 24 hours with 0·1 grm. of the enzyme preparation and 2 c.c. of toluene. Toluene lost through evaporation is replenished. The solution is then heated on a water-bath to boiling for 15 minutes to render the enzymes inactive, and the residue is filtered and thoroughly washed by decantation with water into a carefully calibrated 250 c.c. flask. An aliquot portion (30 c.c.) is taken, 5 c.c. of the mercuric nitrate reagent and sodium bicarbonate are added, followed by filtration and treatment with zinc and hydrochloric acid, as in the preceding abstract. One to 5 c.c. is diluted to 10 c.c. in a pyrex test-tube, reagents are added, and the solution

is compared with a standard colour solution, as before. The dilution factor for mercuric nitrate solution is used, and a blank estimation on 0·1 grm. of taka-diastase is carried out simultaneously. The recovery by this method of 0·25 grm. of potato starch added to (1) spurs and (2) leaves of apple trees in 6 experiments was from 98·4 to 99·4 per cent. Experiments on a secondary hydrolysis with hydrochloric acid and a method for the estimation of other "reserve" polysaccharides are described. P. H. P.

Detection and Estimation of Small Quantities of Cyanogen in Wine. F. Mach and M. Fischler. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 329–337.)—Kolthoff's method (*Zeitsch. anal. Chem.*, 1918, **57**, 3) is the most sensitive for the detection of the cyanogen which may be present in wines after fining with ferrocyanide and iron. To 10 c.c. of the wine are added 20 mgrms. of ferrous sulphate and 10 drops of a solution containing 8 grms. each of sodium carbonate and bicarbonate in 100 c.c.; the mixture is allowed to stand for half an hour and then acidified with sulphuric acid; 2 mgrms. of cyanogen per litre are thus readily detected. For the estimation the aeration method, followed by iodimetric titration, is the best; 50 c.c. of the wine are placed in each of two wash bottles connected in series with two others, the first containing 20 c.c. of 4 per cent. bicarbonate solution saturated with carbon dioxide, and the second 20 c.c. of *N* sodium hydroxide solution. A stream of air is aspirated through the bottles for an hour, and then the sodium hydroxide solution is neutralised by the addition of 20 c.c. of *N* sulphuric acid, and any excess of acid is removed by the addition of a little calcium carbonate; finally the liquid is titrated with 0·01 *N* iodine solution. Working on small quantities of cyanogen the results are always a little low, about 80 per cent. of the true quantity being indicated. Small distributing arms may, with advantage, be fitted to the inlet tubes of the wash bottles. Solutions of tartaric or acetic acids, or wine, always form small amounts of hydrocyanic acid with ferrocyanide at summer temperatures, a trace of this acid is therefore always present after fining with this salt. H. E. C.

Hydrocyanic Acid in Distilled Wines. O. Reichard. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 339–349.)—The fining of wine by ferrocyanide, which was formerly forbidden, is now allowed in Germany; as the result of a number of experiments on distilled wines it is concluded that those fined by this substance almost invariably contain traces of hydrocyanic acid. Details are given of various experiments on the fining of different kinds of wine with ferrocyanide. The presence of traces of hydrogen cyanide cannot now be taken as evidence of the presence of spirit distilled from stone fruit; such addition can only be recognised by the identification of the characteristic aromatic compound or ester, as well as of hydrocyanic acid. The ester may sometimes be identified by shaking out with chloroform. H. E. C.

Hastening the Coloration of Lemons. F. E. Denny. (*J. Agric. Res.*, 1924, **27**, 757–769.)—Commercially mature lemons are frequently green when picked, and, in order to produce the usual yellow colour rapidly, they are placed

in rooms heated by the fumes from kerosene stoves. Investigation showed that the effective colouring agent is an unsaturated hydrocarbon, probably ethylene, although this gas was not positively identified among the products of the combustion. Ethylene in concentration 1:200,000 colours the lemons at 50–60° F. in 5 to 8 days, or in 6 to 10 days at concentration 1:2,000,000; the presence of oxygen is essential. The rate of respiration and the output of carbon dioxide are much increased by the ethylene or stove gas. The absence of physiological or explosive properties in such high dilution render ethylene quite a satisfactory agent for the yellowing of lemons, and, although a large number of other substances were found to have the property in varying degree, they all either injure the fruit or have some deleterious effect. (*Cf.* ANALYST, 1924, 284.) H. E. C.

Estimation of Essential Oils in Certain Flavouring Extracts and of Camphor or Peppermint Oil in Certain Pharmaceutical Preparations. W. W. Randall. (*J. Assoc. Off. Agric. Chem.*, 1924, **7**, 425–430.)—The method in which the essential oil or camphor is precipitated from its alcoholic solution by means of acidified calcium chloride solution and then shaken with petroleum spirit boiling at 40° to 60° C. is attended by various difficulties and inaccuracies, which the author now shows how to avoid. Use is made of: (1) A solution of density about 1·30, prepared by dissolving calcium chloride in water, mixed with about 4 per cent. of its volume of concentrated hydrochloric acid, and filtering perfectly bright the next day; a large bottle provided with a siphon, at the end of which is a tap with a drawn-out tip, forms a convenient container. (2) Redistilled light petroleum spirit boiling at 40° to 60° C. (3) Calibrated 8 per cent. Babcock milk-test bottles. (4) Pipettes of 10, 5, 1 and 0·5 c.c. capacity, the last two being of thick-walled tubing of 2 to 3 mm. bore, drawn out to a long tip, which is bent slightly and filed flat at the end so as to deliver the petroleum spirit smoothly against the inner wall of the neck of the milk-test bottle.

With extracts containing about 5 per cent. of oil, such as orange or lemon extract, or about 3 per cent. of oil, such as peppermint or anise extract, 10 c.c. are pipetted into a clean Babcock bottle, which is then filled to the shoulder with the calcium chloride solution and shaken; exactly 1 c.c. of the gasoline is next run in, care being taken that the tip of the pipette does not touch a wet spot on the neck of the bottle. The latter is stoppered tightly with a soft cork previously wetted with the calcium chloride solution, and afterwards shaken violently for 1 or 2 minutes and centrifuged at fairly high speed for 2 minutes. The cork is then removed and enough calcium chloride solution quickly added to bring the whole petroleum spirit column within the graduated part of the neck, the bottle being then corked, inverted, shaken vigorously and centrifuged again for 3 to 5 minutes. The bottle is then placed upright and the volume of the petroleum spirit column read, the mean graduation between the upper and lower meniscus being taken. The lower meniscus should be flat and sharp, any whitish "collar" indicating lack of sufficient acid in the calcium chloride solution. A parallel estimation in which '0 c.c. of alcohol are used instead of the extract serves to measure the volume of

gasoline delivered by the pipette in terms of the bottle graduation; deduction of this volume from that of the petroleum spirit and oil solution, and multiplication of the remainder by 2 gives the percentage of oil in the extract. For extracts containing 7 to 15 per cent. of camphor or essential oil, such as spirits of camphor or essence of peppermint, 5 c.c. instead of 10 c.c., and the multiplier 4, are used. For extracts containing as little as 2 per cent. of essential oil, *e.g.* nutmeg extract, not more than 0·5 c.c. of gasoline is used to 10 c.c. of the extract.

The factor used may vary somewhat with the composition of the gasoline, which should be tested by an estimation of the oil in a standard 5 per cent. orange extract. T. H. P.

Detection of Aldehydes in Ethers for Anæsthesia. E. Isnard. (*J. Pharm. Chim.*, 1924, **29**, 5–9.)—The methods given by the various pharmacopoeias for the detection of aldehydes in ether differ widely in sensitiveness, and the following three tests are suggested. (1) For ordinary purposes.—Four c.c. of rosaniline bisulphite (Schiff's reagent) added to 10 c.c. of the ether, vigorously shaken 5 or 6 times, and left for 15 minutes, should not produce a rose-violet coloration (sensitiveness 1 in 10,000). (2) For the purest ether.—Ten c.c. of ether shaken with 2 c.c. of Nessler's reagent should not give, in 5 minutes, any red-brown precipitate turning black on adding 0·1 *N* potassium cyanide solution (sensitiveness 1 in 200,000). (3) For the purest ether.—Ten c.c. of ether shaken with 3 c.c. of ammoniacal silver nitrate solution containing sodium hydroxide should not give, in 5 minutes, any brown-black coloration (sensitiveness 1 in 75,000). D. G. H.

Cascara Content of the Wood and Bark of *Rhamnus Purshiana*. R. H. Clark and K. B. Gillie. (*Amer. J. Pharm.*, 1924, **96**, 400–412.)—Physiological tests showed the average potency of extracts from the wood to be 38 per cent. and from the bark 53 per cent., and it is concluded that the wood contains sufficient of the active constituents to make it a practicable source of commercial extracts. The active principle, since complete hydrolysis does not destroy its activity, is probably not a glucoside, although possibly the hydrolytic product of one. Average values obtained for the bark (B) and wood (W) extracts were found to be:—Sp. gr., (B) 1·072 (W), 0·985; total solids, (B) 27·4, (W) 4·69 per cent.; ash, (B) 1·08, (W) 0·27 per cent.; and reducing sugars, before hydrolysis, (B) 5·15, (W) 0·947 per cent.; after hydrolysis, (B) 6·88, (W) 1·5 per cent. D. G. H.

New Reaction for Myrrh. S. Dezani. (*Giorn. farm. Chim.*, 1924, **731**, 5; *Chem. Abstr.*, 1924, **18**, 1177.)—Myrrh gives an intense violet to azure blue colour, persisting for several hours, when treated with a reagent consisting of chloroform (8 parts), glacial acetic acid, (3 parts), and ethyl acetate (1 part), followed by one or two drops of strong sulphuric acid. The reaction is sensitive to 0·5 mgrm. of myrrh. Nineteen other gums and resins examined gave either no coloration or else fugitive yellow, green or red shades.

Identification and Classification by Chemical Methods of Drugs containing Tannin. I. Application of the Goldbeater's Skin Test. C. J. Jordan and A. H. Ware. (*Pharm. J.*, 1924, **113**, 102–104.)—The goldbeater's skin test (*Biochem. J.*, 1922, **16**, 4; ANALYST, 1924, **49**, 25) is regarded as the best single general test for tannins so far devised. If the drug extractive stains the skin at all, the stain will be obtained before immersion in the iron solution, and is practically identical with that obtained by the treatment with ferrous sulphate and 5 per cent. hydrochloric acid. Extractives of certain drugs may give stains which mask, to a certain extent, the tannin reaction, but it is usually possible to distinguish the dyeing and tannin effects, and, at the same time, obtain valuable indications of the presence of natural vegetable dyes. "Dye" stains are not usually greatly enhanced by the iron treatment. Tables are given of (1) drugs giving positive results characteristic of tannin; (2) drugs giving poor or dubious results; and (3) those definitely showing absence of tannin. All the B.P. Codex tannin drugs fall within Class (1) except pyrethrum, which is in Class (2). D. G. H.

New Classificatory Tests for Drugs containing Tannins. A. H. Ware. (*Pharm. J.*, 1924, **113**, 104–105.)—(*a*) *Re-investigation of Stiasny's Test for Phlobotannins.*—The test, carried out as follows, was applied to all the substances classified for the goldbeater's test (see preceding abstract), with confirmatory results. A few drops of 40 per cent. formaldehyde solution are added to a few c.c. of extractive, and subsequently the same number of drops of 10 per cent. dilute hydrochloric acid, and the whole boiled for 1 minute (excess of formaldehyde and acid should be ensured). After cooling, and filtering, the precipitate is treated on the paper with successive portions of water, 90 per cent. alcohol, and aqueous alkali, when a copious residue indicates phlobotannin. To the filtrate are added 1 or 2 drops of 10 per cent. ferrous sulphate solution, and then, drop by drop, a 5 per cent. potassium hydroxide solution (unless the content of chromogenic phenol is very small or none, no precipitate of ferroso-ferric hydroxide occurs). Iron-greening filtrates are yielded by many anthoxanthine bodies; iron-browning filtrates by certain substances containing anthroquinone derivatives; and iron-blueing filtrates by substances containing gallic acid, gallotannins and certain anthocyanins.

(*b*) *New classificatory tests for Tannins with Iodine and Ammonia.*—On boiling the tannin-containing drug extractive with a slight excess of alcoholic iodine solution the following reactions may occur; (1) Phlobotannin drugs yield copious coloured precipitates, the filtrate, in the absence of phenolic bodies, becoming practically colourless on addition of ammonium hydroxide solution, and the precipitates, on treatment with ammonium hydroxide solution, being either soluble, with formation of coloured solutions, (*e.g.* iron-colouring bodies not precipitated by Stiasny's reagent, and non-iron colouring bodies); insoluble (separated into iron colouring and non-colouring); or partly soluble and insoluble. (2) Phlobotannin bodies containing gallic acid or gallotannin, or both, also give precipitates, but the filtrates are highly coloured on addition of ammonium hydroxide solution. (3)

Bodies containing only small quantities of tannin, characterised by reactions due to substances other than tannins. D. G. H.

Assay of Valerian Root and certain other Aromatic Drugs. K. Bullock. (*Pharm. J.*, 1924, **113**, 109–113.)—Fifty grms. of the drug (No. 40 powder) are mixed in a mortar with 50 c.c. of water and 80 grms. of dry sand, extracted for 4 hours with petroleum spirit boiling below 40° C., the solution dried over anhydrous sodium sulphate and filtered, and the bulk of solvent distilled. The residue (3 to 4 c.c.) is dried to constant weight (A) in a current of dry air, dissolved in 15 to 20 c.c. of 70 per cent. alcohol, the fat filtered off, the filtrate and washings transferred to a separating funnel, 20 c.c. of chloroform added, and the whole shaken. Excess of water containing sodium sulphate is added, the whole shaken, the chloroform layer separated, and the diluted alcohol shaken out with 2 successive portions of 5 c.c. of chloroform. The solution is dried, filtered, the solvent removed as before, and the residue weighed (B). Then the percentage of fat extracted is 2(A–B), and that of oleo-resin present in the drug 2B. A complete separation of the oil and resin in the unchanged condition is difficult to bring about, chiefly on account of resinification, but the best method was found to be to dissolve the oleo-resin in chloroform (which prevents bumping and local heating at the beginning) to add excess of glycerin (to prevent oxidation and to bring over last traces of oil; also it contains no water to cause hydrolysis of borneol esters), and to distil *in vacuo*. It is sufficient to distil 30 to 40 c.c. of glycerin for 1 grm. of oleo-resin. The quantity of water present in the drug at the time of extraction has an important effect on the yield of oil, and the fresh root must be dried or completely exhausted with alcohol of at least 98 per cent. strength, and the extract treated with petroleum spirit, preferably with the addition of concentrated hydrochloric acid. D. G. H.

Biochemical, Bacteriological, etc.

The Relation of P_H Value to Tungstic Acid Precipitation of Protein. A. T. Merrill. (*J. Biol. Chem.*, 1924, **60**, 257–259.)—Diphtheria antitoxin serum, peptone free, has been examined, and the protein-free nitrogen estimated by the tungstic acid method of Folin and Wu (*J. Biol. Chem.*, 1919, **38**, 81). At a P_H of about 5·0, which is on the acid side of the isoelectric point of serum globulin, the zone of maximum precipitation of nitrogen occurs. A value of 0·10 per cent. protein-free nitrogen is obtained in the filtrate. An estimation by the trichloroacetic acid method of Greenwald (*J. Biol. Chem.*, 1915, **21**, 61) gave identical results with those obtained by tungstic acid at the P_H of maximum precipitation. In peptone solutions the nitrogen precipitated by tungstic acid, increased considerably as the P_H decreased from 4·0 to 1·0, with a very slight reversal of precipitation at an acidity of 10 per cent. acid solution. The peptone nitrogen is only slightly precipitated at a P_H of 4·0, whilst at this acidity the protein in the serum is completely precipitated. There is no known P_H at which peptide nitrogen is precipitated separately from other nitrogen compounds in peptone solution. A

change in the proportion of tungstic acid to peptone, or the concentration of reagents in solution, affects the quantity of nitrogen precipitated. It is suggested that the tungstic acid precipitate in peptone solutions is affected by the various peptones, bases, amino acids and other organic acids probably present in the peptone. Tables show the results obtained. P. H. P.

Estimation of Iodine in Food, Beverages and Excreta. J. F. McClendon. (*J. Biol. Chem.*, 1924, **60**, 289–299.)—Other methods for estimating minute quantities of iodine are discussed and some points on the difficulties of ashing are considered. Two convenient ways of evaporating about 100 litres of water, keeping it alkaline the whole time, are given. The dry sample is ignited in a boat in a silica combustion tube having a water-cooled stopper at one end, whilst the other end passes into a sodium hydroxide solution. This solution, and the rinsings of the tube, are evaporated to dryness, and the residue powdered, and mixed in a mortar with the powdered ash. The iodides and iodates are extracted by grinding the powder in a little water. This is filtered, and an accurately measured aliquot portion (say 7·5 c.c.) is taken, neutralised with concentrated hydrochloric acid, and the volume made up to 10 c.c. This is then transferred to a separatory funnel (12 c.c.), and shaken with 1 drop of concentrated hydrochloric acid and 1 c.c. of purified carbon tetrachloride. A pink colour denotes the presence of iodate, as well as iodide, in the ash. One drop of 0·1 *N* arsenious acid added and left for 20 minutes reduces any iodate. One drop of nitrosyl sulphuric acid is added to oxidise iodide to iodine and the funnel shaken vigorously for two minutes to extract the iodine. The carbon tetrachloride is run into a glass-stoppered bottle, and centrifuged to remove water droplets. It is then put into one cup of a Bausch and Lomb micro-colorimeter, and into the other cup is put carbon tetrachloride, 1 c.c. of which contains 0·1 mgrm. of pure iodine. The iodine extracted is estimated colorimetrically, and 1 c.c. of carbon tetrachloride again added to the funnel and another extraction made. By repeated extractions practically all the iodine may be recovered, and thus the total amount estimated. Details are given for purifying the carbon tetrachloride and the iodine for the standard solution. A method for ashing foodstuff is described for the estimation of iodine in the presence of large quantities of organic matter. It is possible to detect and roughly to estimate 0·001 mgrm. of iodine in 1 c.c. of carbon tetrachloride, but 0·01 mgrm., or more, is desirable in the sample for analysis. P. H. P.

Polarimetric Method for the Estimation of Diastatic Power. H. C. Gore. (*J. Assoc. Off. Agric. Chem.*, 1924, **7**, 364–367.)—A solution of Lintner's soluble starch containing 2 grms. of the air-dry starch per 100 c.c., and an infusion of the diastatic product to be tested of such concentration that 1 c.c. corresponds with 50 mgrms. of the sample, are prepared. The initial polarisation of the mixture of starch and diastase solutions is determined by mixing 50 c.c. of the starch solution with 0·5 c.c. of concentrated ammonia solution and 0·5 c.c. of the diastase solution in the order named and reading in the polarimeter at 21° C. A mixture of 100 c.c. of the starch solution with 1 c.c. of the diastase solution is left

at 21° C. for a measured interval of time, so chosen that the fall in the polarisation is not greater than 3° Ventzke. A sample of 50 c.c. is then withdrawn, mixed with 0·5 c.c. of strong ammonia solution, left for 25 minutes, and then polarised in a 400 mm. tube at 20 to 21° C. If d represents the fall in polarisation, t the time in hours during which the action of the diastase on the starch proceeds, the diastatic activity on the Lintner scale is expressed by the formula, $L = \frac{100d}{2{\cdot}18t}$, 2·18 being the fall in polarisation observed in a 400 mm. tube produced by 1 c.c. of a diastase solution representing 50 mgrms. of a malt of diastatic power 100 Lintner acting on 100 c.c. of a starch solution containing 2 grms. in 100 c.c. for 1 hour at 21° C. Should the fall in polarisation observed be very small, the remaining solution may be digested for a suitable longer period calculated from the fall in polarisation.

If the results are to be expressed on Lintner's diastase scale instead of the Lintner malt scale, the 1 c.c. of the diastase solution should be so prepared as to represent 1·2 mgrms. of the diastase preparation to be tested. T. H. P.

Behaviour of Insulin towards Fehling's Solution. Knops-Niederhoff. (*J. Pharm. Belg.*, 1924, **6**, 160; *Pharm. J.*, 1924, **113**, 45.)—Insulin can be adjusted to the strength of a given standard by determining the equivalent of 1 unit to dextrose. The insulin is added to 100 c.c. of 1 per cent. dextrose at 38° C., kept at that temperature for 2 hours and boiled, and the proportion of dextrose left estimated by means of Fehling's solution. It was found that, in some cases, urines of patients undergoing insulin treatment contained something which hindered reduction, possibly insulin, and it was necessary to boil with Fehling's solution for 30 minutes to obtain a positive result. In such cases Pavy's solution was used for the estimation after boiling. Such urines may also fail to react to the direct test for acetone, and should be distilled, the ketonic bodies being tested for in the distillate. D. G. H.

Biochemical Detection of Galactose in mixtures of Galactose and Arabinose. M. Bridel and I. Charpentier. (*J. Pharm. Chim.*, 1924, **30**, 33–42.)—The presence of galactose in a reducing mixture containing galactose and arabinose can be shown by first measuring the fall in reducing power and rotation effected by adding emulsin to the alcoholic solution of the sugars, and then removing the arabinose by adding hydrocyanic acid in the presence of a trace of ammonia, whereby the ammonia salt of the *l*-gluconic acid is formed. The ethylgalactoside may then be crystallised out. D. G. H.

Association of Manganese with Vitamins. J. S. McHargue. (*J. Agric. Res.*, 1924, **27**, 417–424.)—The author shows a distinct parallelism between the distribution of vitamin and of manganese in plant and animal tissues. In the plant the manganese is essential for the synthesis of chlorophyll, and a relationship is indicated between its presence and the formation of vitamins. The effect

of the polishing of rice, which is well known to remove the vitamin, is shown by the following results:—Manganese in rice bran, 350; in rice polishings, 100; in unpolished rice, 25; and in polished rice, 10 parts per million. Similarly, wheat bran contains 175 parts, whole wheat 40 parts, and patent flour only 10 parts. The livers of the hog, sheep, and cow contain larger proportions of manganese than do their other organs, but experiments on cod livers showed only traces of manganese. H. E. C.

Preservation of Stock Cultures of Micro-organisms. A. C. Thaysen. (*J. Inst. Brew.*, 1924, **30**, 349.)—The two methods given are designed to maintain, for a long time, strains of micro-organisms, unimpaired in any way, without continual transfer to fresh media. For non-sporing types the following method was found satisfactory:—Sterile media (broth, wort, or whey) are inoculated with a vigorous culture of the organisms to be tested and incubated for 24 to 48 hours, till well established. Then 0·2 grm. of sterile precipitated calcium carbonate is added to each, and the tubes, after being carefully shaken, are sealed and stored at room temperature. The various types tested were:—*B. coli communis*, *B. paratyphi*, *B. fluorescens liquefaciens*, *B. phosphorescens* Bernh., *B. volutans*, *B. Kützingianum*, *Streptococcus acidi lactici*, *Streptococcus hæmolyticus*, *Staphylococcus pyogenes aureus*, a *Saccharomyces* from brewers' yeast, and a red torula. After one year all survived unimpaired in every characteristic, except *Streptococcus hæmolyticus*, which died in less than fourteen days. All the other types retained their bio-chemical and morphological properties to the fullest extent. A parallel experiment, using calcium diphosphate in place of calcium carbonate, showed that the former was not so satisfactory In testing for survival it was found necessary to keep the sub-cultures under observation for about 8 days before finally deciding whether the organism had survived or not.

For sporing organisms carefully cleaned sea-sand is used. Four or five grm. lots are placed in test-tubes and sterilised. Two or three drops of a pasteurised culture, containing a large number of spores, are dropped on to the sand and distributed evenly throughout it. The tubes are placed in the incubator till dry, when they are sealed and stored at room temperature. The types tested were:—*B. mesentericus fuscus*, *B. subtilis*, *B. amylobacter*, *B. aceto-ethylicus*, *Penicillium glaucum*, *Aspergillus glaucus*, and *Rhizopus japonicus*. All were kept for one year, with the result that the sub-cultures made from the sand cultures were found to be distinctly superior to their parent cultures in their enzymic reactions.

R. F. I.

Relation of Bacteria to Growth-promoting Substances. S. R. Damon. (*Amer. J. Hygiene*, 1924, **4**, 408–409.)—Representative organisms of the various bacterial groups—the non-spore-forming bacilli, the spore-forming bacilli, the mucoid bacteria, and the acid-fast bacteria—were grown on a vitamin-free medium, collected and desiccated, and added to the ration of rats on a diet devoid of the water-soluble vitamin. The dried organisms were added in the proportion of 2·5 and 7·5 per cent. of the ration, and the results, as shown by the weight curves

of the animals, indicated an absence of any growth-promoting substance, except in the case of the acid-free bacteria. These organisms, of which *B. timothy*, *B. smegmatis*, and the Mest Bacillus, were used, seem to contain definite growth-stimulating substances, of the type of the water-soluble *B* vitamin, as the weight curves of the animals at once closely approximated those of rats on a diet containing an adequate amount of that substance. Hence, it now seems reasonable to conclude that certain bacteria, at least, produce this vitamin in their growth processes.

Toxicological and Forensic.

Toxicity of Salts of Copper. J. Effront. (*Comptes Rend.*, 1924, **178**, 2152–2155.)—The toxicity of copper salts depends upon the power of absorption of copper by the particular foods present, and the reversibility of the absorption. One hundred grms. of pulp of potato absorbed 4·29 per cent. of anhydrous copper sulphate; leeks, 4·97; beetroot, 7·06; and turnips, 12·36 per cent., when a 7·215 per cent. solution of copper sulphate was used and the concentration of the pulp was 4 per cent. By increasing the dilution of the solution absorption is rendered more complete, and, under certain conditions, the action becomes reversible. For example, in the presence of acid, salts of calcium, and peptones the copper salt is liberated to different extents. Tryptic peptones (1 per cent.) may cause the liberation of as much as 40 per cent. of the copper absorbed. D. G. H.

Organic Analysis.

New Method of Elementary Analysis based on the Measurement of Gaseous Volumes. L. Hackspill and G. D'Huart. (*Bull. Soc. Chim.*, 1924, **35**, 800–803.)—The high results obtained by this method for the percentage of nitrogen (ANALYST, 1923, **48**, 504) are due to air adsorbed by the cupric oxide, and may, therefore, be rectified by re-oxidising the reduced copper, not in air, but in oxygen, and allowing it to cool in this gas; this oxidation is effected in the combustion tube itself. After the second evacuation (for the estimation of the hydrogen) oxygen from a cylinder is introduced by way of a lateral tube furnished with a cock, the temperature being maintained at 400° to 500° C. for some minutes; it is advisable to keep the oxide in oxygen after this treatment.

In order to reduce, as far as possible, the length of time necessary for the collection of the gases, the total volume of the apparatus has been diminished, and the Sprengel pump replaced by a mercury pump. The bore of the silica combustion tube has been reduced to 5 mm., and the substance is mixed, if necessary, with lead chromate. With these modifications the method gives good results, and the whole process occupies rather more than two hours if an accuracy of 1 per cent. suffices for the hydrogen estimation, or three hours if exact figures are required. T. H. P.

Quantitative Separation of Solid and Liquid Fatty Acids. D. Holde, M. Selim and W. Bleyberg. (*Chem. Zeit.*, 1924, **48**, 448.)—A method devised by Meigen and Neuberger (*Chem. Umschau.*, 1922, **29**, 337), depending on the solubility of the thallium salts of liquid fatty acids and the insolubility of the thallium salts of solid unsaturated fatty acids in aqueous alcohol in the presence of strong excess potassium hydroxide has been found to be, on the whole, impracticable in the form suggested. By precipitating the insoluble thallium salts with a 50 per cent. alcoholic solution of the potassium salts, however, it is possible to separate the solid saturated acids (stearic, palmitic and a mixture of both) from the liquid unsaturated acids (primarily oleic acid) quantitatively and in a pure state. The method is not suitable for the separation of solid unsaturated acids (*e.g.* erucic acid), since the thallium salts of these acids behave similarly to those of the solid saturated acids. It might, however, be used for the separation of arachidic and lignoceric acids, on the one hand, and linolic and linolenic acids, on the other hand. P. H. P.

Acetyl Value of Fats. E. Raymond and G. Glot. (*Comptes rend.*, 1924, **178**, 2098–2101.)—Zerewitinoff's method for determining the hydroxyl number of a fat by measuring the volume of the hydrocarbon formed by the action of an aliphatic organo-magnesium compound may be improved by using anisole, which is quite inert towards the Grignard reagent, as solvent for the reacting substances. If the number of mgrms. of potassium hydroxide required to neutralise the free acids of 1 grm. of the fat is denoted by n, and a is the hydroxyl value, the number of hydroxyls, x, contained in 1 grm. is given by $x = a - \frac{n}{56000} = a - 0{\cdot}0000179n$, and the acetyl value by $A = \frac{56000x}{1+42x}$. The index a is calculated by means of the expression $a = \frac{0{\cdot}000162\,VH}{pT}$, where V is the number of c.c. of methane, measured at the pressure H cm. and the absolute temperature T, liberated by p grms. of the substance.

To obtain exact results the fat must be freed from moisture by being kept in a desiccator over sulphuric acid or phosphoric anhydride for 1 or 2 days, and the magnesium methyl halide must be added gradually to the solution of the fat in dry anisole. The reaction vessel consists of a round 50 c.c. flask, surmounted by a very wide neck closed by a rubber stopper, through which pass a delivery tube connected with a Bunte burette, and a pipette fitted with a plug, so that the organo-magnesium compound may be allowed to fall, drop by drop, into the vessel. Correction of the gaseous volumes may be avoided by making all measurements at the ordinary temperature, but, if this is very low, the vessel may require gentle heating. Especially if recently prepared, the organo-magnesium compound rapidly absorbs atmospheric oxygen, but after a time this absorption greatly diminishes, so that the volume of the methane disengaged in a determination becomes constant and the use of an inert gas is rendered unnecessary. Slight

absorption of gas by the organo-magnesium compound may occur, but a correction is easily applied. The time occupied by a determination never exceeds an hour, and the results obtained agree well with those given by the ordinary methods, which are far more tedious. T. H. P.

The Reaction of Iodine with Fats. B. M. Margosches and W. Hinner. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 349–355.)—It is shown that alcoholic or dilute acetic acid solutions of iodine, which are brown, react with the unsaturated acids of fats in the presence of iodic acid as do violet solutions of iodine. Aqueous solutions of iodine in potassium iodide also react quantitatively if some potassium iodate is added; with such solutions the results obtained for the iodine absorption are identical with those with Wijs or Hübl solutions, but the time taken is longer (24 hours are sufficient). The function of the iodic acid is to remove the hydriodic acid which would otherwise interfere with the absorption by the fatty acid. H. E. C.

Influence of Various Solvents on the Hanus Iodine Values of Cottonseed and Coconut Oils. H. J. Bankston and F. C. Vilbrandt. (*Ind. Eng. Chem.*, 1924, **16**, 707–708.)—Chloroform and carbon tetrachloride are the most satisfactory solvents for use in the determination of the iodine values of oils by the Hanus method; of the two, chloroform tends to give more uniform and slightly higher results in the case of cottonseed oil, and variation in the quantity of solvent is without appreciable effect. Ether, ethyl alcohol and benzene are useless as solvents in this method. With oils of low iodine value, such as coconut oil, the effect of variation in the solvent is less marked, but chloroform is the best solvent as regards uniformity in the results obtained. W. P. S.

The Phytosterols of Wheat Endosperm. R. J. Anderson and F. P. Nabenhauer. (*J. Amer. Chem. Soc.*, 1924, **46**, 1717–1721.)—The unsaponifiable matter from the fat extracted from wheat endosperm has been examined. Wheat endosperm contains at least two different sterols, namely, ordinary sitosterol, $C_{27}H_{45}OH$, and dihydrositosterol, $C_{27}H_{47}OH$, m.p. 144–145° C.; $[\alpha]_D^{20}$, + 25·82°. The dihydrositosterol from wheat bran appears to be identical with the saturated sterol that occurs in maize endosperm. The substance exists throughout the wheat endosperm, but the bran is particularly rich in this sterol. Full experimental details are given. A method for separating sitosterol from the saturated sterol by means of acetic anhydride and sulphuric acid with the acetyl derivative is described. The method of Bondzynski and Humnicki (*Z. physiol. Chem.*, 1896–1897, **22**, 396) for separating coprosterol and cholesterol, after brominating a mixture of the two, could not be satisfactorily adapted for the wheat bran sterols. P. H. P.

Estimation of Anhydrous Soap in Lubricating Grease. B. Joachim. (*Ind. Eng. Chem.*, 1924, **16**, 725–726.)—*Free Fatty Acid.*—Ten grms. of the sample are heated with 10 c.c. of alcohol and 90 c.c. of benzene, the hot solution is filtered, and the insoluble portion washed with hot benzene. Twenty-five c.c. of neutral

50 per cent. alcohol are added to the filtrate, and this is titrated with 0·1 *N* sodium hydroxide solution. *Fatty Acids from the Soap.*—Ten grms. of the grease are stirred and heated on a water-bath for twenty minutes with 3 grms. of finely-powdered potassium hydrogen sulphate, the mixture is cooled, extracted with benzene, the benzene extracts are filtered, the filtrate is diluted with alcohol, and titrated. The difference between this titration and the first is a measure of the fatty acids of the soap. *Soap.*—The neutralised solution of the fatty acids is diluted with water, the aqueous layer separated, evaporated to remove alcohol, and then acidified; the liberated fatty acids are extracted with ether and titrated with 0·1 *N* sodium hydroxide solution. This neutral solution is evaporated, and the residue of soap dried and weighed. If metallic soaps are present in the grease, the quantities of the metals must be estimated, and the corresponding quantities of metal soaps then calculated. W. P. S.

Relation between the Durability and Chemical Composition of Woods. L. F. Hawley, L. C. Fleck and C. A. Richards. (*Ind. Eng. Chem.*, 1924, **16**, 699–700.)—The relative resistance to decay of certain woods can be explained by the fact that they contain a larger quantity of substances which are toxic to fungi than do the less durable woods. The aqueous extracts of chestnut, oak, black locust, redwood, etc., are more toxic than are the extracts from maple, birch and alder; the hot water extracts are more toxic than the cold water extracts, and the extract from the heart wood is in each case more toxic than the extract from the corresponding sapwood. There appear to be no grounds for the assumption that the sapwood contains more starch, sugar, gums, etc., than the heart wood, or that these furnish food for fungi and further their growth. W. P. S.

Estimation of Benzene in Petroleum Spirit and Oil of Turpentine. J. Pritzker and R. Jungkunz. (*Chem. Zeit.*, 1924, **48**, 455–457.)—The authors base a method for the estimation of benzene in petroleum or turpentine on its reaction (previously described by Hofmann and Hochtlen, *Ber.*, 1903, **36**, 1149) with nickel ammonium cyanide. The reagent is prepared by mixing 5 grms. of nickel sulphate, dissolved in 20 c.c. of water, with 2·5 grms. of potassium cyanide in 10 c.c. of water, and adding 20 c.c. of a strong solution of ammonia; after the mixture has stood on ice for half-an-hour the crystals are filtered off, and dissolved as required, to form a saturated solution in 50 per cent. acetic acid. On adding this solution to the liquid under test, and shaking for a few minutes, a bluish white powdery precipitate is obtained in the presence of benzene. For quantitative purposes it is convenient to take 10 c.c. of the hydrocarbon mixture, which may contain 1 to 1·2 c.c. of benzene, and shake it for 5 minutes in a stoppered flask with 50 c.c. of the freshly-prepared reagent. The precipitate is filtered off with the aid of the pump, washed first with water, then once with alcohol and once with ether, dried, first by drawing air through it and then over sulphuric acid, and weighed. The precipitate, which has the formula $Ni(CN)_2NH_3C_6H_6$, contains 37·9 per cent. of benzene. H. E. C.

Behaviour of Sodium Sulphoricinate in the Official Method of Tannin Analysis. U. J. Thuan and L. Favre. (*J. Soc. Leather Trades Chem.*, 1924, 346.)—The authors draw attention to the fact that the Official Method is only an indication of the amount of material present in solution which is absorbed by hide powder under the arbitrary conditions laid down, irrespective of whether this material is a real tannin or not. For instance, sulphite-cellulose extract, though giving no precipitate with gelatin, is largely absorbed by hide powder, and thus counts as tannin. Sodium sulphoricinate acts in the same way. If mixed with formaldehyde, the more is absorbed by the hide powder the higher the amount of formaldehyde. The presence of quebracho lowers the quantity absorbed, but if mixed with chestnut extract an emulsion is formed, thus raising the figure for insoluble substances. Analytical results are given in twelve experiments on various mixtures of sulphoricinate etc. R. F. I.

Microchemistry of White Pigments and Inert Bodies as they occur Mixed in Paints. H. Green. (*Ind. Eng. Chem.*, 1924, **16**, 677–680.)—Methods for the necessary preparation of the sample for analysis are given, as well as microchemical tests for the individual pigments; a scheme for the qualitative analysis of a mixture of white pigments is also outlined. For the removal of oil, etc., from a paint, a drop of the sample is spread on a slide with the addition of a few drops of turpentine, the solvent is evaporated at 150° C., and the slide is then immersed in benzene. After a minute or two, the slide is removed and dried; the operation may be repeated, if required. The pigments adhere to the slide, and may be mounted in turpentine or dammar resin for microscopical examination. A portion of the pigment, after removal of the oil, etc., is reserved for microchemical tests. W. P. S.

Inorganic Analysis.

Estimation of Carbon Monoxide. P. Lebeau and Ch. Bedel. (*Comptes rend.*, 1924, **179**, 108.)—The usual reagent for absorbing carbon monoxide (cuprous chloride) has the disadvantage of requiring repeated contact because of the dissociation of the compound formed. The authors claim that this can be overcome by using a suspension of cuprous oxide in sulphuric acid containing β-naphthol. The reagent is prepared as follows:—Ten grms. of β-naphthol are placed in a 125 c.c. flask. To this is added a suspension of 5 grms. of cuprous oxide in a cooled mixture of 95 grms. of concentrated sulphuric acid and 5 grms. of water. The mixture is agitated for several hours and filtered through asbestos. The dark liquor obtained is left several days, decanted from the small deposit formed, and kept for use in well-stoppered flasks. This solution absorbs 18 times its volume of carbon monoxide, has no action on hydrogen, methane and its homologues, and can remain in contact with mercury for a considerable time without change. It is therefore suitable for use in the usual gas analysis apparatus. R. F. I.

Mixed Indicator for Carbonate-Bicarbonate Titrations. S. G. Simpson. (*Ind. Eng. Chem.*, 1924, **16**, 709.)—In titrating a mixture of alkali carbonate and bicarbonate the use of a mixed indicator consisting of thymol blue (thymol-sulphonphthalein) and cresol red (*o*-cresol-sulphonphthalein) is recommended as indicator for the first half of the titration; the colour of the thymol blue disappears, and is followed by a change from pink to orange-yellow, which shade is taken as the end-point, that is, the conversion of carbonate to bicarbonate. Bromophenol blue (tetra-bromphenol-sulphonphthalein) is then added, and the titration continued until the colour changes from blue to green—an indication that the solution is neutralised completely. The end-point in the case of the mixed indicator is much sharper than is that obtained with phenolphthalein. The indicator solutions should be prepared as follows:—Thymol blue, 0·03 grm. +1·42 c.c. of 0·05 *N* sodium hydroxide solution diluted to 25 c.c.; cresol red, 0·91 grm. +0·58 c.c. of 0·05 *N* sodium hydroxide solution diluted to 25 c.c.; bromophenol blue, 0·03 grm. +0·66 c.c. of 0·05 *N* sodium hydroxide solution diluted to 25 c.c. One volume of cresol red solution is mixed with 2 volumes of thymol blue solution, and 3 drops of the mixture are used for a titration; 4 drops of bromophenol blue solution are used for the second titration. W. P. S.

Mercuric Bromates in Analysis. G. F. Smith. (*J. Amer. Chem. Soc.*, 1924, **46**, 1577–1583.)—It has previously been shown by Smith (*J. Amer. Chem. Soc.*, 1923, **45**, 1115, 1417, 1666; ANALYST, 1923, **48**, 462) that the presence of the mercuric ion advantageously alters the usual course of oxidation reactions involving bromate by increasing the oxidation value. Mercuric bromates should be useful as oxidation agents. The preparation of normal and basic mercuric bromates by the interaction of mercuric perchlorate and sodium bromate in acid and practically neutral solutions is described. For the latter, mercuric perchlorate, prepared from mercuric nitrate and perchloric acid, is dissolved in a small volume of water and added slowly, with constant stirring, to a hot, half-saturated solution of sodium bromate. Proportions taken should be calculated from the following equation, with a slight excess of sodium bromate:

$$Hg(ClO_4)_2 + NaBrO_3 + H_2O \longrightarrow Hg(OH)BrO_3 + NaClO_4 + HClO_4.$$

Precipitation occurs upon the first addition of mercuric perchlorate. The solution and crystals are cooled to room temperature, decanted, and the crystals filtered and washed with water, with the aid of centrifugal drainage. The normal mercuric bromate is prepared by treating a hot 3 *N* perchloric acid solution, nearly saturated with sodium bromate, with an equivalent amount of mercuric perchlorate dissolved in 3 *N* perchloric acid; the crystals thus obtained, after cooling the reaction mixture to 0° C., are filtered off and washed with water, with the use of centrifugal drainage. With *N* perchloric acid solutions mixed normal mercuric bromate and hydroxymercuric bromate are prepared. The hydrolysis of normal mercuric bromate results in the formation of basic mercuric bromate and free bromic acid.

$$Hg(BrO_3)_2 + HOH \longrightarrow Hg(OH)BrO_3 + HBrO_3.$$

The relation of hydroxymercuric bromate to known mercuric salts with complex cations is pointed out; and the solubilities of the two bromates in various concentrations of nitric and perchloric acids at 25° C. have been determined. Basic mercuric bromate has been shown to be preferable as a reagent for volumetric bromate reactions because of its stability in 2 *N* nitric and perchloric acids.

P. H. P.

Estimation of Lead in the Presence of Tin and Antimony. E. Stelling. (*Ind. Eng. Chem.*, 1924, **16**, 748.)—Lead may be estimated in alloys containing tin and antimony, without previous removal of these two metals, if advantage is taken of the solubility of tin and antimony oxides in concentrated sulphurous acid and hydrochloric acid. One grm. of the borings is treated with 20 c.c. of concentrated nitric acid and 10 c.c. of water, and evaporated to dryness; the residue is then digested at 60° C. for five minutes with 50 c.c. of water saturated with sulphur dioxide. Twenty c.c. of concentrated hydrochloric acid are added, and the mixture is boiled; the tin and antimony oxides dissolve completely. After the addition of 20 c.c. of concentrated sulphuric acid, the mixture is heated until sulphuric acid fumes are evolved, cooled, diluted with 20 c.c. of water containing 10 per cent. of alcohol and 10 per cent. of sulphuric acid, and the lead sulphate then collected and washed with the same solution. W. P. S.

Volumetric Estimation of Fluorine. W. W. Scott. (*Ind. Eng. Chem.*, 1934, **16**, 703–706.)—The method proposed depends on the precipitation of the fluorine as calcium fluoride. In the case of minerals it is necessary to remove phosphates and sulphates by extraction with hot dilute acetic acid; the dried material is then fused for forty-five minutes with a mixture of sodium carbonate, potassium hydroxide and silica. After cooling, the fused mass is treated with hot water, the solution filtered, and the insoluble portion washed with hot water. The calcium in this insoluble portion may be estimated. The filtrate is heated to boiling and a slight excess of 0·25 *N* calcium acetate solution (12·51 grms. of calcium carbonate and 75 c.c. of glacial acetic acid per litre) are added; the mixture is then rendered slightly acid with acetic acid, heated for five minutes, diluted to a definite volume, and filtered. An aliquot portion of the filtrate is treated with a known volume of 0·25 *N* sodium oxalate solution, the precipitated calcium oxalate is collected, washed, and titrated in the usual way with potassium permanganate solution; the quantity of calcium required to precipitate the fluorine is thus obtained. Owing to the slight solubility of calcium fluoride, and possibly to the formation of a complex compound, the ratio of calcium to fluorine is 40:48, instead of the ratio represented by the formula CaF_2. W. P. S.

Detection and Estimation of small Quantities of Fluorine. R. J. Meyer. (*Chem. Zeit.*, 1924, **48**, 422.)—Lanthanum acetate in presence of ammonium acetate is an extremely sensitive reagent for fluorine; the precipitated lanthanum fluoride, however, always contains adsorbed lanthanum acetate. The precipitate may be dried without decomposition at 130° C. to constant weight. On subsequent

ignition over a Bunsen burner the acetate in the mixture is converted into oxide, whilst the fluoride remains unchanged; the loss in weight is calculated to lanthanum acetate, lanthanum fluoride being found by difference. The results are stated to have been found satisfactory for quantities of fluorine of 0·001 to 0·14 grm.

W. R. S.

Estimation of Sulphur in Coal. H. Bahr. (*Chem. Zeit.*, 1924, **48**, 428.)—The reliability of Eschka's method is stated to be questionable in the case of coals rich in sulphur (over 2 per cent.). The method proposed is based on the alumino-thermic reaction. A mixture of 0·5 grm. of coal, 0·4 of aluminium powder, and 3 of barium peroxide is briquetted; briquetting is indispensable to prevent loss by spirting. The briquette is ignited in a silica crucible covered with a perforated lid; all the sulphur present in the coal is converted into barium sulphide. After cooling, the crucible is treated in a suitable apparatus with dilute hydrochloric acid, and the hydrogen sulphide absorbed in cadmium solution, the sulphide being estimated with iodine and thiosulphate. The whole process may be carried out in 45 minutes.

W. R. S.

Physical Methods, Apparatus, etc.

Application of Fluorescence Phenomena to the Identification of Various Drugs. E. Bayle and R. Fabre. (*J. Pharm. Chim.*, 1924, **29**, 535–543.)—The authors have examined the fluorescence emitted by certain crystalline organic bodies and solutions under the action of ultra-violet rays with defined conditions, using the Georges automatic lamp on a continuous current as the source of light, with a wave length of 3,650 A.U. By this means a differentiation of the constituents of crystalline mixtures, such as novocaine (violet fluorescence) and stovaine (no fluorescence), was easily effected, and the characteristic behaviour of a large number of organic bodies was studied, and in certain cases the length and intensity of the dominant wave length was noted. The results are recorded in a table, and it is shown that the methods employed are capable of many applications, such as, for example, the detection of impurities possessing different fluorescent properties from the substance containing them. Thus 1 part in 2000 of salicylic acid may be detected in milk, and the method will probably prove useful for identifying artificial colouring matters. (*Cf.* Kitching, ANALYST, 1922, **47**, 206.)

D. G. H.

Porous Porcelain Crucibles for Filtrations. G. F. Hüttig and K. Schmitz. (*Zeitsch. anal. Chem.*, 1924, **64**, 224–227.)—The Government porcelain manufactory of Berlin has made successful experiments in the manufacture of porous-bottomed porcelain crucibles to replace the Gooch crucible. They were tested as to their corrodibility, hygroscopicity, constancy in weight, etc., and found to satisfy the requirements of analytical practice. They cannot be used for the filtration of solutions of caustic alkalis, but this is not a serious drawback in ordinary analytical work.

W. R. S.

Reviews.

CHEMICAL REACTIONS AND THEIR EQUATIONS. J. W. D. HACKLE. London: Chapman & Hall, Ltd. Pp. 138. Price 6s. net.

The author claims that this book is the only one of its kind, and in all probability, his claim is justified; the necessity for a volume devoted almost entirely to the construction and use of chemical equations is more open to doubt. There are many text books in use for class purposes which deal adequately with the subject, assuming, of course, that the teacher goes into the matter thoroughly. The book contains an assortment of information on chemical reactions, some of it very elementary in character and some considerably more advanced; by the time the student is in a position to appreciate the latter, the former should be quite superfluous. It is, therefore, difficult to recommend the book to either the beginner or the advanced student; the former could make little of such things as the ionic theory, and the latter should not need instruction on the preparation of simple salts and the adding up of molecular weights; furthermore, the classification of reactions into no fewer than twelve different types and the introduction of compounds of rare elements is surely unnecessary for either class of student. The volume closes with a thirty page appendix containing a displacement series of the elements, an outline of the Periodic System, a list of solubilities, and a glossary of chemical terms—a miscellany but little connected with what appears to be the general purpose of the book. A. F. KITCHING.

PHARMACEUTICAL AND FOOD ANALYSIS. By AZOR THURSTON. Pp. xiv.+416, with 19 Illustrations in Text. London: Chapman & Hall, Ltd. 1923. Price 21s. net.

The name of this book is truly stated above. The title page, but neither the cover nor the "jacket," gives a sub-title of "A Manual of Standard Methods for the Analysis of Oils, Fats and Waxes, and Substances in which they exist; together with Allied Products." The Preface announces that "a subsequent volume is being prepared to make the work a complete guide to the analysis of the more common drugs and foods."

The author has been for the past seventeen years chemist to the Ohio Agricultural Department, so the English reader has to make the best of American procedure in such directions as specific gravities taken at 25° C. (except in the case of alcohol). The drugs that by reason of fatty constituents get dealt with are those of the United States Pharmacopoeia, where camphorated oil and *Sapo Mollis* are made with cottonseed oil in place of our olive oil, where the official Oil of Cinnamon would be known here as the unofficial Oil of Cassia, and where Mercury Ointment is stronger than here. Such divergences are inevitable. In other respects, the author has been too diligent a student of English chemical books

and journals to be unaware of English work, though many of the standards and other criteria used here would have been worth mention.

Our own journal is continuously mentioned in the Bibliography, but one reference is omitted: The four-figure alcohol tables in respect of their weight and volume values are those which Mr. Hehner compiled on the Fownes figure of 0·7938 for absolute alcohol. THE ANALYST celebrated the occasion by, for the first and last time, printing a serial; the Hehner tables were a monthly "feature" for five months in 1880. Mr. Thurston, of course, excludes the Proof Spirit column, but gives "grammes per 100 c.c." instead. The inclusion of this table does not mean that alcoholic liquids are dealt with—they are not—it is simply an appendage to a chapter dealing with twenty-five methods of taking specific gravities.

The author goes stolidly and, on the whole, effectively through the subjects indicated in the sub-title, and the chief failing is the omission of clauses here and there which would have protected the obtuse, the thoughtless and the ignorant, from gleaning wrong impressions. Page 217 reads: "Quite often dairymen determine milk acidity as lactic acid—10 c.c. of milk titrated with $N/10$ NaOH and the result multiplied by 0·9." Page 130 tell us "cocoa husks are roasted and used as cattle food"; a commentary to be found in THE ANALYST, 1920, p. 20, might well replace the statement. A cherry-red stain on "tumeric" (*sic*) paper is all the evidence the author needs that boric acid is present (p. 208), but when the reverse test, that for "tumeric" is required, the addition of alkali to the stain is necessary (p 215). The composition of human milk (p. 178), which occurs in a table of analyses of milk "from different animals," and is "compiled from various reliable authorities," includes percentages of fat as 5·61, and proteins as 1·27. Such peculiarities do certainly occur, as, for example, in an analysis by Richmond (ANALYST, 1908, p. 114), where the fat was 5·17 per cent. and the proteins 1·01 per cent., but it is a peculiar, and not a representative, specimen. The alkali treatment of cocoa is considered with no mention of the individual alkalis used. Treatment of sour milk with ammonia before analysis is advocated, with a foot-note reference to THE ANALYST, 1885, 100. This was a paper by the late Dr. Adams, and was put forward as something preferable to a time allowance for decomposition, when the fat was being estimated by the Adams paper method. The experience of some others, when testing the method, has shown a few good results followed by many anomalous ones, and it seems to be little used in this country now.

In England we are accustomed to find figures for the average fat in cream to be, for example, 47·5 per cent. one year and 53·1 per cent. the next, but Mr. Thurston's analyses show an average of only 24·12 per cent., and the highest fat he records is only 35·21 per cent. (p. 237). However, the Federal standard of 18 per cent. explains much. On the other hand, the range of water in butter is from 5·99 per cent. to 12·59 per cent., and the wettest of twenty-four samples of margarine contained only 11·30 per cent. It is doubtful if any district in England can produce averages for water in either much below 15 per cent. The method

given for the estimation of water in butter consists in drying two grms. "at a temperature of boiling water until a constant weight is obtained." There is no direction as to the time between the weighings from which constancy is taken for granted, but there is a *primâ facie* appearance, at any rate, of inadequate water removal.

To avoid gaps in his book, the author has made some analyses, but his readers have just cause for complaint against the person who supplied him with the sample of "cocoanut" (the spelling appears to be correct for America) oil which gave a Polenske number of 13·11 (p. 161). W. PARTRIDGE.

CHEMICAL SYNTHESIS. STUDIES IN THE INVESTIGATION OF NATURAL ORGANIC PRODUCTS. By HARRY HEPWORTH, D.Sc., F.I.C. Pp. xx. + 243. London: Blackie & Son. 1924. Price 20s.

A very hearty welcome may be accorded to Dr. Hepworth's clear and concise summary of "the more important investigations which have been made by the organic chemist in modern times in the domain of natural organic products."

It is a well written book, and it certainly will do a great deal towards the dispelling of the impression held by some English chemists, that organic chemistry is mainly occupied in the making of organic compounds for Beilstein. There has certainly been a great deal of "beilsteinising" practised in recent years. However, Dr. Hepworth's summary of the classical investigations of Fischer (carbohydrates, purines and proteins), Willstätter (chlorophyll and anthocyanines) and Wallach (terpenes) leaves no doubt that organic chemistry still remains what it was meant to be: the true and honest chemical interpreter of life. It is for this reason that the reviewer was disappointed to find that Dr. Hepworth has devoted a whole chapter to the recent work on photosynthesis, most of which has been disproved, not only in this country, but also in America and Switzerland.

The book is fairly free from technical errors, although the names of some of the authors are wrongly spelt. Thus "Proctor" should read "Procter," "Freudenburg" "Freudenberg," "Zachmeister" "Zechmeister," etc., etc.

M. NIERENSTEIN.

DANGEROUS GOODS. By J. AEBY. Published by the author. Agents: Crosby Lockwood & Son, London. Pp. 320. 1922. Price 30s. net.

The author sets out to enumerate and briefly to describe all substances which may, for any reason, be described as dangerous merchandise from the point of view of shipping. Upwards of 450 different substances are mentioned, and, while it is obviously impossible to make such a work exhaustive, it is suggested that, if a substance is not mentioned in the book, it may be assumed that it presents no danger. Included in the term "dangerous" are substances which are either inflammable, explosive, corrosive, evil-smelling, or poisonous, and those

which, although not dangerous in a strict sense, are liable to contaminate other merchandise.

The book is printed throughout in English, French and German, and there is a comprehensive index, also in the three languages, which gives both chemical and commercial names. For the most part, the merchandise mentioned comprises chemical products, but a number of important articles, such as brewers' grains, which are liable to heat spontaneously, or empty sacks which may be impregnated with nitrate, are mentioned on account of their practical importance. Attached to the name of each article is a brief description of its physical properties, in what way it is dangerous, and notes of any special precautions which are desirable in connection with it. With this information at hand it only remains for the shipper or his advisers to consider the physical state of the product, how it is packed and how it is stowed.

The printing and style of the volume are good, and, so far as the reviewer has observed, the information is accurate, save for a few obvious misprints. While containing few data which are not available to the chemist scattered in other works of reference, the book is sure to be a useful addition to the library of anyone—chemist or otherwise—who has occasion to advise on the subject of dangerous merchandise. H. E. Cox.

SUPPLEMENTARY NOTES ON GRAVIMETRIC ANALYSIS. W. LOWSON. Pp. 54. London: Longmans, Green & Co. 1923. Price 2s. 6d.

Although this is a small book of four chapters, it is filled with valuable information too frequently omitted from the ordinary text-book. Most of the points dealt with might be described as minor, but they are none the less of great importance when real accuracy is required. The title correctly indicates the scope of the work, which should prove useful to the advanced student and, in the more elementary portions, to the beginner also.

The first chapter deals with the choice of apparatus and the ordinary manipulative processes; and the second discusses the occlusion of soluble substances by precipitates; twenty pages are then devoted to notes on typical estimations—notes, the careful observance of which may make all the difference between poor and good results. After a short chapter on the calibration of apparatus, a list of solubilities is given, followed by a table of four figure logarithms. It is a book that can be thoroughly recommended to students. A. F. KITCHING.