

# THE ANALYST

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

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### The Examination of Mixtures of Coconut Oil and Palm Kernel Oil.

### The Determination of Butter Fat in Margarine.

BY G. D. ELSDON, B.Sc., F.I.C. AND PERCY SMITH.

*(Read at the Meeting of the North of England Section, November 13, 1926.)*

SOMEWHAT recently the present authors in a paper dealing with the Reichert-Polenske figures of mixtures of palm kernel oil and other fats (*ANALYST* 1926, 51, 72) pointed out that the relationship between the Reichert and Polenske values of a mixture of fats might give useful assistance in distinguishing between coconut and palm kernel oils, and even within certain limits give some indication as to the relative amount of the two fats present. This suggestion has now been followed up, and the Reichert and Polenske values have been determined for a complete range of mixtures of these two oils. The results obtained are given in Table I below.

It has not been thought necessary to determine the Kirschner value in each case, for the previous papers (*ANALYST*, 1925, 50, 53; 1926, 51, 72) showed, as was indeed previously known, that there is a very definite mathematical relationship between this figure and the amount of coconut and/or palm kernel oil present. For the same reasons mixtures containing butter fat have not been studied, as in this case also the Kirschner value is a linear function of the amount of butter fat present, whilst the effect of the presence of butter fat on the Reichert and Polenske values may be allowed for by finding the approximate percentage of butter from the Kirschner value (in conjunction with the Polenske value) and



In order that the process of finding the approximate percentage of butter fat present in a mixture may be made as easy as possible the following table (Table II) has been compiled.

TABLE II.  
DETERMINATION OF BUTTER FAT IN MARGARINE.

	Butter Fat Per Cent.										
	0	1	2	3	4	5	6	7	8	9	10
Polenske	0.4	0.5	0.5	0.6	0.6	0.7	0.8	0.8	0.9	0.9	1.0
Kirschner	0.2	0.5	0.7	1.0	1.2	1.5	1.7	2.0	2.2	2.5	2.7
Polenske	1.0	1.1	1.1	1.2	1.2	1.3	1.4	1.4	1.5	1.5	1.6
Kirschner	0.3	0.6	0.8	1.1	1.3	1.6	1.8	2.1	2.3	2.6	2.8
Polenske	2.0	2.1	2.1	2.2	2.2	2.3	2.4	2.4	2.5	2.5	2.6
Kirschner	0.5	0.8	1.0	1.3	1.5	1.8	2.0	2.3	2.5	2.8	3.0
Polenske	3.0	3.1	3.1	3.2	3.2	3.3	3.4	3.4	3.5	3.5	3.6
Kirschner	0.7	1.0	1.2	1.5	1.7	2.0	2.2	2.5	2.7	3.0	3.2
Polenske	4.0	4.1	4.1	4.2	4.2	4.3	4.4	4.4	4.5	4.5	4.6
Kirschner	0.8	1.1	1.3	1.6	1.8	2.1	2.3	2.6	2.8	3.1	3.3
Polenske	5.0	5.1	5.1	5.2	5.2	5.3	5.4	5.4	5.5	5.5	5.6
Kirschner	0.9	1.2	1.4	1.7	1.9	2.2	2.4	2.7	2.9	3.2	3.4
Polenske	6.0	6.1	6.1	6.2	6.2	6.3	6.4	6.4	6.5	6.5	6.6
Kirschner	1.0	1.3	1.5	1.8	2.0	2.3	2.5	2.8	3.0	3.3	3.5
Polenske	8.0	8.1	8.1	8.2	8.2	8.3	8.4	8.4	8.5	8.5	8.6
Kirschner	1.1	1.4	1.6	1.9	2.1	2.4	2.6	2.9	3.1	3.4	3.6
Polenske	10.0	10.1	10.1	10.2	10.2	10.3	10.4	10.4	10.5	10.5	10.6
Kirschner	1.2	1.5	1.7	2.0	2.2	2.5	2.7	3.0	3.2	3.5	3.7
Polenske	12.0	12.1	12.1	12.2	12.2	12.3	12.4	12.4	12.5	12.5	12.6
Kirschner	1.3	1.6	1.8	2.1	2.3	2.6	2.8	3.1	3.3	3.6	3.8
Polenske	14.0	14.1	14.1	14.2	14.2	14.3	14.4	14.4	14.5	14.5	14.6
Kirschner	1.4	1.7	1.9	2.2	2.4	2.7	2.9	3.2	3.4	3.7	3.9
Polenske	15.5	15.6	15.6	15.7	15.7	15.8	15.9	15.9	16.0	16.0	16.1
Kirschner	1.5	1.8	2.0	2.3	2.5	2.8	3.0	3.3	3.5	3.8	4.0

This table, which is more or less ideal, is a composite one which has been developed from graphs from the tables given in the two papers to which reference has already been made. Its mode of use will be fairly obvious. When the Polenske and Kirschner values of a fat are known, the horizontal lines in the table are examined until the two figures are found; the figure at the top of the vertical column will indicate the amount of butter fat in the mixture. For example, in the case of a given mixture of fats the Polenske and Kirschner values were 5.6 and 2.5 respectively. The two nearest figures to these are 5.4 and 2.4, from which the interpolated value for the percentage of butter fat is 6.3; the actual proportion added was 6.0 per cent.

An examination of the above Table I will show that the Reichert value due to palm kernel oil alone (mixed, of course, with neutral fat in varying proportions) is in all cases numerically less than the Polenske value, whilst in the case of coconut oil alone the Reichert value is the greater until about 40 per cent. of coconut oil is present. The table has been subjected to critical examination by Mr. H. D. Richmond, to whom the authors are greatly indebted. Mr. Richmond writes as follows: "I think the most that can be expected from your results are, first, it is possible to distinguish coconut oil and palm kernel oil; second, that when the two exist in the same mixture and the percentage of coconut oil is well under 50 per cent., an approximate idea of the relative proportions of the two oils can be deduced within about 15 or 20 per cent. either way."

It should be emphasised that the accuracy of the process depends upon the factors of the oils—chiefly of the butter-fat—which have been used in the mixtures, and also that different mixtures of coconut and palm kernel oil may give identical results, as will be seen from an inspection of the table.

In such cases, therefore, the composition of the coconut oil class part of the oil should be checked by one of the recognised methods, that of Shrewsbury and Knapp (*ANALYST*, 1910, 35, 385), which gives the total amount of such oil, being particularly suitable. Other processes are those of Burnett and Revis (*Ibid.*, 1913, 38, 255) and Stokoe (*J. Soc. Chem. Ind.*, 1921, 40, 57T). It is highly desirable that when the composition of a mixture of oils has been deduced a new mixture should be made having this composition, and the values obtained therefrom compared with those of the original. In this way a very near approximation to the truth may be obtained.

The detection of small amounts of butter in margarines, which is sometimes required, may be carried out by this method, supplemented by the method of Gilmour (*ANALYST*, 1920, 45, 2; 1925, 50, 272). Experiments along these lines have been carried out, and it is hoped to publish the results obtained.

It may not be out of place at this stage to remind workers that practically the whole of our knowledge of the examination of margarine is due to the pioneering work of Cribb and Richards and Bolton, and of Revis and Richmond. The present authors are glad to acknowledge their indebtedness to these workers, and to state that, at best, they can only claim to have confirmed and possibly somewhat extended the earlier work.

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## A Rapid Method for the Sorting of Butters and Margarines.

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(Read at the Meeting, December 1, 1926.)

THE disadvantage of the Reichert process, if comparable results are to be obtained, lies in the close attention that must be paid to the details of the distillation process.

The present paper deals with a rapid method of sorting butters and margarines, and depends, like the Reichert process, upon the butyric acid content. No distillation is involved, and the figures obtained with butters closely approximate to those obtainable from Kirschner determinations. In the case of margarines the figures (with one exception) whilst being somewhat higher than the corresponding Kirschner values, are still, as with butters, lower than the corresponding Reichert values. For twenty butters examined the figures ranged from 20.6 to 26.4, whilst for eighteen margarines the limits were 0.0 and 5.1. (See Tables I and II.)

TABLE I.

*Butters.*

No. of sample.	Reichert (R).	M value.	KR.	KM.	Polenske.	Boric acid. Per Cent.
1	28.0	26.4	—	—	1.2	—
2	27.8	23.4	—	—	2.2	—
3	29.4	22.8	—	—	2.4	—
4	27.0	23.4	—	—	1.8	—
5	28.9	26.3	—	—	1.7	—
6	29.1	25.6	—	—	2.2	—
7	28.5	21.6	—	—	1.9	—
8	27.0	21.6	—	—	—	—
9	28.2	23.0	—	—	1.7	—
10	27.6	22.2	—	—	1.7	0.03
11	—	21.9	—	21.9	—	—
12	—	22.1	—	22.1	—	—
13	28.6	26.2	25.2	26.6	1.5	—
14	—	{ 22.3 } 22.1	—	22.6	—	—
15	—	20.6	—	—	—	—
16	29.0	23.1*	—	—	—	—
17	—	20.6	—	—	—	—
18	—	22.2	—	{ 21.9 } 22.0	—	0.28
19	—	23.2	—	{ 23.2 } 23.1	—	—
20	27.2	22.5	23.1	22.7	—	—

\* Increased to 24.2 by hot water washing of the insoluble acids.

TABLE II.

*Margarines.*

No. of sample.	Reichert.	M. value.	KR.	KM.	Polenske.	Boric acid. Per Cent.
1	5.5	2.5	1.6	1.9	4.6	—
2	4.2	2.8	2.1	1.6	4.6	0.23
3	5.0	2.6	1.1	1.2	3.9	0.15
4	3.9	1.2	1.0	0.9	4.8	—
5	5.8	2.2	1.7	1.7	5.5	0.20
6	5.3	2.4	1.2	1.6	6.5	0.14
7	4.9	3.4	2.6	1.2	4.9	—
8	4.7	1.6	1.3	1.3	4.1	0.27
9	2.3	{ 1.8 1.8 }	1.7	1.4	0.6	0.33
10	8.5	5.1	3.8	3.8	2.8	0.20
11	—	Nil	—	Nil	—	0.33
12	—	Nil	—	Nil	—	0.20
13	—	2.0	—	1.4	—	0.20
14	4.5	3.0	2.2	1.4	1.3	0.22
15	2.5	2.5	2.5	2.5	1.3	0.02
16	5.4	1.6	1.3	1.4	5.9	—
17	—	1.2	—	—	—	0.25
18	—	2.3	—	—	—	0.29

THE NEW METHOD.—The following are the details of the process:—Five grms. of the filtered fat are saponified with 20 c.c. of glycerol soda solution (made by mixing 900 c.c. of pure glycerol with 100 c.c. of a 50 per cent. aqueous solution of sodium hydroxide) and the soap dissolved in 100 c.c. of boiled distilled water. Into the cooled solution 4 drops of 0.5 per cent. methyl orange solution are introduced and sulphuric acid (25 per cent. by volume) added from a burette until the solution is faintly pink. The total volume of the solution and precipitated fatty acids is taken and 100 c.c. filtered off,\* nearly neutralised with 10 per cent. sodium hydroxide solution, and the neutralisation completed with 0.1 *N* sodium hydroxide solution. In this way the sulphuric acid is neutralised, leaving only the soluble fatty acid, which is then titrated with 0.1 *N* sodium hydroxide, after the addition of 0.5 c.c. of 0.5 per cent. phenolphthalein solution. The number of c.c. of 0.1 *N* sodium hydroxide solution taken, less the number required for a blank, is represented as the M value.

The blank is carried out upon 20 c.c. of glycerol soda solution dissolved in 100 c.c. of distilled water (free from carbon dioxide). In measuring the glycerol

\* By using 18.5 cm. paper, the whole of the solution may be transferred at once, thus saving unnecessary expenditure of time in supervision of the filtration.

soda solution, 20 c.c. are poured into a 25 c.c. cylinder, and after the bulk has been transferred to the flask, four drops are allowed to enter, after which the cylinder is removed. In this way a reasonably uniform quantity is used in each case.

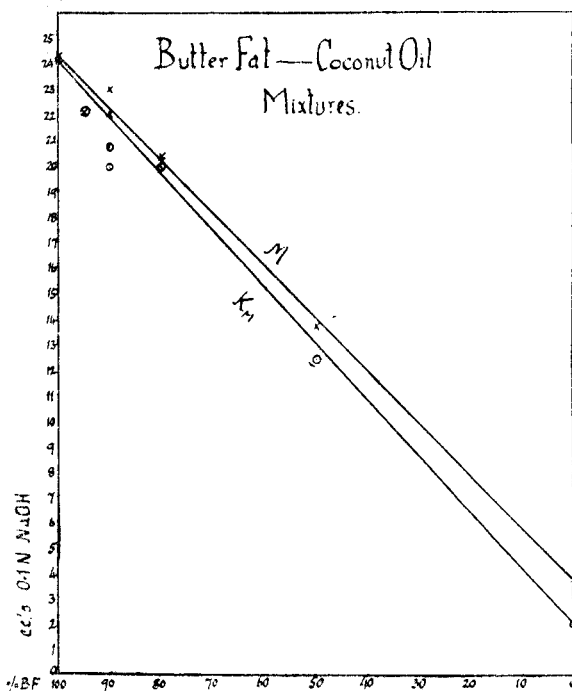
If to the neutral solution, obtained as above from a butter-fat, 0.5 gm. of silver sulphate is added, and a Kirschner determination made, the value obtained in every case is the same, or very nearly the same, as the M value.

SIGNIFICANCE OF THE KM VALUE.—This figure is referred to as the KM value, as distinct from KR, the ordinary Kirschner value. These results seem to indicate that the volatile soluble acids other than butyric are co-precipitated with the insoluble acids, and this theory is to some extent borne out by the fact that when the insoluble acids are washed several times in a separating funnel with hot water, and the washings passed through a filter paper, an increase in the M value results, although even so it does not become equal to the Reichert figure (Butter No. 16). This may possibly be accounted for by the preferential solubility of the soluble acids (other than butyric) in the liquid layer of the higher fatty acids (partition coefficient).

DETECTION OF COCONUT AND PALM KERNEL OILS IN BUTTER FAT.—For margarines, speaking generally, the KM value is lower than the M value. In margarines in which the presence of coconut or palm kernel oil has been indicated by the Reichert–Polenske–Kirschner determinations it would appear that some of the caprylic acid escapes co-precipitation, and this fact, along with the identity of the M and KM values in the case of butter-fats, suggested the possibility of detecting coconut and palm kernel oils, when present in butter-fat, by means of the difference which might exist between the M and KM values in such cases. To this end mixtures of butter-fat and coconut oil were prepared containing 5, 10, 20, and 50 per cent. of the latter, the M and KM values, and also the Reichert–Polenske–Kirschner values, being then determined and the results plotted. (See Table III and Curves.) No difference was observable in the 5 per cent. coconut oil mixture between the M and KM values, and only a slight one (0.4 c.c.) in the 20 per cent. mixture. In the 50 per cent. mixture the difference amounted to 1.3 c.c., no very great amount in view of the proportion of coconut oil present. On the other hand, the 10 per cent. mixture showed a difference of 2.3 c.c.—a figure that was supported by a check determination which yielded a difference of 2.1 c.c. In view, however, of the slight differences obtained with the 20 per cent. and 50 per cent. mixtures, it would be difficult to formulate any definite conclusions. It was thought, however, that a qualitative test might be used, based upon the surmise that silver nitrate (or silver sulphate) added to the solution obtained in the M value determination would produce no turbidity in the case of a genuine butter (owing to the identity of the M and KM values) if sodium hydroxide free from chloride were used throughout, but that a turbidity would ensue if coconut or palm kernel oil were present.

TABLE III.  
*Mixtures of Butter Fat with Coconut Oil.*

	M.	KM.	Difference (M-KM).	Reichert.	KR.	Polenske.
Butter fat, 100 per cent.	24.3	24.1	0.2	28.9	24.8	2.6
Butter fat, 95 per cent.	22.1	22.1	0.0	27.7	23.5	2.7
Coconut oil, 5						
Butter fat, 90	23.0	20.7	2.3	26.7	22.1	3.9
Coconut oil, 10	22.0	19.9	2.1			
Butter fat, 80	20.3	19.9	0.4	25.0	20.1	4.5
Coconut oil, 20						
Butter fat, 50	13.7	12.4	1.3	19.2	13.7	7.9
Coconut oil, 50						
Coconut oil, 100	3.8	2.0	1.8	8.2	2.6	13.4



LIMITATIONS OF THE TEST.—The alcoholic sodium hydroxide used for the saponifications and the aqueous sodium hydroxide solution used for the titrations in these tests were prepared from metallic sodium. The solutions were free from chloride. No turbidity was obtained with silver nitrate or silver sulphate in the case of a genuine butter fat, and only the faintest indication of such with a fat containing 5 per cent. of coconut oil. At a concentration at which an appreciable

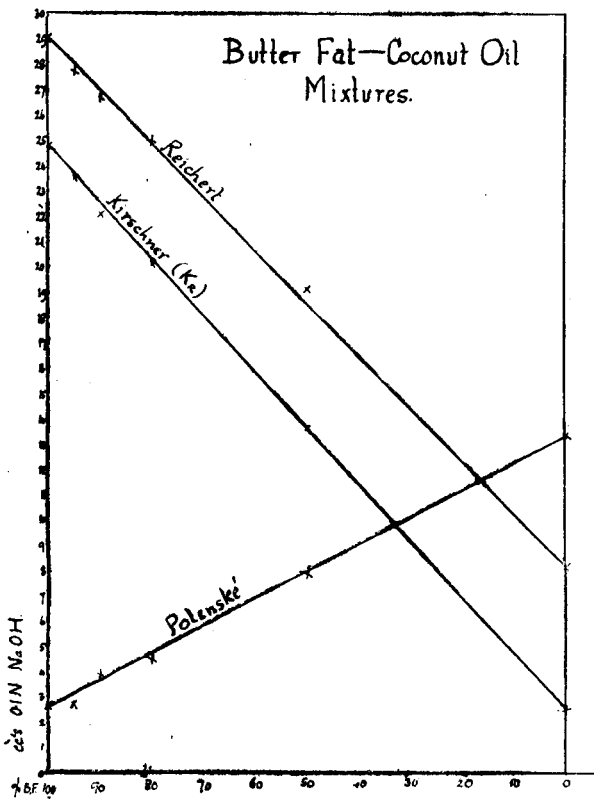


turbidity was obtainable it was found that an approximately equal turbidity was yielded by the solution prepared from a pure butter-fat.

Thus it is not considered that this test can serve to distinguish butter fat in samples containing 5 per cent. coconut oil.

Since these experiments were carried out, some chloride-free glycerol sodium hydroxide solution has been prepared, with results that have supplemented those obtained with the alcoholic sodium hydroxide solution.

Experiment has shown that boric acid has no effect on the M value. A clear filtered margarine fat was tested for boric acid alongside some of the whole margarine; the whole margarine showed the presence of boric acid, whilst the filtered fat gave a negative reaction.



Whilst, as yet, the method here described for examining butters cannot claim the accuracy of the Polenske process, it can be used instead with advantage in the examination of margarines, for the majority of which no butter content is expected, and for these the possibility of a butter fat content in excess of 10 per cent. is, for obvious reasons, extremely small. Of the 20 margarines examined, in only one case did the M value exceed 3.4. In this case (No. 10. M = 5.1) the Polenske-Kirschner figures corresponded with a 10 per cent. butter content. In this case

also, and in several others, the KM and the KR values were identical. Generally, where the M value is 5.0 or over, determinations of both the KM and KR values are recommended.

It has been found that for margarines a period of five minutes is sufficient time for the complete precipitation of the silver salts in Kirschner determinations. This effects a saving in the time of one hour recommended by Kirschner and adopted by Revis and Bolton (*ANALYST*, 1911, 39, 336). Similarly, for butters, thirty minutes were found sufficient. (See Table IV).

TABLE IV.

	Reichert	KR after precipitation lasting—			
		60'	30'	15'	5'
Margarine No. 10	{ 8.5 8.6	3.8	—	4.0	3.9
Butter No. 21	{ 27.8 28.1	22.9	22.7	23.7	—
	M value.	KM after precipitation lasting—			
		60'	30'		
Butter No. 19	23.2	23.2	23.1	—	—

In conclusion, I wish to express my thanks to Mr. Harri Heap for his interest and criticisms.

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## The Determination of Sodium, Potassium and Chlorine in Foodstuffs.

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THE following notes embody the experience obtained in applying certain methods to the analysis of foodstuffs and animal excreta. The modifications described below have been worked out and adopted by us after having tested several methods described in the literature.

**DETERMINATION OF SODIUM AND POTASSIUM.**—One of the methods tested was that described by Kramer (*J. Biol. Chem.*, 1920, 41, 263) for the determination of sodium and potassium in blood, and whilst, in the main, the method outlined below is that of Kramer, we desire to point out two serious discrepancies which occur when applying this method to foodstuffs and to suggest certain modifications to avoid these errors.

Briefly, Kramer's method is as follows: The blood ash is extracted with hydrochloric acid, the sulphates precipitated by barium chloride, and, without previous filtration, ammonia and alcoholic ammonium carbonate are added to precipitate phosphate and all metals save sodium and potassium. The precipitate

is filtered off and washed with the alcoholic ammonium carbonate solution. The filtrate and washings are evaporated to a small bulk, acidified with concentrated hydrochloric acid, and the sodium and potassium weighed as mixed chlorides. The potassium is then determined by the cobaltinitrite method, and the sodium by difference. In testing the method we preferred to weigh the sodium and potassium as mixed sulphates rather than as mixed chlorides.

*Sources of Error.*—(1) In certain analyses serious discrepancies were found in the weights of mixed sulphates from duplicates, and, on examination of the mixed sulphates, they were found to contain both phosphates and magnesium. The presence of the phosphates was undoubtedly due to the fact that, after making the liquid alkaline with ammonia prior to the addition of alcoholic ammonium carbonate, the barium phosphate was not removed by filtration. On the addition of the ammonium carbonate there was a certain amount of interaction between it and the barium phosphate, giving barium carbonate and ammonium phosphate. The latter passed through into the filtrate, with the result that some of the sodium and potassium was weighed as phosphate. This difficulty is overcome if the barium sulphates and phosphates, thrown down by the ammonia, are filtered off and washed, as described below, before the addition of the ammonium carbonate.

(2) The difficulty of complete removal of magnesium is well known. Gooch and Eddy (*Z. anorg. Chem.*, 1908, 58, 427) investigated the method of Schaffgotsch (*Ann. Phys.*, 1858, 104, 482), who described the use of aqueous ammonium carbonate, for the complete precipitation of magnesium. Adhering to his conditions they were unable to effect complete precipitation, but report that, by using an alcoholic solution of ammonium carbonate, they were able to recover the whole of the magnesium as magnesium ammonium carbonate, from a magnesium solution containing 50 per cent. of alcohol. We tested this procedure and proved that it was satisfactory, but found it preferable to evaporate the liquid to dryness before precipitation rather than to add the extra alcohol. It should be noted that Kramer neither evaporates nor adds extra alcohol, and this probably explains the presence of magnesium in the mixed sulphates when using his method.

*Method adopted.*—A suitable quantity\* of the material is ashed at a low temperature, and the ash is twice extracted with hot *N* hydrochloric acid (about 100 c.c. in all being used). The residue is washed with hot water, and the filtrate and washings are made up to 200 c.c. Of this extract, 100 c.c. are heated to boiling, and 5 c.c. of a 10 per cent. solution of barium chloride are added. The mixture is evaporated to about 25 c.c., and then, while hot, made alkaline with strong ammonia and allowed to cool. When cold, the precipitate is filtered off and washed with 2 per cent. ammonia. The filtrate and washings are evaporated just to dryness in a 250 c.c. beaker. To it, when cold, 30 to 35 c.c. of alcoholic ammonium carbonate solution† are added, and the whole is vigorously stirred for

\* A quantity such as will give a final weight of 0.1 to 0.15 gm. of mixed sulphates.

† Gooch and Eddy's solution is prepared by diluting 180 c.c. of strong ammonia (sp. gr., 0.880) to one litre with water and mixing it with one litre of 90 per cent. alcohol. In this mixture 250 grms. of ammonium carbonate are dissolved by warming. When cold the solution is filtered and is ready for use.

about five minutes. The contents of the beaker are transferred to a 100 c.c. flask and the beaker rinsed with more alcoholic ammonium carbonate solution. After thorough shaking, the solution is made up to the mark with the reagent. The mixture is allowed to stand at least two hours, preferably overnight, and then filtered into a dry flask. Fifty c.c. of the filtrate are evaporated to dryness in a weighed vitreous basin, and the cold residue treated with 5 c.c. of redistilled sulphuric acid. The excess of acid is evaporated off on a sandbath, and the residue is heated over a naked flame until quite dry. During this stage it is advisable to scatter on to the residue a little powdered ammonium carbonate, which facilitates the removal of the last traces of sulphuric acid. The dish is then heated in the muffle at a bright red heat for ten minutes, cooled and weighed. From this weight of mixed sulphates and the percentage of potassium the percentage of sodium can be calculated.

*Note.*—If the filtrate obtained after removing the phosphates with barium chloride and ammonia is not evaporated to dryness before adding the alcoholic ammonium carbonate, traces of magnesium are liable to be present in the mixed sulphates.

The following table gives typical results obtained by the modified method. The figures given are from analyses of barley meal with or without the previous addition of sodium chloride.

	Sodium sulphate from sodium chloride added. Grm.	Mixed sulphates	
		Found. Grm.	Calculated. Grm.
Barley meal plus sodium chloride	0.0486	0.1171	0.1176
Barley meal plus sodium chloride	0.0608	0.1296	0.1298
Barley meal alone .. ..	Nil	0.0690	0.0690

**DETERMINATION OF POTASSIUM.**—The determination of potassium can be satisfactorily carried out by the volumetric cobaltinitrite method, as described by Green (*Biochem. J.*, 1912, 6, 69). If strict attention is paid to details very accurate results can be obtained. For the following reasons the determination is preferably made in the mixed sulphates obtained as above rather than directly on the ash extract. Any possible error due to the presence of phosphates, as stated by Kramer, is thus avoided, although personally we have not found any error from this source. The use of an aqueous solution of the mixed sulphates avoids the necessity for adding sodium hydroxide to neutralise the acid of an acid extract. When testing the method we found that samples of sodium hydroxide were liable to contain appreciable traces of potassium, and it is worthy of note that the sodium hydroxide sold to us as chemically pure for analysis contained over five times as much potassium as the commercial stick sodium hydroxide. When working with an ash extract direct it is essential, therefore, that a blank determination should be made with the reagents.

**DETERMINATION OF CHLORINE.**—Weitzel (*Arb. a. d. Reichsgesundh. Amt.*, 1920, 52, 635) has investigated various methods for the determination of chlorine

in foodstuffs, and has shown that wide differences may be obtained according to the methods employed in the preliminary ashing. We desire to draw attention to his results and to emphasise the importance of the procedure at this stage of the determination. The ashing may be effected, according to various workers, by one of the following methods:

- (a) Incineration direct at a dull red heat (Wiley, *Methods of Analysis*, A.O.A.C., 2nd Ed., 1925, p. 225).
- (b) Moistening with 20 c.c. of 5 per cent. sodium carbonate solution and incinerating at a low temperature (Wiley, *ibid.*, p. 43).
- (c) Covering the dried foodstuff with a layer of 10 per cent. of its weight of powdered calcium oxide and ashing at a low temperature (Von Nencki and Schomnow-Simanowski; see Weitzel).
- (d) Mixing the foodstuff with 10–25 per cent. of its weight of calcium oxide, making the mixture into a paste with water and incinerating it at a low temperature.

The following table illustrates the results obtained in the determination of chlorine in some foodstuffs by methods (a), (b) and (d).

*Percentage of Chlorine in Foodstuffs.*

	Percentages found.		
	Method a.	Method b.	Method d.
Palm kernel cake .. ..	Trace	0·1489	0·1666
Scotch barley meal .. ..	0·0037	0·1002	0·1053
Whole barley .. ..	0·0106	0·1105	0·1393
Rye .. ..	Trace	0·0379	0·0433
Fine bran .. ..	Trace	0·0357	0·0424

These results bear out Weitzel's findings. He found losses up to 70 per cent. by direct ashing, as against a loss of 0·5 per cent. by method (d). They suffice to emphasise the importance of proper procedure in the preliminary ashing, as recommended by Weitzel.

The final titration can be satisfactorily carried out on the nitric acid extract of the ash by Volhard's method.

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ABERDEEN.

### THE TERM "CORNFLOUR."

In the ANALYST for August, 1926 (p. 402) a footnote, signed by the Editor on behalf of the Publication Committee, was published, to the effect that "much of the cornflour on the market, however, consists of pure rice starch." With reference to this note we are informed that most, if not all, of the rice cornflour sold in this country is described by the manufacturers themselves as being prepared from rice.—EDITOR.

## 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 FLOCCULATION OF TROUBLESOME PRECIPITATES IN ANALYSIS.

THE colloidal condition of matter may be approached from two extremes: the breaking down of matter in bulk, or the building up from the molecular state. The latter is the method giving rise to finely-divided suspensions or precipitates due to chemical reactions in analytical practice. Close attention to such factors as temperature, time elapsing before filtration, removal of peptising agents, and adequate content of suitable electrolyte, favours the formation of coarse and easily-filtered precipitates.

In this note, however, a less familiar method is mentioned, and it is believed that it is of general application in analytical work, as well as being very simple and efficacious. The method consists in the addition of a small quantity of starch solution to a finely-divided or gelatinous suspension, so as to bring about immediate flocculation.

The author has tried the method with many suspensions of widely different  $P_n$  values and invariably with success. Such diverse systems as calcium carbonate in saturated sodium chloride solution, talc and clay suspensions in water, and certain solids (*Koll. Zeitsch.*, 1921, 28, 233; "Physics and Chemistry of Colloids," London, H.M. Stationery Office, 1921, p. 119) in strongly acid and alkaline media, all respond immediately to the addition of starch, giving coarse flocs which settle readily. The method has been used for many troublesome analytical precipitates, e.g. mercury tungstate.

It is found that the starch is adsorbed by and appears in the precipitate. Since pure starches give only little ash, and since only 1 part of starch per 250 parts of flocculated (dry) solid is the average requirement, it follows that the presence of starch in the settled precipitate has no serious consequences. The author has shown (*loc. cit.*) that a starch solution made up in alkaline solution possesses intense flocculating power, practically independent of the temperature of the suspension treated. Where alkali is not objectionable this type of solution is recommended. The so-called "soluble" starch is not suitable for flocculating. It should be mentioned that, owing to the starch being adsorbed by the flocs, washing of a precipitate is greatly facilitated, progressive settlings being still very rapid, and the starch is not washed out.

The phenomenon of flocculation by this means is a special case of coagulation in colloid systems known as sensitisation (*cf.* Freundlich in Bogue's *Colloidal Behaviour*, New York, 1924, 1, 239, *et seq.*). Normally, organic colloids "protect" suspended matter against flocculation by added electrolytes. However, if the protective colloid is present in but minute amount, the system may be rendered more sensitive to the action of electrolytes, *i.e.* the system is sensitised. In this case the order of mixing is:—

(Suspension + Organic colloid) + Electrolyte,

but with the starch method it is:

(Suspension + Electrolyte) + Organic colloid.

Thus we are not dealing with the usually-accepted scheme of sensitisation. The whole matter is very complex and is receiving detailed treatment in the author's forthcoming book: "The Principles of Applied Colloid Chemistry." It is hoped, however, that the present note will prove of use to analytical chemists.

WILLIAM CLAYTON.

### THE DETERMINATION OF CARBON DIOXIDE IN CARBONATES.

THE method given by Hepburn (ANALYST, 1926, 51, 622) for the determination of carbon dioxide, is very similar, even as regards the apparatus, to the method published by D. Van Slyke (*J. Biol. Chem.*, 1918, 36, 351; abst., *J. Chem. Soc.*, 1919, A. ii, 78).

In that paper it was shown that absorption of carbon dioxide by baryta is practically complete within a few minutes. I have always found it sufficient to allow the absorption to proceed for about 20 minutes, with occasional rotation of the flask. This is obviously preferable to the absorption of 12 to 24 hours suggested in the recent communication.

S. BACK.

While not wishing to detract in any way from the value of the work done by Mr. Hepburn, we feel that some reference should be made to a paper (which Mr. Hepburn appears to have overlooked) by D. D. Van Slyke, who describes a method which, in general principle, is identical with that employed by Hepburn.

Mr. Hepburn has developed this method, and, by substituting standard oxalic acid for hydrochloric acid, has found it possible to titrate the excess of baryta in the presence of the precipitated barium carbonate, the troublesome filtration employed in the Van Slyke method being thus avoided.

Some years ago, while working in the laboratories of Messrs. Thomas Morson & Son, Ltd., the authors, at the instigation of Mr. T. F. Harvey, evolved a modification of Van Slyke's process which was found to be sufficiently accurate for most industrial purposes. In this method the excess of baryta was titrated slowly with standard hydrochloric acid, a quantity of neutral sodium chloride having been added to assist the flocculation of the precipitated barium carbonate.

LESLIE H. TRACE.

CECIL O. HARVEY.

## 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.*

### CITY OF BIRMINGHAM.

#### REPORT OF THE CITY ANALYST FOR THE THIRD QUARTER, 1926.

In the third quarter of the year, 1313 samples were examined. Of the 1133 taken under the Sale of Food and Drugs Acts, 1090 were bought informally (32 adulterated), and 43 under the provisions of the Act (6 adulterated).

MILK.—Twenty-four of the 566 informal samples were adulterated, and 3 of the 38 formal samples. Twenty-three samples taken from 8 different farmers contained less than 11.5 per cent. of total solids.

*Variations of Solids-not-Fat from Day to Day.*—Samples were taken at one of the railway stations from milk consigned by a farmer, who had 17 cows, from August 9 to August 26. Samples of the two meals were taken on 16 days, and the solids-not-fat varied from 8·6 per cent. to 9·3 per cent., the largest variation between successive milkings being 0·4 per cent. The percentages of fat showed more variation, the lowest figure being 3·1, and the highest 4·2.

**EFFECT OF THREE MILKINGS PER DAY.**—It has lately been suggested that if cows are milked three times a day, a notable difference is made in the proportion of solids-not-fat. Experiments to test this were made by the courtesy of a local farmer, who allowed his cows to be milked at irregular intervals under the observation of our inspectors.

Fifteen cows were milked at 4.50 p.m. on the first day, three times on the second day, at 7.10 a.m., 1 p.m. and 7.20 p.m., and at 6.50 a.m. on the third day. The solids-not-fat only varied by 0·3 per cent., but, as was expected, there was a considerable difference in the percentage of fat. The lowest figure, 3·4 per cent., was obtained at a morning milking after an interval of nearly 12 hours, and the highest fat, 5·6 per cent., at a mid-day milking, the interval being nearly 6 hours.

When the cows were fetched up for milking at the unusual time of 1 o'clock, they appeared uneasy, and instead of going straight to their respective stalls, had to be driven there. In spite of being milked at 1 o'clock, they came to the gate at their usual time, about 4.30, and waited there till they were milked at 7.20. In spite of this interference with the cows' regular habits the quality of the milk was hardly affected.

The average of the first two meals was 8.75 per cent. of solids-not-fat, and 4·27 per cent. of fat, and of the next three meals 8·87 per cent. of solids-not-fat and 4·27 per cent. of fat. The first two meals, which were secreted during 24½ hours, yielded 27 gallons, and the second three meals, secreted during 23½ hours, 26 gallons. These results show that milking two or three times a day makes very little difference to the average composition or yield of the milk, and also that the extra labour involved in milking three times a day gives little or no improvement either in quantity or quality.

A similar experiment was made by the Staffordshire County Council on two herds of nine cows, and I am indebted to Mr. E. V. Jones, F.I.C., the County Analyst, for the figures in the following table.

Here, also, the solids-not-fat in the two and three milkings per day were almost identical, and there was very little difference in the percentage of fat. The same table also shows particulars of some experiments made for the Durham County Council at Offerton Hall some years ago. In that case 6 of the 12 cows were milked three times a day for six weeks, and the other six twice a day. For a further six weeks the first 6 cows were milked twice a day and the second six, three times a day.

Here, again, the figures show little difference, and the averages of the amounts of milk obtained in the two and three milkings per day were identical.

#### COWS MILKED TWICE AND THREE TIMES A DAY.

	Solids-not-fat, per cent.				Fat, per cent.			
	Max.	Min.	Average.		Max.	Min.	Average.	
			Two meals.	Three meals.			Two meals.	Three meals.
Birmingham	9·0	8·7	8·75	8·87	5·6	3·4	4·27	4·27
Stafford	9·34	9·16	9·26	9·24	4·75	3·75	4·26	4·13
	9·18	8·97	9·06	9·05	4·36	3·72	4·04	4·07
Durham	—	—	8·80	8·77	—	—	3·36	3·38



In Aberdeen many cows are milked three times a day, and Mr. J. Cumming, the inspector of that city, has published figures relating to three times a day milking. The average solids-not-fat of 18 farms he visited were, for the morning milk, 8.96 per cent., for the noon milk, 8.97 per cent., and for the evening milk 8.97 per cent.

**BENZOIN.**—The B.P. requires that not more than 15 per cent. of this drug shall be insoluble in alcohol. Not one of the six samples complied with this requirement, the amount insoluble varying from 15.5 to 32.2 per cent. In spite of this, I have passed the samples as genuine, as no special request was made for pharmaceutical benzoïn, and as it is sold for industrial purposes as well as medicinal use. The ash of the samples varied from 0.8 to 2.2 per cent., being well within the B.P. limit of 5 per cent.

**BENZOATED LARD.**—Benzoated lard is prepared by heating 3 parts of Sumatra benzoïn with 100 parts of prepared lard. The B.P., though giving tests for the constituents, gives none for the finished product.

The iodine value of 10 of the 12 samples examined varied from 55 to 66, the B.P. limits for lard being 52 to 63. The acid values of the 10 samples varied from 1.5 to 3.7, the B.P. limit for lard being 1.2. Two of the samples had high acid values (13.7 and 12.9, respectively), and rather low iodine values (49 and 51). These samples were rancid; the vendors were cautioned and the articles withdrawn from sale.

Samples of benzoated lard prepared in the laboratory with two different samples of Sumatra benzoïn gave iodine values of 66 and 67 and an acid value of 2.2 and 2.4. A sample made with Siam benzoïn gave values of 68 and 4.9, respectively.

**ATMOSPHERIC POLLUTION DURING THE COAL STRIKE, 1926.**—The amounts of insoluble matter in May and August for the two Bournville gauges were about half the average of the previous two years. On the other hand, there was little difference in the two periods for June, July and September for the Board Room Roof Gauge and in the "K" Block Gauge for June.

The figures of the four gauges, as a whole, indicate that some months have shown an improvement, but that others have been worse than the average, and others, again, have shown only small differences. The coal strike, therefore, has not produced the diminution in smoke pollution, as indicated by the gauges, which might have been expected, and which was found during the coal strike of 1921. I may add that other stations, spread over the country, have shown similar variable results to those of Birmingham.

In July, the Central gauge showed an increase of 19.3 tons of insoluble matter, as compared with the average of the two previous years, while September showed a decrease of 9.1 tons. The other months for this gauge, as well as June and August for the Aston gauge, were similar to the averages of the same months in the previous two years.

J. F. LIVERSEGE.

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## Legal Note.

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

### A STANDARD FOR MEAT AND MALT WINE.

BOWKER *v.* WOODROFFE; BOWKER *v.* PREMIER DRUG CO., LTD.

ON December 16, 1926, an appeal was heard in the Divisional Court (before the Lord Chief Justice, Mr. Justice Salter, and Mr. Justice Talbot) from a decision of the Salford stipendiary magistrate not to convict the defendant (*cf.* ANALYST, 1926, 51, 514).

Mr. T. Eastham, K.C., for the appellant, an inspector under the Sale of Food and Drugs Acts, said that the stipendiary dismissed the information on the ground that there was no recognised standard of quality for extract of meat and malt wine, and that he had acted upon a Scottish decision of 1877. He, counsel, agreed that there was no fixed standard, but that did not say that there was no minimum. He contended that the analyst had set up a reasonable standard.

Mr. Wingate Saul, K.C., for the respondent, asked that the case should be sent back to the stipendiary to be more fully stated.

The Lord Chief Justice, giving the decision of the Court, said that the case must go back for further consideration and re-statement. The stipendiary seemed clearly to have fallen into error when he said that there was no evidence of a standard of quality for extract of meat and malt wine. It was common ground that there was no standard fixed by legislation, but it was no answer to say that therefore there was no standard. The magistrate, after hearing the evidence, ought to decide whether the article purchased was of the nature, substance and quality demanded, and, in order to answer that question, he must look at the evidence to see what the standard was. It was not suggested that there should be any further hearing by the stipendiary.

## Department of Scientific and Industrial Research.

FOOD INVESTIGATION. Special Report No. 28.

BITTER PIT IN APPLES. A REVIEW OF THE PROBLEM.\*

A REVIEW of the present position of knowledge with regard to bitter pit in apples shows that it is not yet clear whether the disease always arises on the tree, or may be developed in the fruit *de novo* in storage, and it is difficult to say whether orchard practices such as light (or heavy) pruning and early irrigation may exercise any real influence, or merely affect the susceptibility of the fruit to the disease. In some varieties of apple bitter pit develops on the tree, and in others only after picking, and in the later case it appears to a greater extent if the fruit is gathered while immature. Cold storage in some cases delays the appearance of bitter pit, but not in others, whilst delay in cold storage lessens its retarding effect. For some varieties of apple storage on the tree appears the best kind of storage for retarding bitter pit development. A critical discussion of the theories of bitter pit, including that of local poisoning, local desiccation, cell rupture by root pressure, and asphyxiation shows that in no case has direct verification been brought forward,

\* Obtainable at Adastral House, Kingsway, W.C. 2. Price 1s. net.

and most of the theories are open to grave objections on theoretical grounds. From the commercial aspect, bitter pit is a particularly insidious source of loss to the industry; it would seem that a combination of late picking with more rapid refrigeration may improve matters, and further experimental work along these lines is needed.

D. G. H.

### THE CLEANING AND RESTORATION OF MUSEUM OBJECTS.\*

IN the two former Reports (ANALYST, 1922, 47, 120; 1923, 48, 173) Dr. Alexander Scott gave an outline of the methods which had been, more or less tentatively, adopted for the cleaning and restoration of museum exhibits of every kind. During the seven years that have passed since this work was first started much experience has been gained as to the relative advantages and disadvantages of the different methods of treatment, and the present Report summarises the knowledge now available, and describes various modifications which have been found to give better results than the processes originally devised. As in the previous Reports, the work is discussed under different headings, and the effects of the methods described are illustrated by a series of excellent plates, showing the various types of objects before and after treatment.

**PRINTS AND PICTURES.**—The safest method of cleaning discoloured and “foxed” prints is the cautious use of bleaching powder and dilute hydrochloric acid. The use of hydrogen peroxide vapour for clearing up engravings, as recommended before (*loc. cit.*), has the drawback that there may still be sufficient moisture present to cause wrinkles in the paper. To prevent this, an ethereal solution of hydrogen peroxide (prepared by shaking commercial hydrogen peroxide with ether) may be used, the solution being applied to the stains by means of a camel’s hair brush. Alcoholic hydrogen peroxide has also been found effective for the removal of mould stains. For the removal of old and coloured varnish no reagent has been found so effective as pyridine.

The use of thymol as a sterilising agent was suggested in the First Report (*loc. cit.*), and experience has proved its value. After fumigation with thymol in a closed box heated to about 45° C., both moulds and their spores on paper are destroyed, and the remains of the mycelia and mould spots may then be removable by means of a small camel’s hair brush.

Writing on other materials, such as wax or metals, can sometimes be made legible by suitable mechanical treatment or by washing with dilute acids. A solution of cellulose acetate in acetone has been found an effective agent for replacing perished size on paper.

**STONE AND EARTHENWARE.**—Repeated analyses of the crystals efflorescing from stone specimens from all parts of the world have shown that they consist chiefly of sodium chloride and sulphate, together with potassium nitrate. Calcium and magnesium chlorides, nitrates and sulphates are also nearly always present, but in small quantities only. It is necessary to remove these salts in some cases by soaking, in others by the application of wet paper pulp, after which the object is dried and coated with celluloid varnish.

**SILVER.**—There is little to add to the methods described in the two earlier Reports (*loc. cit.*). The use of formic acid in various strengths, both warm and cold, has continued to give excellent results in the cleaning of all kinds of silver alloys.

\* THIRD REPORT UPON INVESTIGATIONS CONDUCTED AT THE BRITISH MUSEUM. Pp. 70. With 57 Plates. H.M. Stationery Office. 1926. Price 5s. net.

**IRON OBJECTS.**—When a comparatively thin coating of oxide and a large proportion of coherent metal are present good results, so far as appearance goes, are obtained by boiling the article with stannous chloride solution kept faintly acid with hydrochloric acid. The stannous chloride prevents, to a large extent, the attack of the metallic iron; the free acid attacks the ferric oxide, producing ferric chloride which is reduced at once to ferrous chloride; this does not attack the metal as ferric chloride does. A coating of metallic tin may be formed on the metal, but this, when *thoroughly* cleaned, should protect the underlying iron.

The best method of treating very corroded iron objects is to boil them with dilute (5 per cent.) sodium hydroxide solution and metallic zinc. The caustic soda decomposes the iron chlorides and oxychlorides, and the zinc promotes electrolytically the passage of the chlorine into the liquid and towards the zinc. If only oxides and other compounds of iron remain, the problem is how to make the specimens less friable and more easily handled. For this purpose "Pyruma putty" or kaolin made into a paste with silicate of sodium or potassium have proved satisfactory. Pitted armour has been preserved by cleaning away all rust with a soft iron wire brush (not brass), and then coating the surface with "Duroprene" with the addition of lampblack to take away the rusty-brown tint.

**LEAD.**—Five years' experience has shown that little amendment is required in the methods already given for the preservation of objects of lead. Careful application of dilute acids by means of a brush is usually all that is required. As a rule, it does not matter much whether acetic, nitric or sulphuric acid is used, but when acetic acid is used the final washing must be very thorough.

If it is decided to retain a slight coating of carbonate, the object is dried at 100° C. in an oven, and then placed under the receiver of an air-pump, so that it can be saturated *in vacuo* with a solution of gum dammar in benzene. Subsequently it is drained on blotting paper and allowed to dry.

**COPPER AND BRONZE.**—The so-called "bronze disease" (bright green spots) is caused by absorption of soluble salts from the soil. These are best removed by immersing the object in a strong solution of sodium sesquicarbonate (hot or cold). The method is particularly valuable for articles which have been coated with another metal.

Treatment with Rochelle salt (as described in the former Reports) has been found of great value.

**WOOD.**—Treatment with the sodium silicate solution sold as "P.84" has given promising results. When it is desired to prevent shrinkage glycerin and gelatin, subsequently hardened in formalin solution, may be found serviceable.

**GLASS.**—A method of applying the hydrofluoric test for glass, which leaves no mark behind it, is described. Loss of brilliance in old glass has been found to be due to the presence of excess of alkali which has attracted moisture and carbon dioxide from the air, forming an alkaline liquid which has attacked the transparent silicates in the glass. The best remedy is to treat the glass with dilute (about 1 per cent.) sulphuric acid, and finally to dry it and coat it with thin dammar varnish.

**TEXTILES.**—Chinese paintings on silk have been strengthened by the application of a 2.5 per cent. solution of cellulose acetate in acetone. When the material will stand it, the strengthening solution is best applied by means of a brush. "Duroprene" (diluted with benzene, toluene or xylene) has also been found suitable for strengthening very frail fabrics.

**NOTES.**—Some hints are given on the most suitable appliances and reagents and the best means of applying them. Among the cements, etc., recommended, are several proprietary articles which have been proved to give satisfactory results.

# Bibliography: Standard Methods of Analysis.\*

## LEATHER AND TANNING MATERIALS.

### OFFICIAL METHODS OF THE INTERNATIONAL SOCIETY OF LEATHER TRADES CHEMISTS.

#### VEGETABLE-TANNED SOLE LEATHER.

*J. Inter. Soc. Leather Trades Chem.*, 1926, 10, 411. (J. R. Blockey.)

#### CHROME-TANNED LEATHER.

*Ibid.*, 1925, 9, 508. (R. F. Innes.) *Ibid.*, 1923, 7, 413. (R. F. Innes.)

#### VEGETABLE TANNING MATERIALS.

*Ibid.*, 1926, 10, 30. (G. Hugonin.)

### OTHER METHODS.

#### CHROME-TANNED LEATHERS.

Alumina, *J. Inter. Soc. Leather Trades Chem.*, 1924, 8, 581. (D. Woodroffe.)

Basicity, *ibid.*, 583. (D. Woodroffe.)

Combined acid sulphate, *J. Amer. L. Chem. Assoc.*, 1925, 20, 448.

#### CHROME-TANNING LIQUORS.

*J. Inter. Soc. Leather Trades Chem.*, 1924, 8, 504.

#### LACTIC ACID.

*Ibid.*, 1924, 8, 504.

*Ibid.*, 1926, 10, 257. (U. Thuau.)

#### CUTCH.

*Analyst*, 1925, 50, 162.

#### FAT IN LEATHER.

*J. Soc. Leather Trades Chem.*, 1926, 10, 455. (Colin Russ.)

#### SODIUM SULPHIDE.

*Ibid.*, 1924, 8, 443.

#### ENZYME BATES.

*Ibid.*, 1924, 8, 477.

#### USED LIME LIQUORS.

*Ibid.*, 1924, 8, 247.

#### LIME.

*Ind. Eng. Chem.*, 1926, 18, 389. (J. C. Bailar.)

#### SWEDISH LEGISLATION AGAINST LOADING OF LEATHER AND METHODS OF TESTING. (E. Norlin.)

*J. Amer. Leather Chem. Assoc.*, 1926, 21, 644; 1924, 20, 298.

Abstracted from *Le Cuir Tech.*, 1926, 15, 267, etc.; 1924, 13, 492.

#### CUBE GAMBIE AND INDIAN CUTCH.

*Analyst*, 1924, 49, 379. (R. P. Biggs.)

#### DEGRAS.

*Collegium*, 1924, 329. (E. Last.)

#### RED ARSENIC.

*Leather Chemists' Pocket Book*, p. 36. (Procter.)

The American Leather Chemists' Association publish a special booklet of their methods, reprinted from the reports of several sub-committees published from time to time in the *J. Amer. L. Chem. Association*.

R. F. I.

\* For convenience of reference in analytical work the Publication Committee have decided to publish bibliographies of standard methods of analysis.—EDITOR.

## Ministry of Health.

### Circular 762.

#### SALE OF FOOD AND DRUGS ACTS, Etc.

THE following letter has been sent by the Ministry to Clerks of Authorities administering the Food and Drugs Acts:—

SIR,

1. I am directed by the Minister of Health to request that a copy of the Report of the Public Analyst for the fourth quarter of this year, and a copy of the Report of the Medical Officer of Health on the administration of the Public Health (Milk and Cream) Regulations during the year, may be sent to the Ministry during the ensuing month.

2. It has been represented to the Minister that a few public analysts in reporting on a sample of milk which is found to be deficient both in milk-fat and in other milk-solids, word their certificate in such a way as to imply that the double offence of the abstraction of fat and the addition of water has been committed, whatever the relative degrees of the two deficiencies. After consultation with the Society of Public Analysts, the Minister recommends that in such a case the certificate, while following the general form set out in the Schedule to the Sale of Food and Drugs Act, 1875, should be so worded as to show how much, if any, of the deficiency of milk-fat is presumed to be due to abstraction, allowance being made for the effect of the added water.

3. A new edition of Memorandum 36/Foods has been prepared, embodying in a revised form the matter contained in the previous issue of the memorandum, together with that contained in a number of official circulars. The object of the present edition is to consolidate and bring up to date the principal recommendations of a permanent character which have previously been made as to the procedure of Local Authorities and their officers in the administration of the Sale of Food and Drugs Acts and other legal provisions of a similar character. A copy of the revised memorandum is enclosed herewith.\* The memorandum and this Circular will be placed on sale, and further copies may be obtained directly from H.M. Stationery Office or through any bookseller.

I am, Sir, your obedient servant,

December 20, 1926.

R. B. CROSS (Assistant Secretary).

\* See Memo 36/Foods, *infra*.

#### PROCEDURE UNDER THE SALE OF FOOD AND DRUGS ACTS, Etc. (COMPOSITION AND DESCRIPTION OF FOOD AND DRUGS.)

##### Memo. 36/Foods. (January, 1927).\*

###### I.—LEGAL PROVISIONS.

1. The principal legal provisions relating to the composition and description of food and drugs (apart from provisions as to poisons and other special drugs) in force on the 1st January, 1927, are:—

- (a) The Sale of Food and Drugs Acts, 1875 to 1907, viz.:—Sale of Food and Drugs Act, 1875; Sale of Food and Drugs Act Amendment Act, 1879; Margarine Act, 1887; Sale of Food and Drugs Act, 1899; Butter and Margarine Act, 1907.
- (b) Legislation amending and supplementing the Sale of Food and Drugs Acts, viz.:—Section 10 of the Licensing Act, 1921 (dilution of spirits); Milk and Dairies (Consolidation) Act, 1915; Section 4 of the Milk and Dairies (Amendment) Act, 1922; Section 23 of the Finance Act, 1921 (sending samples to Government Chemist).
- (c) Orders and Regulations made under the Sale of Food and Drugs Acts, viz.:—Order as to Registration of Margarine Factories, etc., 1900; Order as to Registration of Butter

\* H.M. Stationery Office, 1926, price 1d. net.

Factories, etc., 1907; Regulation as to Competency of Analysts, 1900; Sale of Milk Regulations, 1901 and 1912; Sale of Butter Regulations, 1902.

(d) Regulations made under the Public Health (Regulations as to Food) Act, 1907, viz.:—Public Health (Milk and Cream) Regulations, 1912 and 1917†; Public Health (Condensed Milk) Regulations, 1923; Public Health (Dried Milk) Regulations, 1923; Public Health (Preservatives, &c., in Food) Regulations, 1925 and 1926.

2. Most of the legal provisions cited above are enforced and executed by Sale of Food and Drugs Acts Authorities, and it is convenient that they should be treated for the purpose of administration and report as constituting a single group of laws for securing the purity of food.

## II.—ACTION AND REPORTS OF LOCAL AUTHORITIES.

3. The Local Authority is required by statute to send copies of the Public Analyst's Quarterly Reports to the Ministry, and in order to facilitate the work of the Department the reports should be sent every quarter as soon as they are received.

4. The Ministry also desire to receive information respecting the action taken by Local Authorities in regard to each sample not reported as genuine by the Public Analyst, showing what legal proceedings have been instituted and the result of such proceedings. Particulars should be given of any prosecutions of an exceptional character, especially in connection with the adulteration of milk or with the application of any new legislation or Regulations. Where prosecution has not been considered advisable the Ministry should be furnished with a brief statement of the circumstances which have determined the decision, and the precise action which has been taken. The information should be given either on a copy of the Analyst's report or otherwise in such a form as to ensure the identification of the relative sample.

5. Where proceedings in respect of offences committed in any quarter are pending at the time of sending in the report for that quarter, the necessary information as to the result of the proceedings should be transmitted to the Ministry as early as practicable.

6. The Ministry desire to be furnished with particulars of offences other than adulteration, *e.g.* contravention of the labelling requirements of the Regulations mentioned in paragraph 1 (d) of this Memorandum or of the provisions of the Margarine Act, 1887, or the Butter and Margarine Act, 1907, obstructing officers in the discharge of their duty, or refusing to sell. As regards such offences information, as to the action taken should be given on the same lines as that asked for above with regard to adulterated samples.

7. The Ministry would also be glad to be informed of all cases in which samples have been submitted to the Public Analyst, or any other action has been taken with regard to the composition or description of food, otherwise than under the legal provisions referred to in paragraph 1 of this Memorandum (*e.g.* under the Public Health Acts, the Bread Acts or the Merchandise Marks Acts).

8. The Ministry should be furnished with copies of any annual or special reports made to the Authority by the Public Analyst on the general working and administration of the Sale of Food and Drugs Acts and similar matters, or the results of special investigations as to particular foods. The Ministry should also be supplied with a copy of any reports on like matters which may be made by the Medical Officer of Health or any other officer of the Authority. They would be glad to receive such copies as soon as the reports have been submitted to the Authority.

9. In many instances information is given by Public Analysts showing the composition of each sample of milk analysed. This information is valuable, and where it is available the Ministry desire to receive a copy.

## III.—APPOINTMENT OF PUBLIC ANALYSTS.

10. When the appointment of a Public Analyst is submitted for the Minister's approval, particulars of the appointment should be given on the official form.

11. If the person appointed does not already hold an appointment as Public Analyst it is necessary for him to provide satisfactory documentary evidence of competence in analytical chemistry, therapeutics and microscopy.

12. The Minister will ordinarily accept as sufficient evidence of competence in all three subjects the diploma of Fellowship or Associateship of the Institute of Chemistry of Great Britain and Ireland together with a certificate granted by the Institute after an examination conducted by them, in the Chemistry (including microscopy) of Food and Drugs and Water.

13. Where a registered medical practitioner is appointed as a public analyst his medical diploma is ordinarily accepted as sufficient evidence of competence in therapeutics and microscopy, but he is required to furnish evidence of competence in analytical chemistry.

† Revoked as from 1st January, 1928.

14. Where a candidate for the office of Public Analyst does not possess the specific evidences of competence mentioned above, it is necessary for him to produce documentary evidence that he has attained an equivalent standard of competence in each of the three specified subjects. A personal testimonial is of no value as evidence of competence unless it is given by a person who is himself a recognised expert of high standing in the subjects in question and testifies to his personal knowledge of the proficiency of the candidate in these subjects.

#### IV.—COLLECTION AND DISPOSAL OF SAMPLES.

15. The quantity of any article purchased should be sufficient to enable a satisfactory analysis to be made of each of the three portions into which the sample is divided, but should not be so large as to attract special attention. In any case of doubt the Public Analyst should be consulted as to the quantity required.

16. The three portions should be made as nearly equal as possible.

17. In the case of such an article as milk care should be taken to secure an even distribution of the constituent parts of the sample before it is divided. Where milk is sold in bottles it may be useful for this purpose to pour the milk into a larger vessel, and then return a small quantity to rinse out the bottle before the final mixing.

18. The bottles used for liquids should have a narrow neck and should be filled as nearly as possible, since if the samples are shaken in transit the use of bottles much too large for the contents may result in a separation of some of the constituents of the liquids.

19. Such bottles should be closed with new and sound cork stoppers fitting so tightly as to secure the contents with no aid from the wax used for sealing. The sealing should be carried out in such a way as to prevent any attempt to remove the cork. It is, therefore, recommended that the cork should be slit down to a quarter of its length and string drawn through and securely fastened round the neck, the ends afterwards being carried to the top of the cork and sealed thereon.

20. Samples of solid fatty substances such as butter, margarine, lard or dried milk, in which it is important that the proportions of fat and water should be accurately estimated, should be placed without paper (since paper acts as an absorbent) in a dry, wide-mouthed, stoppered bottle, or earthenware jar. It is an advantage to use a screw-capped bottle, provided with a cork-lined metal lid, having a mouth as nearly as possible of the full width of the bottle. The sample can be placed in such a bottle without pressure, and can be easily removed by the Analyst.

21. All bottles should have rounded sides in order to give security to the samples in transit.

22. The labels used should be printed in triplicate, and bear serial numbers so as to avoid any confusion. The vendor should be given an opportunity of verifying the identity of the labels used for the three portions of the samples.

23. In all cases where a screw-capped bottle or any receptacle which cannot conveniently be sealed is used it should be labelled with the necessary particulars, and enclosed in an envelope of stout paper secured at the ends with the Official seal. The serial number and other particulars should be placed both on the bottle and on the envelope.

24. Where cows are milked under the supervision of the sampling officer and a sample of the milk is taken immediately afterwards, the part submitted to the Public Analyst should be marked "Appeal to Cow Sample," or in some other distinctive manner to indicate the circumstances in which it was taken.

25. When a sample of a prescribed medicine is taken, the height of the contents in the bottle supplied by the vendor should be marked in his presence prior to the division of the sample. The bottle so marked should be submitted to the Analyst in order to enable him to determine the total quantity of medicine supplied.

26. The retained portion of any perishable article should be kept at an equable and cool temperature in a dark place.

#### V.—PUBLIC ANALYST'S REPORTS.

27. The Analyst is required by Statute to make Quarterly Reports, and these should be made up to the last day of March, June, September and December respectively.

28. The Analyst should record in his reports the results of the examination of all samples of food and drugs sent to him for the purposes of the Sale of Food and Drugs Acts, &c., including those taken without the prescribed formalities and the special samples of milk (some of which may be taken outside the area for which he is appointed) under the Milk and Dairies (Consolidation) Act, 1915.

29. It is desirable for the sake of uniformity that every report should contain the following particulars (preferably in tabular form\*) :—

\* A suggested tabular form is contained in the Appendix.



- (1) Name or description of article.
- (2) Number of samples of each article examined classified as (a) formal, (b) informal, and (c) private samples; together with (d) total.
- (3) Numbers of samples of each article regarded as adulterated, below standard, or otherwise not complying with prescribed requirements, also classified as in (2);
- (4) Totals of (2) and (3) for all articles.

The following particulars should be given as to the samples regarded as adulterated, &c.:—

- (1) Serial number of the sample;
- (2) Name or description of article;
- (3) Whether a formal, informal, or private sample;
- (4) Nature of the adulteration or irregularity.

30. It is desirable that articles of food which experience shows to be likely to contain preservatives should be examined for preservatives. An indication of the samples which have been so examined should be given under the heading of "Observations" in the main statement of the Report.

31. The Analyst is required to specify in his report the sum paid to him in respect of each analysis. In most cases this can most conveniently be given by a general statement as to the terms of his remuneration.

**APPENDIX.**

**SUGGESTED FORM OF ANALYST'S QUARTERLY REPORT.**

**SALE OF FOOD AND DRUGS ACTS, ETC.**

REPORT of the Public Analyst for the \_\_\_\_\_ of  
upon the Articles analysed by him during the quarter ended the \_\_\_\_\_

**ANALYSES.**

Article.	Number examined.				Number adulterated, &c.			
	Formal.	Informal.	Private.	Total.	Formal.	Informal.	Private.	Total.
<b>Total</b>								

**ADULTERATED SAMPLES, &c.**

Serial Number.	Article.	Whether Formal, Informal or Private.	Nature of adulteration or irregularity.	Observations.

The sum paid in respect of each analysis was.....

(Signed)

Date

# United States Department of Agriculture.

## FOOD INSPECTION DECISIONS

ISSUED AUGUST, 1926.

The following definitions and standards were adopted by the Joint Committee on Definitions and Standards, composed of representatives of the United States Department of Agriculture the Association of American Dairy, Food and Drug Officials, and the Association of Official Agricultural Chemists, at its meeting January 18 to 29, 1926.

### No. 202. DUTCH-PROCESS CHOCOLATE, "ALKALISED CHOCOLATE," AND DUTCH-PROCESS COCOA, "ALKALISED COCOA."

DUTCH-PROCESS CHOCOLATE, "ALKALISED CHOCOLATE," AND DUTCH-PROCESS COCOA, "ALKALISED COCOA," are modifications, respectively, of chocolate and cocoa, in that in their manufacture an alkali carbonate, or other suitable alkaline substance, has been employed.

In the preparation of these products not more than three (3) parts by weight of potassium carbonate, or the neutralising equivalent thereof in other alkaline substance, are added to each one hundred (100) parts by weight of cacao nibs. The finished products conform to the standards for chocolate and cocoa, respectively, due allowance being made for the kind and amount of alkaline substance added.

### No. 203. B. FRUITS AND VEGETABLES.

#### (a) FRUITS AND FRUIT PRODUCTS.

(Except fruit juices, fresh, sweet, and fermented, and vinegars.)

1. **FRUIT** is the clean, sound, edible, fleshy fructification of a plant and is characterised by its sweet, acid and/or ethereal flavor.

2. **FRESH FRUIT** is fruit which has undergone no material change other than ripening since the time of gathering.

3. **DRIED FRUIT** is the clean, sound product resulting from the evaporation of the greater portion of the water from properly prepared fresh fruit.

(a) The term "sundried" is commonly used to designate the product dried without the use of artificial heat.

(b) The terms "evaporated" and "dehydrated" are commonly used to designate the product dried by the use of artificial heat.

4. **"COLD-PACK" FRUIT** is the clean, sound product obtained by packing, in a suitable container, properly prepared fresh fruit, with or without the addition of sugar (sucrose), and maintaining it at a temperature sufficiently low to insure its preservation.

5. **CANNED FRUIT** is the clean, sound product made from properly prepared fresh fruit, with or without water and/or sugar (sucrose).

(a) By processing in a suitable, hermetically sealed container, or

(b) By heating and packing in a suitable container which is then hermetically sealed.

6. **PRESERVE, FRUIT PRESERVE, JAM, FRUIT JAM**, is the clean, sound product made by cooking to a suitable consistency properly prepared fresh fruit, "cold-pack" fruit, canned fruit, or a mixture of two or of all of these, with sugar (sucrose) or with sugar and water. In its preparation not less than forty-five (45) pounds of fruit are used to each fifty-five (55) pounds of sugar (sucrose).

A product in which the fruit is whole or in relatively large pieces is customarily designated a "preserve" rather than a "jam."

7. **GLUCOSE FRUIT PRESERVE, CORN SYRUP FRUIT PRESERVE, GLUCOSE FRUIT JAM, CORN SYRUP FRUIT JAM**, is the clean, sound product made by cooking to a suitable consistency properly prepared fresh fruit, "cold-pack" fruit, canned fruit, or a mixture of two or of all of these, with glucose or corn syrup. In its preparation not less than forty-five (45) pounds of fruit are used to each fifty-five (55) pounds of glucose or corn syrup.

8. **FRUIT BUTTER\*** is the sound product made from fruit juice and clean, sound, properly matured and prepared fruit, evaporated to a semisolid mass of homogeneous consistence, with or without the addition of sugar and spices or vinegar, and conforms in name to the fruit used in its preparation.

9. **GLUCOSE FRUIT BUTTER, CORN SYRUP FRUIT BUTTER**, is a fruit butter in which glucose, or corn syrup, is used in place of sugar (sucrose).

10. **JELLY, FRUIT JELLY**, is the clean, sound, semisolid, gelatinous product made by concentrating to a suitable consistency the strained juice, or the strained water extract, from fresh fruit, from "cold-pack" fruit, from canned fruit, or from a mixture of two or of all of these, with sugar (sucrose).

11. **GLUCOSE FRUIT JELLY, CORN SYRUP FRUIT JELLY**, is the clean, sound, semi-solid, gelatinous product made by concentrating to a suitable consistency the strained juice, or the strained water extract, from fresh fruit, from "cold-pack" fruit, from canned fruit, or from a mixture of two or of all of these, with glucose or corn syrup.

12. **CITRUS FRUIT MARMALADE** is the clean, sound, jelly-like product made from the properly prepared juice and peel, with or without the pulp, of fresh citrus fruit, of canned citrus fruit, or of a mixture of these, by cooking with water and sugar (sucrose). It contains, embedded in the mass, pieces of the fruit peel, with or without portions of the pulp of the fruit.

\* This item has not been revised.

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## ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

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### Food and Drugs Analysis.

**Differentiation of Flours by the Iodine Absorption and by the Rapidity of Sedimentation.** N. A. Trofimuk. (*Z. Unters. Lebensm.*, 1926, 52, 311-318.)

—The absorption of iodine by flour is determined by adding 200 c.c. of water, gradually and with shaking, to 5 grms. of the flour, then shaking the mixture vigorously for 3 minutes, allowing it to stand for 30 minutes and filtering through filter-paper. Fifty c.c. of the filtrate are titrated with a 0.01 *N* solution of iodine in potassium iodide in presence of 2 c.c. of sterile 0.5 per cent. starch paste. Before each titration, the strength of the iodine solution is determined by means of 0.01 *N* thiosulphate solution, and the absorption of iodine by the distilled water used is also measured, the colour obtained in this titration serving as a standard. Since the iodine does not react immediately with the aqueous extract of the flour, the definite time of 1 hour is allowed for completion of the titration. The amount of iodine absorbed is calculated in c.c. of the 0.01 *N* solution per 100 grms. of the dry matter of the flour, determined by drying to constant weight at 100° C. The results thus obtained allow of the classification of flours into two distinct groups—those of monocotyledonous plants absorbing little, and those of dicotyledonous plants absorbing much iodine. For the members of the first group the iodine absorption values are: Millet, 70; rice, 70; wheat, 109; barley, 131; rye, 165; and oats, 178. Bean meal gives the value 703 and pea meal 798.

Further, the physical properties of the proteins present render it possible to identify different flours by the varying rapidity of sedimentation. In a test-tube, 2-3 cm. in diameter, 1 gm. of the flour is mixed by means of a glass rod first with 5 c.c., and then with a further quantity of 7 c.c. of water, the tube being finally

shaken and 10 c.c. of the mixture pipetted into a test-tube, 9.5 mm. wide, in which it occupies a height of 156–160 mm. A curve connecting the height of the sediment (in mm.) with time of settling (in minutes) is then traced. The form of this curve varies widely for different flours, and allows of the detection of 10 per cent. of rye meal, 15 per cent. of oat meal, or 20 per cent. of barley meal in wheat flour. By means of the iodine absorption, the presence of 15 per cent. of barley meal in wheat flour may be detected.

T. H. P.

**Freshness of Eggs.** E. Dinslage and O. Windhausen. (*Z. Unters. Lebensm.*, 1926, 52, 288–291.)—Experimental results show that the change in the specific gravity of eggs during storage is determined far less by the duration than by the conditions of the storage. An important factor is the moisture content of the surrounding air, the loss in density varying inversely with this magnitude. Without knowledge of the conditions of storage, it is quite impossible to judge, even approximately, the age of an egg from its specific gravity.

T. H. P.

**Determination of Benzoic Acid in Egg-Yolk.** E. Waltzinger. (*Chem. Zeit.*, 1926, 50, 949.)—The following procedure, occupying only 4 to 5 hours, yields almost theoretical results. Ten grms. of the yolk are mixed in a small beaker with a little water and then with more water, and transferred to a 500 c.c. flask. After dilution of the liquid to about 400 c.c., and thorough mixing, 42 c.c. of the copper sulphate solution used in preparing Fehling's solution are added slowly and with frequent agitation, and afterwards about 16 c.c. of *N* sodium hydroxide solution. The liquid should then be faintly acid, as otherwise soaps would be formed. The flask is left at rest in a water-bath at 80–90° C. until coagulation of the precipitate occurs, the liquid being then cooled, made up to volume with water and filtered. Of the clear filtrate, 250 c.c. (corresponding with 5 grms. of the yolk), are made alkaline with caustic alkali and evaporated to 20 c.c. in a shallow porcelain dish on wire gauze, the excess of copper being deposited as a brown precipitate and the solution becoming green. The liquid is filtered into a separating funnel, and the precipitate well washed with warm water. The filtrate and washings are acidified with hydrochloric acid and the benzoic acid which separates is dissolved in 40 c.c. of ether, which is completely removed. The residual liquid is shaken vigorously with two successive quantities of 30 and 15 c.c. of freshly distilled petroleum spirit boiling below 80° C. The united ether and petroleum spirit extracts are freed from the aqueous acid liquor, then washed with about 3 c.c. of water, and transferred by means of petroleum spirit into a narrow-necked weighed flask. The solvent is distilled off, and the last traces expelled by means of a current of air, the flask being heated occasionally to 50–60° C. in a drying oven. When the benzoic acid, which should be pure white, attains constant weight, it is dissolved in about 10 c.c. of neutral alcohol, and the liquid titrated with 0.1 *N* sodium hydroxide in presence of phenolphthalein (1 c.c. of alkali corresponds with 0.0122 gm. of benzoic acid or 0.0144 gm. of sodium benzoate). If free fatty acid is present, part of this might contaminate the benzoic acid separated. In such case, the benzoic acid may be removed from the weighed residue by passing a gentle

current of air over the weighed residue heated at 100° C. Commercial egg-yolk is usually preserved with either 5 per cent. of sodium chloride and 1 per cent. of sodium benzoate or with 10 per cent. of sodium chloride and 0.75 per cent. of the benzoate.

T. H. P.

**Specific Characteristics of "Regenerated" Preserved Peas. J. Froidevaux.** (*Ann. Falsif.*, 1926, 19, 536-544.)—The examination was made on the water-insoluble components of the peas, and the material was obtained by washing and draining the peas, placing them in boxes with holes, and immersing the boxes for 16 hours in circulating water, again washing and draining for ½ hour; reducing the peas to a purée, and straining this to remove the outside coverings. It was found that neither differences in the methods of preparation of preserved peas before boxing nor temperature of sterilisation affected the proportion of insoluble matter. In order to find if the preserved peas described as medium (*pois moyens*) could be distinguished from regenerated peas, the insoluble matter of mature dried peas was compared with that of regenerated samples and the following results obtained.

ON DRY BASIS.

	N × 6.25. Per Cent.	Fat. Per Cent.	Mineral matter. Per Cent.	Carbohy- drates. Per Cent.	Hydroly- sable matter. Per Cent.	WATER, Per Cent.
Pois moyens de conserves.						
Paris	23.60	1.93	2.41	72.06	55.29	81.62
	25.92	1.66	1.32	71.10	60.68	78.94
Brittany	23.17	1.77	1.09	73.97	62.75	78.04
	25.40	1.81	1.43	71.36	59.81	76.26
Vend	23.67	1.68	1.24	73.41	67.73	77.44
	24.39	1.56	1.33	72.72	65.29	77.58
Bordeaux	24.12	1.53	1.27	73.08	60.87	81.06
	25.02	1.61	1.26	72.11	64.42	77.74
Pois "Régénérés" de conserves.						
Paris	25.83	1.22	1.77	71.18	59.30	72.90
	25.78	3.15	1.57	69.50	58.33	72.78
	26.51	1.63	1.17	70.69	62.65	73.60
Breton	24.74	1.73	1.97	71.56	59.10	75.74
	Bordeaux	27.73	1.53	1.73	69.01	53.90
Different sources	27.55	1.44	1.52	69.49	52.63	75.72
	24.98	1.52	1.22	72.28	60.54	73.74
	26.07	1.47	1.47	70.99	52.11	76.34
	27.18	1.53	1.53	69.76	57.68	75.87

It is considered that the commercial value of preserved peas is inversely proportional to the drying before boxing. Quality is higher as the nitrogen of the insoluble matter diminishes, and peas with 21 per cent. of protein and 84 per cent. of water are tender, with an excellent taste, but with 23.5 per cent. of protein and

81 per cent. of water the objectionable taste of "preserving" becomes discernible, and 24.7-25.9 per cent. of protein denotes inferior products, with a strong presumption of being prepared from dried peas if the insoluble material retains 76 per cent. water, and the presumption becomes certainty with 25.9 per cent. of protein. (Cf. ANALYST, 1926, 51, 151 and 199.)

D. G. H.

**Determination of Fat in Malted Milk.** E. S. Rose. (*J. Amer. J. Pharm.*, 1926, 98, 595-596.)—A modification of the Werner-Schmid method is suggested as follows:—To 0.5-1 grm. of malted milk are added 5 c.c. of hot water, followed, after emulsification, by 10 c.c. of concentrated hydrochloric acid, and the mixture is placed in a boiling water bath for 5 minutes. After being cooled and shaken for 2 minutes with 25-30 c.c. of a mixture of U.S.P. benzene 2 parts, and washed ether 1 part, the benzene-ether layer is blown off in the usual way, and the extraction repeated twice. The solvent is distilled, and the residue dried and weighed.

D. G. H.

**Determination of Sulphur Dioxide in Sugar Factory Products.** J. P. Ogilvie. (*Int. Sugar J.*, 1926, 28, 644.)—A 400 c.c. flask, provided with a thistle funnel and tap funnel, has a delivery-tube connected with a wider tube (horizontal) containing a cotton wool plug. To this tube is attached, by a tube bent at right angles, an inverted filter funnel having a clamp holding lead acetate paper. About 50 grms. of purest granulated zinc (previously washed with pure dilute hydrochloric acid) are placed in the flask and just covered with water. Fifty c.c. of pure dilute hydrochloric acid (1 : 2) are poured into the tap funnel and allowed to run slowly through the thistle funnel over the zinc, the gases evolved being passed through the lead-impregnated filter paper for 30 minutes. No coloration should appear on the filter-paper. A series of standard stains is now prepared by making the experiment with sugar solutions containing increasing amounts of sodium sulphite, representing 0.0010, 0.0015, 0.0020, and 0.0025 per cent. of sulphur dioxide when five grms. of sugar are used. For an analysis 5 grms. of sugar (or its equivalent in the case of a liquid) are dissolved in water and run on to the zinc through the funnel, followed by the 50 c.c. of pure dilute hydrochloric acid, added slowly as above. The depth of coloration produced on the lead acetate paper is compared with the standard stain.

R. F. I.

**Allantoic Acid in the Green Parts of *Phaseolus vulgaris*.** R. Fosse. (*Comptes rend.*, 1926, 183, 1114-1116.)—The origin of the allantoic acid identified in the green leaves of *Phaseolus vulgaris* has been investigated, and it is pointed out that allantoic acid is readily formed from uroxanic acid, which when dissolved gives, on warming, urea and glyoxylic acid, and, like allantoic acid, is precipitated by acetic xanthidrol, and the dixanthyluroxanic compound by crystallisation in pyridine is transformed into dixanthylallantoic acid. The following facts show that allantoic acid exists as such in the sugar of the green parts of *Phaseolus vulgaris*. Allantoic and uroxanic acids can be readily separated at low temperatures and identified by liberating the pyryle salt and corresponding ureide

by means of mineral acid, adding a few drops of lead acetate solution to the solution of the ureide, and, after removal of mineral acid and pyryle salt, the characteristic crystals of uroxanic acid are seen. The allantoic solution remains clear, and whilst 0.02 gm. of xanthyl uroxamic precipitate will give a large quantity of crystals, 0.2 gm. of the raw xanthylated product from *Phaseolus vulgaris* give no trace of crystals. The hydrolysed xanthylated product of the latter gives, however, positive reactions for allantoic acid. (Cf. ANALYST, 1924, 49, 601; 1926, 51, 152.)

D. G. H.

#### **Water-soluble Content of Calcium and Phosphorus in Cabbage.**

**W. H. and C. B. Peterson.** (*J. Agric. Res.*, 1926, 33, 695-699.)—Determinations of the water-soluble constituents of plants are important when these are to be preserved by natural fermentation processes (e.g. the manufacture of sauerkraut). Twelve samples of cabbage contained 0.038 to 0.053 per cent. and 0.023 to 0.036 per cent. of calcium and phosphorus respectively, the corresponding mean values being 0.046 and 0.028 per cent. An average of 60 and 61 per cent., respectively, of these amounts was water-soluble, but the solubility decreased as the season advanced. For 1924 and 1925 the average values differ by less than 10 per cent.

J. G.

**Vinegar and "Vinegar Essence" containing Formic Acid.** **A. Kreutz and C. Büchner.** (*Z. Unters. Lebensm.*, 1926, 52, 295-298.)—Samples of ordinary vinegar or spirit vinegar containing appreciably more than the permissible (in Germany) limiting proportion of 0.5 gm. of formic acid per 100 grms. of acetic acid are not uncommon. Genuine fermentation vinegar contains only very little formic acid, but artificial vinegar made from carbide, even when controlled during the process of manufacture by means of the permanganate test, may contain as much as 0.3 gm. per 100 grms. of acetic acid. The presence of colouring matter is not capable of accounting for the high proportions of formic acid sometimes found, and the probable addition of preservatives containing this acid is suggested.

T. H. P.

**Food Tests. I. Possibility of Distinguishing between Malt Coffee and Grain Coffee by the Maltol Reaction. II. Blood Sausage with Artificially Coloured Skin.** **T. Merl.** (*Z. Unters. Lebensm.*, 1926, 52, 321-324.)—I. Every one of a large number of samples of malt coffee was found to contain maltol, which was not detected in any sample of grain coffee. The test for maltol is made by heating 10 grms. of the finely ground coffee for a short time to boiling with 20-25 c.c. of chloroform, filtering when cold, shaking 10 c.c. of the filtrate in a small separating funnel with 1-2 c.c. of water, removing the latter, and at once filtering into a centrifuge tube. After addition of 1 c.c. of very dilute ferric chloride solution (1 drop of 10 per cent. solution to 20 c.c. of water), the liquid is shaken vigorously to emulsify it and centrifuged to separate the ferric chloride solution from the chloroform; the upper layer turns violet in presence of maltol. Since the coloration may be due to added salicylic acid, the latter must be tested for by either Lintner's or Jorissen's reaction, which is applied to 10-15 c.c. of distillate

obtained from a mixture of 10–15 c.c. of the liquid under examination with 5 c.c. of phosphoric acid (1·7).

II. Treatment of sausage skin with artificial colouring matter may be detected with certainty by extracting the skin with 50 per cent. alcohol, expelling the spirit from the liquid, acidifying with potassium hydrogen sulphate, and immersing wool, which assumes a greenish tint. When many tests have to be made, it is sufficient to moisten with concentrated hydrochloric acid a small portion of the skin, which turns intense violet.

T. H. P.

**Caffeine in "Yocco."** E. de Wildeman. (*Compt. Rend.*, 1926, 183, 1350.)—"Yocco" has been classified as belonging to the type *Paullinia scarlatina*, Radlk. A number of other types of *Paullinia* from the Botanical Gardens, Brussels, have been examined for caffeine, but this has up to now not been detected in them. *P. scarlatina* is the second type of this group (genus) shown to contain caffeine, the first being *P. sorbilis*, of which the aqueous extract is used as a stimulant in certain parts of South America. (*Cf. ANALYST*, 1926, 51, 465.)

R. F. I.

**The Pyridine Test as a Quantitative Method for the Determination of Minute Amounts of Chloroform.** W. H. Cole. (*J. Biol. Chem.*, 1926, 71, 173–180.)—The reaction of pyridine with chloroform and other substances containing the R-C-halogen<sub>3</sub> group in the presence of strong sodium hydroxide, described by Fujiwara and later by Ross (*J. Biol. Chem.*, 1923–24, 58, 641), has been standardised as a quantitative colorimetric method for the determination of small amounts of chloroform (0·1 down to 0·0001 per cent., corresponding to 0·01256 and 0·00001256 molar), in pure water and in aqueous extracts of animal tissues. The procedure for the preparation of the extracts is given, and the chloroform determination is made as follows:—Into a narrow 10 c.c. test-tube are measured 2 c.c. of 20 per cent. sodium hydroxide solution from a burette graduated to 0·1 c.c. To this are added 1 c.c. of chemically pure pyridine (colourless) from a similar burette, and 1 c.c. of the solution to be tested, measured by a 1 c.c. pipette. The tube is loosely corked to prevent undue evaporation of the pyridine, and, with constant shaking, is immersed above the liquid level for 1 minute in water at 100° C.; then it is transferred to cold water until the temperature of the mixture has fallen to about 20° C. During the cooling, the pyridine and test solution, coloured pink or red if chloroform is present, rise above the alkali, and are removed by a 1 c.c. pipette to a colour comparison tube and are compared with colour standards previously made up to match the colours obtained from known concentrations of chloroform. When all directions are accurately followed, the colour from any one concentration of chloroform may be duplicated regularly. Basic fuchsin made up with 50 per cent. alcohol containing 0·01 per cent. hydrochloric acid is used to match the colours, since the coloured pyridine compound is unstable. Several checks on the reliability of the method are described.

P. H. P.



## Biochemical, Bacteriological, etc.

**The Value of Cocoa and Chocolate as Sources of Protein in the Diet.** H. H. Mitchell, J. R. Beadles and M. H. Keith. (*J. Biol. Chem.*, 1926, 71, 15-31.)—When given to young growing rats at a level of approximately 8 per cent. of crude protein, the nitrogen of cocoa was found to possess an average coefficient of digestibility (corrected for metabolic nitrogen in the faeces) of 38. The average biological value of the absorbed nitrogen of cocoa was 37, though the 15 determinations made varied considerably. An average biological value for milk nitrogen of 84 was obtained, which is almost identical with the values previously reported. A mixture of milk and cocoa nitrogen in the ratio of 1 to 1, also given at a level of approximately 8 per cent. of crude protein, was found to have an average true digestibility of 63 and an average biological value of 70. Some calculations which are given confirm the determined biological value for cocoa nitrogen, and indicate strongly that no marked supplementary relation exists between the nitrogenous compounds of milk and of cocoa. Cocoa, with an average crude protein content of 21.5 per cent., would, according to these investigations, contain 8.2 per cent. of digestible crude protein, and only 3.0 per cent. of "net" protein, available for the replenishment and enlargement of the protein content of the animal body. Evidently cocoa must be classed as an unimportant protein food. The same conclusion applies even more forcibly to chocolate, which contains less crude protein than cocoa. The ingestion of cocoa markedly increased the excretion of creatine+creatinine by rats, as determined by the reaction of Jaffé. An examination of the cocoa by this reaction indicated the presence in it of 0.55 per cent. of creatinine and 0.24 per cent. of creatine. An attempt definitely to isolate and identify creatine or creatinine in cocoa is now being made. P. H. P.

**Nickel and Cobalt in the Pancreas.** G. Bertrand and M. Macheboeuf. (*Bull. Soc. Chim.*, 1926, 39-40, 1646-1648.)—The pancreas is one of the organs richest in nickel and cobalt, and these were respectively found present (expressed as thousandths of a mgrm. per kilo. of dry material) in the pancreas of the cow to the extent of 715 and 357; in that of the calf, 800 and 350; of the horse, 500 and 500; of the sheep, 682 and 341; and of the pig, 213 and 178. Preparations of insulin (extract of pancreas) may contain a hundred times more nickel and cobalt than the glands from which they have been extracted. (*Cf. ANALYST*, 1925, 50, 85.) D. G. H.

**Nephelometric Methods for the Determination of some Sulphur Compounds in Urine.** W. Denis and L. Reed. (*J. Biol. Chem.*, 1926, 71, 205-208.)—Nephelometric methods are described for the quantitative determination of the sulphur compounds in urine. The methods require the minimum of reagents and of time for their use. It is believed that they may be of use in cases in which the quantity of urine available is limited to an extremely small volume. The reagents required are identical with those used for the work on blood, but dilution of the urine to a proper concentration for the total sulphur determination

is necessary. The methods have been checked by a series of comparative analyses made by gravimetric methods. Inorganic and total sulphates have been determined by the methods of Folin (*J. Biol. Chem.*, 1905-06, 1, 131), and total sulphur by the procedure of Benedict (*J. Biol. Chem.*, 1909, 6, 363) with the use of Denis's modification (*J. Biol. Chem.*, 1910, 8, 401) of the oxidation mixture employed in the original method.

P. H. P.

**Methods for the Determination of some of the Non-Protein Sulphur Compounds of Blood.** W. Denis and L. Reed. (*J. Biol. Chem.*, 1926, 71, 191-204.)—Previous work on the inorganic sulphates and ethereal sulphates in blood is described, and shows the diversity of results obtained by different investigators. The authors describe their work, which was carried out in three parts. (1) A comparison was made of the results obtained for the inorganic sulphate fraction, when dialysis, and when various methods of protein precipitation were employed; (2) a series of experiments was carried out to determine whether a fraction of the non-protein sulphur of blood exists in the form of the ethereal sulphates or of neutral sulphur; and (3) an attempt was made to devise micro methods for the quantitative determination of these forms of sulphur. Nephelometric micro methods were devised, and are described, for the quantitative determination of inorganic, total sulphates, and total non-protein sulphur in blood, and analyses are presented which show the distribution of these sulphur compounds in human blood and in the blood of several species of animals. Ethereal sulphates are calculated by difference between the values obtained for total and inorganic sulphates; neutral sulphur by difference between the values obtained for total sulphur and total sulphates.

P. H. P.

**Vitamin Content of Grapes and Grape Wines.** A. Merjanian. (*Z. Unters. Lebensm.*, 1926, 52, 307-311.)—Koopman's experiments on a number of different foodstuffs (*Wiener klin. Wochens.*, 1924) have shown that for the determination of vitamin C, Bezssonoff's reagent (*ANALYST*, 1921, 46, 462; 1924, 49, 594) gives results substantially identical with those yielded by experiments on animals. The author finds that Bezssonoff's reagent indicates this vitamin to be present in varying proportions in grapes and wines of different origins. In young wines the amounts of the vitamin are particularly marked, whereas both still and sparkling wines more than four or five years old are quite free. Grapes lose their vitamin during drying or long storage, and must contain less than the corresponding quantity of fresh grapes, whilst sterile must shows very little, and concentrated must none of the vitamin; this is found in small quantity in pure wine vinegar, but is lacking in wine yeasts.

T. H. P.

**Resistance of Fat-Soluble Vitamins to Hydrogenation.** L. Randoin and R. Lecoq. (*Ann. Falsif.*, 1926, 19, 518-523.)—In order to hydrogenate cod liver oil satisfactorily on a semi-industrial scale with nickel catalyst a temperature of about 180-190° C. is necessary. Such hydrogenated oil (iodine value 28.7) was given to rats showing typical rachitic signs on a Sherman and Pappenheimer

**No. 84** diet, with the result that a slow rate of growth was maintained, but ophthalmic lesions were not always precluded. Hydrogen under pressure did not inhibit the destructive effect of heat on the fat-soluble vitamins. It was found that butter, and particularly summer butter, was not so wanting in antirachitic properties as has been thought, but margarines prepared with hydrogenated oils are inferior to those with natural oils and fats as sources of fat-soluble vitamins. D. G. H.

**On the Growth-Promoting Property of Irradiated Fat in the Diet, of Direct Irradiation and of Cod Liver Oil.** H. Goldblatt and A. R. Moritz. (*J. Biol. Chem.*, 1926, 71, 127-137.)—By two methods an attempt was made to compare the growth-promoting power of irradiated fat in the diet, of direct irradiation and of cod liver oil in order to determine whether irradiated fat in the diet can be used in growth-promotion experiments as a substitute for direct irradiation of the animals. This would be an advantage in the case of small animals, for hooding of the eyes is troublesome and time-consuming, and direct irradiation otherwise induces a conjunctivitis and opacity of the cornea which complicates other eye changes induced by diet, and probably affects the general condition of the animals. Irradiated fat in the diet and direct irradiation possess, to about the same degree, the power to promote gain in weight of rats on a diet deficient in both fat-soluble vitamins (*A* and *D*), but this power is less than that of cod liver oil, which possesses the growth-promoting power to a far greater degree, since it is rich in vitamins *A* and *D*. Thus irradiated oil in the diet can be used in growth promotion experiments as a substitute for direct irradiation of animals, but neither source of radiant energy can act as a complete substitute for cod liver oil unless fat-soluble vitamin *A* is also administered. Radiant energy, administered directly or indirectly, although it prolongs and enhances growth, does not prolong the life of rats on a diet deficient in vitamins *A* and *D*, and does not prevent them from developing xerophthalmia. P. H. P.

**Note on Colorimeter Correction Curves.** S. L. Wright, junr. (*J. Biol. Chem.*, 1926, 71, 209-212.)—Beer's law, the basis of colorimetric determinations, is seldom, if ever, accurate over wide variations in concentration. Generally, a correction curve, prepared experimentally with solutions of known concentration, is used to overcome this error, but it should not be taken too literally. A graph is given which shows variation, among observers, of the colorimeter correction curve for sugar (Folin and Wu (*J. Biol. Chem.*, 1920, 41, 367) method). It is difficult to decide the reason for the variation, but the importance of individual determination of these curves is stressed, and they may be easily prepared. In the author's laboratory, for clinical purposes, a single standard corresponding to 150 mgrms. of sugar is used, as accurate determinations are more important near that point. Attention is called to the high colorimetric correction in creatinine determinations; a graph is shown and reference is made to other methods of overcoming it. P. H. P.

**Action of Neon Light on Bacteria.** A. Philibert and J. Risler. (*Comptes rend.*, 1926, 183, 1137-1139.)—Neon light, the principal radiations of which are

absorbed by methyl violet, had no lethal effect on bacteria, but on sensitised cultures the effect was marked. Cultures of *staphylococcus*, sensitised by a solution of methyl violet, were killed by 1 minute's exposure, whilst 1 hour did not kill the non-sensitised culture. *B. diphtheriae* in sensitised cultures required 5 minutes, *B. pneumococcus* 15 minutes, and *streptococcus* 30 minutes, but 2 hours' exposure had no effect on sensitised *coli bacillus*. Eberth's bacillus, Para A, Para B., *B. pyocyaneus*, *Proteus* and *Shiga* were not destroyed after 30 minutes' irradiation.

D. G. H.

**Testing of Disinfectants by the Rideal-Walker Method.** Q. Moore. (*J. Soc. Chem. Ind.*, 1926, 45, 472-474.)—Rideal-Walker coefficients were determined for three disinfectant fluids, *B. typhosus* (Lister) in three broth culture media being used for the purpose. These were standardised (a) by titration in the cold, acid then being added, and the whole boiled and filtered; (b) by a similar process with titration at boiling point. These solutions had  $P_H$  values of 6.9 and 7.3 respectively, and a third (c) was used which was adjusted to the  $P_H$  value of the Rideal-Walker broth (7.6), and then boiled and filtered. In each case the values obtained increased with the  $P_H$  value, (c) giving results comparable with the accepted Rideal-Walker values. The  $P_H$  value at which the broths are filtered is the chief factor controlling the results obtained.

J. G.

**Bacterial Flora of the Market Oyster.** C. Elliot. (*Amer. J. Hyg.*, 1926, 6, 755-776.)—Samples of fresh market oysters in the shell varied in total aerobic count from 1000 to 800,000 organisms per c.c. of oyster juice, and shell-free and shell oysters kept at laboratory temperatures showed a sudden and maximum rise in total count from the second to the fourth day of storage. The *Bacillus coli* count increased from 4 to 50,000 during the first 14 days (when the oysters were stored in a cool basement), after which spoilage set in and the score dropped. Shell-freed oysters at laboratory temperature very soon became markedly acid (there was little change in those in shells), but later a reversal of action took place until the original  $P_H$  value was regained and maintained. The change lagged 2-3 days behind at ice-box temperature. The principle groups of bacteria found in decomposing oysters are the colon aerogenes, streptococci, water bacteria including the green fluorescent, the yellow pigmented, the non-pigmented, the vibrios composed of comma-shaped organisms, anaerobes (4 groups) and incidental organisms, such as chromogenetic cocci and aerobic spore-formers. Fermentation may be begun by organisms of the colon aerogenes group, but these are inhibited as acidity rises (chiefly due to streptococci). After about 10-12 days the water bacteria increase rapidly, acidity falls, and the oyster meat decomposes, the medium becoming thick and slimy, chiefly owing to yellow pigmented and green fluorescent forms. Non-pigmented water bacteria are present, but do not appear to take part in the decomposition. Anaerobes vegetate profusely in the spoiling oyster and cause evolution of gas, which is apparently their only activity.

D. G. H.

**Bacteriological Study of Ham Souring.** E. A. Boyer. (*J. Agric. Res.*, 1926, 33, 761-768.)—"Ham souring," or the development of a sour or putrid condition in the interior of the ham, is due to organisms possibly present in the

blood and tissues of the living animal. Aerobic organisms isolated were motile and non-motile cocci and rod forms, and chromogenic and non-chromogenic gelatin liquefiers and non-liquefiers. Four anaerobic organisms were tentatively identified as *B. putrefaciens*, *B. histolyticus*, *B. sporogenes*, *B. tertius*. An unidentified glucose-fermenting bacillus was also isolated, which resembled *B. oedematiens*, having the form of a stout, thick rod with non-motile flagella, and large oval spores. The absence of *B. coli*, which abound on the killing-floor, and the fact that most of the above organisms are found in the fresh hams, indicate that souring can be prevented only by prompt and efficient handling at low temperatures rather than by alteration of killing-floor practice. J. G.

**Action of Certain Organic Substances on Alcoholic Fermentation.**  
**E. Mameli.** (*Giorn. Chim. Ind. Appl.*, 1926, 8, 555-564.)—When present in small proportions, the phenoxyacetic acids and their sodium salts, the 3-coumaranones, and the 2:4-diketobenzo-1:3-isoxazines, which are closely inter-related constitutionally and genetically, are able to stimulate the fermentation by fresh pressed brewery yeast of a mixture in equal volumes of 10 per cent. dextrose solution and Willstätter and Steibelt's salt solution, the fermentation being measured by the volume of carbon dioxide evolved in a certain time at 37° C. Recent investigations have shown that the above compounds, in small doses, raise the body temperature without producing local intolerance or toxicity, and experiments to ascertain if they exhibit also vitaminic properties are in progress. Enhancement of alcoholic fermentation to the same degree, namely, about 20 per cent., as is observed with the above compounds, is caused also by small proportions of methyl iodide, chloroform, mannitol or quinol, and less pronounced effects in the same direction are produced by a number of other compounds of various chemical character. Phenol and resorcinol retard fermentation. T. H. P.

**Infection and Temperature Relations of Black Rot of Sweet Potatoes in Storage.** **J. I. Lauritzen.** (*J. Agric. Sci.*, 1926, 33, 663-676.)—Sweet potatoes (Little Stem Jerseys) may become infected with black rot (*Cerastostomella fimbriatum*, Elliot) through the normal skin, but especially at raised areas, slight abrasions, and small rootlets. Infection and growth on sweet-potato agar occur from 9.5 to 34.5° C., the optimum temperature being about 23° to 28° C. The rate of growth of black rot lesions, as measured by their areas, is less rapid than the enlargement of the colonies of the pathogene on sweet-potato agar, but takes place at as low a temperature as 6° C., increasing rapidly above 14° C. until it reaches a maximum at about 25° C., and then declines to zero at 34.7° C. These temperatures vary slightly, however, from one experiment to another. The results show that potatoes which are uninfected when placed in storage, although the pathogene is present, can be kept at 10-12° C. with little danger of infection. At the early stages of infection (11 days) the number of infections is governed by the temperature, the optimum temperature being the same as for the development of the disease itself. Beyond 11 days the influence of temperatures between 14° and 28° C. decreases. J. G.

## Organic Analysis.

**Mercurimetry, a New Method for Volumetric Determinations.** A. L. Jonesco-Matiu. (*J. Pharm. Chim.*, 1926, 118, 533-544.)—Mercurimetry depends on the complete precipitation by salts of mercury of the substance under examination, and subsequent determination of the mercury. The method is regarded as capable of generalisation and has so far been applied in detail to the determination of acetone and alkaloids. *Acetone*.—From 1 to 10 c.c. of the acetone solution, 10 c.c. of mercury solution (50 grms. of mercuric oxide in 500 grms. of sulphuric acid, made up to 1 litre) and 100 c.c. of water are boiled for 20 minutes, left to cool and filtered. The precipitate is washed with water, transferred to a flask, and 25 c.c. of a mixture of 1 part concentrated nitric acid and 2 parts of concentrated sulphuric acid added; the mixture is warmed, and a few drops of 10 per cent. permanganate solution added until a rose pink colour results. The mixture is then poured into 100 c.c. of water, and 12 drops of 10 per cent. sodium nitroprusside solution added, when a turbidity forms, after which 0.1 N sodium chloride solution (1 c.c. = 0.010124 grm. of mercury, or theoretically 0.003348 of acetone, but practically the factor is rather over 0.0028) is added until the solution clears. Results obtained were much more accurate than by gravimetric methods.

*Alkaloids* are precipitated by the Mayer-Valzer mercury reagent (10 grms. of potassium iodide and 15 grms. of mercuric iodide in water), the precipitate dissolved by means of the oxidising acid mixture, and the mercury ion titrated by the Votoček and Kaspárec method, as above. It is necessary to determine the type of salt formed by each particular alkaloid, and the following practical factors were found:—One c.c. of 0.1 N sodium chloride solution was equivalent to 0.0066 grm. of quinine, 0.0140 of strychnine, 0.0083 of morphine, 0.01 of codeine, and 0.0090 of cocaine, *i.e.* from the quantity of mercury fixed by 1 c.c. of a 1 per cent. solution of the alkaloid.

*Albumin*.—Although complete precipitation is obtainable, mercury appears to be fixed by adsorption as well as by combination, and the method of determination has not yet been fully worked out.  
D. G. H.

**Detection of Alcohol Adulterants.** J. M. Haley. (*Ind. Eng. Chem.*, 1926, 18, 1312-1313.)—The following tests are recommended for the rapid detection of the commoner adulterants of ethyl alcohol. *Methanol*.—Five c.c. of a 5 per cent. solution of the alcohol are treated with 2 c.c. of permanganate reagent (potassium permanganate, 3 grms., 85 per cent. phosphoric acid, 15 c.c., water to 100 c.c.); after ten minutes, 2 c.c. of oxalic acid solution (oxalic acid, 5 grms., dissolved in 100 c.c. of 1:1 sulphuric acid) are added, followed by 5 c.c. of Schiff's reagent. A blue coloration develops within ten minutes if the quantity of methanol is not less than 1:7000. *Acetone*.—A red coloration is obtained when 0.5 c.c. of the alcohol solution is treated with 1 c.c. of ammonia mixture (concentrated ammonia, 10 c.c., ammonium sulphate, 30 grms., water, 45 c.c.) and 3 drops of 25 per cent. sodium nitroprusside solution. The test will detect 1 part of acetone in 10,000.

**Benzene.**—Five c.c. of the alcohol are mixed with 5 c.c. of a mixture of equal volumes of 3 per cent. hydrogen peroxide and 4 per cent. sodium nitrite solution, 2 c.c. of 2 *N* sulphuric acid are added, the mixture is boiled for two minutes, cooled, and treated with concentrated sodium hydroxide solution. An orange-red coloration is obtained if benzene is present in toxic quantity. **Alkaloids.**—Three c.c. of the alcohol are mixed with a few drops of sulphuric acid and a drop of alkaloid reagent (phosphomolybdic acid, 3 grms., and ammonium vanadate, 3 grms. dissolved in 100 c.c. of concentrated sulphuric acid). A coloration varying from violet to orange is obtained with as little as 1 part of brucine per 250,000. **Phenol.**—Five c.c. of the sample are treated with 1 c.c. of ferric chloride solution and diluted to 20 c.c. A purple coloration indicates the presence of phenol. **Diethylphthalate.**—Ten c.c. of the alcohol are mixed with 5 drops of 10 per cent. sodium hydroxide solution and evaporated to dryness; 0.5 c.c. of 5 per cent. resorcinol solution is added and the evaporation repeated. The residue is treated with 6 drops of concentrated sulphuric acid, diluted to 20 c.c., and 5 c.c. of 10 per cent. sodium hydroxide solution are added. A yellowish-green fluorescence is obtained if the alcohol contains more than 1 part of diethylphthalate per 10,000. W. P. S.

**Isopropyl Alcohol as a Substitute for Ethyl Alcohol in the Determination of the Acid Values of Fats and Oils.** H. A. Schuette and M. P. Smith. (*Ind. Eng. Chem.*, 1926, 18, 1241–1245.)—Commercial isopropyl alcohol is not anhydrous and consists of a constant-boiling mixture containing about 91 per cent. of alcohol by volume. Distillation following a short digestion with lime gives a practically anhydrous alcohol of boiling point 82.4° C. and specific gravity 0.7855 at 20° C./4° C. The alcohol is freely miscible with water and dissolves fats, oils and waxes. It may be used in place of ethyl alcohol in the determination of the acid values of fats and oils. In the case of samples having a high acidity there is evidence that the results obtained are too high, owing to slight saponification of the oil or fat during the neutralisation. W. P. S.

**Rapid Method for the Determination of Sulphur in certain Petroleum Products.** E. S. Squire. (*J. Soc. Chem. Ind.*, 1926, 45, 466–469.)—The principle of the method is the neutralisation of a sodium carbonate solution by the vapours produced when the oil is burnt, the weighed lamp and contents being re-weighed as soon as the solution is neutralised. An Esling lamp is used, the vapours being drawn off by aspiration. The indicator used depends on the liberation of iodine and the formation of a colour with starch when a solution containing a mixture of an iodate and an iodide is acidified. For oils containing more than 0.25 per cent. of sulphur, 10 c.c. of a *N*/80 sodium carbonate solution containing 38 grms. and 5.3 grms. of potassium iodide and iodate per litre, respectively, are used, and for those containing 0.06 to 0.25 per cent. a *N*/160 solution is used with 25 grms. and 3.5 grms. of the respective salts per litre. In the former case the results are given by direct calculation and agree to within 0.03 per cent. with those given by the gravimetric method, but in the latter, high and low results are obtained for sulphur contents below and above 0.13 per cent., respectively. A correction-curve is

therefore constructed for each piece of apparatus, and the conditions of the experiment kept constant throughout. The mechanism of the indicator reaction is discussed, and the solutions are adjusted so as to ensure that the amount of sulphur dioxide is always sufficient to reduce all the iodine liberated. Each determination takes only 25 minutes.

J. G.

**Comparison of the Deflocculating Power of Soaps by the Carbon Black Test.** R. M. Chapin. (*Ind. Eng. Chem.*, 1926, 18, 1313–1316.)—The carbon black test described by McBain, Harborne and King (*ANALYST*, 1923, 48, 580), and based on the quantities of carbon carried through filter paper, has been slightly modified by the author and used to determine the effect of concentration, temperature, kind of fatty acid, and kind and proportion of alkali on the deflocculating power of soap solutions. The results obtained indicate that at equilibrium colloidal soap is inert, that simple soap anions constitute the active deflocculating agent at the lower concentrations, and that simple soap molecules may constitute the agent at higher concentrations. Further, these two mechanisms are sharply distinct and non-co-operative, and the transformation of colloidal soap into soap crystals may be attended by a distinct increase in deflocculating power. W. P. S.

**Rose Flower Wax.** H. Prophète. (*Bull. Soc. Chim.*, 1926, 39–40, 1600–1610.)—The following figures were obtained on the crude rose wax:—M.pt., 61° C.; water and matter volatile at 110° C., 0.68 per cent.; non-fatty matter, 0.1 per cent.; saponification value, 29.8; iodine value (Hübl), 13; Reichert-Meissl value, 1.35; Hehner value, 97.4; acetyl value (Lewkowitsch), 9; acid value, 3.15. The crude wax had the following composition: Water and non-fatty matter, 0.78; soluble acids, 1.6; insoluble acids: saturated, 10.6; non-saturated, 6.4; un-saponifiable matter, 80.2, made up of hydrocarbons, 56.5, alcohols 20.2 and "acides-alcools" 3.2 per cent. The 20 per cent. of alcohols contained: Pseudocerylic alcohol ( $C_8H_{12}O$ ), 8; isocerylic alcohol, 6;  $C_{10}H_{20}O$  (m.pt. 49.5° C.), 1.5;  $C_{10}H_{20}O$  (m.pt. 43° C.), 1; and an alcohol of m.pt. 16° C., 0.5; whilst the saturated hydrocarbons (51.5 per cent.) comprised triacontane, 6; heptacosane, 15; hexacosane, 8; tricosane, 6; docosane, 6; hencicosane, 2; eicosane, 8; and hexadecane, 0.5.

D. G. H.

**Separation and Analysis of the Volatile Solvents and Thinners of Lacquers.** R. M. Carter. (*Ind. Eng. Chem.*, 1926, 18, 1234–1235.)—The volatile constituents of lacquers are best separated by distillation of about 200 c.c. of the sample from a flask immersed in an oil-bath, but the maximum temperature should not exceed 120° C.; the residue in the flask is then subjected to steam distillation to remove the higher boiling constituents. The two distillates, after being dried, may be mixed or examined separately. The odour, specific gravity and boiling range (fractional distillation) of the distillate often afford information as to the nature of the constituents present. The saponification value of the distillate may be expressed in terms of the most probable ester, as indicated by the odour and boiling range. A measure of the amount of hydrocarbons present is obtained from



the solubility of the distillate in twice its volume of concentrated sulphuric acid ; odour is usually sufficient to identify the hydrocarbon, which, however, may be separated from the acid, neutralised, and its boiling point determined. A high solubility of the distillate in water indicates a high alcohol content, but the presence of ethyl lactate increases the solubility. There is no difficulty in distinguishing between alcohol and ethyl lactate. Solubility in three volumes of saturated sodium chloride solution gives an indication of the amount of methanol, ethyl alcohol and acetone present and these may be identified by suitable tests.

W. P. S.

**Separation and Analysis of Pigments in Lacquer.** F. H. Hopkins. (*Ind. Eng. Chem.*, 1926, 18, 1233.)—When the pigment consists of Prussian blue, chrome yellow or chrome green, 5 grms. of the sample are treated with methylene chloride, the mixture is submitted to centrifugal action, and the liquid portion is decanted. The precipitate, consisting of nitrocellulose and pigment, is placed in an alundum crucible and extracted for three hours with 25 c.c. of a mixture of equal volumes of benzene and ethyl acetate ; the nitrocellulose, plastifiers, and resins are thus removed, and the residue of pigment is dried at 105° C., and weighed. In blue lacquers the pigment is usually Prussian blue and ultramarine. Results of determinations of nitrogen and iron will give data for calculating the quantity of Prussian blue. To distinguish between Prussian blue and ultramarine, a portion of the lacquer is ignited and the ash is tested for iron. Yellow lacquers are usually made with chrome yellows, and the lead may be determined as lead sulphide and the chromium as chromic oxide. Green lacquers are usually coloured with a mixture of Prussian blue and chrome yellow ; determinations of the nitrogen, iron, lead and chromium are required to ascertain the quantity of each of these pigments. White lacquers are made with zinc oxide, zinc sulphide, lithopone, or titanium oxide.

W. P. S.

**Standardisation of Wool.** Henseler. (*Int. Rev. Sci. Pract. Agric.*, 1926, 4, 513–532.)—In the preparation of international wool statistics it is necessary that each country, in sending in its reports, should give details as to the type of wool, *i.e.* its breed, whether washed or not, etc., and weights should be based on a standard moisture content. The methods recommended for determining these points are those in use at the Institute of Animal Breeding, Munich, and are given in detail. In the course of their description the author gives particulars of the more exact examination of wool in the fleece. The most important parts of the sheep from which samples should be taken are the shoulder-blade, flank (at the last true rib) and the middle of the hind leg. The sample should be at least 2.5 cm. in diameter and cut off close to the skin. The samples are examined for :— (1) *Moisture*.—Two and a half grms. of the wool are dried for 2 hours at 98° C., cooled in a desiccator and weighed to constant weight. (2) *Water-solubles*.—From 2 to 3 grms. are washed with tap-water at laboratory temperature till the wash-water is clear. The washed wool is then dried at about 100° C., and weighed to constant weight. (3) *Yield of wool*.—From 2 to 3 grms. of the wool are washed

and then treated with a 5 per cent. solution of sodium carbonate solution at 50–55° C. for 1 hour with one change. The wool is then rinsed till the rinsings are clear, and dried as before. This treatment is followed by extraction with ether in a Soxhlet apparatus, though, as a rule, only traces remain for extraction. (4) *Residue on Ignition*.—This is carried out in a platinum crucible. (5) *Allwörthen reaction with chlorine water*. (6) *Curliness*. (7) *Fineness*.—This is measured by a micrometer at a magnification of 1000 diameters. The wool is embedded in anhydrous glycerin, or, if for permanence, in xylol. (8) Other physical qualities such as durability, tensile strength, elasticity and torsion. The apparatus used is that of Deforden, Dresden.

The making of a great number of micro-measurements becomes in time an impossibility, owing to the strain on the eyes, and a method is recommended whereby measurements of thickness of fibre can be carried out on optical projections of the fibres. Use is made of the trichinoscope employed by the inspectors of slaughter-houses in Munich. The source of illumination is a carbon arc-lamp with clockwork attachment. Immediately in front of the lamp are a lens, a diaphragm, and a screen. The wool is held between two glass plates placed in a glass cell with three divisions, so that samples from shoulder, flank and leg can be examined at the same time. In front of this is the objective which projects the light on to the screen, which is arranged in such a way that the observations can be made at the back of it, thus avoiding shadows being cast by the observer.

Several plates are given of various kinds of wool.

R. F. I.

## Inorganic Analysis.

**Determination of Metallic Lead in Metallurgical Products and Pigments.** D. H. McIntosh. (*Ind. Eng. Chem.*, 1926, 18, 1320–1321.)—The method depends on the insolubility of metallic lead in sodium hydroxide solution and on its solubility in silver nitrate solution. From 1 to 10 grms. of the sample are boiled for five minutes with 50 c.c. of 15 per cent. sodium hydroxide solution; the mixture is then filtered through asbestos, and the residue washed with hot water. Any lead carbonate, oxide, sulphate, silicate, and, to a certain extent, sulphide, which may be present dissolve in the alkali solution. The asbestos filter is transferred to a beaker and boiled for one minute with 10 c.c. of 10 per cent. silver nitrate solution. About 2 c.c. of water are added, and the mixture is again filtered through asbestos and the filter washed with water. The filtrate is evaporated with the addition of 10 c.c. of sulphuric acid, and the lead sulphate then collected on a filter and washed with 50 per cent. alcohol in the usual way. The lead sulphate is dissolved in 10 c.c. of hot saturated ammonium acetate solution, the solution is diluted to 250 c.c., boiled, and titrated with standardised ammonium molybdate solution, tannin solution being used as indicator.

W. P. S.

**Determination of Graphite and Combined Carbon in Pig Iron.** W. A. Burford and W. Bader. (*Z. anal. Chem.*, 1926, 69, 456–457.)—The two determinations can be carried out on one portion by the following procedure. The

sample (1 grm.) is dissolved in 20 c.c. of nitric acid (1 : 1) in a graduated 100 c.c. tube heated in a water bath. Heating is continued until the evolution of gas ceases. The liquid is cooled, and the iron precipitated with 25 c.c. of 30 per cent. sodium hydroxide solution; the volume is made up, and the cold liquid well mixed. After complete subsidence of the precipitate, 15 c.c. of the clear solution are pipetted into an Eggertz tube, and the colour compared with that of a standard treated in exactly the same manner. The balance of the assay is transferred to a beaker, and the precipitate is dissolved by warming with dilute nitric acid; the insoluble residue is collected for the determination of graphite after Ledebur.

W. R. S.

**Precipitation of Alumina by Ammonia.** L. Murawleff and O. Krassnowsky. (*Z. anal. Chem.*, 1926, 69, 389-394.)—Several points bearing on the determination of alumina were investigated, with the following results: Blum's procedure, *i.e.* addition of the ammonia with methyl red as indicator and other directions (*ANALYST*, 1916, 41, 286), is recommended. The precipitate should be washed with hot 2 per cent. ammonium nitrate solution neutralised with ammonia against methyl red, otherwise as much as 3 per cent. of the alumina may be lost in the washings. The addition of macerated filter pulp accelerates filtration and washing, and is in no way prejudicial. The precipitate, after addition of pulp, reaches constant weight after 20 minutes' ignition in platinum over a blast burner. The ignited precipitate is not noticeably hygroscopic.

W. R. S.

**Detection of Molybdenum by Thiocyanate.** F. C. Krauskopf and C. E. Swartz. (*J. Amer. Chem. Soc.*, 1926, 48, 3021-3027.)—The following procedure was worked out:—The solution to be tested is evaporated to dryness with nitric acid, and the cold residue extracted with ammonia. The extract is filtered, neutralised with hydrochloric acid, and treated with 5 c.c. of 10 per cent. potassium thiocyanate solution. If at this point a slight red colour due to iron appears, the solution should be extracted with ether till colourless. A small piece of zinc is placed in the liquid, and strong hydrochloric acid added, drop by drop, till effervescence begins; the red coloration of the reduced molybdenum compound develops on the surface of the zinc. With pure ammonium molybdate solutions the reaction is sensitive to about one part per million. The red compound is soluble in ether, and the determination of the ratio of molybdenum to sulphur in it proves it to be a thiocyanate of trivalent molybdenum. Mercuric chloride, phosphoric acid and organic compounds decrease the sensitiveness of the test.

W. R. S.

**Action of Tungsten Hexachloride on Phenyl Magnesium Iodide.** W. Brydowna. (*Bull. Soc. Chim.*, 1926, 39-40, 1771.)—Tungsten hexachloride reacts vigorously with phenyl magnesium iodide to produce a compound which when decomposed by dilute hydrochloric acid, extracted with ether, and fractionated, yields benzene and diphenyl. The reaction is general, a yield of about 50 per cent. of the di-aryl compound being obtained.

J. G.

**Electrometric Determination of Zinc by Ferrocyanide.** G. G. Reissaus. (*Z. anal. Chem.*, 1926, **69**, 450–455.)—For the description and drawing of the comparatively simple apparatus used and its manipulation, the original paper should be consulted. W. R. S.

**Separation of Radium and Mesothorium I from Barium by Ionic Migration.** J. Kendall, E. R. Jette, and W. West. (*J. Amer. Chem. Soc.*, 1926, **48**, 3114–3117.)—Ionic migration by electrolysis of a column of agar gel (*cf.* ANALYST, 1926, **51**, 647) was applied to barium bromide containing 21 mgrms. of mesothorium per 1000 grms. Considerable concentration of radio-active substance towards the front of the segment was readily produced, which shows that the ions of mesothorium I and its isotope, radium, have a greater mobility than the barium ion. The method is considered to possess practical interest in achieving a rapid concentration of radio-active material. W. R. S.

**Detection of Nitrite, Nitrate, and Sulphite.** E. Eegriwe. (*Z. anal. Chem.*, 1926, **69**, 382–385.)—In the following tests, colour reactions with organic dyes are used:—*Nitrite.*—The solution (1 c.c.), to which a drop of safranin *T* solution (0.03 gm. in 100 c.c.) has been added, is acidified with 2 *N* sulphuric acid. A blue coloration (monodiazosafranin) permits of the detection of 0.02 mgrm. of nitrite ion in 5 c.c. of solution. Excessive quantities of nitrate, chloride, and bromide yield crystalline precipitates. *Nitrate.*—The solution, concentrated by evaporation if necessary, is treated with 3 drops of the above dye solution, a little magnesium powder, and a drop or more of 2*N* sulphuric acid: the reduction produces the nitrite reaction, the froth assuming a violet coloration. Iodides and iodates interfere. *Sulphite.* The solution must be freed from sulphide and polysulphide by cadmium carbonate emulsion, and from hydroxyl ion by saturation with carbon dioxide. A solution of an oxazine dye—“*Echtblau R*” (0.01 gm. in 100 of water)—is added drop by drop, the liquid being stirred after each addition until the violet colour is discharged. A more or less pronounced yellow colour shows the presence of sulphite ion, 0.01 mgrm. in 1 c.c. reacting with 8 drops of the reagent. W. R. S.

**Determination of Soluble Fluorides.** F. L. Hahn. (*Z. anal. Chem.*, 1926, **69**, 385–386.)—The concentrated solution is made alkaline with sodium hydroxide (phenolphthalein), then decolorised with acetic acid; sodium sulphate is added until the solution contains 0.2 gm. of sulphate ion (or more if the fluoride content is high) per 100 c.c. The cold solution is precipitated with excess of calcium chloride solution; the mixed precipitate is easily collected and washed. It is dried, the paper incinerated separately in platinum, and the precipitate ignited gently and weighed. After being moistened with pure, strong sulphuric acid, it is heated, ignited as before, and again weighed: an increase of 0.0010 gm. = 0.000654 gm. F = 0.000689 gm. HF = 0.001344 gm. CaF<sub>2</sub>. W. R. S.

## Physical Methods, Apparatus, etc.

### Improved Meyer Apparatus for Vapour-density Determinations.

**A. Tian.** (*Bull. Soc. Chim.*, 1926, 39-40, 1171-1172.)—A U-tube of suitable dimensions is sealed on to the bottom of the bulb of the Meyer vapour density apparatus, and filled with mercury till the level just enters the bulb. After an experiment the vapour may then be removed completely in 2 minutes by aspiration from the top inlet of the apparatus without the chance that the water in the collecting vessel will run back. The mercury also breaks the fall of the ampoule containing the volatile liquid. J. G.

### Apparatus for the Dehydration of Volatile Liquids. P. Lorientte.

(*Bull. Soc. Chim.*, 1926, 39-40, 1767-1770.)—The liquid to be dehydrated is allowed to drip into an evaporation bulb immersed in a water-bath, and the vapour passed through three dehydrating tubes in series, to a condenser and receiver. The dehydrating tubes contain 250 grms. of the best quality of active lime arranged on 11 perforated shelves, and are maintained at a temperature above the condensation-point of the vapour. Each tube is renewed successively after 70 c.c. of alcohol or 1750 c.c. of ether, benzene, or acetone have been distilled, the renewed tube being replaced at the end of the series. In 8 hours, 4 to 5 litres of absolute alcohol (99.9 to 100 per cent.), or 10 litres of anhydrous ether, benzene, or acetone may be prepared. The distillates are neutral, and the acetone is free from mesityl oxide. J. G.

### Spectrophotometric Determination of Nitrites and Nitrates by Diphenylamine Sulphate. E. Tassilly and R. Savoie.

(*Bull. Soc. Chim.*, 1926, 39-40, 1755-1759.)—The blue colour produced with diphenylamine sulphate may be used for the spectrophotometric determination of nitrites to within 0.1 mgrm. The coloration, and consequently the absorption, are directly proportional to the concentration in the case of nitrites, but not in the case of nitrates. In the latter case it depends on the preliminary conversion of the nitrate into a nitrite, and can be prevented from forming by substances (*e.g.* urea) which destroy nitrites. The reacting substances should be well cooled when mixed, and the reagent renewed after about a month. J. G.

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## Reviews.

PRACTICAL ORGANIC AND BIO-CHEMISTRY. By R. H. A. PLIMMER, D.Sc. Pp. x.+568. London: Longmans, Green & Co. 1926. Price 21s. net.

This useful text-book is sufficiently valued in medical schools (for whose needs it is specially designed) for the new edition to require no lengthy comment. The author's aim is to teach organic and bio-chemistry as one subject by so modifying the treatment of the former that the student shall approach the latter without mentally separating the two subjects.

In the present edition the earlier chapters have been but little altered; in the latter part some sections present in former editions have been omitted to make room for additional matter. The inclusion of a short chapter on vitamins is welcome, as is also a practical section on respiratory exchange, though one regrets the disappearance of a useful chapter on the pigments of the leaf.

Glutathione makes its appearance for the first time in this edition; one would have preferred to see it treated among the oxidising systems rather than among the polypeptides, since its function is of so much more interest to the bio-chemist than its constitution; in any case, the very condensed description of its oxidation-reduction system hardly serves to put the student in possession of the salient facts, whilst the second equation on p. 370 is hard to justify from any knowledge at present to hand. The section on oxidations might have included some allusion to Wieland's well-known views, and a description of Thunberg's methylene blue technique would have been welcome.

The excellent sections on blood and urine analysis are valuable both to the student and the research worker; it is a little surprising, however—in view of the paramount importance of blood sugar determinations—to find the valuable method of Hagedorn and Jensen omitted, whilst the older method of Maclean still holds its place.

Speaking generally, this edition provides a valuable source of much necessary information, and, like its predecessors, will be found on the laboratory shelves of most bio-chemical workers.

MARJORY STEPHENSON.

PLANT PRODUCTS. By S. HOARE COLLINS, M.Sc., F.I.C., and GEORGE REDINGTON, M.Sc. Pp. xiii.+262. London: Baillière, Tindall & Cox. 1926. Price 10s. 6d. net.

This work is published as a Second Edition, though the First Edition appeared under the title of "Plant Products and Chemical Fertilisers." It is somewhat difficult to understand why the latter part of the original title has been dropped, except, perhaps, on account of a companion volume which now bears the title of "Chemical Fertilisers." Nevertheless, the book under review still contains 59 pages devoted to ammoniacal, phosphatic and potash manures, as was the case in the previous issue. Then soils, soil improvers, etc., occupy another 50 pages, so that 110 pages, out of the book's total of 249, deal with subjects one would hardly have anticipated would have been given so much prominence under the new title.

Detailed information concerning a considerable number of fertilisers is contained in the chapter on the nitrogen group of fertilisers, and some prominence is given to the various chemical reactions which occur on the application of many of these to the soil. Not only are the fertilisers described, but their uses are generally outlined, together with notes relative to their action when applied to varying soils. Information generally appears to be essentially non-controversial, and the views expressed are not too emphatic, indicating that the authors have had experience in the waywardness in efficacy of fertilisers applied under varying circumstances.

In dealing with basic slag, the author states that a citric solubility test has a distinct value in its proper place. It is to be noted, therefore, that he is not in agreement with many modern writers who have endeavoured to ridicule the test.

Probably the most interesting section of the book is that dealing with Photosynthesis, 40 pages having been devoted to this important subject. The work of Baly, Heilbron, Barker and Hudson is referred to, and some of their results commented on.

The chapters on the carbohydrates, oils and nitrogenous compounds produced in plants have been slightly extended in the new edition, but information on this important phase of the subject is somewhat limited. Occasionally the authors might well extend their remarks. Under "Sugar," for instance, it is stated that a solution obtained by soaking castor cake in salt water destroys many pests. Obviously, the phrase is intended to mean that this solution, or decoction, is used on the sugar cane, or on the soil, and that the pests to be dealt with are not found in the castor bean itself.

Analytical chemistry is not included in the volume, but many references to papers and works embodying methods of analysis are given.

The volume forms one of a series which is published to give a survey of the chemical industries, and it may be stated to be informative and lucidly written.

F. W. F. ARNAUD.

#### PERFUMES, COSMETICS, AND SOAPS, WITH ESPECIAL REFERENCE TO SYNTHETICS.

By W. A. POUCHER. Second Edition. Vol. II. Pp. xxxii.+406. With 60 illustrations. London: Chapman & Hall. 1926. Price 21s. net.

In this volume the portion of the first edition, dealing with the preparation of natural and synthetic perfumes, and the manufacture of all forms of modern cosmetics, has now been very considerably expanded by the inclusion of a fairly lengthy and interesting chapter on the part played by perfumes in history, and of new sections dealing with toilet soaps and shaving preparations, tobacco flavours, floral cachous, incense and fumigants, whilst the monographs on floral perfumes have been amplified, and extended from 76 to 106 pages, possibly in deference to the criticism of the reviewer of the first edition (*ANALYST*, 1924, 49, 500).

The perfuming of toilet soaps is well described, though the suggested addition of 10 per cent. of terpineol appears excessive, and would probably cause trouble in the plodder; but the treatment of the subject of soap manufacture in the first four pages of the chapter is so condensed and superficial as to be of little value, if not actually misleading, as, for example, in the statement that half-spent lyes separate with nigrés after about a week's standing. The manufacture of transparent soaps cannot be usefully dealt with in eight lines, and, for this class of soap, sugar, castor oil and glycerin are employed in the cold process, not "during the process for ordinary good quality millings." The advice to buy soap chips "on the dry side and add the necessary quantity of water during mixing and prior to milling" appears rather dangerous, as over-dried soap is liable to give a gritty finished tablet. Curd soap, which is usually understood to be an "unfitted"

soap, would not be at all a suitable material for the manufacture of shaving soap powder, and the addition of 20 per cent. of starch or orris root to such a powder seems very excessive, the addition of 2 per cent. being a common practice, to prevent the powder clogging in the sifter.

Under "Tobacco" the perfuming of cigars, cigarettes, leaf tobacco and snuff is well dealt with, and in the following section both the flavouring and colouring of lozenge-made and tablet-made cachous is described. The subject of depilatory pastes has become of increased importance since the recent restrictions on the sale of barium sulphide, and is here dealt with much more fully than in the previous edition, the use of sodium and calcium sulphides in various forms, and of thallium salts and various other depilatories being discussed.

Although the author's treatment of the subject is mostly non-chemical throughout the entire work, the very large number of formulae given for all the various products included should render it useful not only to those engaged in their manufacture, but also the analyst who has to undertake the analysis of such materials.

W. H. SIMMONS.

THE ANALYSIS OF PIGMENTS, PAINTS, AND VARNISHES. By J. J. FOX, O.B.E., F.I.C., and T. H. BOWLES, F.I.C. Pp. x.+179. London: Ernest Benn, Ltd. 1927. Price 16s. net.

Although the paint and varnish industry is one of very great importance in the field of chemical industry, the analysis of its products seems to have attracted comparatively very little attention from scientific writers. All who have been concerned with the analysis of pigments, paints, and varnishes must have been struck by the paucity of reliable published methods, and for this reason the appearance of a book on this subject by two authors so well versed in its practice must be very welcome.

In this volume the methods and processes described have all been actually used and tested by the authors, and it is this fact which gives the book its undoubted value. Too often methods of paint and varnish analysis have been published, the complexity of which far exceeds the reliability of the results obtained, so that after prolonged and difficult work the analyst obtains results which cannot be accepted without grave reservations. This applies particularly to varnish analysis. All such unsatisfactory processes are omitted from this book, and the practical analyst has the welcome feeling of having a foundation of verified processes to work on.

Although the authors rightly acknowledge the importance of physical tests, they wisely refuse to relegate more purely analytical methods to an altogether subsidiary place. Although physical tests may be as necessary as chemical analysis in the valuation of paints, and far more necessary still in the valuation of varnishes, yet the fact remains that the analyst is often called upon to report on the actual composition of a paint or varnish, and analysis must then be undertaken and such conclusions as are possible drawn from it.



The first six chapters deal with the analysis of pigments, and these will be found to include practically all the inorganic pigments likely to be met with. Complete schemes of analysis with full experimental details are given for all the chief pigments, whilst the rarer ones are treated sufficiently fully to enable the analyst to obtain the information required. The experimental details are, for the most part, clearly set forth, though occasionally there is a tendency for the authors to put the cart before the horse, so that the account of the process has to be read through a second time before it is fully understood. It is to be regretted that the pigment dyes and lakes are not included. Within its limits, however, this section of the book is about as good as it possibly could be. The seventh chapter describes the analysis of mixed paints and is excellent, though it might with advantage have been somewhat amplified. Chapter VIII is devoted to the examination of varnishes, and is divided almost equally between their physical and chemical examination, special attention being given to the determination of viscosity. Some readers may be disappointed at not finding more processes described for the separation and identification of the components, but in practice the fairly simple methods here described will usually be found to be the best and most reliable.

An appendix gives some results of specimen analyses in tabular form, and this is followed by a second one giving details of Goldsmith's method for the determination of tung oil in paints and varnishes, and by a third on distempers. The second will be found very useful, but the third one, dealing with distempers, might with advantage be expanded into a short chapter when a new edition is produced. The excellence of this book gives every indication that this will be required before long.

PERCY MAY.

THE ESSENTIAL OILS. By H. FINNEMORE, B.Sc., F.I.C. Pp. xv.+880. London: Ernest Benn, Ltd. 1926. Price £3 10s. net.

A number of authoritative works dealing with essential oils have been published during the past few years. The third volume of Gildemeister and Hoffmann, published in 1922, completed the three volumes of this great work, which, one may at once say, still remains the standard work on the subject. In 1921 and 1922 the two volumes of the 4th edition of *The Chemistry of Essential Oils and Artificial Perfumes* appeared. At the end of 1925 Volume IV of *Allen's Commercial Analysis*, dealing very fully with commercial essential oils, was published, and about the same time, 1925, the *Cyclopaedia of Perfumery* appeared in two volumes, dealing with all the essential oils used in perfumery.

The essential oil chemist must assess for himself the exact meaning to be applied to the statement of the publishers of the work under review, in the circular advertising it, that "This book is the first account of the essential oils published in England since 1918, which assembles and criticises the mass of new data now available."

The work is a bulky volume printed in large type on thick paper, but is actually about half the size, so far as matter is concerned, of Gildemeister and Hoffmann, and is also considerably smaller than *The Chemistry of Essential Oils and Artificial Perfumes*.

The work consists of a series of monographs on the individual essential oils dealt with. This is the course adopted in Volumes II and III of the former work and in Volume I of the latter work just mentioned.

There is no separate treatment of constituents, nor separate chapter on methods of analysis. All the information the author presents is to be found in the individual monographs. The work, so far as one can estimate, contains more detailed figures of the commercial aspect of the various oils than either of the other works on essential oils professes to do. There is a commendable absence of errors of omission and of printers' errors, but, for some reason or other, there are many papers on essential oils and their constituents to which no reference is made, even though they have been available for a considerable time before the publication of the work under review.

On page 141 the characters of Java vetivert oil given, are ascribed to Schimmel's *Bericht* of April, 1924. The author did not, apparently, actually take them from this source, as they are not quite correctly reproduced, and some of the figures (refractive index and acetyl value) are merely represented by blank spaces. A more recent set of figures for Java oil appeared in an English journal in October, 1925, but does not appear to be referred to. An omission difficult to understand is that in reference to a New Zealand *Leptospermum* oil, known there as "Manuka" oil. On page 548 the author makes a four or five line reference to it, based on a publication of 1902. In 1924, 1925, and April, 1926, three long papers on this oil appeared in the *Journal of the Society of Chemical Industry*, but are ignored here.

In a work of this kind one would have expected a much fuller account of the chemistry, as distinct from the analysis, of the essential oils. We have examined the indexed references to bisabolene and nerolidol, and can find no reference to the classic paper of Ruzicka and Capato on the complete synthesis of bisabolene and hexahydrocadaline from nerolidol, published in 1925, and available in English in October of that year.

Under the seven indexed references to farnesol, no reference is made to the paper by Verley on "The constitution of farnesol. Synthesis of a new aliphatic sesquiterpene alcohol, dihydrofarnesol," published in 1924 in the *Bulletin of the French Chemical Society*. The important paper on the new form of fenchone oxime and the characterisation of fenchone in the presence of camphor by Delépine, published in the *Comptes Rendus* in 1924 is not mentioned. Ruzicka and Liebl's paper (1925) on santene is also not mentioned.

These examples, both of essential oils and the chemistry of their constituents, could be considerably increased. The pure chemistry of these bodies is, in the reviewer's opinion, much more fully dealt with in already well-known works.

ERNEST J. PARRY.