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THE ANALYST

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

Death.

WE greatly regret to have to record the death of Dr. Harvey Wiley, of Washington, U.S.A., who had long been an honorary member of the Society.

An obituary will be published in a later issue.

NORTH OF ENGLAND SECTION.

A SUMMER Meeting was held at the Queen's Hotel, Scarborough, on July 5th and 6th. There were twenty-eight in the party, which included a number of ladies. Dr. J. T. Dunn (President) was accompanied by Mrs. Dunn.

On Saturday afternoon a very important and interesting paper was read by Messrs. A. R. Tankard and D. J. T. Bagnall on "Fatalities due to Vitiated Air produced by the Oxidation of Vegetable Refuse." Mr. G. D. Elsdon (Chairman) presided. There followed a discussion in which most of the members participated.

Perfect weather conditions prevailed during the whole of the period, and a very pleasant and sociable week-end was thoroughly enjoyed by all.

Summer Country Meetings were a feature of the early years of the Society, but do not appear to have been held since about the year 1900.

The Micro-Detection of Alkaloids.*

By G. D. LANDER, D.Sc.

THE determination of the limits of detectability of poisons is, in general, a matter of academic interest. Improved technique enables one to detect many of them in quantities, or proportions, in themselves devoid of medico-legal significance. But occasions may arise where such data are valuable, in particular, in cases where the quantity of material available for examination is very limited in amount, or when it is desirable to know whether a drug is present or absent, irrespective of any possible physiological effects.

The technique described in this paper has been elaborated for the detection of "limit" quantities of alkaloids in saliva, but is capable, with obvious modifications, of application to other cases in which material is limited.

It has been well known for many years that drugs, after administration in medicinal amounts, find their way into the saliva, and of all body fluids this is perhaps the best adapted for research for alkaloids by reason of its relatively simple organic composition.

The separation of alkaloids in toxicological practice almost invariably follows the lines of the Stas-Otto process, modified *ad hoc* in special cases. The method of isolation and purification here described was devised some twenty years ago by Professor S. Fränkel, of Vienna, to meet the case of saliva.

The material is extracted several times in the cold with 90 per cent. pure alcohol, *plus* about 5 c.c. of dilute acetic acid, filtered through a fat-free paper, and the alcoholic extract, amounting to 200–300 c.c., evaporated, preferably by distillation in a partial vacuum, to a residue of about 5 c.c. of aqueous liquid. This is extracted with ether, which removes traces of fatty matter, and also small proportions of such purine derivatives as caffeine, and is then evaporated to a "tacky" consistency.

This residue is extracted by rubbing thoroughly with a small rod with successive small quantities of gently-warmed absolute alcohol, passed, after cooling, through a small filter, and gives a total of about 5 c.c. of extract. This extract is taken to dryness in a small dish, and the residue successively extracted by drops of 1 per cent. hydrochloric acid to give, after filtration, about 0·5 c.c. of liquid.

* Another paper on this subject, by Professor Eldin Bey, was read and discussed at the meeting on April 2nd. The Publication Committee have satisfied themselves, by evidence from various sources, that the priority for the principle of the process rests with Professor Fränkel, and that since then methods based upon it have been in constant use in different European countries. They have therefore asked Dr. Lander to give an account of the method which has, for many years past, been used under his direction.

The Committee wish to thank Professor Eldin Bey for drawing their attention to the subject.—EDITOR.

Should this solution be at all dark coloured, it may be purified by adding a drop of dialysed iron, followed, if no precipitate is given, by a scrap of common salt, and again filtering.

This solution is rendered alkaline with sodium bicarbonate and extracted twice with about 3 c.c. of chloroform, and, if considered necessary, with a further 2 to 3 c.c. of benzene. After passage through a double paper these extracts are evaporated to dryness, and the residue taken up with successive drops of 1 per cent. acetic acid, to give, after filtration, from 0·3 to 0·5 c.c. of final extract.

It now remains to test the final solution, in the first place by general alkaloid tests. The selection of reagents and a suitable technique of testing is a matter of personal choice. I have for many years observed these precipitation reactions in capillary tubes. One draws a small quantity of the solution and then of the reagent into a capillary tube. The formation of a precipitate or opalescence is to be observed at the point of union of the liquids. By allowing the column of liquid to move along the tube each open end may be sealed, and the tube can then be moved to the most suitable position for observation, and, moreover, the positive test can be kept until such time as the bulky precipitate coagulates, shrinks, and, if small, becomes no longer visible.

The economy of material is great. It is easy to deliver as little as 0·005 c.c. from a capillary pipette and to observe a positive test therein. Using as much as 0·01 c.c., an average final solution gives enough material for 40 to 50 tests. Naturally when dilution is pressed to the limit there is room for error from personal bias, but it is very interesting to prepare a "limit" test and a similar blank and submit the pair to the unprejudiced observation of an intelligent layman, the limit of observation being in fact the limit of visibility.

Following are some quantitative data worked out several years ago in this connection. Having regard to the sources of error, due to successive dilution of an original standard, to the errors of measurement inherent in the handling of minute volumes, and to variations in individual acuteness of vision and personal bias, no claim to mathematical accuracy is warrantable. But the figures at least indicate the order of magnitude of the quantities involved, and offer a fair estimate of the relative sensitiveness of individual reagents and alkaloids.

The results are expressed in fractions of a milligram, the actual volumes of solution tested ranging from 0·005 c.c. upwards, as measured from a capillary pipette.

Reagent.	Alkaloid.				
	Cocaine.	Strychnine.	Quinine.	Morphine.	Heroine.
Iodine	$\frac{1}{20000}$	$\frac{1}{20000}$	$\frac{1}{20000}$	$\frac{1}{100}$	$\frac{1}{2000}$
Phosphomolybdic acid ..	$\frac{1}{20000}$	$\frac{1}{10000}$	$\frac{1}{5000}$	$\frac{1}{1000}$	$\frac{1}{2000}$
Potassium mercuric iodide	$\frac{1}{20000}$	$\frac{1}{5000}$	$\frac{1}{2500}$	$\frac{1}{100}$	$\frac{1}{500}$
Gold chloride	$\frac{1}{10000}$	$\frac{1}{2500}$	$\frac{1}{2500}$	$\frac{1}{1000}$	$\frac{1}{2000}$
Tannic acid	—	$\frac{1}{2000}$	$\frac{1}{2500}$	$\frac{1}{50}$	$\frac{1}{50}$

The examination of saliva for the purpose of detecting drugs is sometimes of interest in veterinary practice; but clearly, in the case of alkaloids, it is a condition precedent that one must be satisfied that the normal secretion does not contain substances which respond to alkaloid general tests. Out of 16 cases of normal horses taken from my records, all the general tests tried were negative, save 4, which gave very faint opalescences with iodine. Fränkel holds that iodine gives such turbidities with fatty matter, which is probable, since in cases where iodine so behaves, the equally sensitive phosphomolybdic acid gives absolute blanks. A large amount of evidence privately communicated by continental colleagues fully confirms the conclusion that the normal fluid contains no alkaloid or alkaloidal substance.

The identification of an individual alkaloid is here, as in all medico-legal work, a matter for careful consideration, but certain assistance is given by the figures in the above table. For instance, a persistent gold reaction (on dilution) argues for cocaine and against morphine, and it will be seen that morphine and heroine give comparatively weak positives with the general reagents.

One or two notes on the limits of some special tests may be of value. If 0·01 c.c. of a 1 in 20,000 solution of strychnine is dried on porcelain and touched with a minute drop of sulphuric-chromate solution, a perfectly definite positive reaction is given, that is, by 1/2000 mgrm. The corresponding sulphuric-vanadic test is not so good.

Marquis' reagent (formalised strong sulphuric acid) similarly gives a perfectly distinct positive reation with 1/1200 mgrm. of heroine, or of morphine, the Fröhde test being more sensitive but less diagnostic.

If a residue of 1/300 mgrm. of quinine is treated with a minute drop of bromine water and then exposed to ammonia vapour, a positive test is obtained.

In all such limit tests the colours are naturally highly evanescent.

An example from my experimental records will suffice to indicate the nature of the results obtainable. The subject was a light van horse. A control specimen of the saliva gave, in a final solution of 0·4 c.c., negative tests with phosphomolybdic acid, iodine, potassium mercuric iodide, and potassium bismuth iodide; after subcutaneous dosage of 0·1 grm. of strychnine acetate a similar sample of saliva in a final solution of 0·5 c.c. gave positives with each of the above reagents, and also with picric acid. After evaporation to dryness an undoubted positive was given by the sulphuric acid chromate reaction. On the available quantitative data, and yielding to the very natural desire in such cases to estimate a figure, one may hazard the suggestion that in this case about 1/10000 to 1/20000 part of the original dose was contained in the sample.

But it will be readily agreed that in dealing with the living animal the variable factors of time, age, sex, habituation, idiosyncrasy, channel of administration, etc., render arithmetical generalisations both improper and misleading.

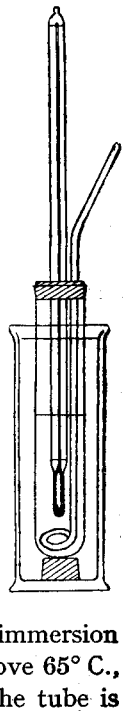
Classification of Cocoa Butter and Its Substitutes by the Freezing-Point Method.

By A. G. AVENT, A.I.C.

The so-called solidification or freezing point method of classifying and evaluating cocoa butter has been known for many years, but it would appear from the published information that little importance has been attached to this particular test. M. Pichard (*Ann. Falsificat.*, **1923**, **16**, 197–215) carried out work on the subject, and concluded that the so-called illipé and kayao butters were different in type, although both belonged to the Borneo tallow group. The result was contrary to the practical experience of the chocolate maker, and it seemed desirable to investigate the matter further.

A method of freezing-point determination is described below, which has been found helpful in the evaluation of cocoa butters, and the determined figures form a useful addition to other recognised chemical constants.

The most important application of the test is in the grading of cocoa butters and butter substitutes according to their suitability for chocolate manufacture. It is also sometimes possible definitely to identify adulteration. The detection of palm-kernel and coconut stearines, for example, presents no great difficulties, but fats from the Borneo tallow group can be mixed with cocoa butter without seriously affecting the resulting physical values. The Borneo tallow group includes fats from *Shorea robusta*, *Shorea stenoptera*, etc., the so-called illipé butters (trade names: Veberine, Kayao, Pontianak-Illipé, etc.). The cooling curves of unmixed cocoa butter, and Borneo tallow, although similar, show sufficient differences for the purpose of identification.



The weight of fat taken and the size of the apparatus have a marked effect on the shape of curve and on the maximum and minimum points.

During the past ten years I have used 75 grms. of fat for the freezing-point method. Occasionally a modified method has been carried out, in which 10 grms. of fat were used. Standard curves were made to correspond with this smaller weight.

Freezing-Point Method (75 Grms.).—A boiling tube, $1\frac{3}{8}\times 8''$, fitted with a cork bored centrally to take a thermometer (0–50° C.) and a glass stirring rod. This tube is fitted into a gas jar, $2\frac{1}{2}''\times 9''$, the boiling tube being held upright by an ordinary clamp.

The 75 grms. of fat are put into the boiling tube, and melted by immersion in a water-bath, the temperature of the fat not being allowed to rise above 65° C., and the fat being stirred until every particle is completely melted. The tube is

then transferred to a large beaker of cold water, and its contents stirred with a thermometer until the temperature is reduced to 40° C., when the tube is removed and wiped free from adhering moisture. The thermometer is now removed, the cork carrying the special thermometer and stirring rod fitted into the tube, and the fat continuously stirred until the thermometer registers 33° C. The tube is then immediately placed in the insulating jar, the space between the tube and the neck of the jar being lightly plugged with cotton wool.

When the temperature has dropped to 31° C., stirring is discontinued and the stop-watch is started; readings are taken at intervals of a minute. The curve can be plotted on graph paper during the test.

The first visible separation of fat crystals is noted, and, after an interval of one minute, the fat is stirred, by means of the bent glass rod, three times during each half minute. This procedure is continued until ten minutes after the maximum temperature is reached. Each kind of fat gives a typical curve, and from the curves a number of values can be tabulated. When taken together and compared with those of other fats, sufficient differences are indicated for classification.

The maximum and minimum temperatures, the difference between them, and the time taken from 31° C. to the maximum temperature, are the most useful values. The gradients of curve slopes have been measured, and theoretical cooling corrections have been applied.

Cocoa butters expressed from normal beans, properly fermented and roasted, give very similar curves. A defective cocoa butter is readily recognised by the depression of maximum and minimum temperatures, together with very slow setting. A noticeable feature of cocoa butter having this type of cooling curve is its high acid value.

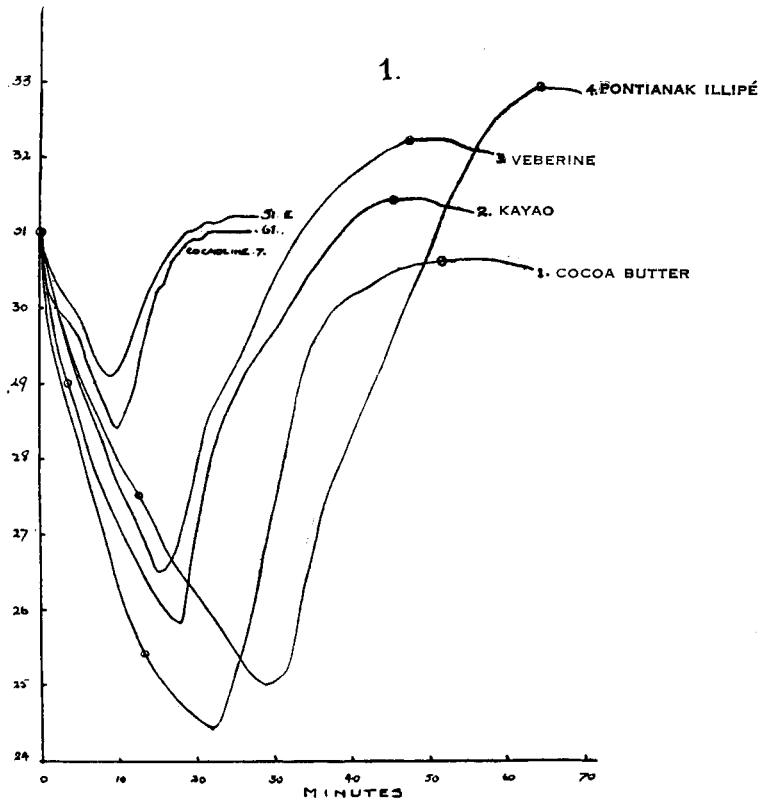
I have examined Borneo tallows giving curves with pronounced depressions from two consecutive bad crops of beans, the third crop being again normal. In this case, however, the acid value change was only very slight. The iodine and saponification values of both the above defective Borneo tallows were quite normal.

When the curves of several normal samples of each sort of fat are compared it would appear that the characteristics shown are sufficient for exact classification, but when ranges of many years' supplies are examined, unfortunately complications occur, and it becomes very much more difficult to decide exactly to what class the fat belongs.

TABLE I.

TYPICAL VALUES FROM CURVES.

Fat.	Max. °C.	Min. °C.	Diff. °C.	Time. Min.	Visible crystallisation. °C.
1. Cocoa butter ..	30·6	24·4	6·2	52	25·4
2. Kayao	31·4	25·8	5·6	46	29·0
3. Veberine ..	32·2	26·5	5·7	48	31·0
4. Pontianak illipé	32·9	25·0	7·9	65	27·5



Graph of Results given in Table I. Various typical fats in 75 grm. test.

The following table shows the variation in the maximum value of cocoa butters, according to the treatment to which the beans have been subjected:

TABLE II.

NORMAL COCOA BUTTER.

	Acid value.	Max. °C.	Min. °C.	Diff. °C.	Time. Min.	Visible crystallisation. °C.
1.	1·8	30·6	24·4	6·2	52	25·4
5.	1·8	30·3	24·2	6·1	64	25·0
6.	1·6	30·5	24·6	5·9	52	26·2
7.	1·2	30·2	24·8	5·4	40	26·3
8.	1·5	30·5	24·4	6·1	72	25·7

These figures may be taken as representing normal cocoa butters from the standpoint of chocolate manufacture.

TABLE III.

COCOA BUTTERS WITH HIGH ACID VALUE AND LOW MAXIMUM TEMPERATURE.

	Acid value.	Max. °C.	Min. °C.	Diff. °C.	Time. Min.	Visible crystallisation. °C.
9.	4·2	29·2	23·5	5·7	65	25·0
10.	4·3	28·8	23·2	5·6	84	24·0
11.	4·5	28·7	23·0	5·7	82	23·5
12.	2·7	29·1	23·4	5·7	64	24·8

The behaviour in manufacture confirms the above figures, which indicate some breakdown of the glycerides.

TABLE IV.

DEFECTIVE COCOA BUTTERS.

	Acid value.	Max. °C.	Min. °C.	Diff. °C.	Time. Mins.	Visible crystallisation. °C.
13.	2·8	29·3	21·0	8·3	114	21·5
14.	3·0	29·3	21·0	8·3	96	21·5
15.	4·8	28·8	—	—	—	22·2
16.	4·8	28·1	21·1	7·0	—	22·9

These butters were found to be quite unsuitable for use in chocolate making.

In addition to the slight variation of the values of cocoa butter expressed from different kinds of beans, the figures obtained from cocoa butter pressed from the same batch of beans varies according to the treatment of the "cocoa mass."

TABLE V.

COCOA BUTTER (75 Grm. Test).

	Acid value.	Max. °C.	Min. °C.	Diff. °C.	Time. Min.	Visible crystallisation. °C.
A. Unalkalised	2·8	30·0	24·8	5·2	52	25·8
B. ,,	2·8	30·0	24·5	5·5	53	26·0
C. Alkalised	3·4	29·6	24·8	4·8	46	26·1
D. ,, and liquid	3·0	29·6	24·0	5·6	57	25·4
E. ,, last fraction	3·4	29·4	23·6	5·8	57	26·8

A. In this case the "Cocoa mass" was run direct into the pots of the hydraulic press, without any of the previous treatment that alkalisation entails.

B. A second untreated sample of the same pressing.

C. The values obtained from butter expressed from the same batch of beans as A and B after alkalisation. A slight breakdown of the glycerides is indicated by the increase in the acid value.

D. Butter from a pressing similar to the alkalised sample C. The freezing point test (75 grms.) was carried out directly on the liquid fat from the press, *i.e.* the first solidification of the fat in the free state.

E. The figures from a curve given by the last fraction expressed from the alkalised mass. Slightly lower maximum and minimum values were noted.

The figures given in Tables II, III and IV serve as a valuable guide in determining whether a butter is fit for use in chocolate.

The contention that the cocoa butters included in Table II are superior to those contained in Tables III and IV was confirmed by the quality of the chocolate made in the works.

A more pronounced variation has been noted in the so-called Illipé group (Borneo tallow). Consignments of butters are labelled by trade names, and it becomes difficult to trace the origin. The consequence is that the values show rather large variations. Butter sold as Pontianak illipé showed an average maximum during 1920–27 of 32·9° C. Samples examined during 1928–1929 showed an average maximum of 30·5° C., with a highest value of 31·9° C.

TABLE VI.

BORNEO TALLOW GROUP.

	Pontianak Illipé.	Acid value.	Max. °C.	Min. °C.	Diff. °C.	Time. Mins.	Visible crystallisation. °C.
17.	1922 Illipé	0·8	33·3	25·5	7·8	57	27·2
18.	1924 ,, ...	0·2	33·1	25·2	7·9	65	27·4
19.	1923 ,, ...	0·2	32·7	25·1	7·9	65	28·8
20.	1922 Finest French illipé	0·9	34·7	26·5	8·2	—	28·7
21.	1929 Illipé	—	31·9	26·8	5·1	63	31·0
22.	1929 ,, ...	—	30·3	25·1	5·2	61	29·8
23.	1929 ,, ...	—	31·5	25·7	5·8	52	30·5
24.	1929 ,, ...	—	30·2	25·9	4·3	68	30·6

The maximum and minimum and visible crystallisation temperatures are all higher than the corresponding values for cocoa butter.

Cocoa butter undergoes super-cooling to a greater extent than Borneo tallow. This is indicated by the separation of fat crystals at a higher temperature in the case of the tallow.

TABLE VII.

DEFECTIVE PONTIANAK ILLIPÉ.

		Max. °C.	Min. °C.	Diff. °C.	Time. Mins.	Visible crystallisation. °C.
89.	1929 ..	27·8	25·3	2·5	31	32
90.	1929 ..	28·4	25·2	3·2	41	33
91.	1929 ..	27·3	25·0	2·3	34	34

The low maximum temperature is an indication that these fats will be defective in use.

TABLE VIII.

VEBERINE (A proprietary article belonging to the Borneo Tallow Group).

		Max. °C.	Min. °C.	Diff. °C.	Time. Mins.	Visible crystallisation. °C.
25.	1924 ..	32·4	26·1	6·3	58	31·1
26.	1924 ..	32·8	26·2	6·6	60	29·2
3.	1925 ..	32·2	26·5	5·7	48	31·0
27.	1925 ..	32·1	26·8	5·3	53	31·0
28.	1925 ..	32·1	26·4	5·7	54	32·0
29.	1929 ..	32·8	25·8	7·0	52	29·8
30.	1929 ..	31·9	25·9	6·0	55	29·9
31.	1929 ..	31·5	26·2	5·3	58	30·6

These show more concordant results, and in each case afford differences sufficient to distinguish this fat from cocoa butter.

Kayao is another product of the Borneo tallow group expressed in France.

TABLE IX.

NORMAL KAYAO.

		Acid value.	Max. °C.	Min. °C.	Diff. °C.	Time. Mins.	Visible crystallisation. °C.
32.	1924 Kayao	1·0	32·7	25·7	7·0	70	25·7
33.	1925 ,,	0·8	30·6	26·0	4·6	70	28·6
34.	1925 ,,	1·0	31·1	26·0	5·1	60	30·7
35.	1925 (Sept.) ,,	—	31·4	25·8	5·6	46	29·5
36.	1929 ,,	—	31·1	25·1	6·0	53	28·6
37.	1928 ,,	—	30·6	25·4	3·2	54	33

The above tables show that the butters of the Borneo tallow group are very similar, and that they are readily distinguished from cocoa butter.

TABLE X.

DEFECTIVE KAYAO.

		Acid value.	Max. °C.	Min. °C.	Diff. °C.	Time. Mins.	Visible crystallisation. °C.
38.	1925, May	0·8	27·9	25·5	2·4	46	31·0
39.	1925, June	1·0	28·0	26·0	2·0	45	31·0
40.	1922, 1.	1·2	27·7	24·6	3·1	—	34·5
41.	1922, 2.	1·2	27·8	24·6	3·2	—	34·5

Even these defective fats are different from both the normal and abnormal cocoa butters.

The following fats give values very different from the preceding types, the outstanding features being the comparatively high minimum temperature and short-time period. The characteristics of palm-kernel and coconut stearine group

can be illustrated by Cocaoline 7, a French product, and an English product which I will call E. Butter.

TABLE XI.

E. BUTTER.

		Max. °C.	Min. °C.	Diff. °C.	Time. Mins.	Visible crystallisation. °C.
42.	1928–1929 E.	30·1	27·7	2·4	33	30·2
43.	,,	31·2	28·9	2·3	21	30·1
44.	,,	31·0	28·7	2·3	25	30·3
45.	,,	30·4	28·0	2·4	30	30·4
46.	,,	30·6	28·3	2·3	23	30·6
47.	,,	30·8	28·8	2·0	20	32·0
48.	,,	30·6	27·8	2·8	25	29·2

One consignment of this type of fat showed the unusual feature of a decided double maximum point. The chemical values were quite normal.

TABLE XII.

	Max. 1. °C.	Min. 1. °C.	Diff. 1. °C.	Max. 2. °C.	Min. 2. °C.	Diff. 2. °C.	T. 1. Mins.	T. 2. Mins.	Visible crystal-lisation °C.
49. 1929 E.	28·7	27·3	1·4	29·9	27·7	2·2	12	29	27·3
50.	29·8	28·0	1·8	30·9	28·5	2·4	13	33	28·0

A cooling curve carried out on the previous delivery and a duplicate test carried out on the same 75 grms. after an immediate re-melting gave the following figures:

TABLE XIII.

E. BUTTER.

		Max. 1. °C.	Min. 1. °C.	Diff. 1. °C.	Time 1. Mins.	Visible crystallisation. °C.
51.	Direct	31·2	29·1	2·1	24	31·6
52.	Remelted	31·2	28·9	2·3	20	31·0

This unusual curve is probably due to the formation of eutectic compounds. The melting points of shavings of fat taken from the different parts of the sample block gave the following figures:

		Incipient fusion. °C.	Complete fusion. °C.
1.	Vitreous fraction	31·0	32·6
2.	,, ,, ...	31·4	34·0
3.	Crystalline fraction	34·2	36·6
4.	,, ,, ...	33·8	35·6
5.	Previous consignment ..	30·4	34·0
6.	,, ,, ..	30·2	33·6
7.	,, ,, ..	30·6	34·0

The separation of the various melting fractions in the abnormal sample could have been brought about by a change in the method of refrigeration during the moulding process.

COCAOLINE 7.—This fat is very similar in use, and in its cooling curve values, to E. Butter. Here, again, the high minimum temperature and short-time period of setting affords means of classification.

TABLE XIV.

Date.	Acid value.	Max. °C.	Min. °C.	Diff. °C.	Time. Mins.	Visible crystallisation. °C.
53. 1920	0·2	30·4	27·9	2·5	27	29·5
54.* 1922	1·2	30·0	27·6	2·4	30	29·8
55. 1923	0·2	30·8	28·6	2·2	30	29·6
56. 1923	0·5	30·1	27·8	2·3	30	29·6
57. 1924	0·2	31·0	28·0	3·0	30	29·8
58. 1925	0·2	30·8	28·3	2·5	—	31·0
59. 1928	—	31·1	28·2	2·9	24	30·0
60. 1928	—	30·9	28·2	2·7	31	31·4
61. 1929	—	31·0	28·4	2·6	22	31·4

* Flaked.

The average and range of the various values have been omitted from tables of all the fats, and typical examples have been given instead. In classifying a fat it is better to correlate the various values of the sample one with the other, rather than to compare each value with previous averages.

From time to time various mixtures of fats have been tested by the cooling-curve method. In the case of genuine milk chocolates, the butter fat extracted with other fats affects the curve. Butter fat, with moisture and curd removed, and veberine gave the following values:

TABLE XV.

	Max. °C.	Min. °C.	Diff. °C.	Time. Mins.	Visible crystallisation. °C.
62. Cow's butter	24·8	23·9	0·9	43	24·6
63. Veberine	32·3	26·1	6·2	65	31·8

A mixture of Veberine and milk fat in the proportions which would occur in a milk chocolate (Veberine, 26·3; milk fat, 3·7 per cent.) gave a depression of the maximum of Veberine by 2·3° C., and of the minimum 0·8° C.

	Max. °C.	Min. °C.	Diff. °C.	Time. Mins.	Visible crystallisation. °C.
64.	30·0	25·3	4·7	65	29·5

The cooling curve of cocoa butter is not distorted to any great extent by admixture with fats of the Borneo tallow group.

TABLE XVI.

		Max. °C.	Min. °C.	Diff. °C.	Time. Mins.	Visible crystallisation. °C.
63.	Veberine	32·3	26·1	6·2	65	31·8
6.	Cocoa butter	30·5	24·6	5·9	52	26·2
64.	57 per cent. (63) Veberine 43 ,, ,, (6) cocoa butter	31·1	25·4	5·7	61	27·3
65.	29 per cent. (63) Veberine 71 ,, ,, (6) cocoa butter	30·5	23·0	7·5	69	24·3

The effect of palm-kernel and coconut stearines, however, is very marked, a depression of values well below that of either of the constituents being observed.

TABLE XVII.

		Max. °C.	Min. °C.	Diff. °C.	Time. Mins.	Visible crystallisation. °C.
66.	Illipé special	31·9	26·0	5·9	38	—
67.	E. butter	30·0	27·4	2·6	32	—
68.	Illipé 60 per cent. .. .. E. 40 ,, ,, ...	29·1	23·2	5·9	54	—
69.	Veberine	30·7	25·0	4·7	61	31·4
43.	E. butter	31·2	28·9	2·3	21	30·1
70.	Veb. (69) 33·3 per cent. .. .. E. (43) 66·6 ,, ,, ..	27·5	24·8	2·7	50	25·3
71.	Veb. (69) 66·6 ,, ,, ... E. (43) 33·3 ,, ,, ..	27·8	23·2	4·6	64	25·8
72.	Cocoa butter	29·6	24·2	5·1	52	—
37.	Kayao	30·6	25·3	5·3	54	—
59.	Cocaoline 7	31·1	28·2	2·9	24	30·0
73.	C.B. 38 per cent. .. Kayao 38 ,, ,, .. Cocaoline 7 24 ,, ,, ..	27·4	23·2	4·2	70	—

The mixtures in the above table appear rather haphazard, the reason being that the original curves were plotted during quite separate investigations. The effect of mixing one type of fat with another is sufficient to show that, with controls, a considerable amount of assistance can be obtained when examining unknown mixtures.

FREEZING POINT METHOD (10 grms.).—In order that smaller quantities of fat could be examined, cooling curves were made, with the use of only 10 grms.

A test tube, $5'' \times \frac{3}{4}''$, fitted with a bored cork to take a 0–50° C. thermometer and with a small groove cut in the side for a wire stirrer, is fitted into another tube ($7'' \times 1''$) and supported by wire suitably twisted.

A similar procedure of melting and stirring was then carried out and the results were plotted as before.

TABLE XVIII.

COCOA BUTTER.

		Max. °C.	Min. °C.	Diff. °C.	Time. Mins.	Visible crystallisation. °C.
74.	Accra	28·2	22·5	5·7	20	—
	,, re-melted	28·2	20·8	7·4	27	22·6
75.	Accra	28·6	23·3	5·3	24	—
76.	San Thomé	29·1	24·2	4·9	22	—
77.	Cocoa butter (solvent extraction)	28·8	22·0	6·8	32	23·0
78.	Accra (laboratory extraction)	29·0	24·6	4·4	16	acid value 1·0

Cocoa butter differs from the other fats in respect of re-melting. It was found that on re-melting the fat in the tube, immediately after the first test was completed, and repeating the usual procedure, the minimum temperature was 1·7° C. lower than in the first test. The other fats did not show this difference.

TABLE XIX.

		Max. °C.	Min. °C.	Diff. °C.	Time. Mins.	Visible crystallisation. °C.
79.	Illipé	29·0	24·5	4·5	20	27·8
80.	Veberine	28·4	25·5	2·9	17	—
81.	Kayao	28·6	24·7	3·9	18	29·7
	,, re-melted ..	28·8	24·2	4·6	20	29·8
82.	Kayao	30·6	24·8	5·8	25	—
83.	E. Butter	30·8	28·0	2·8	10	29·8
	,, ,, re-melted ..	30·8	28·3	2·5	7	—
*49.	E. Butter	30·5	27·0	3·5	9	29·9
*50.	E. Butter	30·6	27·8	2·8	8	—
84.	Cocaoline 7	31·2	28·5	2·7	9	—
85.	,, ,, ...	30·5	26·3	4·2	9	28·0
86.	Cocoa Butter, 50 per cent. (Accra No. 74) .. Veberine 80, 50 per cent.	27·8	23·5	4·3	25	25·1
87.	Accra 74, 50 per cent. E. Butter 83, 50 ,, ,,	24·0	21·0	3·0	36	22·2
88.	Veberine 80, 50 per cent. E. Butter 83, 50 ,, ,,	25·9	21·0	4·9	36	27·0

* The fats Nos. 49 and 50 gave double maximum and minimum points with the 75 grm. test, but in the 10 grm. test showed only a single minimum value lower than a normal E. Butter, the cooling effect of the room on the smaller quantity of fat preventing the latent heat from influencing the curve.

The cocoa butters differ from the other fats by the relative lowness of the minimum temperature.

When sufficient quantity of sample is available the 75 grm. method is preferable because it is not influenced so much by the temperature of the room, and any abnormalities are more easily detected. However, the 10 grm. method

is sufficiently sensitive to be of use when only a limited amount of fat is available, especially when examining fats extracted from chocolates. The effect of the room temperature on cooling-curves made by either method is not very great, provided the temperature is kept within reasonable limits.

In order to obtain 10 grms. of fat, three Soxhlet extractors, each containing 10 grms., are charged with the chocolate, which is then extracted with petroleum spirit (b.pt. 40°—60° C.) for at least 10 hours. Provided that all traces of solvent are removed from the fat, the freezing-point curve is not distorted.

The more divergent the cooling curve of a fat is from that of cocoa butter the less suitable the fat is for chocolate making.

On the other hand, the similarity between the cooling curves of cocoa butter and any other fat is an indication that the latter can replace cocoa butter in manufacture. The most satisfactory substitute in use is Borneo tallow, and the contention that the cooling curve is an indicator of suitability is upheld in this instance. The chocolate moulding and refrigerating temperatures do not give rise to any unusual defects when a proportion of Borneo tallow is used, and the contraction of a chocolate so made is good.

When replacing cocoa butter by Borneo tallow a rather larger percentage of the substitute is required to obtain a chocolate of standard fluidity at moulding temperatures. This is probably associated with the higher temperature of visible crystallisation of the Borneo tallow.

The distortion from the normal of a cocoa butter cooling curve indicates the presence of a substitute. As is the case with other tests for the detection of Borneo tallow in admixture with cocoa butter, the effect of a defective Borneo tallow on the mixture would be misleading.

Apart from the question of cost, Borneo tallow is added for the purpose of "hardening" chocolate. A defective tallow appears to have the opposite effect. The relatively low maximum temperature of such a fat is an indication of "softness."

The Effect of Temperature on the Sulphur Dioxide Content of Corn Syrup in Mixtures of Sugar and Corn Syrup.

By R. HAROLD MORGAN, B.Sc., A.I.C.

Introduction.—Among the foodstuffs mentioned in Schedule I of the Public Health (Preservatives, etc., in Food) Regulations, 1925, which may contain preservative, are corn syrup and sugar, two substances which form the basis of present-day confectionery.

Both corn syrup and sugar are bleached during manufacture, and the maximum permissible amounts of sulphur dioxide which may be present are 450 and 70 parts per million, respectively.

It has been the practice among some confectionery manufacturers to disguise the use of inferior sugar and lack of care in boiling (which produce yellow-coloured products) by bleaching their goods by means of sulphur dioxide in the form of sulphite. A common bleaching agent is a concentrated solution of sodium bisulphite, saturated with sulphur dioxide. The Ministry of Health deprecates this practice, and has declared such further additions of preservatives (equivalent in this case to "bleachers") illegal.

A corn syrup containing originally the maximum amount of sulphur dioxide allowed (*viz.* 450 parts per million), when boiled, loses a certain amount of sulphur dioxide, as the gas is driven away by heat, and also the bisulphite present is decomposed as follows: $2NaHSO_3 = Na_2SO_3 + SO_2 + H_2O$.

The amount of sulphur dioxide evolved depends apparently on the time and conditions of heating. The longer the time, the less the amount of sulphur dioxide remaining in the final product.

It has been stated above that no addition of bleaching agent is allowed beyond that originally introduced, in the manufacture of the raw material, which, in the case of corn syrup, is 450 parts per million. Therefore, when confectionery products, which are sugar and corn syrup mixtures, are boiled to temperatures ranging from 240–320° F., the residual amount of sulphur dioxide must be less than that initially present in the proportion of corn syrup in the mixture, assuming the concentration of sulphur dioxide to be the maximum permissible amount.

Public Analysts administering the Food Laws, and especially the prohibition of the use of bleaching agents, have been unable to confirm their suspicions that bleaches are being used, when these are added in calculated amounts so as not to leave a residual percentage of sulphur dioxide greater than that corresponding to the presence of a corn syrup containing 450 parts per million of sulphur dioxide.

The object of the investigation was to find whether any relationship exists between the time of heating and the residual amount of sulphur dioxide, while endeavouring, as far as possible, to obtain results comparable with those from products produced commercially. The experimental work of the paper has been carried out to ascertain what residual amount of sulphur dioxide can be expected when corn syrups are boiled to various temperatures without the addition of bleaching agents during the heating process.

COMMERCIAL PRACTICE AND LABORATORY EQUIVALENT.—Three methods of heating are used commercially, *viz.* coke fires, compressed air and gas, and vacuum pan boiling. A laboratory equivalent was based on the following data (confirmed by actual practice):

(*a*) Heating on coke fire takes a longer time to reach a pre-determined temperature than either of the other two methods. Consequently, the loss of sulphur dioxide is always greater than with the compressed air and gas method.

(*b*) Vacuum pan boiling also shows a greater loss of sulphur dioxide than does compressed air and gas heating, due to the suction of the vacuum.

Consequently, the best laboratory equivalent to commercial practice was arranged on the lines of the second method, *viz.* heating over a large and powerful gas flame. This was confirmed by analyses of sugar and corn syrup mixtures heated to definite temperatures in the laboratory apparatus, and of similar mixtures heated to the same temperatures over forced draught burners on a commercial plant. The results obtained for the amount of residual sulphur dioxide in each case corresponded very closely.

To economise in gas and to prevent discoloration, as is done in industrial practice, the heating was carried out as rapidly as possible.

EXPERIMENTAL METHOD.—A copper pan, well tinned inside, was placed on a large tripod stand over a powerful triple burner. As corn syrup cannot be boiled alone owing to frothing, the usual industrial mixture of sugar and glucose was employed. The effect of the presence of the sugar is noted later. A common proportion of sugar to corn syrup is 3: 1, and so 1½ lbs. of sugar and ½ lb. of corn syrup were put in the pan, together with one-third of a pint of water to allow of easy solution without burning. The water boils off and does not affect the analytical results. A thermometer was inserted into the pan and the whole heated rapidly to the required temperature, a careful note being made of the time of heating. The mixture was stirred at the beginning of the heating, and towards

the end the flame was reduced somewhat, to prevent local burning of the boiling syrup. When the desired temperature was reached, the boiling mass was poured on to a cool slab which had been rubbed over with mineral oil to prevent sticking, and the solid was analysed when it had become cold. In each case duplicate results were obtained.

Analytical Methods.

Corn Syrup.—The corn syrup used was of the usual commercial variety supplied to confectioners. This consists of a mixture of maltose, dextrose, and dextrin in water, the last mentioned amounting to 17 or 18 per cent. of the total mass. To provide a definite basis on which the sulphur dioxide can be calculated, the total solids were determined by making up a 10 per cent solution, obtaining its density in a specific gravity bottle, and dividing by the factor 3·86. This industrial determination is based on the fact that the specific gravity (1000) of a 1 per cent. sugar solution is 1003·86, and gives rise to the following equation:

$$\text{Total solids} = \frac{\text{Sp. gr. of solution} - 1000}{3{\cdot}86}.$$

For confirmation, the total solids were also determined by obtaining the refractive index of a 15 per cent. solution and calculating according to the following formula:

$$\text{Total solids} = \frac{n_D^{20} - 1{\cdot}3330}{f} \quad (f = 1{\cdot}43 \text{ for 10–20 per cent. solutions}).$$

The initial sulphur dioxide content of the corn syrup was determined by the method given below.

Sugar.—The sugar used was Tate and Lyle's stoved granulated sugar, and is a particularly good quality for sugar boiling, having a low ash content. It was practically free from sulphur dioxide. Experiments with other varieties showed that commercial sugars do not contain appreciable amounts of sulphur dioxide, and where any positive reactions were noted, the sulphur dioxide was in such a state of combination as to be unaffected by the heating action of the process. Determinations of the total solids gave results practically equivalent to 100 per cent. of solids.

With regard to the presence of sulphur dioxide in sugar, it might be of interest to note that I was informed by Messrs. Tate and Lyle that the substance was regarded as an impurity, and every effort made to eliminate it during the process of manufacture. This is particularly interesting, as the Food Regulations permit sulphur dioxide to be present in sugar in amounts not exceeding 70 parts per million.

Sugar and Corn Syrup Products.—The resulting cold solid left after the heating process was analysed, firstly, for total solids, according to the methods mentioned above; and, secondly, for sulphur dioxide.

I was investigating various methods for the determination of sulphur dioxide when the Ministry of Health published its report on the Determination of Sulphur Dioxide in Food (Reports on Public Health and Medical Subjects, No. 43, 1927). The method advocated in that Report was then adopted, with a few modifications. The modified method was similar, in most respects, to that published afterwards in THE ANALYST (1928, **53**, 118).

The weighed solid, together with phosphoric acid, was placed in an inclined flask, and in an atmosphere of carbon dioxide, and the sulphur dioxide was driven off by heating, through a still-head into a condenser fixed vertically. An adapter of the "bubbler" type conducted the gas from the lower end of the condenser into a neutral solution of hydrogen peroxide. The acid formed was titrated against standard alkali, bromphenol blue being used as an indicator, and the sulphur dioxide evolved was calculated on the basis of percentage on the dry solids in the sugar and corn syrup product. About 15 minutes' heating was found sufficient to drive away all the sulphur dioxide present in the solid. Duplicate results were obtained in each case. The following tables summarise the results obtained from five series of experiments, in each of which a different commercial corn syrup was used, together with the granulated sugar mentioned above.

TABLE A.

Corn syrup (H 832). Total solids 82 per cent.

Sulphur dioxide content = 370 parts per million.

Initial sulphur dioxide content of sugar and corn syrup mixture = 97 parts per million on the dry solids.

Temp. in °F.	Time of boiling. minutes and seconds.	Percentage of solids.	Residual sulphur dioxide in parts per 1,000,000.	Average.	Residual sulphur dioxide, per 1,000,000 of dry solids.	Sulphur dioxide evolved, per 1,000,000 of dry solids.
Initially					97	
240	8–00	88·1	60	59	68	29
	8–00	88·2	58			
250	9–15	90	57	55	60	27
	9–30	90·8	53			
260	10–00	93·1	45	44	48	49
	10–00	93·2	43			
270	11–00	94·6	35	34·5	37	60
	11–15	94·7	34			
280	12–30	95·7	27	26·5	28	69
	12–30	95·8	26			
290	12–45	96·1	23	21·5	22	75
	13–00	96·2	20			
300	13–30	97·4	13	12·5	13	84
	13–45	97·5	12			

TABLE B.

Corn syrup (J 23). Total solids 82·6 per cent.

Sulphur dioxide content = 375 parts per million.

Initial sulphur dioxide content of sugar and corn syrup mixture = 98 parts per million on the dry solids.

Temp. in °F.	Time of boiling. Minutes and seconds.	Percentage of solids.	Residual sulphur dioxide in parts per 1,000,000.	Average.	Residual sulphur dioxide, per 1,000,000 of dry solids.	Sulphur dioxide evolved, per 1,000,000 of dry solids.
Initially					98	
240	7–75	88·0	62	61	70	28
	8	88·2	60			
250	9–45	90·9	57	57	64	34
	9–30	90–8	57			
260	10–	93·1	47	47	51	47
	10–15	93·2	47			
270	11–15	94·8	38	36	38	60
	12–	94·9	34			
280	12–15	95·9	27	27·5	29	69
	12–15	95·9	28			
290	12–30	96·0	23	21·5	23	75
	12–30	96·2	20			
300	13–30	97·1	14	13·5	14	84
	14	97·1	13			

TABLE C.

Corn syrup (H 504). Total solids 83 per cent.

Sulphur dioxide content = 416 parts per million.

Initial sulphur dioxide content of sugar and corn syrup mixture = 109 parts per million on the dry solids.

Temp. in °F.	Time of boiling Minutes and seconds.	Percentage of solids.	Residual sulphur dioxide in parts per 1,000,000.	Average.	Sulphur dioxide evolved, per 1,000,000 of dry solids.	Residual sulphur dioxide, per 1,000,000 of dry solids.
Initially					109	
240	8–30	88·1	65	65·5	74	35
	8–45	88·2	65			
250	10–0	91·2	59	58·5	64	45
	10–15	91·3	58			
260	11–0	92·8	56	54·5	58	51
	11–30	93·0	52			
270	11–45	94·6	44	43·5	46	63
	11–45	94·8	43			
280	12–0	95·6	35	34·5	37	72
	12–0	95·7	34			
290	13–0	96·4	25	24	26	83
	13–0	96·3	23			
300	13–15	97·6	19	18	19	90
	13–30	97·8	17			

TABLE D.

Corn syrup (J 32). Total solids 83 per cent.

Sulphur dioxide content = 341 parts per million.

Initial sulphur dioxide content of sugar and corn syrup mixture = 88 parts per million on the dry solids.

Temp. in °F.	Time of boiling. Minutes and seconds.	Percentage of solids.	Residual sulphur dioxide in parts per 1,000,000.	Average.	Residual sulphur dioxide, per 1,000,000 of dry solids.	Sulphur dioxide evolved, per 1,000,000 of dry solids.
Initially					88	
240	7–15	88·1	55	54·5	62	26
	7–30	88·1	54			
250	9–00	90·6	49	49·5	54	34
	9–15	90·6	50			
260	10–30	93·6	39	39·5	43	45
	10–00	93·3	40			
270	11–00	94·6	33	31·5	33	55
	11–15	94·7	30			
280	11–30	95·3	25	23·5	25	63
	12–00	95·4	22			
290	12–15	96·00	16	15·5	16	72
	12–15	96·00	15			
300	12–30	97·5	11	11	12	76
	13–15	97·6	11			

TABLE E.

Corn syrup (H 451). Total solids 82 per cent.

Sulphur dioxide content = 351 parts per million.

Initial sulphur dioxide content of sugar and corn syrup mixture = 92 parts per million on the dry solids.

Temp. in °F.	Time of boiling. Minutes and seconds.	Percentage of solids.	Residual sulphur dioxide in parts per 1,000,000.	Average.	Residual sulphur dioxide, per 1,000,000 of dry solids.	Sulphur dioxide evolved, per 1,000,000 of dry solids.
Initially					92	
240	7–45	88·1	57	56	64	28
	8–15	88·1	55			
250	9–00	90·5	53	51	56	38
	9–15	90·6	49			
260	10–00	93·5	42	41	44	48
	10–00	93·4	40			
270	11–00	94·8	35	34	36	56
	11–15	94·8	33			
280	11–30	95·3	28	25	27	65
	11–45	95·5	22			
290	12–30	96·0	20	19	21	71
	12–30	96·1	18			
300	13–00	97·6	13	11·5	12	80
	13–00	97·7	10			

TABLE F.

RELATION BETWEEN TEMPERATURE OF BOILING AND PERCENTAGE OF DRY SOLIDS IN RESULTING MASS.

Temperature of boiling. °F.	Percentage of dry solids. A.	B.	C.	D.	E.	Average.
240	88·1	88·1	88·2	88·1	88·2	88·1
250	90·4	90·8	91·2	90·6	90·5	90·6
260	93·1	93·2	92·9	93·4	93·4	93·2
270	94·6	94·8	94·7	94·6	94·8	94·7
280	95·7	95·9	95·6	95·4	95·4	95·5
290	96·1	96·1	96·3	96·0	96·0	96·1
300	97·4	97·1	97·7	97·5	97·6	97·5

RESULTS AND INFERENCES.—(1) It is seen from the experimental figures that in each series of results the residual amounts of sulphur dioxide diminish with rise in the temperature to which the mixture is boiled.

(2) When the residual sulphur dioxide contents, based on dry solids, are plotted against the temperatures of boiling in each series, five practically regular curves are obtained, which are of a similar nature.

These curves show that the amount of sulphur dioxide evolved increases with the temperature to which the sugar and corn syrup mixture is boiled.

Similarly, the sulphur dioxide contents of the various corn syrup mixtures at the same temperature are plotted against the sulphur dioxide contents of the original mixtures before heating. Regular curves are obtained, showing that a relationship exists between the temperature of boiling and the sulphur dioxide evolved.

(3) Table F shows that the dry solids content at each temperature approximates the same value in each series of experiments. A determination of the dry solids will indicate the temperature to which a sugar and corn syrup mixture has been boiled.

The results of the investigations are of particular importance to the Public Analyst examining samples of confectionery with a view to ascertaining whether bleaching agents have been added during the process of manufacture. A method is indicated by which can be ascertained the probable maximum limit of sulphur dioxide which could be present in a sample of confectionery made up of sugar and glucose. This necessitates plotting a curve as mentioned in (2) above, *viz.* residual sulphur dioxide content against temperatures of boiling.

The proportions of sugar and corn syrup in the sample can be obtained by analysis, while the temperature to which the mixture has been boiled will be indicated from the amount of dry solids present. Experiments have shown that dry solid content depends only on the temperature of boiling, irrespective of the initial amount of water present. Assuming that the corn syrup contains the maximum permissible amount of sulphur dioxide, the corresponding curve can be

drawn which will give the maximum amount of sulphur dioxide in parts per million of dry solids that we should expect to be present at various temperatures. Such a curve will, at least, give a good idea of the sulphur dioxide content limit.

I hope, in the future, to publish further results, dealing with the effect of variations in the time of boiling on the residual sulphur dioxide.

The Determination of Cadmium and Copper in Spelter and Zinc Ores by Rapid Internal Electrolysis.

By ELLA M. COLLIN, B.Sc., A.I.C.

(*Read at the Meeting, April* 2, 1930.)

The separation of zinc and cadmium as sulphides is always attended with a certain difficulty, as the slight difference in acid concentration necessary for the precipitation of the two sulphides necessitates a repetition of the process two or more times. A rapid quantitative separation of these two metals may, however, be effected by the Internal Electrolysis method devised by Dr. Sand, already used for the separation of bismuth and lead, and fully described in preceding papers (Analyst, 1930, 309). For the separation of zinc and cadmium the anodes were of zinc, and were made by wrapping several thicknesses of zinc foil round a glass tube flanged at both ends. A stout piece of tinned copper wire with platinum foil at one end was soldered on at the top for making connection with the cathode. A dilute solution of zinc sulphate (5 per cent.), acidified with sulphuric acid, was used in the anode compartments.

Preliminary experiments on a solution containing only zinc and cadmium showed that cadmium can be satisfactorily deposited from a sulphate solution of low acidity at a temperature of about 70° C. in 12 to 15 minutes. The *p*H of the electrolyte was measured either electrometrically or by a capillator to determine the optimum value for the deposition, as the cadmium is not deposited if the *p*H is too low, and zinc hydroxide tends to be precipitated if it is too high. The buffer solution acetic acid/sodium acetate was employed to obtain small variations in acidity. The results are recorded in Table I.

These experiments were carried out in a solution containing 5 grms. of zinc sulphate.

TABLE I.

Cadmium added. Grm.	Cadmium found. Grm.	*p*H value.
0·0045	Nil	2·4
0·0045	0·0004	2·8
0·0045	0·0010	3·0
0·0045	0·0036	3·6
0·0045	0·0046	4·0
0·0014	0·0014	4·0
0·0028	0·0027	4·0
0·0090	0·0089	4·0
0·0045	0·0044	4·8

These results show that the lower limit for the *p*H for the deposition of cadmium is 4, but subsequent determinations were made in solutions having a *p*H between 4·5 and 5·5.

The use of methyl orange as indicator for the adjustment of the acidity of the solution prior to the deposition of cadmium is convenient, as its colour change takes place between *p*H 4·5 and 5. It was found that a solution of the required acidity could easily be reproduced with the aid of an acetate buffer, the necessity of measuring the *p*H value of each solution before electrolysis being thus obviated. A solution containing 5 grms. of zinc, as sulphate, with known amounts of cadmium in a volume of about 300 c.c. was first made neutral to methyl orange with ammonia. Two drops of a 2 per cent. solution of sulphuric acid and 5 c.c. of a 5 per cent. solution of sodium acetate were then added, the solution turning yellow after the addition of the latter. Each of the following determinations was made in a fresh solution made up in this way, the values of the *p*H recorded showing that the conditions necessary for the deposition of cadmium are readily reproduced, and can be relied on within the limits required. The electrolysis was carried out at a temperature of about 70° C.

TABLE II.

Cadmium added. Grm.	Cadmium found. Grm.	*p*H value.
0·0062	0·0062	5·8
0·0269	0·0272	5·4
0·0045	0·0045	4·8
0·0117	0·0123	5·0
0·0067	0·0065	5·0
0·0018	0·0018	5·0
0·0014	0·0011	5·0
0·0028	0·0027	5·5
0·0042	0·0044	5·5
0·0014	0·0014	5·3
0·0029	0·0031	
0·0037	0·0038	
0·0050	0·0050	5·5
0·0070	0·0066	5·8
0·0105	0·0098	5·0
0·0057	0·0055	5·4

In order to make this separation of zinc and cadmium applicable to the analysis of ores and spelter it is necessary to consider the effects of other metals which may be present.

COPPER.—The deposition of copper from sulphate solutions is not usually satisfactory. It was found with this apparatus that in a solution of zinc sulphate containing an excess of 20 c.c. of 2 per cent. sulphuric acid in a volume of 300 c.c. the deposition of cadmium is prevented, but copper is deposited, although the deposit is not of a good colour, and results slightly in excess of the theoretical are usually obtained. After the removal of the copper the acidity of the solution can be re-adjusted and the cadmium be deposited.

Experiments in nitric acid solution showed that, although a good copper deposit is obtained from a solution containing an excess of 10 c.c. of 10 per cent. nitric acid, it is not possible to deposit the cadmium afterwards from the same solution, even when the acidity has been re-adjusted to a *p*H value of 5.

Experiments were made with a solution containing 5 grms. of zinc, as nitrate, and an excess of 10 c.c. of 10 per cent. nitric acid, the copper being deposited first at a temperature of 70° C. and the electrolyte then re-adjusted with ammonia, sulphuric acid and sodium acetate as previously described. The following results were obtained:

TABLE III.

	Amounts added. Grm.	Amounts found. Grm.	
Copper	0·0050	0·0050	
Cadmium	0·0070	0·0037	
Copper	0·0050	0·0045	
Cadmium	0·0042	0·0030	*p*H 4·4
Copper	0·0050	0·0050	
Cadmium	0·0049	0·0014	*p*H 4·2
Cadmium	0·0028	0·0007	*p*H 5·0 (0·5 grm. of hydroxylamine hydrochloride added)
Copper	0·0050	0·0049	
Cadmium	0·0043	0·0022	
Cadmium	0·0043	0·0005	

It was found that the most satisfactory way of separating copper and cadmium present together in solution is to deposit the copper first from a sulphate solution containing an excess of 20 c.c. of 2 per cent. sulphuric acid in a volume of about 250 c.c. If the amount of copper present is more than 3 mgrms., the deposit can be dissolved in nitric acid, 10 c.c. of 10 per cent. nitric acid added in excess, and the solution, after dilution to 100 c.c., electrolysed at a temperature of 70° C., when an excellent deposit is obtained. The original sulphate solution, after the removal of copper, is re-adjusted with ammonia, sulphuric acid and sodium acetate, as previously described, and electrolysed for cadmium.

TABLE IV.

	Amounts added. Grm.	Amounts found. Grm.	
Copper	0·0050	0·0049	
		0·0047	after re-electrolysis in nitric acid solution
Cadmium	0·0040	0·0041	
Copper	0·0100	0·0104	
		0·0095	do.
Cadmium	0·0043	0·0044	
Copper	0·0050	0·0048	do.
Cadmium	0·0044	0·0044	

ANTIMONY.—Experiments with solutions of zinc sulphate containing known amounts of copper, cadmium and antimony showed that antimony is not deposited with either the cadmium or copper, under the conditions to be given for analysis.

IRON.—Iron does not interfere with the electrolysis of copper and cadmium in this apparatus if present only in small quantities, but if there is a relatively large amount of iron the cadmium deposit will contain traces of it, and there is also a tendency for some of it to be precipitated as hydroxide in the feebly acid solution from which cadmium is deposited and to cause mechanical contamination of the cathode.

Attempts were made to determine the amount of iron deposited with the cadmium by dissolving the deposit in nitric acid and matching the colour formed with potassium thiocyanate against a standard solution, but in most cases the amount was too small and the test not sufficiently accurate.

Unless the amount of iron present is small, and it is not required to be determined, it is better to precipitate it prior to the electrolysis of copper and cadmium. In the case of ores, where one grm. is taken for analysis, the amount of zinc present can be held in solution by the addition of 5 to 8 grms. of ammonium chloride, and the iron be precipitated by ammonia, but in the case of spelters, where a large amount of zinc is dealt with, an excess of ammonia is added to re-dissolve the zinc hydroxide. Before the precipitation, 5 c.c. of a 10 vol. solution of hydrogen peroxide is added to convert all the iron to the ferric condition, any excess of the peroxide being removed by boiling the solution prior to filtration. It is usually necessary to make a double precipitation of iron, and, for its quantitative determination, better results are obtained by final titration with permanganate than by weighing as oxide.

In the experiments recorded in the first section of Table V, the cadmium (and copper if present) was first deposited from a sulphate solution containing 5 grms. of zinc, as previously described, and the iron precipitated from the electrolyte by the addition of excess of ammonia. In the second section the iron was precipitated before the electrolysis.

TABLE V.

IRON PRECIPITATED AFTER THE ELECTROLYSIS OF COPPER AND CADMIUM.

Expt. No.		Amounts added. Grm.	Amounts found. Grm.
1	Copper	0·0050	0·0046
	Cadmium	0·0072	0·0078 (deposit contained iron, also some precipitated as hydroxide during electrolysis)
	Iron	0·0085	Not determined
2	Cadmium	0·0056	0·0057 (contained trace of iron)
	Iron	0·0085	Not determined
3	Cadmium	0·0042	0·0041
	Iron	0·0085	Not determined
4	Cadmium	0·0056	0·0057
	Iron	0·0085	Not determined
5	Cadmium	0·0042	0·0042
	Iron	0·0017	0·0020 (weighed as oxide)
6	Cadmium	0·0042	0·0043
	Iron	0·0038	0·0038 (weighed as oxide)
7	Cadmium	0·0049	0·0044
	Iron	0·0068	0·0074 (weighed as oxide)
8	Cadmium	0·0042	0·0045 (contained iron)
	Iron	0·0112	0·0122 (weighed as oxide)
9	Copper	0·0080	0·0084 (deposit free from iron)
	Iron	about 0·1	Not determined as some was precipitated during electrolysis
	Cadmium	0·0050	0·0080 (contained much iron)

IRON PRECIPITATED PRIOR TO THE ELECTROLYSIS.

	Added. Grm.	Found. Grm.
Cadmium	0·0042	0·0040
Iron	0·0102	0·0114
Cadmium	0·0050	0·0049
Iron	0·0085	0·0088

ANALYSIS OF SPELTER.—The analysis of spelter is carried out as follows:—Ten grms. of the sample are attacked with 50 c.c. of 10 per cent. sulphuric acid in the cold, preferably overnight. This is insufficient acid to dissolve all the metal, and the residue is filtered off (F_1), washed with water, and dissolved in 10 c.c. of 20 per cent. nitric acid. If tin is present, it is precipitated, and the solution is diluted to 40 c.c., boiled, and then filtered after being allowed to settle. If necessary, the tin can be determined in this precipitate by titration with iodine in the usual manner. After the removal of any tin present, the filtrate is evaporated

with 10 c.c. of 20 per cent. sulphuric acid until fumes appear; this removes the nitric acid and precipitates any lead present. The residue is taken up with water, and the lead sulphate allowed to settle and filtered off. If required, it is ignited and weighed. The filtrate from the lead is combined with the original solution (F_1), 5 c.c. of a 10 volume solution of hydrogen peroxide added and excess of ammonia (sp. gr. 0·880). The solution is boiled to destroy the excess of peroxide and to coagulate the iron precipitate, which is allowed to settle, filtered off, and washed with 2 per cent. ammonia. It is usually advisable to dissolve the iron precipitate in a little hydrochloric acid and to repeat the precipitation. The iron may be ignited and weighed as oxide, but it is preferable to determine it by titration with permanganate. The solution, after the removal of the iron, is just neutralised with sulphuric acid (1: 1), and an excess of 20 c.c. of 2 per cent. sulphuric acid added. It is then electrolysed for copper at a temperature of 70° C. in a volume of about 250 c.c. The deposit of copper is weighed, and may be dissolved in nitric acid and re-electrolysed, as described above. The solution, after the removal of copper, is re-adjusted and electrolysed for cadmium, as previously described.

ORES AND RESIDUES.—The treatment of ores and residues differs only in the initial attack and in the use of ammonium chloride to keep the zinc in solution during the precipitation of iron if a small sample is taken for analysis. Direct attack with nitric acid or hydrochloric acid, followed by evaporation with sulphuric acid, is usually sufficient to decompose the sample, the lead sulphate being filtered off and the analysis continued as for spelter.

TABLE VI.

RESULTS OF ANALYSES.

	Sample number.		Electrolytic method as above.	Ordinary method. Separation of Cu and Cd by H_2S. Cd determined as $CdSO_4$. Cu determined by iodide method.
			Per Cent.	Per Cent.
Spelter	1 two trials	Copper	Trace Trace	Trace
		Cadmium	0·04 0·045	0·06
		Iron	0·13 (weighed as oxide) 0·09 (titrated)	0·10
Spelter	2 contained much tin	Copper	0·052 0·046	0·06
		Cadmium	0·031 0·025	0·028
		*Iron	0·17 (titrated) 0·26	0·25
Spelter	3	Copper	0·072	0·07
		Cadmium	0·104	0·10
		Iron	1·02 (titrated)	1·14

TABLE VI —*continued.*

	Sample number.		Electrolytic method as above.	Ordinary method. Separation of Cu and Cd by H_2S. Cd determined as $CdSO_4$. Cu determined by iodide method.
Spelter	4	Copper	0·014	0·015 (two trials by 0·01 different
		Cadmium	0·136	0·141 chemists) 0·15
		Iron	0·154 (weighed as oxide)	0·074* 0·15
Ore	5	Cadmium	0·70	0·69 0·72
Ore	6	Cadmium	2·90	2·85

* This difference in iron figure is probably due to variations in the sample.

I wish to thank Dr. Sand for his continued interest in this work.

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LONDON, E.C.3.

Notes.

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

THE DETECTION OF OXALIC ACID.

IN THE ANALYST (1930, p. 322) statements are made concerning the extraction of oxalic acid from solution by means of ether.

Oxalic acid, in the presence of free mineral (hydrochloric) acid, dissolves very appreciably in ether, especially when the oxalic acid solution is concentrated. But, in the absence of free mineral acid, oxalic acid dissolves only sparingly in ether. If to 100 c.c. of a 0·1 per cent. solution of oxalic acid 20 c.c. of concentrated hydrochloric acid are added, three extractions of ether will extract about one-fifth of the oxalic acid in solution. An ethereal extraction process, therefore, may be used, under certain circumstances, as a qualitative test for oxalic acid.

The microscopic examination of precipitated calcium oxalate is also a useful test for an oxalate, the crystals being characteristic.

F. W. F. ARNAUD.
J. W. FLINT.

SESSIONS HOUSE,
MAIDSTONE.

THE FATE OF APOMORPHINE AFTER SUB-CUTANEOUS INJECTION.

THE following experiments were carried out with a view to settling a point raised by defending counsel in a forensic case:—

A man was accused of possessing morphine illicitly, and of swallowing the suspected drug in order to defeat the police. The stomach was washed out and the washings examined in the usual way for the presence of alkaloids, the final extraction being made from an ammoniacal solution by means of a mixture of chloroform and alcohol. The presence of an alkaloid was proved, and positive reactions for morphine (or one of its derivatives) were obtained by means of the Marquis reagent and the Denigès–Oliver* test (in which a red colour is developed in a solution containing morphine or heroine by adding hydrogen peroxide, ammonia, and a trace of copper or copper salt to liberate oxygen).

The defending counsel alleged that injections of apomorphine had been recently given to produce emesis, and suggested that the presence of morphine, or a derivative thereof in the stomach, was due to this.

The contention appeared to have nothing to recommend it, for the following reasons:—

(1) If apomorphine had been present in the stomach-wash, its presence would have been indicated by a violet colour in the chloroform-alcohol mixture.

(2) There is no evidence that apomorphine is converted into morphine in the animal organism; on the contrary, the injection of apomorphine is not followed by symptoms of narcosis.

(3) According to Cushney (*Pharmacology and Therapeutics*, 7th Ed., p. 437) and other authorities, the emetic action of apomorphine is due to action on the medulla, and, moreover, "Apomorphine is not excreted into the stomach like morphine."

Nevertheless, it was considered advisable to be able to give a positive opinion, and to this end the experiments were made.

Four rabbits, approximately equal in weight (*circa* 1700 grms.), were chosen; each received a subcutaneous injection and, after being allowed to live for one hour, was killed.

The injections were, respectively:—(1) 160 mgrms. of morphine hydrochloride; (2) 160 mgrms. of apomorphine hydrochloride; (3) 160 mgrms. of apomorphine hydrochloride; (4) 6 mgrms. of apomorphine hydrochloride.

There was, of course, no emesis in any case.

In experiments (1) and (2) the *stomachs and their contents* were extracted by the Stas-Otto process, while in (3) and (4) the *contents of the stomach only* were extracted. In each instance the extracting solvent was a mixture of alcohol and chloroform in presence of ammonia. In experiments (2) and (3) the chloroform-alcohol layer showed the colour characteristic of apomorphine. Only the extracts from (1) and (2) gave precipitates with the usual alkaloid precipitants and also the colour reactions of the morphine group.

No colour reactions could be obtained from the extracts from (3) and (4), except the purple colour of the immiscible solvent in (3). From the stomach contents of No. (4) no indication of any derivative of morphine could be obtained, although the rabbit received the usual therapeutic dose for a man.

* As a result of wide experience in testing for derivatives of opium, I believe these two tests to be, in practice, a sufficient indication of the presence of alkaloids of the morphine group (with the exception of the ethers of morphine, which do not give the Denigès-Oliver reaction), provided the presence of an alkaloid has been proved.

Further, the alkaloid extracted from (2) was completely soluble in a small quantity of pure chloroform in the cold, there being not sufficient residue to give a colour even with the Marquis reagent, showing that the apomorphine extracted was quite free from morphine. The conclusions are, as might have been predicted:

(1) No considerable quantity of apomorphine reaches the *interior* of the stomach after subcutaneous injection, even when given in enormous doses.

(2) The colour in chloroform is a very delicate test for apomorphine.

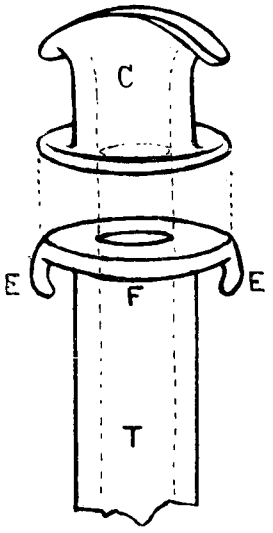
(3) No part of the apomorphine injected is converted into morphine.

F. BAMFORD.

MEDICO-LEGAL DEPARTMENT,
CAIRO.

A MODIFIED GUTZEIT ARSENIC APPARATUS.

THE apparatus described below is a modification of that given in the British Pharmacopoeia, 1914, and by Hill and Collins (*Chem. and Druggist*, 1905, 548). As various analysts have pointed out, the stains produced by this apparatus are often irregular in shape, and are, therefore, difficult to compare. Improved forms of the apparatus have been suggested (Harvey, *Chem. and Druggist*, 1905, 168; Hibbert, *J. Soc. Chem. Ind.*, 1916, 672; Stubbs, White and Cribb, ANALYST, 1927, **52**, 699), but that described below is simpler than those recommended by these workers. In addition, it possesses the merits that:—



(1) It is entirely composed of glass, and is, therefore, readily cleaned by chemical methods.

(2) The stains formed are all of uniform size and each is of uniform tint over its whole surface.

(3) The loss of arsenic, through escape of the evolved gases round the paper cap, is reduced to a negligible minimum (*vide infra*) by making these gases to pass through the paper by diffusion.

The vessel for the generation of the arsine may be selected by the analyst, for the important part of the apparatus is the device for the securing of the mercuric chloride paper over the top of the tube.

The tube, T, is 20 cm. long, with internal bore 5 mm. and external diameter 8 mm. A somewhat stout tube is used, because the working of it into the required shape, without distortion of the internal dimensions, is much easier with a thick tube than with one whose wall is thin.

The flange, F, at the top, is made without widening the internal diameter more than is necessary, and is then ground down to form a perfectly plane surface with the hole of the original bore. Two ears, E, are sealed on to the flange.

A disc of mercuric chloride paper is cut out with a cork borer and secured between the ground surfaces of the cap and the tube. The cap, C, which is about 2 cm. high, is prepared in the same way as the tube, with an identical flange, whilst the upper end is worked into a double-lipped shape, as shown in the diagram. The cap and the tube are held together by a pair of rubber bands (conveniently

cut from a piece of rubber tube) passing beneath the ears and resting in the hollow formed by the two lips.

It has been shown by the following series of experiments that the loss of arsine by leakage through the joint is negligible:—

(1) Test stains were prepared in the usual way with 0·01, 0·008 and 0·006 mgrm. of arsenious oxide in the apparatus.

(2) Similar stains were made with the same amounts of arsenic, the same samples of zinc and hydrochloric acid, but with the joint made gas-tight by means of a ring of paraffin wax. In each experiment the corresponding stains were identical.

The apparatus has been in use for 2 years in the laboratories of Messrs. Baird and Tatlock (London), Ltd., and has been found to give reliable results.

The author desires to express his thanks to the above company for facilities to prepare and investigate this apparatus, and for permission to publish the description. It should be noted that the above design has been registered (No. 754,404).

A. J. Linsey.

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Cross Street, Hatton Garden, E.C.

Notes from the Reports of Public Analysts.

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

COUNTY OF LANCASTER.

Annual Report of the County Analyst for the year 1929.

During the year the total number of samples examined was 5327, of which 4993 were submitted under the Food and Drugs Act (140 adulterated).

Milk.—Of the 2757 samples examined, 4·2 per cent. were adulterated; there were 86 "appeal to the cow" samples.

In two cases in which a prosecution was instituted, it became obvious that the deficiency in fat was due to carelessness in not stirring the milk, and the curious defence was suggested that the vendor thought that he was not allowed to stir the milk if the sample was required by an inspector. He was ordered to pay the costs in each of two cases, a total of £7 7s. 3d.

"*Old Milk.*"—A sample which was sold to the inspector as "old milk" contained only 1·3 per cent. of fat. There appears to be some evidence that this term is used locally as synonymous with skimmed milk, but, as the usage is by no means general, the sample was classified as adulterated, and the vendor was warned that in future such milk must be sold as skimmed milk.

Skimmed Milk.—One sample, sold as "skimmed milk," contained 4·2 per cent. of fat. The composition of this sample might have been due to skimmed milk having been allowed to stand for a long time, so that the bulk of residual fat had risen to the surface, or, possibly, to the vendor not being sure of the composition of his milk and describing the sample as skimmed milk, to prevent the possibility of a prosecution. In any case, the circumstances were not considered satisfactory, and the Local Authority concerned has been advised to draw the attention of the vendor to these points.

One sample, described as "Machine-skimmed milk," was received during the year which contained 2·1 per cent. of fat, whilst a further sample described as "Skimmed milk" contained 3·7 per cent. of fat. Neither of these samples was, of course, correctly described. It is suggested that the descriptions were given to them by vendors, who were uncertain of the quality of their milk, and thought that such a declaration to the Inspector would save them from trouble. It is very doubtful whether they would be so described to any but an official purchaser.

The Warranty Defence.—In one case the vendor of an adulterated milk produced a warranty, and the Magistrates decided that he had sold the milk in the same condition in which he received it. A case against the farmer who supplied the milk was then taken, and the Magistrates held that the warranty was given in good faith and discharged the defendant.

This case well shows the difficulty of obtaining convictions where the warranty defence is pleaded, unless both defendants are tried by the same Court and at the same time. In the case under discussion, milk which was, no doubt, adulterated was being sold to the public. The retailer pleads warranty, which is accepted, and he is discharged. At the subsequent hearing, on a charge of false warranty, the person giving the warranty contends that the milk was not sold to the Inspector in the same condition in which it was received. This plea may also be accepted, and the second defendant is discharged, so that, although it is common ground that the milk has been adulterated, no one is punished for the offence, and the public is not protected as the Act intended. In such instances the case against both defendants should be heard together. The Court would first of all decide whether the milk was adulterated, and, having decided that point, would proceed, if necessary, to settle the responsibility for the adulteration, between the vendor of the milk and the giver of the warranty.

Canned Cream.—Ten samples of canned cream contained from 21·2 to 25·6 per cent. of fat, whilst one contained 39·9 per cent. The manufacturers state that it is not possible to prepare a canned cream containing more than about 25 per cent. of fat. The difficulty seems to have been overcome by the manufacturer of the sample mentioned above containing 39·9 per cent. of fat. As the sale of tinned cream is becoming very general, and as it is not unlikely to be sold in competition with fresh cream, it would appear to be very desirable that labelling regulations, along the lines of those now in force for condensed milk, should be laid down.

Tartaric Acid.—One sample consisted entirely of cream of tartar. The remaining 10 samples contained no arsenic, whilst the amount of lead varied from 0 to 15 parts per million.

Ammoniated Quinine Tablets.—Ammoniated quinine tablets are prepared with the object of supplying a solid medicament which shall take the place of the ordinary ammoniated tincture of quinine. They are composed of quinine and ammonium carbonate with the binding materials necessary to prepare the tablet. They are usually prepared of such a strength that one tablet is equivalent either to a teaspoonful or half a teaspoonful of the tincture. There is no difficulty in

preparing the tablets of the correct strength, so far as quinine is concerned, but unless the tablets are very carefully prepared and stored in hermetically sealed bottles, there is a very great risk of deficiency in ammonium carbonate, even though the correct amount had originally been present.

One sample was found to be deficient to the extent of 90 per cent. of ammonia. On this being pointed out to the manufacturers, they replied that it was not possible to manufacture a tablet containing ammonium carbonate which would retain its strength unimpaired on keeping. Another sample of tablets, prepared by a different maker and received on the same day, was found to be practically of the correct strength, due, at least in part, to the fact that care had been taken in packing to counteract the volatile nature of this substance. The makers have undertaken to add a statement to their labels that the tablets are likely to be deficient in ammonia.

A third sample was found to be deficient to the extent of 90 per cent. of the correct quantity of ammonium carbonate, and the attention of the persons concerned was called to this deficiency, and to the cause thereof.

In addition to the variation in ammonia, such tablets vary in their proportion of quinine. Some of them contain an amount equivalent to a teaspoonful of the B.P. tincture, whilst others contain an amount equivalent to half a teaspoonful. This is not particularly objectionable in those cases where the strength is clearly stated on the label, but it is by no means always done, and it would appear to be desirable that some uniform practice should be adopted.

LABELLING OF PHOSPHATE FERTILISERS.—In the case of the phosphate fertilisers examined, the amount of phosphate given in the Statutory Statement was stated in terms of calcium phosphate, in place of phosphoric acid, as required by the Act. As the composition of the articles was quite satisfactory, this cannot be regarded as a serious offence, but such a method is undesirable, as, unless the method of presenting the results is taken into account by the purchaser, mistakes may very easily arise.

G. D. ELSDON.

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.

"CRYSTAL" SYRUP SOLD AS GOLDEN SYRUP.

ON June 7th a firm of Burslem grocers was summoned at Hanley for selling "Crystal" syrup as golden syrup. The Inspector said that he bought a pound of golden syrup, and while he was sealing the samples he was informed that the assistant, in ignorance, had supplied him with "Crystal" syrup instead of golden syrup.

The report of the Public Analyst showed that the syrup contained 175 parts per million of sulphur dioxide, whereas the maximum allowed in golden syrup is 70 parts per million.

In reply to the Stipendiary Magistrate, the Inspector said that there was not much difference between the prices of golden syrup and "Crystal" syrup.

The Magistrate said that he did not think that there had been any intention to defraud, but the fact remained that something had been sold which was not what it purported to be. He imposed a fine of £2 2s., with £3 3s. costs.

LABELLING OF PORT WINE.

A FIRM of wine and spirit merchants was summoned at Margate, under the Merchandise Marks Act for applying a false trade description to bottles of Australian wine sold at the shop on March 15th.

Mr. R. T. Monier-Williams, appearing on behalf of the Port Wine Shippers' Association, said that the proceedings were taken under the Anglo-Portugal Commercial Treaties Act, which was passed in 1914, and stipulated that only wine coming from a limited area in Portugal could be termed "port wine." With the sale of any port wine imported into this country a certificate of origin, issued by the Portuguese Government, was necessary.

In this case the words "Port Royal Australian" were the large words upon the label. The letterpress was immaterial, although the word "brand" was printed in small letters beneath the word "royal." He submitted that the words were so placed to suggest a connection between port and Australia.

Mr. F. G. Bray, for the defence, submitted that there was nothing to prevent a man naming his wine after a particular place—Port Royal. He contended that the label in no way contravened the Act.

The Magistrates imposed a fine of £1 on each summons, a total of £9, with £5 5s. costs.

Department of Scientific and Industrial Research.

REPORT OF THE FOOD INVESTIGATION BOARD FOR THE YEAR 1929.*

SECTION A. MEAT.—Investigations have been conducted on the loss of bloom during the transport of New Zealand frozen mutton and lamb; the factor of quality in the freezing of meat; physiology of rigor mortis, and the freezing of tissues.

Since the alteration in colour of ox's muscle on drying may either produce an increase in depth of colour (*e.g.* on hanging uncovered for 24 hours at ordinary temperature and humidity), or a decrease in colour with a withered appearance of the muscle (seen for example in frozen mutton or lamb), and similar changes may be observed when there is no chemical change in the pigment (*e.g.* on drying in pure nitrogen), the changes are believed to be due to alteration in the optical properties of the muscle. In one case a gel-like superficial coating is formed on drying a large muscle, and the change in transparency will increase the depth of light penetration, and this, with the increased concentration of pigment, leads to an increase in depth of colour. Under other conditions the interspaces of strands and fibres of dried tissues may be filled with air, when there is a decrease in colour.

* Obtainable at H.M. Stationery Office, Adastral House, Kingsway, W.C.2. Pp. 146. Price 2s. 6d. net.

Rancidity of Fats.—Work dealing with changes in fats during storage includes an examination of rancidity tests in use; and, while the Kreis and Schiff tests have been found useful, a new test for detecting rancidity in the earliest stages has been elaborated whereby the oil or fat is heated with a solvent mixture of glacial acetic acid and chloroform (2:1 by volume) in the presence of solid potassium iodide. The tube is filled with nitrogen, to prevent oxidation, and the steady evolution of chloroform vapour prevents diffusion of oxygen back into the tube. On cooling, the reaction mixture is poured into 5 per cent. potassium iodide solution and titrated with 0·002 per cent. thiosulphate solution. With beef or mutton fat the sensitiveness is about 20–30 times greater than that of the Kreis test on a similar quantity of material. This method has also been used for following the effect of light on fats. It was seen that the process of oxidation had little effect upon the free fatty acidity of the fat, titrations up to 60 c.c. of thiosulphate per grm. only resulting in increases of acidity of 0·2 to 0·3 per cent. (as oleic acid). Once oxidation has started, it proceeds, after removal of the source of light, at a velocity which depends upon the amount of active oxygen already present. Samples of fat from different animals may show different rates of oxygen absorption, and the catalytic action of light does not vary directly with intensity, but chemical action decreases to an approximately corresponding extent to the decrease in intensity of light up to a certain point, after which, reduction in intensity produces relatively much less change in the resulting catalytic activity. Very little change occurred in frozen mutton and lamb fat after 18 weeks' storage, except that, in the case of a carcase that had been frozen and had been brought out of store from time to time and allowed to sweat, oxidation had commenced, although the Kreis and Schiff tests were still negative. The investigation into the yellowing of the abdominal fat of frozen rabbits points to the conclusion that the "yellow fraction" arises from the oxidation of the linoleate glycerides, and that this reaction may be catalysed by a tissue oxidase, water and haemoglobin, and also that the pigment is an unsaturated ketonic acid, possibly α-β-di-keto acid.

Crystallisation of Fats.—Thin films of mutton and beef fats, heated to 50° C. and cooled slowly, develop doubly-refracting rosette-shaped crystals; but, when cooled rapidly in mercury at –15° C., the crystals are wholly microcrystalline. If the fats are cooled slowly in the region of the setting point, needle-shaped crystals may be obtained, probably composed of the higher-melting, fully-saturated glycerides. Thin layers of fat cooled rapidly are less opaque than if cooled slowly, and photometric experiments on layers 0·06 mm. thick showed that the transparency of the fat on slow cooling is only 65 per cent. of that cooled in mercury at –20° C. Beef fats have little tendency to supercool, but temperature affects velocity of crystallisation. Fat, melted at 60° C., plunged in mercury at –15° C. for 3 minutes, warmed rapidly in mercury at 20° C. until the thermometer in the fat registered 20° C., and placed in a thermos flask at 20° C., showed a rapid rise in temperature to 22·2° C., due to heat of fusion of the still liquid fat, which crystallises rapidly at 20° C. This evolution of heat gradually diminishes on storage, being eliminated completely when tubes of fat are stored at –15° C., 0° C. and +12° C. for 2, 1·5, and 0·3 hours, respectively.

Bacteriology.—An investigation has been started to determine the extent to which carcases are exposed to bacterial contamination in slaughter-houses during killing and dressing, and bacterial contamination of carcases in a well-conducted cold store has been found to be practically negligible, and the air was purer than in many well-ventilated rooms. But bacteria were present in the ice round the pipes and in the dirt, etc., on the floor, a surprisingly large number being actinomyces. These grow well on potato, Czapek's agar, cellulose, sugar, etc., but

less well on media rich in protein, and are peculiarly adapted to spreading in cold stores. Fresh minced beef, heavily inoculated with a strain of *coli* at 37° C. and 20° C., and a control sterile autolysis at the same temperature, showed that even under optimum conditions there is a lag of some 40 hours at 37° C., during which the bacteria multiply slowly at the expense of the soluble nitrogenous compounds and then grow vigorously, so that over 50 per cent. of the total nitrogen of the proteins was broken down in six days at 37° C. Cooling of the carcases immediately after slaughter is advised.

SECTION B. FRUIT AND VEGETABLES.—The biochemical study of senescence in apples has been continued, and the effect of climatic conditions on storage life; loss of water from apples in relation to humidity, and the effects of humidity on their storage, were further studied. The effects of acetaldehyde on the growth of moulds has received considerable attention, and, according to the concentration in which it is present, this substance may retard or inhibit germination (3 to 5 parts in 10,000) or growth of spores, or may kill them. Killing is a function of concentration, temperature and the time of exposure.

Other work reported on includes the storage life of pears in artificial atmospheres, and wastage of fruit in commerce and during transport overseas.

SECTION C. PIG PRODUCTS.—This section includes the freezing and storage of mild-cured bacon; the scientific basis of curing, and the effect of sodium chloride on the superficial micro-flora of meat.

SECTION D. BIOLOGICAL ENGINEERING. *Corrosion of Tin.*—Experiments on the corrosion of tin, iron and tin-iron couple in 0·5 per cent. solutions of citric acid buffered with sodium citrate, a range of H ion concentration from *p*H 2·41 to *p*H 5·54, were carried out in the presence and absence of oxygen. Experiments were made with tin plate itself, usually over periods of about two weeks, and the main factors influencing corrosion are the acidity of the foodstuff with its unequal effect on the corrosibility of the two metals in the presence and absence of oxygen; the presence of oxidising or depolarising agents (*e.g.* anthocyanin pigments) which encourage attack on both metals; presence or absence of accelerators or inhibitors; the fact that tin compounds tend to become concentrated in the solid portions, which encourages further attack on the tin and lessens the inhibiting effect of tin salts on the corrosion of iron; and the presence or absence of protective coatings, lacquers or enamels on the inside of the tin. A large area of exposed tin has a protective action on the iron, and in lacquered cans the iron and tin are initially exposed in more equal amounts, and corrosion then more resembles that of plain iron. Lacquer protects red- or purple-coloured products from the action of tin, which turns them blue, but the lacquering must be as perfect as possible.

SECTION F. FISH.—*Methods of Preservation.*—Experiments at sea on the preservation of haddock on long voyages have shown that the most satisfactory method tried is to brine-freeze to equilibrium at a temperature of 0° to −5° F. After storing at −10° F. for six weeks a cure was obtained with good texture, glossy pellicle and elastice translucent flesh, the flaps being only slightly discoloured and the eating quality good. Air freezing to equilibrium at 27° F., and storing for six weeks before canning, produce a good eating quality with roughter surface and more discolouration. Freezing in brine at 0° to −6° F. and storing at 20° F. is unsatisfactory, as is air freezing in cold store at 20° F., unless curing is effected within 14 days.

SECTION G deals with researches conducted at the National Physical Laboratory under the direction of the Engineering Committee.

SECTION H includes research at the Imperial College of Science and Technology for the Director of Food Investigation, connected with chemical and biological work on fruit. D. G. H.

REPORT OF THE WATER POLLUTION RESEARCH BOARD FOR THE YEAR ENDED 30TH JUNE, 1929, WITH REPORT OF THE DIRECTOR OF WATER POLLUTION RESEARCH.*

THIS is the second Annual Report (ANALYST, 1929, **54**, 107), and the investigations noted in the first have been carried further. The monthly summaries of current literature on water pollution research, made primarily for the guidance of the Board, are now being printed and are on sale at H.M. Stationery Office, beginning with the first number of Volume III for January, 1930 (*cf.* ANALYST, 1930, 195).

BEET SUGAR FACTORIES EFFLUENT.—Modifications in methods of operation have been made at several factories in accordance with the conclusion in the first report that waste waters should, as far as possible, be re-used. A factory dealing with 1,000 tons of beet a day would use 3 to 4 million gallons of water; 2·5 to 3·5 million gallons from the condensers, used again for condensing or for transport of the beet; transport and beet washing water (often as above), 2·5 to 3·5 million gallons; diffusion and pulp press water, 300,000 to 500,000 gallons; and water for conveying spent lime to lagoons, 40,000 to 80,000 gallons. Processes of screening and sedimentation for removing spent solids from the transport and washing waters have proved satisfactory at certain factories on a large scale, re-use of the water thus being made possible. In some factories the cossettes are extracted by continuous diffusion, whereby no waste press or diffusion water is produced. Prospects of solving or alleviating the problem of the disposal of beet sugar factories' effluents are, therefore, good.

Investigations of the process of biological filtration of the effluent at Rothamsted included the general survey of the organisms involved, with the object of selecting those of sufficient importance to merit further study. Where the filter media were not changed the film and bacterial population had to be built up again during the campaign 1928–1929, since the great majority of bacteria isolated in 1927–1928 were non-spore-formers. In 1928–29 smaller quantities of humus were discharged. "Springtails" (*Achorutes*) were scarce, and sewage fly (*Psychoda*) was not abundant till after the end of the campaign. Bacterial platings were made from samples of gelatinous material scraped from the medium of the filters at Colwick, and from each plating 50 colonies were taken for examination, about 800 cultures being thus collected. These are being tested on gelatin and on 7 sugars. It seems probable that, in the purification by biological filtration, oxidation of the sugars is by stages, each stage being brought about by a particular group of organisms.

SURVEY OF THE RIVER TEES.—The survey was not begun till April, 1929. Sixteen stations on the upper reaches have been selected for systematic investigation, and others for occasional examination. During April and May, flowering plants were rare in the upper reaches; mosses were not abundant; *Lemanea* (filamentous algae) occurred in the swiftest stretches, quantities of *Cladophora* were present below the junction of the Skerne, and a considerable quantity of sewage fungus in the Skerne itself. The most varied zoological collection was taken at Low Middleton. In the tidal reaches, determinations are made of salinity, dissolved oxygen, nitrite, ammonia, dissolved oxygen absorbed at 18° C. in 5 days; tar acids, phosphates, iron, opacity, and *p*H values, and zoological and botanical collections are being made. It is hoped to calculate the volumes of water moving in different parts of the estuaries at various states of the tide; the rates of surface

* Obtainable at H.M. Stationery Office, Adastral House, Kingsway, W.C.2. Price 9d. net.

and sub-surface flow, the quantity of fresh water reaching the sea on each tide; and the time taken for fresh water to travel from Stockton to the sea.

BASE EXCHANGE OR ZEOLITE PROCESS OF WATER SOFTENING.—Experimental work at Teddington is to provide information on (1) the exchange values of typical commercial base-exchange materials, (2) the variations in the properties of different batches of particular commercial products; (3) the variations in exchange value, with different rates of flow of water; (4) the bulk densities of different materials and the volumes of the interstitial spaces, with definite grades of sizes; (5) the changes which result from various methods of regeneration, with solutions of salt of different concentrations; (6) the comparative amounts of material lost by disintegration; and (7) the solubilities of the materials in water under various conditions of operation of the process. (*Cf.* ANALYST, 1930, 46.)

CO-ORDINATION OF RESEARCH.—The Research Sub-Committee, appointed by the Institution of Gas Engineers in June, 1926, to consider methods of overcoming difficulties associated with disposal of liquor effluents from gas-works, has issued three reports. Experiments at Hinckley have shown that modifications in the usual methods of cooling and scrubbing the effluent can reduce the proportion of thiosulphate and thiocyanate, and the removal of the heavier tar from the hot crude coal gas before the condensation of liquor will result in a reduction of the higher acids in the effluent. These latter may also be much reduced by use of the electrostatic tar precipitator, but this caused an increase of monohydric phenols. At Coventry four percolating filter beds have been introduced for biological purification of gas liquor effluent. These were matured with sewage and are now treating effluent, without sewage, diluted with effluent from the filters.

D. G. H.

Fuel Research.

THE REACTIVITY OF COKE (III). INFLUENCE OF IRON COMPOUNDS.*

IRON compounds which are capable of easy reduction to the metal have an accelerating effect on the reaction between carbon dioxide and coke (ANALYST, 1929, **54**, 471). During the course of a reactivity determination, this reducible iron is present as the metal at the reactivity I stage, and as ferrous oxide at the reactivity II stage. The activating influence of metallic iron is large, that of the oxide small.

If the reducible iron is removed or rendered inert, close approximation to the reactivity III value may be obtained without prolonged passage of carbon dioxide. This may be effected either by extraction of the coke with mineral acids or by treating it with hydrogen sulphide, silicon dioxide, titanium oxide, or alumina, each of which combines with the iron and renders it inert. Analytical determinations reveal a relationship between the amount of reducible iron present in a coke and the type of its reactivity curve.

Study of equilibrium conditions in the system, carbon dioxide—carbon monoxide—iron—ferrous oxide, furnishes an explanation of the phenomenon of reactivation, and verifies the conclusions drawn with regard to the catalytic effect of iron in the metallic state. Variation of the temperature, or $CO:CO_2$ ratio, prevailing during the reaction renders it possible to cause transformation of iron from the oxidic to the metallic state or *vice-versa*, with consequent activation or deactivation

* Technical Paper No. 25. Obtainable at H.M. Stationery Office, Adastral House, Kingsway, W.C.2. Price 2d.

of the coke. The phenomenon of reactivation, an auto-activation occurring with some cokes, during the passage of nitrogen at a high temperature, is due to reduction of iron oxide to the metal by the coke, and is observed only with cokes containing the largest proportions of reducible iron.* Metallurgical cokes contain some iron which is soluble† but not reducible, but the amounts of both the soluble and the reducible forms may be increased by a process of oxidation at 450° C. Possible catalytic effects, due to inorganic constituents other than iron, are of limited extent. Reactivity I represents the reactivity value of a coke under such conditions that the ash can exert its full catalytic effect, whereas reactivity III is the value obtained when the iron has been converted to ferrous oxide, or otherwise rendered inert, and is a close approximation to the ash-free reactivity value of a coke. T. H. P.

* "Reducible iron" here means iron in the metallic state or as oxide.

† "Soluble" means readily extracted by mineral acids.

The National Physical Laboratory.

REPORT FOR THE YEAR 1929.*

THE volume contains the usual reports of the Executive Committee and Director, statistical comparisons of tests made in the period 1927 to 1929, a list of publications for 1928 and 1929, and detailed reports of the Departments of Physics, Electricity, Metrology, Engineering, Aerodynamics, and Metallurgy, and of the William Froude National Tank.

PHYSICS.—PHYSICAL CONSTANTS AND STANDARDS.—A calorimeter, suitable for specific heat determinations of up to 160 grms. of heavy, powdered materials, is described, the principal features of which are an electrical heating-coil, arranged so that the heat is generated within the mass of the material, and a special stirrer with large helicoidal blades, which alternately lifts and drops the powder when rotated, and so ensures uniform mixing. The thermo-couples are attached to the blades themselves, and the cold-junction rotates with the stirrer.

Work has been continued on the specific heats of gases at high temperatures (*cf.* ANALYST, 1929, **54**, 340) by the velocity of sound methods, and use has been made of a piezo-electric quartz crystal as the source of sound. The m.pt. of palladium has been determined by two methods, with a probable accuracy of ±2° C., and the results fully justify the value of 1555° C. adopted for the purpose of the International Temperature Scale. International comparisons of the thermo-couple scale are also in progress, while the identity has been established, to within 0·01° C., between the platinum resistance-thermometer and the thermodynamic scale for the range 0 to 100° C.

REFRIGERANTS.—Latent heats of sulphur dioxide between −15° and +25° C., and of pentane, have been determined by balancing the heat required for evaporation of a known weight of liquid by a measured amount of electricity. The specific volumes of a number of refrigerants have been determined, and also the rate of heat transmission by cold pipes measured in a closed circuit wind-tunnel. Other allied problems studied include humidification of air by water-sprays, apparatus for the production of streams of air containing known low concentrations

* Department of Scientific and Industrial Research. Obtainable at Adastral House, Kingsway, W.C.2. Pp. 298. Price 11s. net.

of vapour of essential oils, thermal resistivity of heat insulators, and pipe-line flowmeters. Water has been determined in fogs by vaporisation of the suspended water in an electric heater and measurement of the resulting humidity on a wet- and dry-bulb hygrometer.

RADIOLOGY.—In view of the international adoption of the Röntgen unit of X-ray intensity (r), the standard electroscope previously used for calibrating barium platinocyanide pastilles has had itself to be calibrated in terms of a standard ionisation-chamber. The pastille-dose in "r" units is given by $273CVPd^2/228{,}000\,vTd^2p$, where C is the capacity, V the voltage rise, v the effective volume of ionised air (at room temperature and pressure), d the distance from the focal spot to the effective centre of ionised volume, dp twice the focal distance of the pastille, T (°A.) the temperature, and P the pressure (mm. of Hg.). The four new secondary radium standards re-tubed by the Government Laboratory were found to contain approximately 13·1, 25·7, 60·0, and 106·5 mgrms. of radium element.

Work on industrial X-ray crystal-analysis has been extended to tungsten-magnet steels (with special reference to changes produced by heat and to carbide formation), single metal crystals, electrical insulating materials and the grain-size of paints.

COLORIMETRY.—Confirmation has been obtained of the utility of the colorimetric method of heterochromatic photometry, and measurements are in progress on the 57 standard colours scheduled for ready-made paints by the B.E.S.A.

At the request of the Atmospheric Pollution Research Committee an apparatus has been designed to indicate approximately the total energy and variations in energy-distribution of the spectrum of daylight.

ELECTRICITY.—STANDARDS.—Variations in laboratory standard-cells made up from 0·1 N acid have been tested continuously since 1925, and a variation from the mean of 10 parts per million established.

PHOTO-ELECTRICITY.—In view of the increasing uses of photo-electric cells, investigations on the laws of photo-electricity have been made, with special reference to the validity of Talbot's law, colour-sensitiveness, colour transmission of glass and the recording of daylight and sky-illumination.

METROLOGY.—There has been an increase of 50 per cent. in the amount of glass-ware sent for test, though tests on weights have decreased. An apparatus is being prepared to calibrate a standard steel end-gauge (1 m. or 1 yard in length) in terms of the wave-length of the cadmium red line. At present, devices are being designed to determine the necessary temperature corrections. Optical block-gauges have, however, been measured in terms of krypton radiations, the wave-lengths of which are themselves known in terms of the metre, and agreement obtained with the mechanical method to within 10^{-6} inch. Assistance has been given in the preparation of specifications for volumetric glassware for the Committees on Dairy Research and Standardisation of Tar Products Tests, and reference is made to an article by V. Stott (*Nature*, 1929, **124**, 622) on the use of the millilitre in place of the c.c.

Jaeger's method for the measurement of surface tension has been applied to density determinations. Two glass jets, connected with a common source of air-pressure, are placed in cylinders containing distilled water and the liquid to be investigated, respectively. The depths of the jets are adjusted by raising or lowering the cylinders till bubbles just begin to issue simultaneously from both, and the ratio of the changes in heights gives values of the density accurate to within ± 5 units over the range 0·65 to 1·5 grms./ml.

METALLURGY.—Attempts to prepare pure beryllium by decomposition of the vapour of the iodide have been replaced by sublimation *in vacuo* (see Sloman, *J. Soc. Chem. Ind.*, 1929, **38**, 309T.). Spherically-ground ball and socket joints have been found very satisfactory in place of rubber tubing in the determination of hydrogen in steels by exhaustion methods, and water-cooled caps fitted with such joints have been adopted for the ends of combustion and reaction tubes. The hydrogen reduction method has been found reliable for determinations of oxygen present in metals as oxide of iron or of other easily-reducible metals, so long as accurate temperature control is employed. The cupferron reagent has been used successfully for the determination of iron in alloys for use at high temperatures.

J. G.

New Zealand.

SIXTY-SECOND ANNUAL REPORT OF THE DOMINION LABORATORY.

IN his annual report for the year 1928, the Dominion Analyst (Dr. J. S. Maclaurin) states that 5455 samples from various Government Departments were examined during the year, of which 3621 were for the Health Department, 461 for the Customs, 303 for the Mines Department, and 114 for the Post and Telegraph Department.

CUSTOMS.—Most of the samples were examined to determine their classification for tariff purposes, but some to ascertain whether they complied with the Regulations under the Sale of Food and Drugs Act. They included a number of flours, which were tested for "artificial bleaching." The result has been that imported flour is now free from chemical bleaching.

JUSTICE.—The 42 samples examined included medicines, rat poison, and exhibits relating to cases of suspected poisoning.

Meta Fuel Tablets.—Samples of these were examined, and, in view of possible grave danger to children, the desirability of a clear indication as to their poisonous properties was brought to the notice of the Director-General of Health (*cf.* ANALYST,).

COMPARATIVE VALUE OF LEMONS.—Comparative tests were applied to 10 representative lemons from each of four countries: (1) New Zealand; (2) New South Wales; (3) South Australia; (4) California. The following results, *inter alia*, expressed in terms of the weight of fruit taken, were obtained:

	(1).	(2).	(3).	(4).
Volume of juice in 100 grms. of lemons (c.c.) ..	26·6	43·5	44·0	27·7
Weight of juice in 100 grms. of lemons (grms.) ..	27·6	45·4	46·0	28·5
Weight of citric acid in 100 c.c. of juice (grms.) ..	6·1	6·6	7·2	6·2
Weight of citric acid in 100 grms. of lemons (grms.)	1·4	2·9	3·2	1·7

These results indicated that the South Australian lemons were the best, and the New Zealand lemons the poorest.

SEA WATERS AND BATH WATERS.—Comparative analyses were made of the sea water from various parts of Auckland Harbour and of the water from various sea-water swimming baths. The following are typical of the results obtained:

		Parts per 100,000.					
		Chlorine in chlorides.	Nitrogen in nitrates.	Nitrogen in nitrites.	Ammoniacal nitrogen.	Albuminoid ammonia.	Oxygen absorbed. Four hours at 80° F.
256.	(1) Auckland harbour, Tiri ..	1993·0	nil	nil	0·0012	0·0048	0·045
	(2) ,, ,, sewer outlet	1831	,,	,,	0·1840	0·0320	0·110
873.	(1) ,, ,, Shelly Beach	1682·7	,,	,,	0·0064	0·0150	0·123
	(2) ,, ,, Prince's Wharf	1895·7	,,	,,	0·0025	0·0100	0·042
887.	Tepid baths (six weeks in use)	1327·7	0·06	0·20	0·0350	0·0137	0·112
923.	Tepid bath	1228·3	0·15	0·25	0·0385	0·0140	0·147
994.	Tepid bath	1917·0	nil	nil	0·0053	0·0090	0·040

and total solids also indicated dilution by admixture with sewage. The figures showed that 256 (2) was very badly polluted, and the low chlorides for 873 (1) and (2) indicated considerable dilution, which might be due to the normal tidal washing of the upper harbour, and not to contamination by sewage as such. The sample No. 887 showed evidence of considerable pollution.

THE CRYOSCOPIC MILK TEST.—The milk supply of Christchurch attracted considerable public interest during the year. This led to a close examination of the regulations relating to milk, more particularly from the legal standpoint. On the chemical side the cryoscopic test for the detection of added water in milk was made the subject of a lengthy test case, resulting in a decision in favour of the test.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Determination of Egg in Ice Cream. N. C. Smith. (*J. Assoc. Off. Agric. Chem.*, 1930, **13**, 272–291.)—The method described depends on the quantitative recovery of lipoid phosphorus from the ice cream by the Roese and Gottlieb (Mojonnier) procedure for extracting the lipoids, followed by a colorimetric determination of the phosphorus. After addition to the ice cream (amount not stated) of water, ammonia and alcohol, the material is extracted with two quantities of 25 c.c. of ether and two of petroleum spirit, the solvents being evaporated and the residue dried in a steam oven and dissolved in dry chloroform. The solution is filtered through cotton wool placed in the stem of a small funnel and made up to 50 c.c. with chloroform or 95 per cent. alcohol. A suitable aliquot part is evaporated to dryness with 1 c.c. of 50 per cent. magnesium nitrate solution in a platinum dish on a steam bath, the residue being very carefully ignited with a few drops of nitric acid and then strongly ignited to remove all carbon and nitrogen peroxide. The white residue is dissolved completely in a small quantity of water and 2 c.c. of

sulphuric acid (1 : 1), the clear solution being made up, with washings, to 20 c.c. in a 200 mm. test-tube. Two c.c. of 15 per cent. sodium bisulphite solution, containing 0·5 per cent. of hydroquinone, is then added, followed by 2 c.c. of a solution prepared by dissolving 25 grms. of ammonium molybdate in 300 c.c. of water and 200 c.c. of dilute sulphuric acid (125 c.c. of water, 75 c.c. of concentrated sulphuric acid). The mixture is shaken and the tube placed for 15 minutes in a boiling water-bath and then cooled in running water. The blue liquid thus obtained is made up with water to a definite volume and compared in a colorimeter with standard tubes prepared similarly from small volumes of a solution containing 0·2193 grm. of potassium dihydrogen phosphate per litre (1 c.c. corresponds with 0·05 mgrm. of phosphorus); this solution may be preserved by addition of a few drops of chloroform. Colorations obtained from 0·05–0·1 mgrm. of phosphorus are most satisfactory for comparison.

Taking 50 as the average percentage of total solids in fresh egg yolk, 0·7777 grm. of lipoid phosphorus as the amount in 100 grms. of water-free commercial yolk, and the content of lipoid phosphorus in commercial (*i.e.* containing no egg) ice cream as 2·56 mgrms. per 100 grms., the percentage of dry egg yolk in the ice cream is 0·129 (A–2·56), where A represents the number of mgrms. of lipoid phosphorus found in 100 grms. of the ice cream. No factor is applied for loss of lipoid phosphorus, the average recovery by the above procedure being 96 per cent. A large number of analyses have been made and, even with aged or pasteurised material, the percentage of egg yolk was found to agree sufficiently well with the amount added. T. H. P.

Estimation of the Age of Flour. W. Hartmann. (*Z. Unters. Lebensm.*, 1930, **59**, 364–379.)—Samples of flour and bread (33·3 grms.) were extracted for 6 hours with boiling petroleum spirit or chloroform, and the refractive index at 40° and acid value (c.c. of 0·1 *N* sodium hydroxide solution required for titration, with phenolphthalein as indicator) of the extract determined. Flours recently mill-ground had acid-values of 54 to 76 and 46 to 64 when containing 60 per cent. of rye and wheat flour, respectively, while the corresponding figures for flours of the same composition, ground in the laboratory and left for 24 hours, were 44 and 39. Bleached flours were not included. Extractions, after various intervals, showed that the acid-value increases with time of storage, rapidly at first, to about 240 after 15 months. The refractive index, gluten-nitrogen and lecithin phosphate contents fall on storage at a rapid, particularly in the initial stages. The acid value and refractive index of the unground grain, however, show little alteration on storage. Reductions in refractive index from 69 to 56 and from 79 to 58 were observed for bread baked from rye and wheat flours, respectively, while the acid value was increased in the latter case only. The lecithin phosphate fell from 6 to 1·3 mgrms. per 100 grms. of flour in both cases. The bacterial contents were also determined on samples 5 to 15 months old; and in the presence of growths of *Oidia*, small amounts of an unsaponifiable wax-like substance were obtained. In view of statements that flour is no longer suitable for baking purposes after 6 months' storage, it is

suggested that the maximum permissible acid values in such cases should normally be 120 to 150 for 60 per cent. rye flours, and 90 to 100 for 60 per cent. wheat flours.

J. G.

Formol Titration of Lemon Juices. A. Niethammer. (*Z. Unters. Lebensm.*, 1930, **59**, 420.)—The method of Tillmans and Kiesgen (ANALYST, 1927, **52**, 417) was applied to (1) pressed lemon juice, (2) natural lemonade, and (3) three samples of commercial (artificial) lemonade. The amounts of 0·1 *N* sodium hydroxide solution required by 10 c.c. of liquid were 110·4, 90·2 and 28·0 to 30·0 c.c. for direct titration, and 2·0, 2·3 and 0·6 to 0·8 c.c. for "formol" titration, respectively. The latter method, therefore, gives a good indication of the nature of the juice.

J. G.

Use of Lead Acetate in the Determination of the Acidity of Fruit Products. B. G. Hartmann and F. Hillig. (*J. Assoc. Off. Agric. Chem.*, 1930, **13**, 259–265.)—Phenolphthalein is found to be a satisfactory indicator for organic acids, and neither the natural colouring matter of a fruit product nor added colouring matter in moderate quantities interferes with the end-point of the titration. The available acidity of a fruit product is not obtained by either direct or electrometric titration, owing to incomplete ionisation of the fruit acids. When, however, lead acetate is added to a solution of a fruit acid the neutral lead salt of this acid is formed and the equivalent quantity of acetic acid liberated; moreover, a substantial portion of the colouring matter is removed with the precipitated lead salts. The procedure proposed for determining the acidity of fruit products is hence as follows:

The reagents required are prepared by (1) diluting 15 c.c. of concentrated nitric acid to 1 litre with boiled distilled water; (2) dissolving 100 grms. of normal lead acetate in 300 c.c. of boiled distilled water containing 10 c,c. of glacial acetic acid, boiling for 10 minutes, cooling, making up to 1 litre with boiled distilled water, and filtering. If the material to be analysed contains insoluble solids, a sample solution is prepared as directed in Methods of Analysis (*Assoc. Off. Agric. Chem.*, 1925, 209); fermented liquids are freed from alcohol, and carbonated soft drinks are boiled to expel carbon dioxide. In a 250 c.c. measuring flask, 200 c.c. of the sample solution or 25 c.c. of the fruit juice (diluted to about 200 c.c.) are shaken with 20 c.c. of the nitric acid solution and then with 20 c.c. of the lead acetate solution, the liquid being then made up to 250 c.c. and filtered. The filtrate is treated with dry potassium oxalate (not a large excess) to precipitate lead and refiltered; 100 c.c. of the filtrate is titrated with 0·1 *N* sodium hydroxide in presence of phenolphthalein, the reading obtained being corrected for that given by a blank test with the reagents alone.

This method gives theoretical results with organic acids and their salts, and with phosphoric acid and phosphates of different types, the third hydrogen atom of the phosphoric acid being titrated. No difficulty is experienced in securing a sharp titration end-point when the above method is applied to jams, jellies and fruit juices.

T. H. P.

Chemical and Physical Properties of Bright Lager Beers. E. Remy. (*Z. Unters. Lebensm.*, 1930, **59**, 402-406.)—The full chemical and physical properties of eight beers of different origins are tabulated, and are shown to fall within narrow limits for this type of beer. The mean values obtained include alcohol 3·19 by weight, extract 4·88, mineral matter 0·227, and total nitrogen 0·069 per cent. (of which 14·2 per cent. was formol-titratable), ratio of amino nitrogen to total nitrogen 1:7·04, *p*H value 4·42 to 4·60, refractometer reading 39·6 (beer) and 19·4 (alcoholic distillate) at 17·5° C. The formulae, extract$=3{\cdot}3\ [s_1+(s_1-s_2)]$ and alcohol$=2{\cdot}7\ s_2$, where s_1 and s_2, respectively, are the relative surface-tensions determined in Traube's stalagmometer of the beer and alcoholic distillate (prepared by distillation of 75 c.c. from 100 c.c. of beer and dilution to 100 c.c. at 15° C.), gave results in good agreement with those obtained by other methods. No tyrosine or tryptophane was detectable, and only a weak colour was obtained with Bezssonow's reagent for vitamin-*C* (see, however, Glassmann and Posdeew, ANALYST, 1929, **54**, 432). The *p*H value is best determined colorimetrically on the 10-fold diluted beer, with bromcresol green (*p*H 4·1 to 5·3) as indicator. J. G.

Neutral Fat of Beef Liver and Other Tissues. W. R. Bloor and R. H. Snider. (*J. Biol. Chem.*, 1930, **87**, 399–413.)—Previous work by various investigators on the fat of beef liver and other tissues is discussed. The fatty acid contents (below those required for true fat) found for the "neutral" fat fractions of beef tissues and beef liver have shown that something other than fat was contained in the fractions. In the case of the liver an accurate knowledge of the nature of the fat is especially desirable because of the important place assigned to that organ in present conceptions of fat metabolism. A more critical examination of the fat fraction has, therefore, been made, and attempts to isolate and examine the real neutral fat of beef livers and certain other beef tissues are described. The neutral fat of beef liver was found to be considerably more unsaturated than the deposited fat or the fat of the other organs examined. This finding is in agreement with that of Kennaway and Leathes (*Proc. Roy. Soc. Med.*, 1909, **2**, **136**), and supports the conception of Leathes (Leathes and Meyer-Wedell, *J. Physiol*, 1909, **38**, 38), of the liver as a desaturating organ for the fatty acids. However, the possibility cannot be excluded that the presence of more highly unsaturated fats in this organ may be due to selection of these substances from the blood, possibly as part of the protective function of the liver. The percentage of fat in most of the livers examined was variable and small, both absolutely and in relation to the phospholipid content; for this reason the phospholipid to fat ratio was high (average about 86: 14). Occasional high fat values in normal samples of all tissues emphasise the normal variability of the fat of the liver and other organs. Whereas the fat content of the organs was variable, the phospholipid content was quite constant for each organ, and characteristic of the organ. The iodine number of the liver phospholipid was found to be lower than that of the other organs examined.

P. H. P.

Occurrence of High-Molecular Fatty Acids in Linseed and Soya-Bean Oils. J. Grossfeld. (*Z. Unters. Lebensm.*, 1930, **59**, 412–418.)—Seven linseed oils and one soya-bean oil were examined by the author's method (ANALYST, 1930, 138), the fatty acids being fractionally precipitated with lead acetate from alcoholic solution. The results were obtained in terms of arachidic and erucic acids from the formulae, 25·9 (42·98–k) and 14·5 (42·98–k), respectively, where k is the potassium perchlorate value (*id.*, 1930, **55**, 138). It was concluded from the results that in each case the potassium salt, which forms a gel in mixed alcohol and ether, represents a fatty acid of molecular weight higher than that of stearic acid, and that stearic acid, when present, occurs only in very small quantities. A mean value of 9 per cent. was found for the palmitic acid content from the difference between the percentages of total solid fatty acids and erucic acid. J. G.

Influence of the *p*H Value on a Colour Reaction of Adrenaline. H. Berry and B. Gouzon. (*Compt. rend.*, 1930, **21**, 1239–1241.)—If adrenaline is dissolved in water by means of hydrochloric acid and a steel pin introduced into the solution, a series of colour changes occurs according to the *p*H of the solution, and the time for the colour to attain its maximum varies with the acidity of the solution. With *p*H 3 or less (strongly acid) 1 hour, pale green; *p*H 3 to 7 (feebly acid) about 1 minute, blue violet; *p*H 7 or over (neutral or alkaline) about 25 minutes, red mauve. The reaction is still distinct for 1 in 20,000 solutions. If nickel, chromium, zinc, copper, tin, or platinum is used, the reaction is negative. D. G. H.

Lead Tetrachloride as a Reagent for Alkaloids. Microchemical Characterisation of Cocaine and Strychnine. V. Arreguine and F. Amadeo. (*Ann. Chim. analyt.*, 1930, **12**, 165–168.)—The lead tetrachloride reagent is prepared by decomposing lead sub-acetate solution with hydrochloric acid, filtering, washing, and adding the solid thus formed in excess to concentrated hydrochloric acid. When cold, a few grms. of potassium chlorate are cautiously added to the mixture, when the lead dissolves to form a yellow solution of the colour of picric acid. Although the reagent thus prepared only decomposes slowly, it is best to use it freshly prepared. An immediate crystalline precipitate results when it is used with dilutions of 1 in 400 of cocaine and 1 in 1600 of strychnine nitrate; precipitation in 10 minutes with 1 in 1600 of cocaine; and turbidity with 1 in 3200 of strychnine, and the reaction is negative with greater dilutions. Working with a microscope, quantities down to 1 in 10,000 of either alkaloid may be detected, since the crystals are characteristic. Precipitates are also formed after varying times with certain other alkaloids, but they cannot be detected in such great dilution as above. D. G. H.

Determination of Caffeine in Decaffeinated Coffee. W. F. Allen. (*J. Assoc. Off. Agric. Chem.*, 1930, **13**, 265–272.)—When the Fendler and Stuber method (ANALYST, 1916, **41**, 88) and the Power and Chesnut method for determining caffeine (ANALYST, 1919, **44**, 342) are applied to decaffeinated coffee, a considerable error is introduced if the final residue is assumed to be caffeine alone.

The caffeine in this residue may be determined either by subliming it on to the upper part of a two-piece sublimation tube, the lower part of which is heated at 180–190° C., or by determining the nitrogen present by the Kjeldahl method. The most accurate results are obtained by a combination of these two methods. Either a modification of Pregl's micro-Kjeldahl apparatus or a macro-Kjeldahl apparatus, using 0·01 *N* solutions for the titration, may be employed, but the apparatus must be previously steamed out thoroughly until the blanks vary by not more than 0·3 c.c. of the acid. T. H. P.

Biochemical.

Fastness of Dyes to Perspiration [Composition of Perspiration]. C. C. N. Vass and B. A. McSwiney. (Fastness Test Committee.) (*J. Soc. Dyers and Colourists*, 1930, **46**, 190.)—The committee reports on an investigation on the composition of human perspiration, with particular reference to reaction, ammoniacal and amino-acid nitrogen, urea, glucose, chlorides and phosphorus. The samples of perspiration were all obtained under conditions as nearly sterile as possible from rheumatic, stout, and normal males and females during treatment at the Royal Baths, Harrogate. The extremes and averages of the analytical figures were as follows:—

	*p*H value.	Urea N. Mgrms. per 100 c.c.	Glucose. Mgrms. per 100 c.c.	Chlorides as NaCl. Grms. per litre.
14 normal males—				
Extremes ..	5·1–7·35	11·1–32·9	9·4–26·4	2·65–5·01
Average ..	6·14	21·4	12·6	3·70
10 normal females—				
Extremes ..	6·35–6·80	13·6–27·7	12–35	2·23–3·87
Average ..	6·57	19·23	20·0	3·00

It was found that figures varied considerably in any one individual. The perspiration from stout subjects was generally higher in glucose. That from rheumatic subjects showed no marked divergence from the normal. The changes which occur in the organic constituents of sweat on standing are shown to be due to bacteria. R. F. I.

Simple Test for Laevulose in Glucides. S. Tashiro and E. B. Tietz. (*J. Biol. Chem.*, 1930, **87**, 307–310.)—During an attempt to obtain a permanent pink colour for a method for the determination of bile salts in the blood by the Pettenkofer reaction, Tashiro (*J. Biol. Chem.*, 1925, **63**, 64) observed that laevulose and carbohydrates that contain the laevulose molecule behaved quite differently from other carbohydrates, in respect to both the delicacy and the speed of the reaction; it thus appeared that a reverse Pettenkofer test might be applied to a method of detection of laevulose in much the same manner as is done in the Molisch test, with the bile salts instead of α-naphthol. The method devised gives a clear demarcation between the laevulose reaction and that of other carbohydrates in

ordinary conditions, and is useful when only a small amount of the sample is available for a test. In a small test-tube (1 cm. in diameter) is placed 1 c.c. of 0·1 per cent. fresh aqueous solution of the ordinary ox bile salts; 1 c.c. of concentrated sulphuric acid is added, the tube is shaken immediately, and a drop of a solution to be tested is quickly added to the mixture. (The temperature is an important factor. Under these conditions, before the addition of the sample drop, the temperature is about 85° C.) An immediate production (in about 15 seconds) of pink or purple colour on the top of the solution indicates the probable presence of laevulose, free or in combination. The minimum concentrations of the carbohydrates containing laevulose that give a positive test have been found to be 0·03 per cent. for laevulose, 0·03 per cent. for inulin, 0·06 per cent. for saccharose, 0·09 per cent. for melezitose, and 0·1 per cent. for raffinose. Therefore (ignoring the water containing laevulose that give a positive test have been found to be 0·03 per cent. for laevulose, 0·03 per cent. for inulin, 0·06 per cent. for saccharose, 0·09 per cent. for melezitose, and 0·1 per cent. for raffinose. Therefore (ignoring the water eliminated in the condensation of the hexose to form these higher carbohydrates), the test quantitatively detects laevulose if the drop contains roughly 0·00001 grm. or more laevulose in free or combined form, *i.e.* the concentration must be $M/600$ or more. There is no reason to suppose the reaction is specific for laevulose, as all the sugars give the test if the concentration is high enough, *e.g.* glucose at 10 per cent., maltose 8 per cent., glycogen 8 per cent., starch 8 per cent., and dextrin 2·5 per cent. Free glucose is, therefore, 300 or more times less sensitive than laevulose. Galactose gives the test at 4 per cent., lactose at 8 per cent. and mannose at 3 per cent. concentration. Preliminary applications of the test have been made with a few substances. The undiluted plasma of the blood collected before breakfast gives no laevulose reaction, even if taken from a severely hyperglycemic patient. The plasma of a normal person, whose blood before the ingestion of 150 grms. of saccharose was negative, gave a strongly positive reaction 1 hour after, when diluted with an equal amount of distilled water; this indicates the presence of at least 0·06 per cent. laevulose in the blood, either free or partly in the form of saccharose. The test was negative with samples of human and cow's milk. Although normal plasma does not contain enough to give the test, normal urine appears to contain about 0·06 per cent. laevulose, an amount which does not seem possible in the light of recent research. A sample of thymonucleic acid gave the test in a concentration about 10 times that necessary for laevulose. Within the concentrations described, and with the elimination of such compounds as furfural (which gives this reaction), it is possible to single out the presence of laevulose and probably other ketone sugars (?) with this test. P. H. P.

Colorimetric Determination of Guanidine Bases in Blood. J. J. Pfiffner and V. C. Myers. (*J. Biol. Chem.*, 1930, **87**, 345–355.)—A study has been made of the colorimetric reaction of certain guanidine bases with alkaline oxidised nitroprusside and ferricyanide. The method of Weber (*Proc. Soc. Exp. Biol. and Med.*, 1927, **24**, 712) for the determination of guanidine bases in blood

has been modified to exclude interference by creatine. For the new method 50 c.c. of Folin-Wu blood filtrate are pipetted into a 150 c.c. Erlenmeyer flask, 3 to 4 drops of 10 per cent. sodium hydroxide solution are added, followed by 0·5 grm. of blood charcoal (Merck's purified by acid). The contents of the flask are mixed and filtered through a moist filter (Whatman No. 40), and the flask is thoroughly drained. The filter is allowed to drain for 5 minutes, and then returned to the original flask. Twenty-five c.c. of 95 per cent. ethyl alcohol containing 0·5 c.c. of *N* hydrochloric acid are added to the contents of the flask. The flask is tightly stoppered, shaken, and allowed to stand overnight, and the alcoholic solution is then filtered. In an evaporating dish 20 c.c. of the filtrate are dried on a water-bath between 80–90° C. The residue is taken up in 10 c.c. of 0·2 *N* hydrochloric acid, the deep Pyrex dish covered with tin-foil, and autoclaved for 30 minutes at 120° C., when the contents of the dish are again brought to dryness on a water-bath at 80–90° C. The last traces of acid are dissipated by the addition of 1 or 2 c.c. of absolute alcohol and subsequent evaporation. The residue is taken up in 4 c.c. of distilled water and 1 c.c. of alkaline ferricyanide reagent is added. Methylguanidine standards are made up simultaneously. The solutions are centrifuged and colour comparison made within the second 5 minutes after addition of the reagent. A series of standards containing 0·01, 0·02, 0·03, 0·04, 0·05, 0·06, 0·07, and 0·2 mgrm. of methylguanidine base is made up in matched test-tubes (12×100 mm., marked at the 4 c.c. level) from a solution containing 1 mgrm. of methylguanidine base per 10 c.c., and diluted with distilled water to the 4 c.c. mark. To each is added 1 c.c. of the alkaline ferricyanide reagent. If the unknown indicates a concentration of less than 0·07 mgrm. of methylguanidine, the amount is determined by inspection, but if greater it is matched against a 0·2 mgrm. standard in a micro colorimeter, and the amount of methylguanidine present is determined by reference to a proportionality curve. The amount found multiplied by 25 gives the amount of methylguanidine in 100 c.c. of blood. The percentage recovery with the modified technique is about the same (average 75·5 per cent.) as that with Weber's method corrected for creatine. The chloride content of the residue is doubtless one factor that inhibits the colour development. The modified method has been applied to a few normal and hypertensive blood samples and the results obtained are given. P. H. P.

Factors Determining the Ergosterol Content of Yeast. I. Species. C. E. Bills, O. N. Massengale and P. S. Prickett. (*J. Biol. Chem.*, 1930, **87**, 259–264.)—Ergosterol is known to be widely distributed in traces in both plant and animal kingdoms, but the fungi, and particularly the yeasts, remain its practical source. Few studies have been reported on the factors which determine the ergosterol content of yeast. In 1927 it was observed that the bakers' yeast in use as a possible source of commercial ergosterol varied greatly in sterol content. Care is exercised in the manufacture of yeast to keep it uniform; thus it seemed that the ergosterol content must be extremely sensitive to the conditions of culture. This supposition was supported by a later observation that another yeast, fresh

from a brewery, contained scarcely a trace of ergosterol, yet, grown in an aerated wort, it became an excellent source. The authors' scheme was to grow in a given medium a variety of yeasts, and to select from these the most promising species for studies upon the influence of cultural conditions. They aimed not only at developing commercial ergosterol production, but also at studying the significance of ergosterol in the metabolism of fungi. It was found that the method of drying the yeast influences the yield of ergosterol. Of the 29 yeasts investigated, 14 were cultivated only once, whilst the 15 which showed interestingly high ergosterol content were run twice or more. In these repeated runs the average variation in the yield of dry yeast per litre of medium was 13 per cent., and in ergosterol content of dry yeast 19 per cent. A table shows that different species of yeast, similarly cultivated, may differ enormously in ergosterol content. *Saccharomyces logos* contained but a trace, whilst *Saccharomyces carlsbergensis* yielded 2 per cent. Different strains of one species, *Saccharomyces cerevisiae*, ranged from 0·2 to 1·4 per cent. All yeasts of high ergosterol content grew well in culture, but not all which grew well showed high ergosterol content; in fact, the two most prolific growers, *Mycoderma sp.* and a strain of *Saccharomyces cerevisiae*, contained a low percentage of ergosterol. In duplicate runs of the same cultures it was observed in 37 out of 53 cases that the runs which gave the heavier yield of yeast also showed the higher percentage of ergosterol. The yield of yeast is markedly influenced by the intensity of the aeration during cultivation. Microscopic examination, to which the cultures were subjected in case of bacterial contamination before being harvested, indicated that much of the precipitate produced by autoclaving was re-dissolved during cultivation, but a little of this sediment remained in the centrifuged yeast. Spectrographic analysis, which was carried out, is probably the most accurate method for the determination of small quantities of ergosterol. Heretofore the variations in the ergosterol content of yeast have been attributed largely to differences in the conditions of culture. The experiments of the authors show that the different yeasts exhibit decidedly different capacities for the elaboration of ergosterol, capacities which may be attained or repressed by manipulation of the cultural conditions. Some yields of ergosterol obtained commercially ran parallel with the values calculated for the flask cultures. P. H. P.

Bacteriological.

Fungi causing Mildew in Cotton Goods. L. D. Galloway. (*J. Text. Inst.*, 1930, **21**, 277T.)—The majority of mildews in cotton goods belong to the two genera *Aspergillus* and *Penicillium*, but *Fusarium*, *Mucor* and other *fungi imperfecti* are common. The principal source of infection is the raw cotton itself. Mildew spores and mycelium are easily distinguished from cotton fibres by their being readily stained with the dyestuff cotton blue. The effects of mildew on cotton goods are: (1) A musty smell; (2) staining; (3) production of acid or alkali, and (4) tendering. Staining is the most important. Staining may be due to masses of coloured spores or pigment formation by hyphae or perithecia. Acid is produced

from starch or sugar by *Aspergillus niger* but not by a cladosporium. Alkali is formed by the growth of a cladosporium on unbleached cotton free from starch. Grey cloth exposed to high humidities rapidly becomes alkaline, but this is due to bacteria and not to mildew. Tendering is due to the cellulose being attacked by the enzymes produced by certain fungi. Damage to the cuticle is readily shown up by the Congo red method, even before loss of tensile strength can be detected.

So far as classification is concerned, the use of the system of Thom and Church obviates any difficulty in the case of the types *Aspergillus*, but the classification of the *Penicillia* is on a less firm basis, though the monograph by Biourge is very full. A sugar-gelatin medium therein described is invaluable for differentiating species by virtue of the remarkable colours formed. R. F. I.

Inhibitory Action of Certain Substances on the Growth of Mould Fungi. R. G. Fargher, L. D. Galloway and M. E. Probert. (*J. Text. Inst.*, 1930, **21** 245T.)—The antiseptic used most widely in preventing mould growth on cloth is zinc chloride. The amount required (0·8 per cent. on the weight of the cloth) is not always practicable. An extended series of tests has been carried out on the effect of 161 compounds on 25 types of mould fungi. The substances tried included inorganic compounds (those of aluminium, copper, mercury, thallium, zinc), organo-metallic compounds containing mercury, arsenic, selenium, and organic compounds with derivatives such as phenols, *p*-nitrophenol, acetanilide, naphthylamine, quinoline, toluidine, aldehydes, aromatic amides, etc. Of the compounds examined, that which appeared to combine the greatest number of desirable properties for general use in the cotton industry was the sodium salt of salicylanilide. Salicylanilide alone is only sparingly soluble in water (0·005 grm. per 100 grms. of water), whereas the solubility of the sodium salt is 31·7 grms. at 25° C. The method of preparation is given, and examples of the effect of structure on fungicidal power are quoted. Fungicidal power and bactericidal power are not necessarily concomitant. Sodium salicylanilide, for example, is a poor bactericide. R. F. I.

Toxicological and Forensic.

Detection of Mercury Poisoning after Burial. A. Sartori. (*Chem. Ztg.*, 1930, **54**, 461–462.)—In a case of double suicide in which mercury poisoning was suspected, one of the bodies was exhumed after 5 weeks, and the separated organs (which were alkaline to litmus) heated on the water-bath with pure hydrochloric acid, small amounts of potassium chlorate being added at intervals. The resulting pulp was filtered, washed, free chlorine removed by a stream of carbon dioxide bubbled through the filtrate, the excess of acid in which was then neutralised with sodium hydroxide solution, and the mercury separated by means of hydrogen sulphide. J. G.

Detection of Luminal in Urine. W. Koenig and H. Kluge. (*Chem. Ztg.*, 1930, **54**, 451.)—The sample is acidified with tartaric acid within 24 hours of taking, extracted with ether, and the extract washed with 30 c.c. of water and

evaporated to a syrup. The residue is dissolved in hot water, charcoal added, filtered, and the cool filtrate made alkaline with sodium hydroxide and extracted with ether (A). The aqueous solution is acidified with sulphuric acid, solid sodium bicarbonate added till the reaction is just alkaline to litmus, and again extracted with ether (B). Finally, the acidified aqueous residue is extracted with ether (C). On evaporation, A gives an oily, unidentified residue which may contain some luminal (phenyl-ethyl-barbituric acid). B gives a residue, from which, after extraction with hot water, treatment with charcoal, filtration, and evaporation to 1 c.c. in a micro-beaker, crystals of luminal are obtained. Needle-shaped crystals of hippuric acid separate from C after the same treatment. The crystals are identified by their m.pt. alone, and when mixed with pure luminal, and by the reactions with sodium carbonate, silver nitrate, Millon's reagent and ammonium phosphate (Gadamer, *Lehrbuch der Toxikologie*, 1924, 457). Only 2 per cent. of a dose of 0·8 grm. was recovered in the urine collected over a period of 3 days, and traces were still detectable after 10 days. J. G.

Toxicity of *Bikukulla formosa* (Western Bleeding Heart). O. F. Black, W. W. Eggleston and J. W. Kelly. (*J. Agric. Res.*, 1930, **40**, 917–920.)—*Bikukulla formosa* is found in shady woods from British Columbia to Oregon and other parts, and is the fourth member of the genus to be reported on, one of which, *B. cucullaria*, was found to contain an intensely poisonous alkaloid. An extract with 80 per cent. alcohol weakly acidified with acetic acid was made from dried tops of *B. formosa*, and this was treated with lead acetate, the acidified filtrate partially neutralised, concentrated and made strongly alkaline with sodium carbonate, and the precipitate filtered off. The precipitated bases and residues from the ether extractions were united and treated with hot benzene, and 1 per cent. of the crude basic material was isolated. This was a light brown amorphous gummy mass, responding to the usual alkaloid tests and showing no tendency to crystallise. The lethal dose was 2·5 to 5 mgrms. for a mouse of about 20 grms. weight, and death was apparently due to respiratory paralysis. *Bikukulla formosa* is to be regarded as a poisonous plant, and a potential danger to livestock. D. G. H.

Organic Analysis.

Linolic Acids and their Oxidation by Per-acids. W. C. Smit. (*Rec. Trav. Chim. Pays-Bas*, 1930, **49**, 539–551.)—The literature on this subject is discussed. Van Loon (Thesis, Delft, 1929) gives, in some cases, the name "apparent iodine value" to the Wijs iodine value, and considers that it has little significance if nothing is known of the relative amounts of the Wijs reagent and of the oil examined. If a curve is constructed showing the apparent iodine values obtained by using various amounts of an abnormal oil, such as Chinese wood oil, with a constant amount of Wijs reagent, a single determination with any other sample of similar oil will suffice to show if this sample complies with the fixed standards. It appears likely that each substance having a conjugated system of double linkings has a characteristic curve which may be used in this way. T. H. P.

Quantitative Oxidation of Double Linkings in Oils and Fats by Peracetic Acid. New Method of Determining the Degree of Unsaturation. W. C. Smit. (*Rec. Trav. Chim. Pays-Bas*, 1930, **49**, 691–696.)—Nametkin and Abakumovsky's method of determining unsaturated compounds in mixtures by means of a chloroform solution of perbenzoic acid (*J. prakt. Chem.*, 1927, **115**, 56) has proved unsatisfactory, but good results are obtained when peracetic acid is used. This acid is prepared by careful addition, to 1 part of 30 per cent. hydrogen peroxide solution at 0° C., of 4·5 parts of acetic anhydride containing 1 per cent. sulphuric acid, the temperature being kept below 10° C. This solution contains from 2 to 3 equivalents of active oxygen; it contains no water and practically no diacetylperoxide. (The latter is formed by the action of the acetic anhydride on the peracetic acid when the water has disappeared, and that is the reason for adding the anhydride slowly to the hydrogen peroxide.) Two c.c. of this reagent required, for example, 1 c.c. of 0·1 *N* permanganate (for H_2O_2), 51 c.c. of 0·1 *N* thiosulphate directly after addition of potassium iodide (for $CH_3CO.O.OH$), and 1 c.c. of 0·1 *N* thiosulphate after total hydrolysis [for $(CH_3CO.O)_2$]. The peracetic solution is diluted with pure glacial acetic acid to normal strength. Using this solution, the oxidation of the oils is complete after 16 hours at the ordinary temperature, the reagent being stable under these conditions.

To 5 c.c. of the peracetic solution such quantity of the unsaturated material is added that the excess of the oxidising agent is about 100 per cent. The quantities of hydrogen peroxide, peracetic acid, and diacetyl peroxide together are determined before and after the oxidation by pouring the liquid into an excess of potassium iodide and 4 *N* sulphuric acid, allowing the reaction to proceed for an hour, and titrating with 0·1 *N* thiosulphate; subsequent heating of the solution to 50° C. for a few moments should give no further coloration.

Tested in this way, oleic and linolic acids, ethyl linolate, and olive, safflower, linseed, soya bean, castor and rapeseed oils give unsaturation values which, when calculated as iodine values, yield results agreeing closely with the Wijs iodine values, and, for the first three compounds, with the theoretical values. T. H. P.

Inorganic Analysis.

Separation and Determination of Mercury by means of Cupferron. A. Pinkus and M. Katzenstein. (*Bull. Soc. Chim. Belge*, 1930, **39**, 179–195.)—Solutions of mercurous nitrate are quantitatively precipitated by cupferron. The precipitate is white, heavy, and settles well. The solution (0·2 to 0·3 grm. Hg in 100 c.c., and not more than 0·5 *N* in nitric acid) is stirred while being treated, drop by drop, with a fresh 5 per cent. cupferron solution (2·5 c.c. of reagent for 0·1 grm. Hg). The precipitate is collected at once under slight suction, washed with 150 c.c. of 0·5 per cent. cupferron solution, and dissolved on the filter in hot *N* nitric acid. The resulting solution is received in a sand-blasted platinum dish and electrolysed near the boiling-point with 2 to 2·5 amp. at 4·5 to 5 volt. A disc anode and a small rotating glass stirrer are used. The deposit is washed without

interruption of the current, and dried over calcium chloride in an atmosphere saturated with mercury vapour. Divalent mercury is not precipitated by cupferron, but the reaction cannot be utilised for its separation from univalent mercury. The separation of the mercurous ion from silver, lead, cadmium, aluminium, chromium, manganese, nickel, cobalt, and zinc is satisfactory. W. R. S.

Delicate Tests for Copper. I. M. Kolthoff. (*J. Amer. Chem. Soc.*, 1930, **52**, 2222–2226.)—Feigl's reagent for silver (ANALYST, 1928, **53**, 615) is not specific for that metal, as mercury and copper were found to react with the rhodanine. A test which will detect copper in distilled and tap water is as follows: 10 c.c. of liquid are treated with a few drops of 2 per cent. hydrazine sulphate solution, an excess of 1 to 2 c.c. of 6 *N* ammonia, and 0·2 c.c. of 0·02 per cent. alcoholic rhodamine solution. After standing for 5 minutes, the solution is acidified with 30 per cent. acetic acid. A distinct red-brown colour is given by less than 0·0003 grm. Cu per litre, the blank showing a yellow to brownish-yellow tinge. Copper may be thus detected in presence of other metals except silver and mercury. Iron interferes unless phosphoric acid is substituted for acetic acid, but the coloration is then less stable. Another test for copper consists in adding 1 c.c. of *N* sodium acetate solution containing 7 c.c. of *N* acetic acid per 100 c.c., 0·2 to 0·3 c.c. of alcoholic 0·1 per cent. dimethylglyoxime solution, and 1 c.c. of saturated potassium periodate solution (0·35 per cent.) to 10 c.c. of liquid to be tested. A violet-red colour develops after 5 minutes' standing. The reaction is claimed to be sensitive to 0·0001 grm. per litre; it permits of the detection of copper in distilled and in natural waters containing lime and magnesia. W. R. S.

Indicators for the Reaction between Ceric and Ferrous Ion. N. H. Furman and J. H. Wallace, Jr. (*J. Amer. Chem. Soc.*, 1930, **52**, 2347–2352.)—In the titration of ferrous salt with ceric sulphate (ANALYST, 1928, **53**, 404) or *vice-versa*, methyl red forms an excellent indicator. When ceric salt is titrated with ferrous sulphate, the solution is added rapidly until the colour becomes faint; methyl red is then added, followed by 25 c.c. of sulphuric-phosphoric acid mixture (150 c.c. of each acid made up to one litre); a yellowish colour is produced. The addition of ferrous sulphate is then continued until a violet end-point is obtained. The colour change is much more rapid than that of diphenylamine (*id.*, 1930, **55**, 408). In the reverse titration (ferrous salt by ceric sulphate) it is likewise best to add the indicator after the reaction is nearly completed. One drop of 0·1 *N* ceric solution (0·05 c.c.) is subtracted from the reading: the results then coincide with the potentiometric ones.

The indicators erioglaucine and eriogreen (*id.*, 1929, **54**, 437) give a rose-red colour with a very slight excess of ceric sulphate; in presence of ferric salt the colour produced is orange or pale rose. The colour change is not quite instantaneous, and no correction is required. The three indicators can be used in chloride as well as in sulphate solution, and in the presence of mercurous and mercuric chlorides. W. R. S.

Determination of Total Nitrogen in Calcium Nitride. W. Lepper. (*Z. anal. Chem.*, 1930, **80**, 331–334.)—The decomposition of calcium nitride by the present method can be accomplished in half-an-hour. Five grms. of sample are mixed in a dry 500 c.c. Jena flask with 1 grm. of copper sulphate crystals, 10 to 20 grms. of powdered potassium sulphate, and 50 c.c. of strong sulphuric acid. The vessel is heated for 15 minutes over a small flame until the water is expelled and the strong reaction has taken place, then boiled for another 15 minutes. The mass is dissolved in water, the solution made up to 500 c.c., and 100 c.c. distilled with sodium hydroxide as usual. Addition of sodium sulphide is unnecessary. Should the caustic alkali contain any nitrate, no zinc powder should be added in the distillation. W. R. S.

Detection of Cyanogen Iodide in Iodine. S. Morris, E. B. Callaghan and L. Dunlap. (*J. Amer. Chem. Soc.*, 1930, **52**, 2415–2417.)—The method used by Baxter (*id.*, 1905, **27**, 876) has been developed. The aqueous suspension of iodine was treated with hydrogen sulphide, and the resulting mixture of aqueous hydriodic and hydrocyanic acids fractionally distilled from a flask fitted with an ordinary water-cooled condenser, the volume of liquid in the still being kept approximately constant. Successive 10 to 12 c.c. portions of distillate were tested by the Prussian-blue reaction, and it was found that the hydrocyanic acid concentrates in the head-fractions. After 2 hours' boiling, the distillates gave negative tests. Repeated re-distillation of an initially large head-fraction renders the detection of traces of cyanogen iodide possible; by taking 902 grms. of iodine and re-distilling the head-fraction ten times, the authors detected the presence of 0·00015 per cent. of cyanogen. W. R. S.

Microchemical.

Capillary Analysis. Identification of Small Amounts of Formaldehyde with Dimethyl-hydro-resorcinol. L. Kofler and H. Hilbck. (*Mikrochemie*, 1930, **8**, 117–120.)—The test utilises the capillary properties of filter paper; long strips (20 cm. long and 1 cm. wide) are used, of which about 1 cm. at the end is shaped to a tip, 3–4 mm. wide. Behind the shaped portion one drop of "dimedon" is added from a platinum loop. The formaldehyde solution, of which 0·1–0·5 cm. is sufficient, is placed in a micro pointed test tube, and the strips of filter paper dipped, point downwards, in the liquid. Formaldimedon is formed where the two liquids meet. On then adding distilled water to the tubes it ascends the filter paper, drives up the last portion of formaldehyde, and also washes the compound formed. The paper is then cut, so as to leave only the portion containing the formaldimedon, and dried. It is heated to 130° C. in Klein's micro melting-point apparatus (Klein, *Mikrochemie Pregl-Festschrift*, 1929, 192), when it sublimes, forming characteristic needles and prisms, melting at 189° C. With 0·1–0·5 c.c. the limit of dilution of the formaldehyde solution for the test is 1: 20,000. J. W. B.

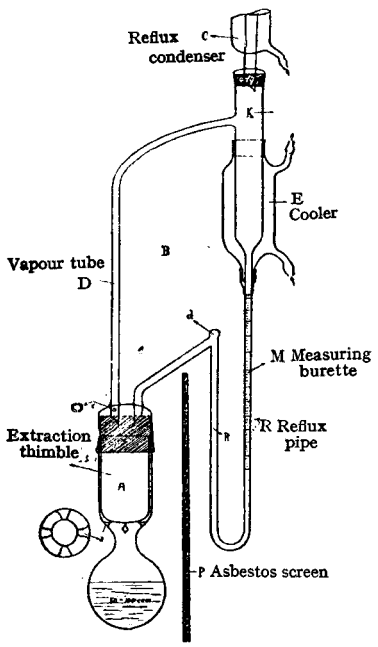
Micro-Determination of Halogens and Metals in Organic Compounds. H. H. Willard and J. J. Thomson. (*J. Amer. Chem. Soc.*, 1930, **52**, 1893–1897.)—The method is adapted from the macro method (*J. Amer. Chem. Soc.*, 1930, **52**, 1195). The organic matter is oxidised with fuming sulphuric acid and the halide oxidised by permanganate or hydrogen peroxide to free halogen, which is distilled into alkaline arsenite and precipitated as silver halide. Samples of 15–25 mgrms. are used, and are weighed on a balance accurate to 0·01 mgrm. (Bunge air-damped balance, type 4 D.M.). The apparatus, which is made of Pyrex glass, consists of a 25 c.c. flask provided with two small condensers, and connected with two absorption bulbs, joined in series (the first of 50 c.c., the second of 25 c.c. capacity) by means of ground-glass joints, 7 mm. inside diameter. A small dropping funnel is fused to the top of the higher condenser (apparatus obtainable from Arthur H. Thomas Co., Philadelphia). For the determination of chlorine or bromine the compound is weighed into a glass micro beaker, 6 × 7 mm., and both are transferred to the decomposition flask, and 0·5 grm. of potassium persulphate, a few glass beads and a little copper sulphate added. The apparatus is connected up and water passed through the condensers. The absorption flask contains 0·1 grm. of arsenic trioxide (to reduce halogen to halide), 1 grm. of chlorine-free sodium hydroxide and 15 c.c. of water. Through the dropping funnel 4 c.c. of fuming sulphuric acid containing 20 per cent. of sulphur trioxide are added, and the mixture boiled gently for 15 minutes, when 5 c.c. of concentrated sulphuric acid are added. When the acid becomes colourless an excess of saturated permanganate solution is added (about 2 c.c.) and 5 c.c. of water. The condensers are then emptied and the solution boiled until sulphur trioxide fumes appear. The apparatus is now disconnected, and the contents of the absorption bulbs transferred to a 100 c.c. beaker and neutralised with dilute nitric acid (sp. gr. 1·10), methyl orange being used as indicator. An excess of silver nitrate is added, then 2 c.c. of concentrated nitric acid (sp. gr. 1·42). The solution is boiled and filtered through a filter crucible with porous porcelain bottom, washed with 1 per cent. nitric acid, then with acetone, dried at 135° C. and weighed. Liquids are weighed in thin-walled bulbs. Chloroform or carbon tetrachloride may escape before complete reaction with sulphur trioxide, but if a quartz tube, 20 cm. long, is connected between the condenser and decomposition flask, and heated in the centre, the organic halide gas is decomposed. For iodine compounds no persulphate is added, and 10 per cent. hydrogen peroxide is used instead of permanganate. Metals may be determined in the solution from which the halogen has been removed. In this case copper sulphate need not be added. Test analyses with various organic compounds gave percentage results for chlorine, bromine and iodine agreeing within the second place of decimals with the theoretical amounts. J. W. B.

The Cobalt Thiocyanate Reaction for the Detection of Cobalt and Thiocyanate. I. M. Kolthoff. (*Mikrochemie*, 1930, **8**, 176–181.)—The test may be used for cobalt in the presence of up to 400 times its amount of nickel. On mixing one drop of the cobalt solution on a depression on a porcelain spot plate

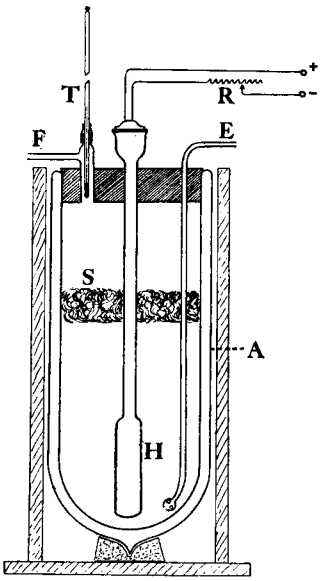
with 5 drops of a saturated solution of potassium thiocyanate in acetone, a green colour develops, visible with 0·5μ grm. of cobalt. Ferric iron is rendered inert by adding excess of fluoride, but cupric copper must be removed by hydrogen sulphide, or by separation as cuprous thiocyanate. The thiocyanate is preferable to the cyanate as a reagent. To test for thiocyanate, a drop of the test solution is evaporated with a small drop of cobalt sulphate, and the residue is moistened with acetone. The formation of a distinctly green solution indicates the presence of thiocyanate. Nitrite interferes. J. W. B.

Apparatus, etc.

Determination of Water and Crude Fat in Substances rich in Fat by means of Trichloroethylene. A. Heiduschka and G. Neumann. (*Chem. Ztg.*, 1930, **54**, 271–272.)—Determination of water and fat in materials like linseed by drying, followed by extraction with ether, occupies about ten hours, whilst Prjanischnikow and Telnow's method of simultaneous extraction with benzine and isobutyl alcohol and distillation of the water (*Z. anal. Chem.*, 1929, **76**, 161) is attended by various disadvantages. Satisfactory results are readily obtained in this way if trichlorethylene is used as solvent, as the condensed water separates easily without emulsifying, and is only very slightly soluble in the solvent. In the apparatus recommended, the extraction thimble A rests on four supports in the neck of the extraction flask, which is closed by a ground-on cap and connected thereby with the condensers and the measuring burette M. The vapour tube D has two small lateral holes at its lower end, and the condensed water and trichloroethylene are cooled further by the cooler E, through which water at a temperature not exceeding 10° C. must be passed. In this way the liquid is cooled to 20–25° C. before it enters the graduated burette, the diameter of which need not be more than 4·6 mm. in order that the trichloroethylene may pass through the collected water. The reflux pipe R becomes wider at *d*, and the condenser C is capped by a calcium chloride tube to prevent access of atmospheric moisture to the apparatus. The flask is charged with 50 c.c. of the solvent, and the extraction is complete in 4 hours. The residue in the extraction thimble is dried at 95–100° C. and weighed. The weight of water corresponding with any given volume in the burette is determined beforehand by a series of experiments in which 0·3, 0·4, etc., grm. of water is added to the flask with the 50 c.c. of solvent, and its volume read



in the burette after distillation. With their apparatus, the authors obtained, for the weight of water corresponding with one scale division of the burette, values ranging from 0·7895 to 0·8000 grm., the mean being 0·7951 grm.; the maximum error in the percentage of water involved by using this mean value would be 0·08, if the amount of substance extracted were 5 grms. The method gives results in close agreement with those obtained by the Soxhlet method, using dried material and ether, and by Prjanischnikow and Telnow's method. T. H. P.



Constant Temperature Preheater. S. T. Bowden. (*J. Soc. Chem. Ind.*, 1930, **49**, 257–258T.)—The preheater is intended primarily for use with the refractometer and polarimeter, and consists of a cylindrical Dewar flask (A), 6×20cm., in a wooden box fitted with a rubber stopper through which a 300 watt electrical heater (H) of the immersion type passed, and which is connected with the mains through a variable manganin resistance (R). Water enters the vessel by the inlet tube E, which has a small bulb with several orifices so that the water is swirled round the heater. The outgoing water leaves at F, where a thermometer (T) is placed. A thick wad of thin copper gauze (S) is fixed to the heater about two-thirds up the vessel, and this ensures the equilibrium of the temperature of the water before it leaves the vessel and obviates the use of any stirring arrangements. A sliding resistance (R) enables the final adjustment of temperature to be made, and by suitable alteration in the flow of water and the heating current the usual ranges to temperature may be obtained. With a water flow of 600–700 c.c. per minute the issuing water may be maintained at 25° C. with less than 0·05° variation. D. G. H.

Reviews.

MICROSCOPIC PHARMACOGNOSY. By W. MANSFIELD, A.M., Dean, Albany College of Pharmacy, Albany, N.Y. 8vo. Pp. x + 211. New York: J. Wiley & Sons; London: Chapman & Hall. 1929. Price 15s. net.

In his preface the author states that "this book is written to provide an authoritative text in Microscopic Pharmacognosy for students of pharmacy and as a reference book for pharmacists and drug analysts in government, manufacturing and private laboratories." The book consists of a series of 88 full-page line drawings representing characteristic structures visible in the powders of a corresponding number of drugs and drug adulterants; each illustration is faced

by a page of descriptive matter stating briefly the nature of the structures represented in the drawing. The subject of Pharmacognosy, as studied from a microscopical standpoint, is, therefore, only very partially covered by the matter in the book; in fact, the author has produced what is really an atlas illustrating 88 powdered drugs and adulterants, so that it can hardly claim to be more than a very partial attempt to deal with a subject covering so wide a field as "Microscopic Pharmacognosy." The particular powders chosen for illustration appear to have been selected upon a principle that is rather difficult to understand. The resulting choice has included a number of rather rare drugs, such as mullein, and coltsfoot leaves, thyme and boneset herbs, red clover flowers, sumac berries, tonga rhizome, horehound, etc., while many commoner and more important drugs, such as belladonna, henbane, tea and bearberry leaves, santonica flower heads, cloves, indian hemp, coriander, almonds, linseed, cascara, rhubarb, jalap, etc., are omitted; no starches are described or figured. From a certain point of view it may be considered useful to include the more unusual drugs and to exclude many of the commoner ones, which can be found adequately dealt with elsewhere, but one hardly expects to find such omissions from "an authoritative text in Microscopic Pharmacognosy." The drawings have all been made to scale, so that they are comparable with one another and give a correct idea of the relative sizes of the cells and other structures represented; it is, however, an unfortunate oversight that the magnification used is nowhere stated.

When one comes to look more closely into details, it is evident that, while much useful information is given about each of the 88 drugs by means of drawings and description, in several instances characteristic features have been overlooked and the drawings themselves lack that refinement of finish which distinguishes careful drawings from rapid sketches. The drawings are of about the same quality as might be produced by a good student in the course of ordinary class work, and the magnification is also about that at which a student would make his sketches. The notes upon the structure of the hairs of Nux Vomica and of Ignatius Beans seem to indicate some misunderstanding of their construction; in the drawing of the epidermis of Cubebs, the characteristic prismatic crystals are omitted. No microchemical tests are given, either for the tissues or their contents, and there is no indication of those characters which are most diagnostic of each drug.

T. E. WALLIS.

ANNUAL REPORTS ON THE PROGRESS OF APPLIED CHEMISTRY. Vol. XIV for 1929. Issued by the Society of Chemical Industry. Pp. 754. Price to members, 7s. 6d.; to non-members, 12s. 6d.

The months pass quickly by, and another of these invaluable Annual Reports is to hand long before the preceding volume has lost its interest or its importance.

The present Report has, except for the omission of an article on "Explosives," similar subject-matter to the previous year's volume (see ANALYST, 1929, **54**, 772),

and the reviewer has again to comment on the valuable summaries of recent progress in the various branches of applied chemistry given so generously in this publication; though, indeed, this is only what one expects on perusing the list of authors of the twenty-four sections of the Report. The information packed into these pages is not only well compiled, but is in many instances fortified by apt and critical comments drawn from the writer's experience.

Under "General, Plant and Machinery," it is stated that the year 1929 has been marked by the conclusion of several important international agreements in the chemical industries, particularly in the nitrogen industry. A draft convention prepared by the League of Nations, and now before the various Governments for consideration, maintains that authors of scientific discoveries should have a share in the profits derived from them when such discoveries are capable of practical utilisation. Workers have been engaged on the general question of satisfactorily eliminating dust and sulphur compounds from large volumes of flue gases, and it is probable that the solution entails in all cases efficient cleaning of the coal, treatment of the flue gases to remove grit, and scrubbing to catch acid constituents. In connection with boiler waters, the conditioning of the water and the mechanism of scale formation are subjects dealt with in this section. The results of investigations on the heat-insulating properties of various materials used in cold storage construction are given, and the employment of tantalum in corrosion-resisting apparatus is advocated.

In the section on "Fuel" there is shown throughout the influence of a more enlightened policy regarding atmospheric pollution, an aspect of the fuel problem which now constantly asserts itself wherever combustion is involved. Thus the new power stations, the use of pulverised fuel, coal cleaning, steam-raising, low-temperature carbonisation, and aspects of domestic heating are all discussed more or less in relation to this important issue—the problem of lessened pollution of our town air. There are valuable sub-sections on the origin and chemistry of coal, on its spontaneous combustion, and its evaluation and analysis,the last-named including methods for the determination of moisture-content—for example, by extraction with methyl alcohol and determination of the dilution of the solvent.

"Mineral Oils," "Rubber," "Leather and Glue," "Soils and Fertilisers," with many others, form subjects of sections which are most interesting and informative, but space forbids more than this bare mention. "Colouring-Matters and Dyes" are dealt with by describing the ten years' progress since the termination of the War. This section shows the vast strides made in the colour industry, the success of research into new series of colouring-matters, and the progress made in the production of dyes with enhanced value, such as exceptional fastness to light. Recent researches have simplified the work of the dyer.

In the section on "Fibres and Textiles, etc.," cellulose acetate "silks" are included under the description "Rayon," in spite of the fact that certain manufacturers of the acetate "silk" do not acknowledge this generic name as applied to their product. The statement quoted here, on the findings of the U.S. Bureau

of Standards, that white cotton and linen will transmit ultra-violet light as well as white viscose "silk" or white cellulose acetate "silk," is not only contrary to the advertisement writer, as the author of the article states, but is apparently contradictory of previous work on this question.

The analysis and testing of the physical properties of various fibres—natural and manufactured—find special mention in several portions of the volume, and the analyst will find many valuable hints, throughout the Report, on most of the subjects with which the various writers deal in their reviews. An interesting paragraph on the wetting agents used in processes connected with the treatment of fibres appears in the book, and the same section discusses a protective colloid stated to prevent the separation of insoluble soap salts with hard water, and a new anti-moth agent for woollen goods, fast to washing and light treatments.

An important part of the review of "Iron and Steel" is that dealing with corrosion, in which the results of the latest work are recorded. This subject is also dealt with in the sections on "Non-Ferrous Metals" and the "Electro-Chemical Industries."

The book contains a comprehensive and highly interesting account of the technology of "Oils, Fats and Waxes," with much important matter in connection with their examination and analysis. The article on "Foods" and other sections again show how vitamin-chemistry has become all-important in assessing the value of manufactured ("processed") foods. The determination of the freezing-point of fresh milk appears to provide a helpful method for the determination of extraneous water; while the refractive index of milk-serum has been shown by recent work to be of no assistance in doubtful cases of adulteration, though the contrary view is still held by some workers. A method is described by which maltol, which, like salicylic acid, gives a violet coloration with ferric chloride, may be distinguished from the latter substance.

The writer of "Sanitation and Water Purification" ably reviews the methods of analysis of sewage effluents, the mechanism of the chlorination of water, the disposal of sewage sludge and many related subjects. Sections on "Fine Chemicals" and on "Photographic Materials and Processes" conclude the volume, which is provided with subject and author indexes.

The value of this comprehensive survey to all chemists cannot be exaggerated. These reports have secured a sure place in the libraries of all who desire to keep abreast of the literature, and who know how hopeless a task this is, in the midst of a busy life, without such help as these Reports give. The errors noted are few and unimportant. It might be useful to readers if the Editor would, in future issues of these Reports, provide a page of explanatory notes on the abbreviated name references to the journals cited, and if it were made clear to "foreign" readers what the precise significance of the letter "B" is in the footnote references to the British Chemical Abstracts.

ARNOLD R. TANKARD.