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THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS

Honours

THE President and Council of the Society have congratulated the following members on the Honours conferred upon them on the occasion of His Majesty's Coronation:

Dr. ROBERT HOWSON PICKARD, F.R.S., *President of the Institute of Chemistry*—Knight Bachelor.

Lt.-Col. CLIVE NEWCOMB, M.D., F.I.C.—Companion of the Most Eminent Order of the Indian Empire (C.I.E.).

Mr. ANDREW MORE, F.I.C., *Deputy Government Chemist*—Companion of the Imperial Service Order (I.S.O.).

Sir Robert Pickard has also been congratulated on his appointment as Vice-Chancellor of the University of London.

NORTH OF ENGLAND SECTION

THE Eighth Summer Meeting was held at the Prince of Wales Hotel, Scarborough, from June 4th to 7th. There was an attendance of sixty, including many ladies.

The Chairman (Mr. Arnold R. Tankard) presided, and among those present were the following:—President (Dr. G. Roche Lynch); Past-Presidents (Dr. B. Dyer with Mrs. Dyer, Dr. J. T. Dunn with Mrs. Dunn, Mr. E. R. Bolton and Mr. J. Evans); Hon. Secretary (Mr. L. Eynon); Editor of THE ANALYST (Dr. C. A. Mitchell); Chairman of the Scottish Section (Dr. J. F. Tocher), Hon. Secretary (Mr. J. B. McKean); Mr. E. M. Hawkins, Dr. H. E. Cox and Miss Bradford.

The Chairman extended a cordial welcome to all present, especially those from the South and from Scotland. Dr. Dyer responded for the members from the South and Dr. Tocher for the Scottish members.

On Saturday morning Dr. J. J. Fox, O.B.E., F.I.C., Government Chemist, gave an address entitled, "The Functions and Usefulness of Analytical Chemists." Prof. T. P. Hilditch took part in the ensuing discussion. A vote of thanks to Dr. Fox

for his address, moved by Dr. Roche Lynch and seconded by Dr. Dunn, was carried unanimously.

A resolution was unanimously passed expressing the greetings and affirming the loyalty of the Section to the Parent Society; it was proposed by Prof. W. H. Roberts and seconded by Mr. J. G. Sherratt.

Telegrams of greeting were received from Mrs. Chaston Chapman, Miss Elliott and Dr. E. B. Hughes.

The President expressed the regret of the members at the absence of Mrs. Tankard through illness.

Dr. Dyer conveyed the thanks of the meeting to the Honorary Secretary (Mr. J. R. Stubbs) for arranging the meeting. The help of the following members in carrying out the arrangements is gratefully acknowledged:—Mr. W. G. Carey, Mr. W. F. Elvidge, Mr. T. W. Lovatt, Mr. C. R. Louden and Mr. F. J. Smith.

On Sunday afternoon the party proceeded by motor over the moors to Whitby, where afternoon tea was taken.

The following is an outline of Dr. Fox's address:

Analytical Chemistry

By J. J. Fox, O.B.E., D.Sc., F.I.C.

Dr. Fox reminded his audience that this country lagged behind America and the Continent in the absence of a chair of analytical chemistry. The reason for this was not clear, unless it were due to a want of appreciation of the scope and purpose of analytical chemistry, which, after all, was an important part of the foundation of practical chemistry. He pointed to the work published in the established journals of analytical chemistry and asserted that it was of the same quality as that arising from other branches of chemistry.

Analytical laboratories were continually engaged in investigations, as the published work indicated. The application and extension of the methods of physical and general chemistry to analytical purposes was illustrated by various examples in actual use in industrial and other laboratories. The speaker indicated the necessity for the analyst to keep in touch with advances in other branches of chemistry, and also pointed out that most practising analysts were, in their own spheres, specialists in some branch of chemical knowledge. It was well-known to most of the audience that the analyst was continually in the position of having to realise the necessity of examining the basis of his operations. He was rarely able to accept, without examination, the methods he found described. This meant a large diversion of his activities in the direction of general investigation, unless he was content to remain dormant in his methods.

Whenever a little spare time became available, it was an amusing pastime to select a paper from The Analyst or one of the other journals of a similar character, put in a mathematical expression or two, and see how far the paper, so revised, met the requirements of "pure" chemistry; it was likewise instructive.

Obituary

LAWRENCE JOHN DE WHALLEY

By the death of Lawrence John de Whalley, on January 9th, 1937, our Society loses one of its oldest members.

De Whalley was born in 1853 in Lancashire, and received his early education there. In 1874 he won a Royal Exhibition at the Royal School of Mines, London, and for the next three years was a student at the School, where he attended the lectures of Frankland, Guthrie and Huxley, and was trained in practical chemistry by Valentin. In 1877 he took his B.Sc. degree at the University of London, and in 1879 became honorary assistant to Frankland at the Royal School of Chemistry (into which the Royal School of Mines had by then developed). About the same time he was also appointed lecturer on chemistry at Whitgift School, Croydon, and for many years he continued to be associated with that school as examiner in chemistry.

In 1889 de Whalley turned from the teaching to the industrial side of chemistry, and became chief chemist at the tar works of Messrs. Forbes, Abbot and Lennard at East Greenwich. While there he devised what afterwards developed into the first continuous still. This historic piece of apparatus, the precursor of many oil stills of its type all over the world, is not only in existence but is still working.

In the following year de Whalley succeeded his brother-in-law, the late John Joseph Eastick, as chief chemist to Messrs. Abram Lyle & Sons, later incorporated with Messrs. Tate & Lyle, Ltd., sugar refiners, of Plaistow Wharf, Victoria Docks, London, and he remained with them for the rest of his professional life, until his retirement in 1930.

De Whalley was a great authority on sugar refining, and although most of his work was unpublished, he inaugurated and perfected many improvements in refinery processes and control. He was a pioneer in this country in the use of kieselguhr for sugar filtration, and was probably the first to associate microscopic structure of the diatoms with rate of filtration. He was responsible for the introduction of affination at Plaistow Wharf, and made many improvements in the manufacture of golden syrup.

He took part in international conferences on sugar analysis in this country and on the Continent, and was Chairman at the Conference of Sugar Chemists (the Raffinose Conference) in 1910, in Berlin, when he contributed some work on raffinose in beet sugars. During the Great War he was a representative at the Royal Commission on Sugar Supply. He attended the opening meeting of the recent International Commission for Uniform Methods of Sugar Analysis in August of last year.

He was elected a member of this Society in 1882, and regularly attended its meetings and dinners until a few years before his retirement.

He was an original member of the Society of Chemical Industry, and an abstractor for the sugar section of that Society's journal from 1896 to 1915. In 1911 he was elected a Fellow of the Institute of Chemistry, and he was for many years a Fellow of the Chemical Society. One of the founders of the Chemical

Club, he was a member of the first committee, and also took an interest in the founding of the Institution of Chemical Engineers.

De Whalley was a good mathematician and linguist and a lover of the classics, which he read in the original Latin and Greek. He spoke French, German, Russian and Polish, and was often engaged in the translation of technical works, even to within a week of his death.

In addition to his scientific work, de Whalley found time to interest himself in many other spheres, and may be remembered by some as Honorary Secretary for many years, and later President, of the Cheerybles Musical Society. Freemasonry occupied some of his time, and he held high rank in the Grand Lodge of England.

From early boyhood until his death he was a student. His country upbringing, with biological training under Huxley and geological excursions with Seeley, had made him an ardent nature-lover. His many interests, the clear way in which he expressed his thoughts, and his remarkable memory, made him a most entertaining companion.

A quiet, modest, lovable man of unassuming manner, he was liked and admired by all who knew him. He was at all times ready with kindly help and advice for those who sought it.

De Whalley married, in 1883, the only daughter of Z. Eastick, an early gasworks chemist, and leaves a widow and twelve sons and daughters, nine of whom are married, and nineteen grandchildren. He will be sadly missed by his many friends.

H. C. S. DE WHALLEY

Quantitative Microscopical Analysis of Feeding-stuffs

I. Determination of Rye, Wheat and Barley Starches in Mixtures. "Ground Oats" Mixtures.

BY J. G. A. GRIFFITHS, B.A., PH.D., A.I.C.

(*Read at the Meeting, April* 7, 1937)

THE determination of the nature and the proportions by weight of the ingredients of an animal feeding-stuff sometimes involves the identification and determination of the proportions by weight of each of several products from the grains of different cereals. Chemical analysis usually affords only very general guidance, and recourse must be had to microscopy.

It is essential, before deciding upon a quantitative microscopical method, to identify the ingredients of a mixture, and the first part of this paper, which has particular reference to the adulteration of ground oats, is therefore concerned with the microscopical identification of products derived from the grain of oats, rye, wheat and barley, together with other starches which may be found in feeding-stuffs, and the presence of which may sometimes lead to confusion. In the second

part of the paper (p. 513) the "lycopodium and starch grain" counting method has been extended, with the object of determining in mixtures the proportion by weight of each of two different ingredients of which the distinctive particles used in the counting process have closely similar characteristics, differing, perhaps, only in respect of the maximum size of the particles, *e.g.* the starches of wheat and barley. A detailed account is given of a means whereby it is possible to determine the respective proportions by weight of ground endosperm (flour) or comminuted whole grain of rye, wheat or barley in a feeding-stuff containing the starches of one or two of these cereals. Details are given, in Part II (p. 519), of a simple microscope projector technique of general application for the accurate counting and classification of particles according to size or other characteristics.

A. Factors controlling the Microscopical Appearance of Cereal Products

The following points concerning the microscopical examination of a mixture containing products derived from the grain of oats, rye, wheat and barley are relevant:

In the process of threshing, the husk is removed from wheat and rye so that the ground whole grain and products derived therefrom will contain material characteristic of the kernels only, that is, the bran and the contents of the grain. On the other hand, threshing does not remove the husk from oats and barley grain, so that the ground products of these cereals will contain material characteristic of the husk, bran and interior of the grain. Oat meal for human consumption is made by comminuting oats from which the husk has been removed, and such a product, which, if unfit for human consumption, may be used in animal feeding-stuffs, consists of bran particles and starch. The flours (ground endosperm) of wheat, rye, oats or barley, when unfit for human food or when very cheap, may be used in foods for animals. These flours consist of starch with only traces of bran tissue (including hairs). These traces, however, are of diagnostic value.

In principle, therefore, it is possible to determine whether a particular cereal ingredient of a mixture is a flour (ground endosperm) or a meal (comminuted whole grain) or a branny or husky by-product by determining the ratio of the weight of the characteristic bran fibre to that of the corresponding starch present.

Thus, if a sample of ground oats is found to contain lenticular starch of rye, wheat or barley, and if this starch is accompanied by only traces of tissue characteristic of the bran of these cereals, it is concluded that the cereal, which, incidentally, is identified by these traces, is present as flour. If, however, the cereal has been added in the form of a meal produced from the whole grain, the proportion of characteristic bran (and husk) tissue with respect to the corresponding starch will be correspondingly greater than with a flour. Similarly, an offal will show a greater proportion of bran or husk tissue, or both, than a meal made from the whole grain.

Some Diagnostic Features.—The microscopical appearance of cereal products is dealt with in detail in the standard works of Winton, Greenish, König, Moeller, and others. In general practice, the following features have been found to have the greatest diagnostic value:

OATS.

(1) *Husk*: (*a*) Occasional pieces with characteristic "*saw edge*" of closely packed short hairs.
(*b*) *Spongy parenchyma* of the flowering glume. Characteristic irregularly perforated appearance which differs from that of corresponding barley tissue.

(2) *Bran*: (*a*) *Geminate hairs* of epicarp.
(*b*) *Long hairs*. Tapering towards both ends (contrast with wheat).

(3) *Starch*: (*a*) Approximately *spheroidal aggregates*, about 40μ in diameter, distinguished from rye, wheat and barley starch grains by the faintly defined lines of demarcation between the angular grains and also by the evident differences in shape revealed on slowly rotating the aggregates under the microscope by gently moving (pressing on) the cover-slip with a needle.
(*b*) *Angular grains*. Less than 12μ in diameter. A few *spindle*-shaped and *dumb-bell*-shaped grains.

RYE.

(1) *Bran*: (*a*) *Cross cells of the pericarp*. Thick and characteristically "beaded" long walls and smooth unbeaded short walls (contrast with wheat).
(*b*) *Hairs*.

(2) *Starch*: Lenticular, generally 25–50μ in diameter. Some grains have *stellate hila*. Many small grains, a few *hat* or *bell-shaped* (very rare in wheat). (*See also* p. 514.)

WHEAT.

(1) *Bran*: (*a*) *Cell walls* are often distinctly brown.
(*b*) *Cross cells of pericarp*. Thick and "*beaded*" walls (*cf.* rye).
(*c*) *Hairs*.

(2) *Starch*: Lenticular, generally 25–40μ in diameter. Many small grains. (*See also* p. 514.)

BARLEY.

(1) *Husk*: *Spongy parenchyma* of flowering glume. Cells have irregular, approximately rectangular outline. (*Cf.* Oats.)

(2) *Bran*: (*a*) *Aleurone layer*; 2 to 4 cells deep, thereby differing from other cereals. Cells about half the size of those of wheat and rye.
(*b*) *Cross cells of pericarp*. Thin, unbeaded walls, double layer.
(*c*) *Hairs*.

(3) *Starch*: Lenticular, generally 20–30μ in diameter. Many small grains. (*See also* p. 514.)

OTHER STARCHES FOUND IN FEEDING-STUFFS.

(1) *Tapioca or Manihot starch* approximates to an irregular spherical form, generally truncated, less than 35μ in diameter, and may be confused with cereal lenticular starch. The latter, however, appears discoid when rotated under the microscope, and, further, each manihot starch grain shows, in contrast to rye, wheat and barley, a distinct cross when viewed by polarised light through crossed Nicols.

(2) *Ground-nut or pea-nut* starch is globular and less than 15μ in diameter.
(3) *Pea, bean, lentil.*
(4) *Sago, potato, arrowroot.*
(5) *Maize, dari* (sorghum), *rice, millet.*

In general, there will be found with these starches tissues of the parts of the plants from which the starches have been derived. For present purposes, further consideration of these starches has been omitted, but, when they are present, their characteristic features enable them to be identified, and analogous methods to those described later may be applied for determining their proportions by weight in the samples.

B. Quantitative Microscopical Analysis of Mixtures

Before undertaking a quantitative microscopical investigation of a sample it is essential to make a qualitative examination and to identify the ingredients.

Binary Mixtures.—The technique for determining microscopically the proportion by weight of one ingredient of a binary mixture has been described by Wallis,[1] and is outlined in the British Pharmaceutical Codex[2] (*see also* p. 519).

The following experiments with ground oats adulterated with wheat flour illustrate the application of the method to a feeding-stuff:—The sample (0·2000 g.) was mixed with 0·0400 g. of lycopodium, and 0·2000 g. of a standard mixture of ground oats and wheat flour (80 : 20 parts by weight) was mixed with 0·0400 g. of lycopodium. Appropriate counts (9 fields per slide) of the lycopodium spores and of the starch grains greater than 10μ in diameter were made, with the following results:

Sample mixture:

Slide I. $\dfrac{\text{Total wheat starch grains}}{\text{Total lycopodium spores}} = \dfrac{1263}{752} = 1{\cdot}68$

Slide II. Ditto. $\dfrac{482}{295} = 1{\cdot}63$

} Mean 1·655

Standard mixture:

Slide I. $\dfrac{\text{Total wheat starch grains}}{\text{Total lycopodium spores}} = \dfrac{1126}{578} = 1{\cdot}95$

Slide II. Ditto. $= \dfrac{673}{350} = 1{\cdot}92$

} Mean 1·935

$\therefore$ Wheaten flour in sample $= \dfrac{1{\cdot}655 \times 20}{1{\cdot}935} = 17$ per cent. by weight.

This method is also directly applicable to ground oats and other feeding-stuffs adulterated with rye flour or ground whole rye or with barley meal or, in general, with any single adulterant which has an ingredient of suitable particle size and can be recognised microscopically. The same method can be used to determine the composition of ternary, or other, mixtures only if each ingredient has particles with easily distinguishable characteristics.

TERNARY MIXTURES.—The method described above is not directly applicable to a ternary mixture containing rye and wheat or barley starches, for the following reason:—The characteristic (lenticular) starch grains of these three cereals approximate to the form of circular discs, and, although the average diameters of the starch grains are rye>wheat>barley, the following table shows that there is considerable overlapping, and therefore it is not possible to identify, by means of dimensions alone, an isolated starch grain in a mixture of these cereals unless the diameter exceeds the maximum for wheat.

	Diameter		
	Smallest	Average	Largest
	μ	μ	μ
Barley ..	2–7	20–30	40
Wheat ..	2–9	25–40	50
Rye ..	3–10	25–50	60

In the examination of these mixtures it is neither convenient nor necessary to deal with grains smaller than 10μ in diameter. A simple count, with respect to lycopodium, as described above, of the total number of starch grains greater than 10μ does not give a true measure of the percentage by weight of the total rye plus wheat or barley endosperm, or comminuted whole grain, in the sample, since the number of starch grains per unit weight of these products (generally) differs from cereal to cereal. The consequences of disregarding this point are illustrated later (p. 516). It is therefore necessary to determine the relative numbers of the starch grains of each cereal present.

GROUND OATS ADULTERATED WITH PRODUCTS FROM THE GRAIN OF RYE AND WHEAT OR BARLEY.—The ingredients of the mixture having been identified by the preliminary microscopical examination, it is necessary to decide which features of each particular ingredient are suitable for the quantitative work, it being assumed that such features have not been distorted or modified by heat or other treatment. In certain mixtures the starches are chosen as the basis of the determinations.

Rye starch differs from the starches of wheat and barley in two microscopical features of diagnostic value, *viz.* the hilum and the large size of a proportion of the grains.

(1) *The Hilum.*—Whereas the hilum of wheat and barley starch seldom appears larger than a small dot, centrally situated in the disc, the hilum of a small proportion of rye starch grains is stellate. In the samples of rye flour examined, the numerical proportion of starch grains, greater than 10μ in diameter, with stellate hila varied from 5 to 11 per cent. (average 7·5 per cent.) (see Table II).

(2) *Large Diameter of some Rye Starch Grains.*—Of the total number of starch grains of diameter greater than 10μ, in the samples of rye flour examined, 6·5 to 9 per cent. (average 7·5 per cent.) had a diameter greater than 40μ, whereas, in samples of wheat flour, less than about 1 in 300 of the total number of starch grains greater than 10μ in diameter exceeded 40μ in diameter. Barley starch grains do not exceed 40μ and seldom exceed 35μ in diameter (see Table III).

APPLICATION OF CRITERIA.—If, therefore, in a cereal meal containing lenticular starch grains there are found some starch grains with stellate hila, it is strong presumptive evidence that rye is present, and this is confirmed if there are lenticular grains of diameter greater than 50μ (*cf.* Winton[3]). Considering only lenticular grains of diameter greater than 10μ, if the numerical proportion with stellate hilums is less than 5 per cent. of the total number of starch grains, then wheat or barley or both may be present, and if the numerical proportion of grains of diameter greater than 40μ is less than 6·5 per cent. of the total number of grains, then wheat or barley starches, or both, are probably present with the rye. The observation that the numerical ratio of these grains of diagnostic value falls below the values usual for rye may be the first indication obtained that a small proportion of wheat or barley starch is present. Such an indication must be confirmed by a very thorough examination of the fibre, since a small proportion of wheat flour or barley meal will contribute only a very small proportion of fibre easily distinguishable from that of oats.

THE EXTENDED "LYCOPODIUM AND STARCH GRAIN" COUNTING METHOD.—The proportions by weight of endosperm or comminuted whole grain of rye and wheat (or barley) in a sample are determined as follows:—A mixture (of known composition by weight) of the sample and lycopodium is submitted to a counting process, by the technique described in Part II, in which the total number of lenticular grains of diameter greater than 10μ is determined. Lenticular grains with stellate hila, and lenticular grains with diameters greater than 40μ and 50μ are separately counted. With the appropriate factor derived from determinations with mixtures of known composition, the proportion by weight of rye flour (endosperm) or meal (comminuted whole grain) in the sample can be calculated either from the numerical proportion, with respect to lycopodium, of the grains with stellate hila, or, more accurately, from the similarly defined proportion of grains of diameter greater than 40μ.

Any surplus of "greater than 10μ, less than 40μ starch," beyond the requirement of the content of the rye grain product calculated from the data, is then used for calculating the proportion by weight of wheat (or barley) flour or comminuted whole grain, by means of the appropriate factors (see Tables II and III).

The following example of a sample of ground oats adulterated with the flours of rye and wheat illustrates the method. A mixture of 0·1000 g. of lycopodium and 0·8000 g. of the sample was made, and 9 fields per slide were counted (see Part II, p. 519, for technique).

TABLE I

No. of lycopodium spores (L) in 9 fields	No. of lenticular starch grains			No. of lenticular grains with stellate hila
	S>10μ	S>40μ	S>50μ	
225	537	11	2	14

Of the starch grains of diameter greater than 10μ (S>10μ), the low percentages of grains which are greater than 40μ in diameter (S>40μ), and also of those with stellate hila, show that lenticular starch other than that of rye is also present. A careful microscopical examination of the fibre showed that the other adulterant was wheat flour.

The calculations require the following basic data:

In mixtures of *equal* weights of lycopodium and rye flour it was found that each lycopodium spore corresponds to 0·73 to 0·92, average 0·80 [= factor $(R_f>10\mu)/L$], rye starch grain of diameter greater than 10μ. The analogous factor for wheat flour, $(W_f>10\mu)/L$, has values between 1·75 and 1·95 (average 1·87).

From the data in Table I, $(S>10\mu)/L=537/225=2{\cdot}387$ starch grains per lycopodium spore.

If all 537 starch grains are assumed to be rye, the weight percentage of rye flour in the sample is deduced by applying the factor $(R_f>10\mu)/L$, thus:

$$\frac{\text{Wt. of rye flour}}{\text{Wt. of lycopodium}} = \frac{(S>10\mu)/L}{(R_f>10\mu)/L} = \frac{2{\cdot}387}{0{\cdot}80} = 2{\cdot}98$$

i.e. in the lycopodium and sample mixture there are 2·98 parts by weight of rye flour for each part of lycopodium.

But 8 parts of the sample were mixed with 1 part of lycopodium, therefore 8 parts of the sample contain 2·98 parts by weight of rye flour = 37 per cent. by weight of rye flour.

If, however, it is assumed that all the lenticular starch counted is derived from wheat flour, there are $\frac{(S>10\mu)/L}{(W_f>10\mu)/L} = \frac{2{\cdot}387}{1{\cdot}87} = 1{\cdot}28$ parts by weight of wheat flour in 8 parts of the sample, *i.e.* 16 per cent. by weight of wheat flour.

The large difference between the percentages by weight of total adulteration calculated from the $(S>10\mu)/L$ data by assuming that the adulterant is either wholly rye flour or wholly wheat flour shows that the proportion of total adulteration cannot be stated until it is known which adulterant is present, or if both, their relative proportions (see p. 517). A method for calculating such proportions from data similar to those in Table I is shown in Tables II and III.

CALCULATION OF PROPORTIONS OF RYE AND WHEAT FLOURS IN THE SAMPLE.—(1) *By means of Starch Grains with Stellate Hila.*—The data are taken from Table I. The 537 starch grains, $S>10\mu$, consist of R rye starch grains plus W wheat starch grains, and R in Table II is calculated from the number of starch grains, 14 (Table I), with stellate hilum and the proportion of such grains in pure rye flour.* From the respective starch grain to lycopodium (L) ratios, R/L and W/L, are calculated $\frac{R/L}{0{\cdot}8}$ and $\frac{W/L}{1{\cdot}87}$, the parts by weight of rye flour and wheat flour, respectively, in 8 parts of sample.

(2) *By means of Starch Grains greater than* 40μ *in Diameter.*—Since among approximately 300 grains of wheat starch of diameter greater than 10μ there is one of diameter greater than 40μ, allowance must be made for this when computing the proportions by weight of rye and wheat grain products from starch-size data if a preliminary calculation, where no allowance is made, shows that the relative number of wheat starch grains warrants the modification of the number of large grains used in calculating the content of rye product.*

*An analogously determined correction is necessary in the case of wheat and barley starch mixtures if the governing diameter chosen is 35μ. The necessity for a correction does not arise with mixtures of rye starch and barley starch if the governing diameter chosen is 40μ, nor would it arise with mixtures of rye starch and wheat starch mixtures if the governing diameter chosen were 50μ, but the numerical proportion of rye starch grains of diameter greater than 50μ is too small to give the order of accuracy here attained unless a very large number of grains are counted.

TABLE II

	Rye flour				Residue as wheat flour				Total wt. of adulteration Per Cent.
	Total No. of rye starch grains $>10\mu$, (R)	R/L	R/L / 0·8	Wt. in sample Per Cent.	No. of wheat starch grains $>10\mu$, (W)	W/L	W/L / 1·87	Wt. in sample Per Cent.	
Min.	$\frac{14\times100}{11\cdot0^*}=127$	0·564	0·705	8·8	537−127=410	1·822	0·974	12·2	21
Av.	$\frac{14\times100}{7\cdot5^*}=187$	0·831	1·039	13·0	537−187=350	1·556	0·832	10·4	23
Max.	$\frac{14\times100}{5\cdot0^*}=280$	1·244	1·555	19·4	537−280=257	1·142	0·611	7·6	27

* Considering rye starch grains of diameter greater than 10μ, the minimum, average and maximum numerical proportions of grains with stellate hilum are 5, 7·5 and 11·0 per cent. respectively (see p. 514).

TABLE III

The symbols have the same significance as in Table II

	Rye flour				Residue as wheat flour				Total Wt. of adulteration Per Cent.
	Total No. of rye starch grains $>10\mu$, (R)	R/L	R/L / 0·8	Wt. in sample Per Cent.	No. of wheat starch grains $>10\mu$, (W)	W/L	W/L / 1·87	Wt. in sample Per Cent.	
Preliminary									
*Av.	$\frac{11\times100}{7\cdot5}=147$	0·653	0·816	10·2	537−147=390	1·73	0·927	11·6	22
Corrected for wheat starch $>40\mu$									
*Min.	$\frac{10\times100}{9}=111$	0·493	0·616	7·7	537−111=426	1·893	1·012	12·6	20
*Av.	$\frac{10\times100}{7\cdot5}=133$	0·591	0·739	9·2	537−133=404	1·796	0·960	12·0	21
*Max.	$\frac{10\times100}{6\cdot5}=154$	0·685	0·856	10·7	537−154=383	1·702	0·910	11·4	22

* Since the preliminary calculation and the results in Table II indicate 257–410 wheat starch grains, it is concluded that only 10 of the 11 grains of diameter greater than 40μ are due to rye. Of rye starch grains of diameter greater than 10μ, the minimum, average and maximum numerical proportions of those with diameter greater than 40μ is 6·5, 7·5 and 9 per cent., respectively (p. 514).

Bearing in mind the variations in the characteristics of natural products, the results in Tables II and III are consistent with one another, and show that the composition of the mixture is within a narrow range as compared with the extreme values which would be deduced by supposing that the whole of the starch was either that of wheat or rye. It is clear, however, that by choosing a factor for rye flour in the range 0·73 to 0·92 in place of 0·80 and a factor for wheat flour in the range 1·75 to 1·95 in place of 1·87, the range within which the composition falls is increased somewhat, and the calculated minimum proportion of total adulteration by weight would become approximately 19 per cent. in place of the values tabulated.

The results obtained, as described above, should be checked by repeating the counting process with another slide and then with a fresh lycopodium and sample suspension. In order to enhance the accuracy of the result, standard mixtures of approximately the composition deduced should be submitted to exactly the same mixing and counting processes as the sample, and the figures so obtained used to calculate the most probable composition of the sample.

The factors used in the calculations given above were derived from the examination of ranges of pure cereal products on the market about the time the analysis was made. It is to be expected that the characteristics of the cereals will depend upon the variety of the species, upon the locality where grown, and upon the climatic conditions existing during the growth and ripening of the grain. It follows that seasonal variations in the characteristics of grain are to be expected. It is, therefore, highly desirable that the analyst should obtain his own factors from an examination of genuine commercial grain or grain products on the market at the time of the analysis. It is not unlikely, therefore, that an extended examination of pure cereal products from all over the world will reveal a wider range of variation than has been found in the samples that I have examined, particularly in respect of the proportion of rye starch grains with stellate hila.

In view of the comparatively large variations found in the characteristics of the natural products, it has been considered unnecessary to introduce small corrections for the variations in the moisture-content of these air-dry materials. However, since the proportion by weight of moisture is about 11 per cent. (generally between 8 and 14 per cent.), it is desirable, for comparing results obtained at different times, to determine ratios on samples and mixtures at a definite moisture-content when mixed with lycopodium. When the moisture-content of a sample differs much from 11 per cent. the equivalent of the quantity used in the mixture can be readily calculated on an 11 per cent. moisture basis.

The directly determined chemical composition of the sample should be compared with that deduced from the microscopically determined composition, using accepted values of the chemical compositions of the ingredients. Any disaccord between the results, after allowance has been made for variations in the chemical composition of natural products, generally points to the presence of an ingredient which has been overlooked.

In discussing the extension of the "lycopodium and starch grain" count method to ternary and other mixtures, it may be noted that Wallis[4] determined the percentage composition of a binary mixture of wheat and barley flours by making use of the difference between the maximum sizes of wheat and barley starches. Bearing in mind the differences in principle and technique between the methods used by Wallis and that used here, it appears that the respective values recorded for the numerical proportions of wheat starch grains of diameter greater than 40μ in wheat starch and wheat flour (endosperm) are not inconsistent.

The method, illustrated above, is applicable to ground oats and other feeding-stuffs adulterated with rye flour and barley meal, or wheat flour and barley meal, and, in general, to the determination of the percentages by weight of each of two adulterants which have ingredients of suitable microscopical characteristics differing in as little as only one particular, that of maximum size of the particles.

II. A Microscope Projector Technique

To ensure as accurate as possible a count of a small proportion of large starch grains among many smaller grains, it is essential to have a considerable number of starch grains, say more than 50, not overcrowded, so that the grains will be flat, in each field counted, and an approximately equal number of lycopodium spores to act as a basis of reference. Nine fields are counted per slide, arranged as in Fig. 1, or if greater accuracy is required, 25 fields, arranged as described by Wallis.[5]

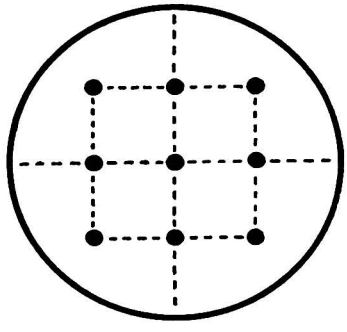

Fig. 1

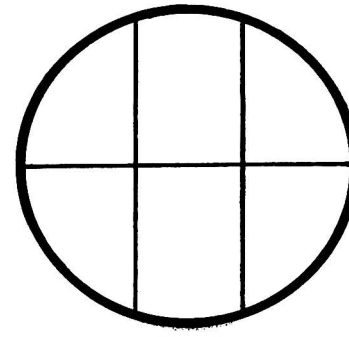

Fig. 2

It is a matter of some difficulty to divide into the appropriate ranges of size and to count all the starch grains, together with the admixed lycopodium spores, in a sufficiently densely covered microscope field when viewed through the eyepiece of a microscope. For this reason, a method has been devised whereby the image of the field is projected upon a vertical white screen. The microscope is tilted so that the axis of the optical system is horizontal, and a 30 candle-power Point-o-lite lamp and lens replace the mirror and lamp as source of illumination. The ordinary eyepiece is replaced by a projector lens, and the microscope is situated at such a distance from the screen that, with an 8-mm. objective, starch grains 50μ in diameter have an apparent diameter of 15 mm. This is checked by projecting on to the screen with this system a scale graduated in 0·01 mm. For convenience of counting, the field is divided into sections by means of fine wires stretched across an aperture placed between the projector and the screen (Fig. 2).

For distinguishing between grains falling within the different ranges of size, discs or loops of appropriate sizes are attached to the ends of rods in such a way that the discs can be brought easily into contact with the images on the screen. Thus, a solid disc of diameter 3 mm. is used to show whether starch grains are of diameter less than 10μ, whilst circular loops, 12 mm. and 15 mm. in diameter, of fine blackened wire, serve to show whether starch grains exceed 40 and 50μ in diameter, respectively. A transparent scale may also be used.

It is essential that adequate definition should be secured, for the following reasons:

(1) Complete aggregates of oat starch with circular outline may be mistaken for lenticular starch grains, with or without stellate hila, if the definition is poor. With good definition, the boundaries of the grains in the aggregates are clearly defined, and there is no confusion with lenticular grains having stellate hila.

(2) In commercial grinding and milling processes, lenticular grains very occasionally become flattened and their diameter considerably increased without rupture of the grain. Such objects may simulate large rye starch grains, and, if included in the total of large starch grains counted, lead to error. With good definition, such grains are easily recognised, as they have a very faint outline and an unsubstantial appearance, compared with the strong outline and robust appearance of the normal grains. These very occasional "ghosts" are neglected.

MOUNTING THE SAMPLE.—With the apparatus set up as described, the plane of the slide supporting the specimen is vertical, so that, unless the starch and lycopodium mixture is suspended in a highly viscous medium, the grains drift downwards, thus making accurate counting, etc., impossible. The difficulty is overcome in the following manner: The dry cereal and lycopodium mixture is mixed *thoroughly*, by rubbing gently with a flat-ended glass rod in a small beaker or mortar to break up aggregates of starch grains without smashing the grains. A sufficient quantity of water is then *thoroughly* incorporated, by gentle rubbing, to make a suspension of a soupy consistency. A small drop of this material is deposited on a slide, any small fragments of fibre are carefully removed by means of forceps, and a small drop of a commercial liquid glue is completely incorporated by means of a needle with the material on the slide. The quantity of material on the slide should be just sufficient to form a very thin permanent film covering the whole of the cover-slip when gently pressed to the slide. If the film is too thick, the suspended matter drifts across the field of view when the slide is supported in the projector. Under correct conditions the lenticular starch grains are oriented with their planes parallel to the plane of the glass slide.

There are at the present time several microscope projectors on the market, and one of these can be used with advantage in place of the microscope arrangement described above. In general, the stage of the projector is in a vertical plane when the projected image is received on a vertical screen, and hence the technique described above is applicable to these circumstances. If, however, the stage is in a horizontal plane, a less viscous mounting medium, such as described by Wallis (*loc. cit*), may be used.

SUMMARY

(1) A description is given of an extension of the lycopodium and particle count method to the quantitative microscopical analysis of certain ternary mixtures for which the simple method is inadequate.

(2) The microscopical determination of the proportion by weight of rye flour (or ground rye grain) in mixtures, including adulterated ground oats, containing starch-bearing products derived from wheat or barley grain, is described.

(3) Of rye starch grains of diameter greater than 10μ in diameter, there were found in rye flour

(*a*) Between 5 and 11 per cent. with stellate hila.

(*b*) Between 6·5 and 9 per cent. with a diameter greater than 40μ, whilst only a very small proportion of wheat starch grains of diameter greater than 10μ (about 1 in 300) exceeded 40μ in diameter.

(4) A microscope projector technique has been developed whereby the counting and classification of particles according to size, or other characteristic, are easily and accurately carried out.

I wish to thank Dr. J. J. Fox, the Government Chemist, for kind permission to publish this paper, and Mr. A. More for his helpful interest.

References

1. T. E. Wallis, Analyst, 1916, **41**, 357; *Analytical Microscopy*, p. 126 *et seq.*
2. *British Pharmaceutical Codex*, 1934, p. 1592.
3. A. L. and K. B. Winton, *Structure and Composition of Foods* (Chapman and Hall), Vol. I, p. 259.
4. T. E. Wallis, *Pharm. J.*, 1922, [IV], **55**, 82.
5. —— Analyst, 1935, **60**, 520.

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Discussion

Mr. Wallis congratulated Dr. Griffiths on this work. He must have had a strenuous time in doing these very numerous counts, for in producing figures that could be used for the purposes of a standard test it was necessary to take special care. He was also glad to see a new worker in this particular field; he, personally, owed a debt of gratitude to Dr. Griffiths for entering it. He thought that Dr. Griffiths had been wise to study these different types of starch rather than to attempt to deal with the number of granules in starch, as had been attempted in Germany. He was also wise in choosing a minimum of 10μ in making the counts. He (Mr. Wallis) liked the projection method very much. He did not know whether, in doing the work, Dr. Griffiths had been able to arrive at any figure for the variation in the numbers occurring in any given material; such a figure would be quite interesting. He, personally, had found in materials such as linseed and senna that the natural variation in the material, due to different habitats and conditions under which they were grown, was somewhere about 15 per cent. It would be very interesting to know whether cereals showed similar variations.

Mr. L. Eynon thought that Dr. Griffiths had partly solved the difficult problem of distinguishing quantitatively between rye, wheat, barley and oats. He asked whether the results obtained by this method had been compared with those given by colorimetric methods, more particularly those for rye and wheat starches.

Dr. Dyer said that the so-called "stellate hilum" of rye starch granules had sometimes puzzled him. He had noticed that often only quite a small minority of rye granules bore this characteristic appearance. He had doubt as to whether this appearance was properly described as a "stellate hilum" or whether it was merely the result of the bursting of the starch granules through over-ripeness.

Dr. D. W. Kent-Jones did not agree with the figure of 11 per cent. as the average moisture-content in materials of this sort; that was not his experience, and he thought that it ought to be 2 or 3 per cent. higher. Also, he did not approve of the suggestion of getting information from the percentage of gluten that could be washed out from such materials. The presence of other substances would alter the amount of gluten that could be washed out, so that a false conclusion might be indicated. He suggested that the test, however excellent, was a complicated one and, in view of the fact that high fees were not paid for such work, he would like to know how long such an analysis took.

Dr. Griffiths, replying, remarked that in the range of factors that he had given there was an indication of the range of natural variation. There was, of course, an experimental error which came into these determinations, but his figures showed that in rye flour, of the starch grains greater than 10μ in diameter

between 6·5 and 9 per cent. exceeded 40μ in diameter and 5 to 11 per cent. had stellate hila. The variation indicated by the first range was close to that referred to by Mr. Wallis, that of the second range was somewhat larger.

He, personally, had had no experience of the colorimetric methods mentioned by Mr. Eynon, but he understood from others who had, that they were not very reliable.

Dr. Dyer had raised a very interesting point. It seemed to be quite a possibility, if there were a central hilum in the grain, that during ripening the material had contracted, leaving these fissures. It was partly for this reason that he considered that the counting of starch grains with stellate hila afforded less reliable data than the counting of starch grains greater than 40μ in diameter. With regard to the method of analysis of rye middlings and wheat middlings, he did not know the method adopted by Dr. Dyer, but, naturally, the starch count method would give only the proportion of rye flour and wheat flour present, and then the proportion of each fibre present must be determined by some other process. He had done some work in connection with the determination of wheat fibre by a counting process, using the very characteristic "beaded" tissue of wheat. There was another alternative—if the proportion of wheat endosperm in the mixture were high ($>$ 50 per cent.) one could obtain a certain amount of gluten. Wheat flour was the only flour that gave gluten. The uncertainty of the factor to be used for converting gluten to wheat flour made the result an order of magnitude rather than a definite quantity.

In reply to Dr. Kent-Jones, Dr. Griffiths said that the moisture figure given was the average he had found for samples examined in the laboratory. It was quite easy to convert the factors to any other moisture-content which one might find. The gluten test was suggested only as being a very rough guide. It was agreed that the amount of gluten that could be washed out depended upon the other materials present. Some mixtures containing as much as 30 to 50 per cent. of wheat flour gave completely unsatisfactory results. Regarding the question of cost, he admitted that this was a tedious method. It was adopted to obtain a trustworthy figure when a figure had to be given and when no other method, shorter and quicker, was available.

The Determination of Boric Acid in Foodstuffs

By ROBERT S. ALCOCK, M.A., Ph.D.

The volumetric method of Thomson[1] for the determination of boric acid in foodstuffs has been shown to be liable to considerable errors when applied to samples of food containing large proportions of phosphates or of fat and small proportions (such as those found naturally in certain fruits) of boric acid. The errors were traced to co-precipitation of calcium borate during removal of phosphates, incomplete removal of phosphate, or volatilisation of boric acid with glycerol derived from fat during the ashing of the food in an alkaline condition. Monier-Williams[2] modified the process by removal, first, of calcium salts and, secondly, of phosphoric acid as magnesium phosphate, so as to avoid the first two causes of error. These causes of error were reduced to a minimum by another modification of the process,[3] based on work in this laboratory, in which the exact conditions of alkalinity required for precipitation of calcium phosphate and retention in solution of calcium

borate were specified; the removal of fat before ashing the sample was a part of this modification, of which other later investigators have made slight changes.

As the conditions for removal of phosphate require great care and the results obtained by titration of the boric acid may be vitiated by accidental presence of phosphoric acid or carbonic acid, it is desirable to have a confirmation of the results by some test specific for boric acid. At one time all results obtained in this laboratory[4] were confirmed by a method involving ashing of the sample with excess of alkali, solution of the ash in a known excess of hydrochloric acid, addition of alcohol and turmeric solution in definite proportions, and comparison with known amounts of boric acid treated in the same way. This method was generally satisfactory, but failed when applied to meat products or to articles containing appreciable proportions of iron.

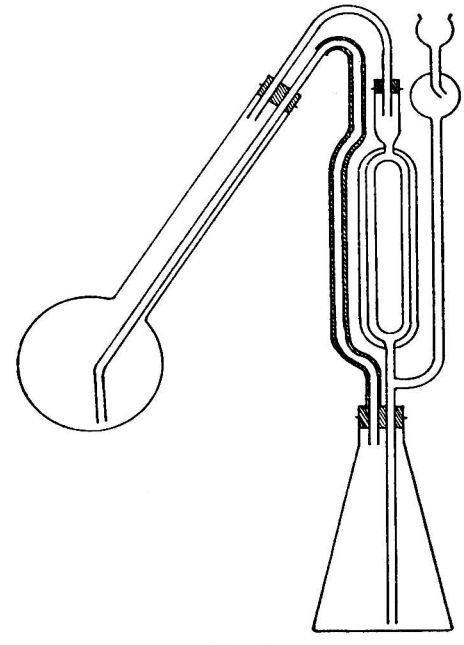

Fig. 1

The present method was developed for the determination of boric acid in phosphate-rich ashes and makes use of the ready esterification of boric acid with methyl alcohol, distillation of the methyl borate and final titration of the boric acid as in the Thomson process.

APPARATUS.—The distillation is carried out in the apparatus here illustrated (Fig. 1). The acidified ash is boiled with methyl alcohol in a 300-ml. Kjeldahl flask, and the vapours are carried over into a double-surface condenser, the condenser being in a vertical position and the Kjeldahl flask inclined at 30° to it. The exit tube is elongated and dips below the surface of alkaline methyl alcohol in a 400-ml. conical flask. This flask stands on a tripod and gauze, and is strongly heated by a Bunsen burner. It is closed with a rubber stopper which carries, besides the condenser exit, another wide glass tube passing through a second hole in the bung of the Kjeldahl flask down into the methyl alcohol. The ascending limb of this tube must be lagged. This is conveniently done by enclosing it in a rubber tube.

Immediately above the stopper of the conical flask is a side-tube sealed on to the condenser outlet. This runs up close to the condenser and carries a splash-bulb and funnel. It serves to maintain atmospheric pressure inside the apparatus, to prevent possible losses through bumping in the receiver flask, and to make possible the washing out of the lower part of the condenser outlet.

PROCEDURE.—From 40 to 50 g. (or less when large amounts of boric acid are anticipated) of the sample are moistened with 10 ml. of 2 *N* sodium hydroxide solution. If much fat is present, it should be removed by solution in ether, as previously described.[3] The water is evaporated on a steam-bath, and the sample is ashed; there is no need to burn away all the carbon. The ash is transferred

from the dish into the Kjeldahl flask, as little water as possible being used. The dish is finally washed with a few ml. of dilute sulphuric acid, and the ash in the flask is dissolved in a further quantity of the acid by warming. This removes most of the carbon dioxide and dissolves the lumps of ash. Methyl red is added, and 30 per cent. sodium hydroxide solution is run in until the colour changes to yellow. The liquid is concentrated to one or two ml. over a Bunsen flame, with continuous agitation. After cooling, 60 ml. of methyl alcohol and 1 ml. of methyl red solution are added.

Conc. sulphuric acid is then dropped in until, after shaking, the solution is strongly acid to the indicator. The flask is then attached to the apparatus, as is also the 400-ml. conical flask containing 0·5 ml. of *N* sodium hydroxide solution and a few drops of phenolphthalein solution. The Kjeldahl flask is heated and, when sufficient alcohol has collected in the conical flask, that too is strongly heated, so that the vapour bubbles vigorously through the acid liquid in the Kjeldahl flask. The flame beneath that flask is then adjusted so that 15 to 20 ml. of alcohol remain. If during the distillation the colour of the phenolphthalein in the conical flask is discharged, a further 0·5 ml. of sodium hydroxide solution is added.

After half-an-hour the flame beneath the Kjeldahl flask is replaced by a beaker of cold water, and as much of the methyl alcohol as possible is distilled up into it, and may be recovered later. The conical flask is removed, and the condenser tube, both inside and out, is washed into it with water. The remaining methyl alcohol is boiled off, and the solution is then made just acid to methyl red with *N*/10 sulphuric acid and boiled for a further five minutes to remove carbon dioxide. After cooling, the acidity is re-adjusted with *N*/20 sodium hydroxide solution until it is just not acid to methyl red. More phenolphthalein is added, and, after addition of 1 g. of mannitol, the titration is carried to the phenolphthalein change. A blank determination is made with water in place of a distillate. This result is usually of the order of 0·1 ml. of *N*/20 sodium hydroxide solution (1 ml. of sodium hydroxide solution ≡ 0·0031 g. boric acid).

Conditions of Distillation.—The conditions governing the distillation are illustrated in the following tables:

TABLE I

30 mg. of boric acid; 60 ml. of methyl alcohol; distilled 30 minutes

Acid	Methyl alcohol strength Per Cent.	Boric acid recovered Per Cent.
H_2SO_4	90	99·5
H_2SO_4	95	99·5
H_2SO_4	100	99·5
H_3PO_4	90	82·0
H_3PO_4	100	96·0

Table I shows that, given a sufficient strength of acid (excess of sulphuric over phosphate in the ash) amounts up to 10 per cent. of water in the alcohol do not influence the rate of distillation. When phosphoric acid is used the rate is affected.

TABLE II

1 g. of "artificial ash" plus boric acid; 60 ml. of methyl alcohol; excess of sulphuric acid; distillation 30 minutes

Boric acid mg.	Titration (less blank) $N/20$ NaOH ml.	Apparent boric acid mg.	Percentage error
0	0·05	0·15	—
5	1·65	5·12	2·4
10	3·28	10·15	1·5
30	9·77	30·18	0·6

Table II indicates the error likely to arise from phosphoric acid being carried over in the vapour. An artificial ash was made of equal weights of calcium chloride, kieselguhr, sodium sulphate and disodium phosphate. One g. of this mixture was placed in the distillation flask for each experiment. The phosphate coming over corresponds to some 0·15 mg. of boric acid (3 p.p.m. on 50 g. of sample). In practice, the figures for actual ashes seem to be less than this. Such a degree of accuracy is seldom required, but when very small amounts of boric acid are involved—of the order of 1 mg.—the distillation should be followed by the usual precipitation, after adding a drop or two of 10 per cent. calcium chloride solution. The phosphate comes over as a result of esterification, not through splashing, as more appears when a small amount of phosphate, acidified with sulphuric acid, is in the distillation flask, than when syrupy phosphoric acid itself (2 ml.) is used for acidification. Sulphuric acid is used, as the lower pH accelerates the distillation.

TABLE III

Boric acid; 60 ml. of methyl alcohol; sulphuric acid

Boric acid mg.	Time Minutes	Boric acid recovered mg.	Recovery Per Cent.
10	5	8·1	81
10	15	10·0	100
20	15	19·8	99
20	15	20·0	100
30	15	25·7	86
30	20	28·8	92
30	20	29·5	98
30	30	29·8	99·3
30	30	30·0	100

Table III summarises the experiments on the time required for distillation. In every instance the liquid in the receiver flask was boiled as vigorously as possible. It is seen that up to 20 mg. come over quantitatively in 15 minutes, while 30 mg. require longer. Half-an-hour's distillation is a usual routine time, as amounts of more than 30 mg. are seldom met with. If, however, on titration, more than 10 ml. of $N/20$ sodium hydroxide solution are required, the distillation can be carried on for a further half-hour period, when the remainder, if any, will be recovered.

TABLE IV

Sample	Weight g.	Boric acid added mg.	Titration (less blank) $N/20$ NaOH ml.	Boric acid found mg.	Recovery Per Cent.
Currants A	40	0	1·55	4·8	—
,, A	40	20	8·00	24·8	100
Flour B	50	0	0·3	0·9	—
,, B	50	20	6·7	20·8	99·5
,, B	50	40	13·3	41·2	100·7

Table IV gives two examples of the many determinations that have been made on various materials. It will be seen that the boric acid added to the sample before ashing was quantitatively accounted for in the final titration.

SUMMARY.—A method for the determination of boric acid in foodstuffs, using the distillation of methyl borate followed by a mannitol titration, is described.

A continuous steam-distillation apparatus, used in the method, is illustrated.

My thanks are due to Dr. J. J. Fox, the Government Chemist, for permission to publish this work.

REFERENCES

1. R. T. Thomson, ANALYST, 1896, **21**, 64.
2. G. W. Monier-Williams, ANALYST, 1923, **48**, 413.
3. ANALYST, 1923, **48**, 416.
4. A. Scott-Dodd, ANALYST, 1929, **54**, 15.

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The Determination of Tartaric Acid as Lead Tartrate

BY C. H. MANLEY, M.A., F.I.C.

(*Read at the Meeting of the North of England Section, December* 14, 1935)

IN 1934, when undertaking the full analysis of a baking powder containing a mixture of rice, sodium bicarbonate, tartaric acid and acid potassium tartrate (concerning the percentages of which there had been some divergence of opinion), I sought for a new method for the determination of the total tartrate present, as an alternative to the usual one involving separation as acid potassium tartrate and subsequent titration with $N/10$ alkali.

The intention was, if possible, to weigh the tartaric acid in the form of one of its metallic salts. Of these, the lead salt, $PbC_4H_4O_6$, is one of the most sparingly soluble, its solubility at 20° C., according to Cantoni and Zachoder,[1] being nil. Like calcium tartrate, however, it is soluble both in acids and alkalis, but it was found possible to effect quantitative precipitation at room temperature from a solution previously rendered neutral to phenolphthalein.

In this method the use of the comparatively large amount of alcohol required in the precipitation and washing of acid potassium tartrate prior to its titration with standard sodium hydroxide[2] is dispensed with.

Lead acetate proved unsuitable as a precipitant, the results obtained with a neutralised solution of tartaric acid being in excess of those obtained by titration with standard alkali. Satisfactory results, however, were obtained with a sufficient excess of lead nitrate solution, after a two hours' precipitation period, in a solution neutralised to phenolphthalein.

At first no special attention was paid to the quantity of water used for washing the lead tartrate free from soluble salts, the object being to continue with a cold water wash until the filtrate showed no cloudiness with potassium chromate. It was subsequently found, however, that for the successful application of the method, a limited quantity of water must be used, as, notwithstanding the statement of Cantoni and Zachoder[1] to the contrary, lead tartrate was discovered to possess a slight solubility at 18°–20° C. amounting to 2 mg. per 100 ml. Washing with a cold saturated aqueous solution of dried lead tartrate effected no improvement.

The composition of the lead tartrate produced by the prescribed method was checked by conversion into lead sulphate (Pb in $PbC_4H_4O_6$: theory, 58·4 per cent.; found, 58·1 per cent.).

The following procedure was adopted, first, in testing the proposed method by employing it to assay samples of tartaric acid and acid potassium tartrate of known purity ; and later, in applying it to the determination of the total tartaric acid in baking powders :

(*a*) TARTARIC ACID.—Two g. were dissolved in water, and the solution was made up to 200 ml. Several quantities of 20 ml. (= 0·2 g.) were made neutral

TABLE I

TARTARIC ACID

Purity = 99·75 per cent. by titration of 0·5 g. with 0·2 *N* NaOH (mean of 99·6 and 99·9 per cent.).

Twenty ml. of a 1 per cent. solution (= 0·2 g.) used for each experiment.

Expt.	Precipitant	Time	Weight of lead tartrate g.	Tartaric acid, per cent. By titration	Tartaric acid, per cent. By lead tartrate method
42*b*	15 ml. of 5 per cent. $Pb(NO_3)_2$	½ hour	0·465	Average 100·3	98·3
43	,, ,,	1 ,,	0·466		98·3
40	,, ,,	2 hours	0·471		99·6
44	,, ,,	3 ,,	0·468		99·0
46	,, ,,	16 ,,	0·471		99·6
42*a*	,, ,, $Pb(C_2H_3O_2)_2$	3 ,,	0·480		101·5
39	,, ,, $Pb(NO_3)_2$	2 ,,	0·476		100·7*

* Precipitate washed with 50 ml. of saturated lead tartrate solution.

to phenolphthalein by titration with 0·2 *N* sodium hydroxide solution in 100-ml. beakers, and in each case 15 ml. of 5 per cent. lead nitrate solution were added, and, after stirring, the white flocculent precipitates were left to stand for varying

times. After this the precipitate was transferred to a Gooch crucible and washed with 50 ml. of water at 18°–20° C., the filtrate being tested with potassium chromate after 30 ml., 40 ml., and finally 50 ml. had passed through. By this time a constant opalescence was registered. By using the blue ring test for nitrates in a separate experiment with diphenyl-benzidine sulphate as reagent,[3] it was concluded that the excess of lead nitrate was removed after the passage of 35 ml. of water. The lead tartrate was dried to constant weight at 105° C. The factor for tartaric acid/lead tartrate is 0·423. The results obtained are given in Table I, together with the result obtained by substituting lead acetate for lead nitrate as precipitant. Ten ml. of 5 per cent. $Pb(NO_3)_2$ solution were found insufficient; 15 ml. were then used and found sufficient.

(*b*) ACID POTASSIUM TARTRATE.—Three g. were treated with hot water and phenolphthalein and neutralised by titration with 0·2 *N* sodium hydroxide solution. The solution thus obtained was cooled to room temperature and made up to 200 ml. Twenty ml. of this solution were then treated with 15 ml. of 5 per cent. lead nitrate solution, after which the procedure was identical with that followed in (*a*).

The results obtained are given in Table II, and, like those in Table I, they show that 2 hours are sufficient to ensure complete precipitation.

TABLE II

CREAM OF TARTAR

Purity = 100·0 per cent. by titration of 1 g. with 0·2 *N* NaOH (twice).
Twenty ml. of a 1·5 per cent. solution (= 0·3 g.) used for each experiment.

Expt.	Precipitant	Time	Weight of lead tartrate g.	Acid potassium tartrate per cent.	
				By titration of 0·3 g.	By lead tartrate method
49	15 ml. of 5 per cent. $Pb(NO_3)_2$	2 hours	0·566	99·3	100·0
47	,, ,,	16 ,,	0·563		99·5
50	,, ,,	2 ,,	0·564*		99·7*

* Precipitate washed with 50 ml. of saturated lead tartrate solution.

BAKING POWDER. — Following several preliminary experiments, the standardised lead tartrate method, with slight modification, was used for the determination of the total tartrate present in baking powders containing tartaric acid or acid potassium tartrate, or both, as the acid principle.

The presence of tartrate was first ascertained by treating about 1 g. of the baking powder with water, filtering, adding ammonia and a crystal of silver nitrate, and heating in the water-bath.[4] Other portions of the filtrate were tested for *phosphate, sulphate and chloride, any of which, if present, would interfere with the accuracy of the determination.*

If the sodium bicarbonate were exactly balanced in the baking powder by its equivalent of acid component, the lead tartrate could be precipitated directly from the aqueous solution freed from the rice or other starchy filler. As this

is rarely so, however, the sodium bicarbonate being usually in slight excess, the following procedure was necessary:

One g. of baking powder was treated with a few ml. of cold water, and the starchy filler was filtered off on a weighed Gooch crucible, the filtrate being then made up to 50 ml. in a graduated flask. Twenty-five ml. of the filtrate were rendered just acid to methyl orange with 0·2 *N* nitric acid, the solution was boiled to remove carbonic acid and cooled, and 0·2 *N* sodium hydroxide solution was added to neutrality to phenolphthalein. Ten ml. of 5 per cent. lead nitrate solution were added, after which the normal procedure was followed.

For comparison, the tartrate was determined in the other 25 ml. of the tartrate solution by the method hitherto usually recommended, *viz.* precipitation as acid potassium tartrate and subsequent titration with 0·1 *N* sodium hydroxide solution.

The accuracy of this latter method was checked by employing it to assay the tartaric acid and acid potassium tartrate used in (*a*) and (*b*); these when titrated with 0·2 *N* sodium hydroxide solution had each shown approximately 100·0 per cent. With 0·2 g. of each compound the results were 99·0 per cent. for the tartaric acid and 98·7 per cent. for the acid potassium tartrate, indicating an error of approximately 1 per cent.

As the maximum figure for the total tartaric acid in a baking powder rarely exceeds 20 per cent., a 1 per cent. deficiency in this case would show 19·8 per cent. instead of the theoretical 20·0 per cent.—a comparatively small divergence. Actually, in two baking powders prepared in the laboratory the percentages of tartaric acid found both by the lead tartrate and the acid tartrate methods were slightly higher than theory. The results in Table III are given without the application of any correction, and indicate the close agreement between the old and the new methods.

TABLE III

TARTARIC ACID IN BAKING POWDER

0·5 g. used for each experiment

No. of baking powder	Exp.	Weight of lead tartrate g.	Tartaric acid, per cent. By lead tartrate method*	Tartaric acid, per cent. By acid pot. tartrate method	
(1)	60/61	0·242	20·5	20·7	Theory: 20·3
(2)	62/63	0·244	20·6	20·8	
(3)	56/57	0·216	18·3	18·3	
(4)	58/59	0·179	15·1	15·3	
(5)	64/65	0·245	20·7	20·7	
(6)	73/74	0·146	12·3	12·6	
(7)	75/76	0·127	10·7	11·1	
(8)	77/78	0·271	11·5	11·3	

* Percentage of tartaric acid $= W \times \frac{100}{0{\cdot}5} \times \frac{150}{355} = W \times 84{\cdot}6$, where W = weight of lead tartrate from 0·5 g.

If, instead of using 1 g. of baking powder, the extract from 2 g. is made up to 100 ml., 50 ml. of this may be used for the detection and determination of

potassium, if present, the solution being acidified with dilute hydrochloric acid, and evaporated with platinum tetrachloride solution. From the potassium chloroplatinate obtained, the percentage of acid potassium tartrate may be calculated, and from this and the total tartrate the percentage of free tartaric acid is obtainable.

In the above Table, No. 5 was the only one containing acid potassium tartrate in addition to tartaric acid as the acid principle. In exceptional cases, when the weight of lead tartrate is less than 0·18 g., it is recommended that the determination should be repeated with a weight of baking powder sufficient to give such a concentration of tartaric acid in 25 ml. of extract that a minimum of 180 to 200 mg. of lead tartrate is obtained. This avoids a tendency towards low results, as in No. 6, in which, owing to the smaller quantity of precipitate, only 35 ml. of water were used for washing.

References

1. H. Cantoni and F. Zachoder, *Bull. Soc. Chim.*, 1905, [iii], **33**, 754; quoted in Thorpe's *Dict. of Applied Chem.*, 1926.
2. *A.O.A.C. Official Methods of Analysis*, 3rd Edition, 1930, p. 121; 4th Edition, 1935, p. 186.
3. G. W. Monier-Williams, ANALYST, 1931, **56**, 397.
4. A. F. Lerrigo, ANALYST, 1926, **51**, 180.

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An Improved Method for Determining Ethyl Alcohol in the Presence of Acetone

BY C. R. HOSKINS, B.Sc., PH.D.

THE necessity for an accurate method of determining ethyl alcohol in the presence of acetone has long been recognised. Two methods, involving the preliminary removal of the acetone, which have frequently been used in this laboratory, are those of Macoun[1] and of Hoff and Macoun.[2] The first depends on condensing the acetone with benzaldehyde in the presence of potassium hydroxide, the excess of benzaldehyde being subsequently removed. Whilst this method removes acetone quantitatively, an appreciable proportion of ethyl alcohol is lost; indeed, Macoun had observed losses amounting to as much as 4 per cent. of the alcohol present—losses for which no satisfactory explanation was offered. The second method removes acetone by condensing it with paraformaldehyde in the presence of alkali. Whilst material losses of ethyl alcohol have not been observed with this method, a disturbing factor has been the formation of traces of methyl alcohol.

If an aqueous alcohol solution containing acetone is distilled with excess of acid mercuric sulphate solution, the acetone is precipitated as a complex mercury compound (Denigès),[3] but the alcohol is not recovered quantitatively. The losses of alcohol in this operation have been studied, and the technique described below has been found to make these losses negligible.

METHOD.—The following solutions are necessary:—(1) Acid mercuric sulphate containing 50 g. of mercuric oxide, and 125 ml. of conc. sulphuric acid per litre. (2) Potassium oxalate, approximately 300 g. per litre. (3) Sodium formate, approximately 100 g. per litre.

The amount of acetone in the alcohol solution must be ascertained approximately. A rapid method is that of Adams and Nicholls,[4] which measures colorimetrically the indigo formed on condensing the acetone with *o*-nitrobenzaldehyde in the presence of alkali.

This having been done, an amount of alcohol solution containing not more than 1 ml. of acetone is introduced into a flask of approximately 750 ml. capacity. For each 0·1 ml. of acetone to be removed are added 25 ml. of the acid mercuric sulphate solution and 0·2 ml. of the sodium formate solution. The flask is connected with an efficient condenser for refluxing, and its contents are heated (see below), with frequent agitation, at such a rate that clouding takes place in approximately ten minutes. The flame under the flask is then lowered so as to keep the solution at about the same temperature for ten to twelve minutes. A few ml. of distilled water are poured down the condenser to wash any acetone which may have condensed there into the flask. At the end of the ten to twelve minutes, excess of potassium oxalate solution—12 ml. for each 100 ml. of mercuric sulphate—is poured through the condenser to precipitate the excess of mercuric sulphate as mercuric oxalate. The flask is cooled, and the condenser is again rinsed with distilled water, after which the flask is removed and connected with an ordinary distilling apparatus. Since the final volume of liquid may be as much as 400 or 500 ml., it is advisable to collect at least 150 ml. of distillate and to redistil this, preferably from an alkaline solution, to a smaller volume.

The formation of the mercury complex takes place at approximately 80° C. By avoiding any appreciable rise above the clouding temperature of the solution, and by precipitating the excess of mercuric sulphate when condensation with the acetone is complete, the oxidising action of the mercuric sulphate on the alcohol is greatly reduced. The addition of sodium formate, which appears to be preferentially oxidised by the mercuric sulphate, also slightly lessens the oxidation of the alcohol during the initial heating period.

The following table of results shows how small is the loss of ethyl alcohol. In each experiment acetone was added to 100 ml. of ethyl alcohol solution of known strength, and the mixture was treated as described. All normal precautions against loss of alcohol were taken, distillates being collected by means of an adaptor dipping below the surface of the liquid in the receiver. The distillate from the mercuric oxalate precipitate occasionally gave a slight aldehyde reaction with Schiff's reagent ; and although the effect of this on the specific gravity of the distillate was negligible, the opportunity was taken, when redistilling to the original volume of 100 ml., of removing the aldehyde by distilling from alkaline silver nitrate. Specific gravities were determined at 60°/60° F., the volume of ethyl alcohol being obtained from the official Spirit Tables. Each distillate was tested for the presence of acetone, and in the rare cases in which it was found, the amount was determined colorimetrically. In the table the amounts of alcohol recovered have been corrected, where necessary, for the acetone present, it being

assumed as a near approximation that a 1 per cent. by vol. acetone solution has the same specific gravity as a 0·7 per cent. by vol. ethyl alcohol solution.

Original solution			Final solution			Loss of alcohol	
Acetone ml.	Specific gravity at 60°/60° F.	Alcohol ml.	Specific gravity at 60°/60° F.	Acetone ml.	Alcohol ml.	ml.	Per Cent.
0·2	0·99317	4·75	0·99318	0·02	4·73	0·02	0·4
0·2	0·98681	9·81	0·98692	nil	9·72	0·09	0·9
0·2	0·98160	14·51	0·98172	nil	14·39	0·12	0·8
0·6	0·99311	4·80	0·99318	nil	4·74	0·06	1·2
0·6	0·98737	9·33	0·98750	nil	9·22	0·11	1·2
0·6	0·98160	14·51	0·98169	nil	14·42	0·09	0·6
1·0	0·99317	4·75	0·99318	nil	4·74	0·01	0·2
1·0	0·98681	9·81	0·98696	nil	9·68	0·13	1·3
1·0	0·98160	14·51	0·98172	0·04	14·59	0·14	1·0

A few experiments were carried out to determine the loss of methyl alcohol under the same conditions. The results of three of them are given in the following table. The percentages of methyl alcohol were obtained from the tables of Klason and Norlin.[5]

Original solution			Final solution			Loss of alcohol	
Acetone ml.	Specific gravity at 60°/60° F.	Alcohol ml.	Specific gravity at 60°/60° F.	Acetone ml.	Alcohol ml.	ml.	Per Cent.
1·0	0·99358	4·50	0·99389	nil	4·28	0·22	4·9
1·0	0·98626	10·03	0·98655	nil	9·80	0·23	2·3
1·0	0·98050	14·70	0·98090	nil	14·36	0·34	2·3

It will be observed that the losses with methyl alcohol are greater than with ethyl alcohol, but there is evidence that part, if not all, of this increased loss occurs during distillation.

For purposes of comparison, acetone was removed from an ethyl alcohol solution by the method now described and by the two other methods mentioned. The methyl alcohol formed in the paraformaldehyde method was estimated colorimetrically by means of Schiff's reagent after oxidation of the methyl alcohol to formaldehyde. In each instance 1 ml. of acetone was removed from an ethyl alcohol solution of sp.gr. 0·98683 at 60°/60° F., containing 9·79 ml. ethyl alcohol in 100 ml.

	Final solution				Loss of ethyl alcohol	
Method	Special gravity at 60°/60° F.	Acetone ml.	Methyl alcohol ml	Ethyl alcohol ml.	ml.	Per Cent.
Mercuric sulphate	0·98699	0·04	nil	9·63	0·16	1·6
Paraformaldehyde	0·98697	nil	0·06	9·62	0·17	1·7
Benzaldehyde ..	0·98727	0·08	nil	9·36	0·43	4·4

In view of the loss of alcohol and the considerable time necessary for its operation, the benzaldehyde method must be considered inferior to the other two. There appears to be little to choose between the mercuric sulphate and the paraformaldehyde methods where methyl alcohol formation is not a disadvantage; but its

presence, even in traces, is undesirable—indeed, inadmissible if it be necessary to decide whether the alcohol present has been methylated or not.

The new method has been used for several months in a section of this Laboratory with consistently trustworthy results.

I am indebted to Mr. G. F. Sheppard for much valuable criticism, and to Dr. J. J. Fox, the Government Chemist, for permission to publish this method.

References

1. J. M. Macoun, *J. Soc. Chem. Ind.*, 1928, **47**, 43T; Abst., ANALYST, 1928, **53**, 235.
2. R. W. Hoff and J. M. Macoun, ANALYST, 1933, **58**, 749.
3. G. Denigès, *J. Pharm. Chim.*, 1899, **9**, 7; Abst., ANALYST, 1899, **24**, 92.
4. C. A. Adams and J. R. Nicholls, ANALYST, 1929, **54**, 2.
5. P. Klason and E. Norlin, *Arkiv Kemi, Min., Geol.*, 1907, **2**, No. 27.

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The Determination of Bismuth as Phosphate

BY W. R. SCHOELLER, PH.D., F.I.C., AND D. A. LAMBIE, B.Sc., A.I.C.

(*Read at the Meeting, May 5, 1937*)

A METHOD for the determination of bismuth in high-grade ores, in which the metal is weighed as phosphate, has been described by Schoeller and Waterhouse.[1] The process has been extensively used by us, and has lately been the subject of further investigation, the results of which are given in this paper.

What caused us to study the method afresh was the observation that, whilst very serviceable on the whole, it was found occasionally to give fluctuating results, more particularly with substantial amounts of bismuth; in such cases, the deviations from the observed mean were either positive or negative, reaching but usually not exceeding 0·5 per cent. These irregularities were eventually proved to be due to contamination of the phosphate with sulphate, derived from the bismuth sulphide by oxidation with nitric acid.

PRECIPITATION OF BISMUTH PHOSPHATE IN PRESENCE OF SULPHATE.—The co-precipitation of sulphate is illustrated in Exps. 1 to 4, in which a uniform quantity of sulphuric acid was added to solutions of bismuth nitrate prior to precipitation of the phosphate. The washed phosphate precipitate was decomposed with sodium carbonate solution, and the acidified filtrate precipitated with barium chloride.

Exp.	Bismuth taken g.	Sulphuric acid added g.	Sulphuric acid found in $BiPO_4$ g.
1	0·0523	0·1000	0·0020
2	0·1020	0·1000	0·0039
3	0·3044	0·1000	0·0112
4	0·5025	0·1000	0·0180

In these experiments the sulphate-content of the unignited precipitate is very nearly a linear function of the quantity of bismuth.

When bismuth phosphate is precipitated from an acid solution containing sulphate, we must assume that it carries down a sulphate comparatively low in bismuth, for the bismuth result at first shows a positive error; on continued ignition, the sulphate is converted into oxide, and the error becomes negative. Such is the case in Exps. 5 to 8, in which a known amount of bismuth was dissolved in nitric acid, the solution precipitated with hydrogen sulphide, and the sulphide converted into phosphate according to Schoeller and Waterhouse's directions:

Exp.	Bismuth taken g.	Bismuth found		Error	
		1st weighing g.	Last weighing g.	1st Wt. g.	Last Wt. g.
5	0·0556	0·0561	0·0554	+0·0005	−0·0002
6	0·1023	0·1030	0·1021	+0·0007	−0·0002
7	0·3032	0·3065	0·3021	+0·0033	−0·0011
8	0·5395	0·5441	0·5375	+0·0046	−0·0020

These tests show that, for quantities of bismuth of the order of 0·1 g., the error incurred in Schoeller and Waterhouse's procedure is negligible. With large amounts of bismuth, however, the absolute error becomes appreciable.

As the assay of bismuth ores unavoidably involves sulphide precipitation as a step in the separation procedure, and subsequent solution of the sulphide in nitric acid (with partial oxidation of the sulphur to sulphuric acid), we argued that precipitation of the bismuth from the resultant solution by means of sodium carbonate, and solution of the washed precipitate in nitric acid, would yield a nitrate solution free from sulphate, from which pure bismuth phosphate would be precipitated. This improved method, which we are applying to the determination of bismuth in high-grade ores, is as follows:

AUTHORS' METHOD.—A quantity of powdered ore equivalent to less than 0·3 g. of metallic bismuth is digested with strong hydrochloric acid, with the object of decomposing any wolframite and galena present. The acid is evaporated to a small bulk, strong nitric acid is added, and the contents of the beaker are evaporated almost to dryness on a steam-bath. The nitrates are converted into chlorides by two evaporations with strong hydrochloric acid. The dry residue is digested with 5 ml. of strong hydrochloric acid, which is then diluted with 20 ml. of hot water, the insoluble matter being filtered off and washed with warm *N* hydrochloric acid. The filtrate is boiled with 1 to 2 g. of very fine pure iron wire; when the solution has become colourless, the boiling is continued for another 20 to 30 minutes, hot water being added towards the end so as to reduce the acidity to less than 0·5 *N*. The metallic precipitate is filtered off by decantation on a filter containing a small spiral of iron wire, washed with boiling water, returned to the beaker, and dissolved in hot hydrochloric acid and bromine.

The filtrate from the iron precipitation should always be tested for complete precipitation as follows:—It is saturated with hydrogen sulphide, any precipitate being collected and reserved (see below, A). The main solution is freed from bromine by evaporation, diluted, and treated with hydrogen sulphide. The

precipitate is collected, washed free from iron with acidulated hydrogen sulphide water, returned to the beaker, and digested for an hour on a hot plate with yellow sodium sulphide solution. The insoluble fraction is collected on the filter previously used, and washed with hot 3 per cent. sodium sulphide solution.

The alkaline filtrate is boiled with 10 to 15 g. of ammonium sulphate and left to settle.[2] Any precipitate is collected and heated, together with precipitate A above, with nitric and sulphuric acids until the former is expelled and copious fumes of the latter are given off. The residual acid, while still warm, is diluted with 1 : 1 and then with *N* sulphuric acid; any lead sulphate is filtered off, and the filtrate is tested for bismuth by the colorimetric iodide method.

After having been extracted with sodium sulphide, the sulphide precipitate is returned to the beaker, the paper is cleaned with hot nitric acid (1 : 1), and the precipitate is digested hot with strong nitric acid until the residual sulphur has fused into transparent globules. The nitrate solution is filtered through a small pad of filter-pulp, which is washed with 5 per cent. nitric acid. The filtrate is neutralised with sodium carbonate, of which an excess of 1 g. is added, as well as 0·5 g. of potassium cyanide, and the solution is boiled for a short time. The precipitate is allowed to settle completely, collected on a tight pad of filter-pulp, and washed with 2 per cent. sodium carbonate solution containing cyanide. It is advisable to treat the filtrate with hydrogen sulphide water, and to test any precipitate for bismuth, as with A above. The carbonate precipitate and pad are returned to the beaker, and heated with not much more than enough nitric acid to effect solution. The solution is once more filtered through a small pad of filter-pulp into a 600-ml. beaker, the pulp suspended in the liquor being gathered and squeezed with a glass rod; washing is effected with 5 per cent. nitric acid.

The filtrate (less than 100 ml.) is now ready for phosphate precipitation. It is left to cool, treated with 1 : 1 ammonia till turbid, cleared with 2 ml. of strong nitric acid, heated to boiling, and precipitated with 30 ml. of 10 per cent. diammonium phosphate solution (for 0·25 to 0·3 g. Bi), added, drop by drop, from a burette during continuous agitation. The first portion of precipitant is added very slowly; this results in a coarsely crystalline precipitate. When precipitation is complete, the liquid is diluted with 300 ml. of boiling distilled water and left to settle for an hour on a hot plate. The precipitate is collected on a tared porous porcelain crucible and washed with hot 2 per cent. ammonium nitrate solution containing a few drops of nitric acid per litre. Alternatively, the precipitate is collected on a 9-cm. No. 40 Whatman filter, and washed as before. The paper may be dried and ashed separately, but we have actually found this precaution unnecessary; in serial analyses we transfer the wet filter to a tared porcelain crucible, dry the contents on a hot plate, and gently heat them on an asbestos mat until the paper has charred. The carbon is burnt away at low temperature. The ignition is completed on a triangle over the full flame of a Bunsen burner. The weighed precipitate should again be ignited and weighed.

If the filtrate from the phosphate precipitate is tested with hydrogen sulphide it should remain colourless, but occasionally a negligible pale-brown coloration is produced.

NOTES ON THE PROCESS.—(i) We prefer to operate on quantities of bismuth

not exceeding 0·25 or 0·3 g., because the accuracy is not increased by the use of large amounts. On the contrary, the precipitation conditions realised by our procedure tend to give closer results when the quantity of metal is kept below 0·3 g.

(ii) When the sulphide precipitate is digested with yellow sodium sulphide for the purpose of extracting arsenic and antimony, a small, variable amount of bismuth is usually found in the filtered extract. This is recovered by boiling with ammonium sulphate, the sodium sulphide being converted into the ammonium salt, in which bismuth sulphide is insoluble.

(iii) Alkali cyanide is added in the precipitation of sulphate-free bismuth carbonate by sodium carbonate, in order to extract any copper and silver which may be present.

(iv) After several years' practical experience, we are satisfied that the phosphate method is the most accurate and convenient process for the determination of bismuth in high-grade ores. Hillebrand and Lundell, who give preference to the gravimetric determination as bismuth oxide,[3] assert that the phosphate method should not be considered in accurate analyses, no reason being given by them for its rejection. On the other hand, Schoeller and Waterhouse's procedure for the precipitation of the phosphate is reproduced in the compilation of select methods for use in reference analyses, published by the Committee of German Metallurgical Chemists.[4]

Our reasons for giving preference to the phosphate method are, that the phosphate is the most tractable bismuth precipitate known, being "a white, heavy, crystalline precipitate resembling lead sulphate, quite insoluble in water or in very dilute nitric acid, depositing and filtering quickly, unchanged by ignition, and not easily reduced."[1] The ignition is carried out, as described above, in ordinary or in porous porcelain crucibles. The use of ordinary crucibles and paper filters is an invaluable advantage in serial work.

The determination of bismuth as oxide, on the other hand, involves its precipitation by means of ammonium carbonate. The basic carbonate precipitate is by no means an ideal one as regards settling, filtering, and washing. It is also liable to contamination with basic sulphate; this can be corrected by re-precipitation, but such work has to be conducted with great care, as bismuth carbonate is slightly soluble in ammonium carbonate, a fact admitted by Hillebrand and Lundell. The filtrates from the carbonate precipitation should therefore be tested for bismuth. The greatest drawback of the oxide method is the ignition of the precipitate. Bismuth oxide cannot be ignited in contact with filter fibre, and must therefore be separated from the dried filter, the part adhering to the paper being dissolved in nitric acid and the solution evaporated to dryness. The oxide is fusible, and in the molten state rapidly destroys the glaze of porcelain crucibles. Hence, Hillebrand and Lundell advise ignition in platinum, a rather unattractive procedure, on account of the risk of accidents to the crucibles and the number of costly vessels required in serial work.

SUMMARY.—Bismuth phosphate, precipitated from solutions containing sulphate, is liable to be contaminated with sulphate, which loses sulphur trioxide on ignition. A modified phosphate method for the assay of high-grade bismuth ores is described, in which the interference of sulphate is overcome by an intervening

precipitation with sodium carbonate. The advantages of the phosphate method over the gravimetric determination as bismuth oxide are discussed.

References

1. W. R. Schoeller and E. F. Waterhouse, ANALYST, 1920, **45**, 435.
2. C. Boy, *Metall. u. Erz.*, 1935, **32**, 163.
3. W. F. Hillebrand and G. E. F. Lundell, *Applied Inorganic Analysis*, New York, 1929, 189, 192.
4. *Ausgewählte Methoden für Schiedsanalysen und kontradiktorisches Arbeiten*, Gesellschaft Deutscher Metallhütten-und Bergleute e.V., Berlin, 2nd Edition, 1931, 271.

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Discussion

Dr. B. S. EVANS said that the authors had mentioned the presence of sulphur trioxide in the phosphate precipitate; it was not quite clear whether this replaced P_2O_5 or whether it was simply adsorbed in some way. It was amazing how much sulphuric acid could be precipitated in this manner; considering that there was only 0·1 g. of sulphuric acid present, it meant that 0·5 g. of bismuth precipitated contained about 20 per cent. of it.

Dr. Schoeller had also referred to the elimination of sulphuric acid by the precipitation of bismuth carbonate, and later on he spoke of the objection to the determination as oxide because it had first to be precipitated as basic carbonate, which was liable to contamination with basic sulphate. If that were so, one would think that the method wanted much care in avoiding that contamination of basic sulphate in basic carbonate.

Dr. Schoeller, with characteristic modesty, had not mentioned a paper recently published in *Industrial and Engineering Chemistry* in which the authors put forward a method that seemed to be substantially that of Schoeller and Waterhouse. (They had not claimed an original method.) The figures given confirmed Schoeller and Waterhouse's original paper to a very gratifying extent.

Dr. SCHOELLER, replying, said that regarding the mode of combination of the sulphur trioxide in the precipitate, he was unable to give any definite information. All he could say was that at the beginning of the ignition there was a positive error, and at the end a negative one. This led to the inference that an acid sulphate was precipitated with the phosphate, and that the conversion of the sulphate into oxide by stronger ignition caused the negative error. The use of the modified method described in the present paper would do away with the slight inaccuracy.

As to the contamination of the carbonate precipitate with sulphate it should be borne in mind that Hillebrand and Lundell used ammonium carbonate as a precipitant, whilst the authors used the sodium salt. The former, being weaker in its action, might yield a precipitate not altogether free from sulphate, which would contaminate the oxide to be weighed; hence double precipitation might be necessary. The authors, on the other hand, used a stronger alkali, and the bismuth carbonate thus obtained was converted into phosphate—an operation in which traces of sulphate, if any still remained, would not interfere.

The amount of bismuth that went into the sodium sulphide filtrate and was recovered by ammonium sulphate was very small; it might amount to 2 mg. It seemed to vary according to the presence or absence of such elements as arsenic, antimony and copper. The determination of the recovered bismuth by the colorimetric process was convenient and accurate, and much to be preferred to a gravimetric method.

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 DETERMINATION OF SMALL QUANTITIES OF STRYCHNINE IN THE PRESENCE OF CAFFEINE (COMPOUND SYRUP OF GLYCEROPHOSPHATES)

SIMMONDS'S method (ANALYST, 1914, **39**, 81) for the determination of small quantities of strychnine in the presence of a large excess of quinine, which depends on the solubility of quinine ferrocyanide and the insolubility of strychnine ferrocyanide in strongly acid solution, can be readily adapted for mixtures of caffeine and strychnine, such as are obtained by extraction from Compound Syrup of Glycerophosphates B.P.C.

The mixed caffeine and strychnine can be extracted from the syrup by adding a large quantity (5 g.) of citric acid to 50 ml. of syrup and, after making the solution alkaline with ammonia, extracting five times with chloroform. After drying, the residue of anhydrous caffeine and strychnine is weighed.

To meet the present problem, the procedure of the original method can be simplified, since it is found that caffeine does not appear to be co-precipitated with the strychnine; hence re-precipitation is unnecessary.

The following results of some experiments made to ascertain this point show the simplicity of the method:

Solution A: 2·500 g. of anhydrous caffeine in 250 ml. of water.

Solution B: 20 ml. of Liquor Strychninae Hydrochlor. B.P. (containing 0·8622 per cent. w/v of strychnine) sufficiently evaporated to eliminate alcohol, and made up to 50 ml.; 3 ml. of dilution = 0·0103 g. of strychnine.

Solution C: Freshly-prepared 5 per cent. potassium ferrocyanide solution.

Solution D: 25 per cent. v/v sulphuric acid.

		Strychnine
1.	25 ml. A + 5 ml. C, overnight	No precipitate
2.	25 " A + 5 " C + 1 ml. D, overnight	" "
3.	25 " A + 5 " C + 10 " D, "	" "
4.	25 " A + 3 " B + 1 " C, "	Trace
5.	3 " B + 25 " water + 1 ml. C + 1 ml. D, overnight	0·0100 g.
6.	25 " A + 3 " B + 1 ml. C + 1 ml. D, "	0·0101 g.
7.	25 " A + 3 " B + 1 " C + 10 " D, "	0·0102 g.
8.	25 " A + 3 " B + 1 " C + 1 " D, 4 hours	0·0098 g.
9.	25 " A + 3 " B + 1 " C + 1 " D, 4 hours, re-precipitated overnight	0·0095 g.

The most suitable procedure for the determination would appear to be: Dissolve the weighed caffeine and strychnine from 50 ml. of syrup in 25 ml. of water to which is added 1 ml. of 25 per cent. v/v sulphuric acid, warming if found necessary. Cool, add 1 ml. of freshly-prepared 5 per cent. potassium ferrocyanide solution, stir to induce precipitation and leave for some hours, preferably overnight. Filter through a 7-cm. No. 1 filter-paper and wash well with water slightly acidified with sulphuric acid. Place the funnel in the neck of a separator, pierce the tip of the filter-paper, and wash in its contents successively with 10 ml. each of 10 per cent. ammonia, water and chloroform, added from the precipitation

beaker. Extract three times with chloroform, evaporate with 5 ml. of alcohol, dry the residue, and weigh.

For re-precipitation, only slight washing of the precipitate is necessary. After chloroform extraction, re-extract with 30 ml. of water containing 1 ml. of 25 per cent. v/v sulphuric acid, in three portions. Boil the acid extracts to eliminate chloroform, and re-precipitate with 1 ml. of 5 per cent. potassium ferrocyanide.

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OPTICAL ACTIVITY OF PREPARATIONS OF SQUILL

IN a recent communication from this laboratory (ANALYST, 1937, p. 192) it was pointed out that both Tinct. Scillae B.P. 1932 and Acetum Scillae B.P. 1932 showed a marked laevorotatory optical activity.

The quantitative aspect of this matter has now been investigated and it has been shown that Tinct. Scillae B.P. 1932 has an average optical rotation of −2·0°, whilst that of Acetum Scillae B.P. 1932 is −3·5°. The optical activity of the Acetum thus approaches twice that of the Tincture, although the same amount of Squill is used in the manufacture of both these preparations. As it is generally recognised, from the therapeutic point of view, that the Tincture is the more active preparation, it would thus appear that the higher optical activity is associated with the lower therapeutic value.

It was considered possible that the development of optical activity might be due to hydrolysis of the squill glycosides giving rise to a laevorotatory sugar. The following experiment was therefore carried out in order to see whether this hydrolysis could be carried further:—A sample of Tinct. Scillae B.P. 1932, known to be of full physiological activity and having an optical rotation of −1·8°, was treated with 1 per cent. of hydrochloric acid at laboratory temperature, and measurements of the optical rotation were made from time to time. It was found that the optical rotation slowly increased, reaching a maximum of −4·3° in two weeks. A simple pharmacological test carried out on the treated sample gave indications of decrease in physiological activity; although an isolated experiment, this was probably significant.

A sample of Acetum Scillae B.P. 1932 similarly treated showed a rise in optical rotation from an initial value of −3·5° to a maximum of −4·65°.

LEO MCGRAGHAN

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MESSRS. BRADY & MARTIN, LTD.
NEWCASTLE UPON TYNE, 2

DYESTUFFS FOR DEVELOPING LATENT FINGER-PRINTS

FURTHER experiments have been made on the applicability of Waxoline dyestuffs for the development of latent finger-prints, by dusting the dyestuff powder over the article under examination, and then fixing the impression of the finger-print by exposure to acetic acid fumes and to steam (*cf.* ANALYST, 1937, 192). The following (I.C.I.) Waxoline colours are particularly suitable for this purpose and cover a variety of shades:—Waxoline Yellow OS, Waxoline Orange AS, Waxoline Red AS, Waxoline Violet 2BS. An appropriate dyestuff can therefore be selected according to the colour of the object under examination, so that the impression of the finger-print will show up in contrast; for instance, the pale yellow dyestuff would be suitable for dusting over such articles as japanned tin boxes.

H. A. THOMAS

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THE DETECTION OF INHALED HYDROCYANIC ACID

THE use of hydrocyanic acid for fumigation purposes has become very prevalent, not only for ships but also for warehouses and private houses. Reports on the subject have been issued from time to time (*cf.* Reports on Public Health and Medical subjects to the Ministry of Health, Nos. 19, 60 and 72, and ANALYST, 1931, **56**, 46).

We were asked to examine the viscera from a case of suspected poisoning by hydrocyanic acid, and we were successful in showing the presence of this substance in the blood of the deceased. It has been suggested to us that there are very few cases on record where hydrocyanic acid has been found in the blood of those who have died by inhaling an atmosphere containing a very small amount of this gas, and that our results may be of interest to others.

An elderly couple, each over 70 years of age, were found dead in a house next door but one to a house in which fumigation with hydrogen cyanide had been carried out. The fumigation had commenced at about 9.20 a.m., but the bodies were not found until about 10.30 p.m., although there was evidence to show that both persons were alive and apparently well at noon.

The intermediate house was used as a buffer house with windows and doors open all day long. Furthermore, although all the houses were "attached," the one in which the fatalities took place was built five years after the others in the row, and there was no obvious sign of intercommunication; neither did a smoke test yield any positive result.

When the viscera were received, comparatively small quantities were examined for hydrocyanic acid, but with negative or inconclusive results. We then operated by steam distilling 250 ml. of the blood from the lungs, until 15 ml. of distillate had been collected. On two separate 5-ml. quantities we carried out the Prussian blue and sulphocyanide reactions as described below. In each case we obtained slight but definite reactions. The amount of cyanide found was certified as approximating to one milligram of hydrocyanic acid per litre of blood. The stomach contents were free from all but the smallest trace of hydrocyanic acid.

Blank distillations, carried out before the addition of the blood, and after the first 15 ml. of distillate from the blood had been collected, gave negative results.

The verdict at the inquest was accidental death due to cyanide poisoning.

PRUSSIAN BLUE TEST.—About 5 ml. of the distillate are made alkaline with two or three drops of 5 per cent. sodium hydroxide solution. To this are added two drops of 5 per cent. ferrous sulphate solution and one drop of 3 per cent. ferric chloride solution. The mixture is then allowed to stand for two minutes, gently heated, and acidified with dilute hydrochloric acid. Sufficient hydrochloric acid only is used to clear the liquid; there should not be a great excess of acid. Traces of cyanide may show only after fifteen to thirty minutes, but on standing overnight a blue precipitate settles out.

SULPHOCYANIDE TEST.—About 5 ml. of the distillate are heated with one drop of yellow ammonium sulphide solution for fifteen minutes on the water-bath. Dilute nitric acid is then added until the yellow colour of the solution is (just) entirely destroyed. Ferric alum solution is then added, drop by drop, until the maximum colour is obtained.

G. D. ELSDON
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The determination of traces of cyanide in cases in which death has resulted from the inhalation of hydrocyanic acid presents difficulties, for the amount of the poison present may be extremely minute and, owing to its volatile nature, it tends to disappear. As the writers of the above note have shown, the only chance of detecting cyanide lies in the examination of blood or lung tissue, and material

should therefore be sent to the analyst in a well-stoppered bottle as soon as possible.

Under the most favourable conditions it sometimes happens that even the Prussian blue and sulphocyanide tests fail. In those circumstances the following procedure, which has proved successful in my hands, may be tried. The steam distillate, which should be collected in a conical flask, is treated with a few drops of basic lead acetate solution to fix the sulphur compounds which are always present in the distillates from viscera, and especially in those from lung tissue. A few drops of dilute sulphuric acid are then added until the liquid is faintly acid. A drop of silver nitrate solution of about $N/10$ concentration is placed on a microscope slide, and this is inverted over the top of the conical flask and left in position in a warm place for at least 30 minutes, after which it is removed and covered with a cover-glass; microscopic examination may then reveal the presence of the characteristic crystals of silver cyanide. Alternatively, or in addition, the alloxan test may be tried (*cf. Ann. chim. anal.*, **1921**, **3**, 179; Abst., ANALYST, **1921**, **46**, 334). With both these tests I have obtained positive results on more than one occasion with distillates which had failed to give either the Prussian blue or the sulphocyanide reaction. I would add, however, that I am not satisfied that either of these tests establishes absolute proof of the presence of cyanide, although, taken in conjunction with all the facts of the case concerned, they will leave little doubt in the minds of most people.

In connection with the investigation of this class of cases I would observe that the post-mortem findings should in any given instance reduce the question down to the alternatives of carbon monoxide and hydrocyanic acid poisoning. A blood examination will establish the presence or absence of carbon monoxide. Finally, in the absence of carbon monoxide, positive reactions with either or both of the tests mentioned above should establish beyond all reasonable doubt the cause of death.

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Report of the Essential Oil Sub-Committee to the Analytical Methods Committee

REPORT No. 13

THE DETERMINATION OF ESTERS

THE Essential Oil Sub-Committee makes the following recommendations for the determination of esters in essential oils. Numerous experiments carried out by members of the Sub-Committee have shown that, unless the conditions of the tests are standardised somewhat closely, considerable variations in results between different operators and in different laboratories are likely to occur.

The following reagents are required:

Alcohol (90 per cent. by volume).

Alcoholic potassium hydroxide: approximately $N/10$ prepared by diluting alcoholic potassium hydroxide, approximately $N/2$, with 90 per cent. (by volume) alcohol and standardising by titration with $N/10$ acid using phenolphthalein as indicator.

Alcoholic potassium hydroxide: approximately $N/2$ prepared by dissolving 33 g. of potassium hydroxide of reagent purity in 1 litre of 95 per cent. (by volume) alcohol, allowing to stand, and decanting or filtering the clear liquid.
Sulphuric acid or hydrochloric acid: $N/2$.
Phenolphthalein: 0·2 per cent. solution in 60 per cent. alcohol.

METHOD OF DETERMINATION.—Two g. of the oil, or other suitable quantity so that the amount of alkali added is at least double that required for saponification, are weighed into the saponification flask; 5 ml. of freshly well-boiled and neutralised alcohol are added, and the free acid is titrated with $N/10$ alcoholic potassium hydroxide solution, using 0·2 ml. of phenolphthalein solution as indicator (*Note* 1). The result of the titration is calculated to acid value (mg. of KOH required for the neutralisation of 1 g. of the oil). To the neutralised solution in the flask are then added 20 ml. of $N/2$ alcoholic potassium hydroxide solution, and the whole is boiled under a reflux condenser for 1 hour (*Note* 2), at the end of which time the excess of alkali is titrated immediately with $N/2$ acid, using an additional 0·5 ml. of phenolphthalein solution as indicator. At the same time a blank determination is carried out by boiling for 1 hour under a reflux condenser 5 ml. of alcohol, 20 ml. of $N/2$ alcoholic potassium hydroxide, and 0·2 ml. of phenolphthalein solution, and then titrating immediately with $N/2$ acid after the addition of a further 0·5 ml. of phenolphthalein solution. In the case of bergamot oil, there may be a re-appearance of the pink colour on standing; this should be ignored (*Note* 3).

The difference between the two titrations is calculated to the percentage of esters in the usual manner, using the appropriate factor. Those for the most commonly occurring esters are as follows:

Bornyl acetate	0·0981	Santalyl acetate	0·1311
Geranyl acetate	0·0981	Methyl salicylate	0·0760
Linalyl acetate	0·0981	Geranyl tiglate	0·1181
Menthyl acetate	0·0991	Linalyl benzoate	0·1290

The following should be noted:

Note 1. With oil of wintergreen and oil of sweet birch, the free acid should be determined in a separate experiment by shaking 5 g. of the oil with 25 ml. of water, and titrating with $N/10$ aqueous sodium (potassium) hydroxide solution, using 1 ml. of *phenol red* (0·04 per cent. in 20 per cent. alcohol) as indicator. The saponification should be carried out without the preliminary neutralisation of the free acid, and from the volume of alkali required the equivalent of that used in the separate determination of the free acid should be deducted.

Note 2. With oil of wintergreen and oil of sweet birch, the boiling should be continued for an hour and a half.

Note 3. It is proposed to deal with the examination of bergamot oil in a later report.

RESULTS.—The members of the Sub-Committee have from time to time carried out determinations in their own laboratories on samples of various ester-containing oils circulated for this purpose. In the accompanying table are shown the results obtained on eight samples of such oils. From a consideration of these results amongst others we are of opinion that the maximum variation in the percentage of esters estimated by this method should not exceed $\pm 0{\cdot}7$ per cent.

Results of Ester Determination in Oils

Member	Lavender Linalyl acetate Per Cent.	Bergamot Linalyl acetate Per Cent.	Bergamot Linalyl acetate Per Cent.	Bourbon geranium Geranyl tiglate Per Cent.	Pine Bornyl acetate Per Cent.	Pepper-mint Menthyl acetate Per Cent.	Spike lavender Bornyl acetate Per Cent.	Methyl salicylate Per Cent.
1	42·6	40·4	37·5	24·5	37·3	8·0	2·7	100·0
	42·6	40·4	37·3	24·6	37·3	8·0	2·5	99·9
			37·8	24·9	37·3	7·9	2·3	
2	42·1	40·2	37·7	25·2	37·5	7·3	2·4	—
	42·2	40·3						
	42·3	40·3						
	42·4	40·4						
		40·3						
		40·2						
		40·4						
3	41·6	—	38·3	25·2	37·6	7·7	2·3	99·6
			38·0	25·2	37·4	7·8	2·1	
4	42·2	40·7	38·0	24·4	37·2	7·2	2·1	99·1
	42·4	40·9	37·7	24·4	37·2	7·3	2·0	99·2
5	42·1	40·4	37·7	25·6	37·2	7·8	2·3	99·7
	42·4	40·5	38·1	25·1	36·6	7·5	2·2	99·5
6	42·6	40·4	—	—	—	—	—	—
	42·3	40·5						
7	42·7	41·4	—	—	—	—	2·7	99·2
	42·8	41·5					2·6	
8	41·9	40·5	38·0	25·2	38·1	7·7	—	99·2
9	—	40·9	37·6	25·0	37·7	7·7	2·6	99·6
		40·5	37·8	25·5	37·5	7·4	2·3	99·0
		41·3	38·1	25·1	37·5		2·2	
		40·6			37·8			
Range ..	41·6–42·8	40·2–41·5	37·3–38·3	24·4–25·6	36·6–38·1	7·2–8·0	2·0–2·7	99·0–100·0
Mean ..	42·3	40·6	37·8	25·0	37·4	7·64	2·35	99·6
Standard deviation	0·32	0·31	0·26	0·37	0·30	0·26	0·23	0·31

(*Signed*)

W. H. Simmons (*Chairman*), C. T. Bennett, S. W. Bradley, L. E. Campbell, Thos. H. Durrans, T. W. Harrison, Ernest J. Parry, John H. Seager, Gilbert E. Smith, J. Sutherland, T. Tusting Cocking (*Hon. Secretary*).

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 LEEDS

ANNUAL REPORT OF THE CITY ANALYST FOR 1936

THE total number of samples examined was 2218, of which 138 were taken informally. Nine of the informal, and 161 of the formal samples were adulterated.

POTTED BEEF.—Two of 7 samples were adulterated, one containing 48 per cent. and the other 49 per cent. of starchy filler; in each case the amount of starch was 4·6 per cent. It is contended that such products should be sold as "Potted beef pastes." The wholesale vendor of the first sample (which was taken in course of delivery to the retailer) was prosecuted and found guilty, but the case was dismissed under the Probation of Offenders Act on payment of 14s. 6d. costs.

No proceedings were instituted in connection with the second sample, as the wholesaler had intimated at the time of the sale that the preparation was meat paste.

DUST IN LEEDS, HALIFAX AND HUDDERSFIELD.—A second series of exposures, in which soft paraffin wax was used instead of prepared lard, was made during the six months October, 1935, to April, 1936, and the results obtained are given in Table I.

TABLE I

Station	Total dust		Arsenic oxide		Lead		Copper	
	mg. per sq.ft.	cwts. per sq.mile	mg. per sq.ft.	cwts. per sq.mile	mg. per sq.ft.	cwts. per sq.mile	mg. per sq.ft.	cwts. per sq.mile
LEEDS								
*No. 1. Centre (Market Buildings) ..	1700·0	933·0	0·582	0·320	5·190	2·850	0·970	0·533
No. 2. Residential (Spring Bank) ..	526·0	289·0	0·216	0·119	0·563	0·309	0·216	0·119
No. 3. Industrial (Goodman Street) ..	112·0	61·5	0·043	0·024	0·193	0·106	nil	nil
No. 4. Residential (Knostrop)	1160·0	637·0	0·304	0·167	0·990	0·544	1·270	0·697
No. 5. Centre (Market)	6050·0	3320·0	2·880	1·580	18·300	10·050	2·520	1·384
HALIFAX								
No. 1. Centre (Over Clock Chamber) ..	5930·0	3255·0	1·422	0·780	14·690	8·000	1·900	1·044
No. 2. Industrial (Public Library) ..	2070·0	1136·0	0·634	0·348	25·500	14·000	5·620	3·085
No. 3. Residential (Northowram Hall) ..	942·0	516·0	0·427	0·234	11·200	6·150	0·642	0·353
HUDDERSFIELD								
†No. 1. Industrial (Disused Mill Room) ..	160·0	87·8	0·013	0·007	0·086	0·047	nil	nil
No. 2. Centre (Market Hall Clock Tower)	3180·0	1745·0	1·810	0·994	4·350	2·390	14·800	8·130
No. 3. Residential (Ravensknowle) ..	1770·0	972·0	0·532	0·292	2·180	1·198	1·330	0·730

* Tampered with—apparently not seriously. † Removed and placed vertically in a window.

Owing to the widely different conditions under which the plates have been exposed, any general comparison between the quantities found in different areas would give a false impression. Thus, as an illustration from the Leeds deposits, we know from the rain-gauge deposits that Hunslet usually gives figures about treble those at Headingley, yet the dust collected on the plate at Goodman Street, Hunslet, is only about one-fifth of the amount collected at Spring Bank, Headingley. Similarly, of the two plates exposed at Market Buildings, Leeds, one has collected three and a half times as much dust as the other, owing to differences in the manner of exposure.

Fortunately, three plates (one plate in each town) were exposed under similar conditions, namely, in the open air, but protected from rain, and these may be compared one with another, although the areas in which they were exposed were of different types. The Leeds area was in the centre of the town, and the Halifax and Huddersfield areas were residential. The figures for these three plates are given below:

	Cwts. per square mile			
	Total dust	Arsenic oxide	Lead	Copper
Leeds—Market Roof ..	3320·0	1·580	10·050	1·384
Halifax—Northowram ..	516·0	0·234	6·150	0·353
Huddersfield—Ravensknowle	972·0	0·292	1·198	0·730

As one would expect, the Leeds plate collected the largest amount of dust and of arsenic, lead and copper; the Huddersfield plate collected nearly twice as much dust as that at Halifax, but contained only about the same amount of arsenic, one-fifth the amount of lead and twice as much copper.

Proportions of Arsenic, Lead and Copper.—The figures in Table II are of similar order to those obtained from the six Leeds dusts examined in 1932 (*cf.* ANALYST, 1933, **58**, 471).

TABLE II

	Arsenic oxide		Lead		Copper	
	Per Cent.	p.p.m.	Per Cent.	p.p.m.	Per Cent.	p.p.m.
LEEDS						
*No. 1. ..	0·034	342	0·306	3055	0·057	570
No. 2. ..	0·041	410	0·107	1070	0·041	410
No. 3. ..	0·038	384	0·172	1725	nil	nil
No. 4. ..	0·026	262	0·085	854	0·109	1095
No. 5. ..	0·047	476	0·302	3025	0·041	416
HALIFAX						
No. 1. ..	0·024	240	0·248	2480	0·032	321
No. 2. ..	0·031	307	1·234	12340	0·272	2720
No. 3. ..	0·045	453	1·188	11880	0·068	682
HUDDERSFIELD						
†No. 1. ..	0·008	80	0·053	534	nil	nil
No. 2. ..	0·057	570	0·137	1370	0·465	4650
No. 3. ..	0·030	300	0·123	1230	0·075	752

* Tampered with—apparently not seriously. † Removed and placed vertically in a window.

This table is more useful than Table I for purposes of comparison because the proportions of the metals given are independent of the amount of dust collected, and therefore all the plates can be compared one with another.

Arsenic is found in greatest proportion in the centres of Leeds and Huddersfield, and in the residential area of Halifax, Huddersfield having the greatest percentage, and Leeds and Halifax being about equal.

Lead is found in greatest proportion in No. 2 (Industrial) and No. 3 (Residential) at Halifax, the percentages being far greater than on any other plate and so high that they call for some explanation. In Leeds and Huddersfield the centre of the town contains the highest percentage of lead, Leeds containing more than Huddersfield. Copper is also found in an unusually high percentage in the central area, Huddersfield. Halifax has the next largest percentage, in an industrial area, and Leeds has the third largest percentage in No. 4 area, which is near a copper works.

C. H. MANLEY

CITY OF BIRMINGHAM

REPORT OF THE CITY ANALYST FOR THE FIRST QUARTER, 1937

THE total number of samples analysed was 1,465, of which 1,422 were bought informally.

"HOME-MADE" "FULL FRUIT STANDARD" JAM.—A sample of blackcurrant jam was labelled "Full Fruit Standard," and in another place as "Home-made." It is unlikely that a small shopkeeper producing a home-made article would be a signatory to the agreement with the Food Manufacturers Federation. Nevertheless, there is nothing illegal in the use of the Federation label by a person making home-made jam, and as no proof was forthcoming of the origin of the sample, no action was taken.

COMPOUND BISMUTH LOZENGES.—In response to a demand for this article, for which the B.P. description specifies lozenges containing 0·15 g. of bismuth carbonate, 0·15 g. of magnesium carbonate and 0·3 g. of calcium carbonate, tablets were supplied of a heart shape which were seriously deficient in all three ingredients. The vendor stated that he kept stocks both of these tablets and of the official lozenges and apologised for having supplied the former in error. These were asked for by customers as "heartshape bismuth tablets," and were supplied by a wholesale firm for sale as "indigestion tablets." He promised to take steps to ensure that no confusion should arise in the future.

SODA MINT TABLETS.—This article is contained in the B.P. Codex under the name of Compound Sodium Bicarbonate Tablets, and one of the synonyms is "Soda Mint Tablets." Each tablet should contain, according to the Codex formula, 0·324 g. of sodium bicarbonate and 0·008 g. of ammonium bicarbonate, together with small amounts of saccharin and oil of peppermint. One sample contained the correct weight of sodium carbonate but no ammonium bicarbonate. A second sample, bought from the same shop a month later, was found to be of the correct B.P.C. quality. No action was taken.

ARTIFICIAL VINEGAR.—Seven informal samples, sold as vinegar, consisted of artificial vinegar. In each case the sampling officer visited the shop and explained the difference between the two articles and the conditions under which they should be sold. Artificial vinegar is often sold wholesale as "pure vinegar," and some retail vendors may not be aware that what they are buying under this name is really the synthetic product.

A sample of malt vinegar, bought informally, consisted of artificial vinegar, and an official sample bought two days later, had the same composition. The stock barrel was labelled "Guaranteed absolutely pure vinegar." On a blackboard in the shop, however, was written in chalk, "Pure Malt Vinegar, 2d. pint." The vendor was prosecuted and fined 20s. H. H. BAGNALL

METROPOLITAN BOROUGH OF CHELSEA

ANNUAL REPORT OF THE PUBLIC ANALYST FOR 1936

OF the 400 samples analysed, 301 were taken formally and 4 of these were adulterated. Eight of the 99 informal samples were adulterated.

CITRUS FRUIT POWDERS.—A sample of Orangeade Powder and one of Lemon Barley Powder were each reported as adulterated, as they contained tartaric acid instead of citric acid, the acid natural to citrus fruits.

MEDICATED SWEETS.—A sample of iodine and blackcurrant pastilles contained 0·046 per cent. of iodine. A sample of linseed, liquorice and chlorodyne tablets

contained neither morphine nor prussic acid, which would have rendered the seller liable under the Dangerous Drugs Act and the Pharmacy and Poisons Act, but the sale of these sweets indiscriminately to children is dangerous. Sometimes, liking the strong flavour, they eat too many; on the other hand, there is the risk, which is perhaps greater—that some people take so-called medicated sweets or wines with impunity, and think that they can do the same with the properly medicated preparation obtained from a druggist. T. McLACHLAN

Department of Scientific and Industrial Research

THE INVESTIGATION OF ATMOSPHERIC POLLUTION

REPORT AND OBSERVATIONS IN THE YEAR ENDED MARCH 31ST, 1936*

THE 22nd Report on Atmospheric Pollution embodies, as in previous years (*cf.* ANALYST, 1936, **61**, 257), the Report of the Standing Conference to the Co-operating Bodies; the Report of the Atmospheric Pollution Research Committee; the Report of the Superintendent of Observations, which includes tables and graphs; an Appendix on Observations on Ultra-violet and Visible Rays in Rochdale, and the General Deposit Tables. Photographs are also included of the Experimental Apparatus for measuring Sulphur in Air in relation to Wind Directions, the Standard Lead Peroxide Gauge for estimation of sulphur in the atmosphere, and the combined apparatus for measuring sulphur dioxide (by the volumetric method) and suspended impurity.

The number of the deposit gauge stations has been increased during the year from 98 to 115, and the number of automatic filters from 14 to 15, whilst the number of lead peroxide cylinders exposed has decreased from 45 to 42. The special intensive survey of pollution for which preparation has been in progress for some time has been arranged to take place in Leicester. Renewed interest has been taken in obtaining records of sulphur dioxide concentration, and the apparatus now designed (photograph) records the frequency of the winds from each of 8 sectors, and at the same time exposes a corresponding sector of the lead peroxide fabric, which can subsequently be analysed by sectors. An apparatus constructed to measure the conductivity of rain water is shortly to be tested. With a view to finding the best way to estimate the amount of dust in a stain obtained by drawing air through filter-paper, the possibility of calibrating optical densities of stains by direct weighing is being explored.

In considering the general trend of atmospheric pollution over a period of years, as shown by the tables and graphs, there is definite ground for belief that the smoke abatement movement has had real effect. It has to be remembered that increases in pollution in certain cases may be due to increased industrial activity. Taken generally, the total deposit curves show a large decrease in pollution before the industrial depression set in. Suspended matter, on the other hand, although showing an improvement in the air in certain cases, in others indicated a marked increase in impurity. Sunshine observations show that winter sunshine in London, compared with that at Kew, has risen from 20 to 52 since 1881.

LIME IN DEPOSIT.—A table is included in the Report showing the deposit of lime in metric tons per sq. km. with the same figure expressed as per cent. of total solids. A large addition to the number of stations estimating lime occurred in September, so that comparison for the months April to August is not possible in most cases. For the summer months the lime (as per cent. of total solids) averaged 7·1, and for winter (October–March) 5·57.

* Published May 19th, 1937, pp. 128. Obtainable at Adastral House, Kingsway, W.C.2. Price 6s. net.

MEASUREMENT OF *p*H.—In all stations at Burnley the water was consistently acid except for one month; Coventry showed consistently alkaline water. Two Liverpool stations had acid water, but two other Liverpool stations showed alkaline water on a few occasions. Salford's water was acid (with *p*H under 5·5 throughout the year), as also that of Wakefield, whilst at Southport it was slightly alkaline in 2 months, and Rochdale's acid water only once had a *p*H exceeding 5·5. There was a greater tendency towards acid water during the winter. The highest alkalinity was found during June and July last year, and this year during May and July.

Acidity (as sulphuric acid) and *Alkalinity* (as ammonia), estimated by titration, do not necessarily agree exactly with the *p*H values, and the table indicates constant acidity in nearly all stations. The only stations showing constant alkalinity were Hove, with a five month's record, and Kingston-upon-Hull all the year, whilst Birmingham (West Heath) was alkaline for every month except February. From the table the tendency appears to be toward increased acid pollution.

AUTOMATIC FILTER.—In general, higher impurity is shown in winter than in summer, December–February being the worst and May–September being the best periods. The highest concentration of impurity at any of the stations was recorded for Stoke-on-Trent, where the summer minimum was about twice the winter maximum at Cardiff. In order to bring out the trend of the automatic filter records over a period of years, where a sufficiently long series of records is available, the average hourly concentration of sooty matter for the whole summer and winter have been calculated and graphs drawn. For some stations definite improvement is indicated; for example, London generally shows improvement except for Victoria Street, but in some places there is increase in pollution, *e.g.*, Cardiff and particularly Coventry.

SULPHUR POLLUTION.—*Volumetric Method.*—Sulphur in the air is at once seen from the tables to be a winter product. London showed the highest average pollution, but the highest concentration figure for the year was for Salford (Regent Road) in December, but as this was more than three times any other figure for Salford it was doubtless due to some specific cause. It must be remembered that measurement of sulphur concentration is made at a particular place and does not denote conditions at any great distance from that place. Returns obtained by the lead peroxide method were made from 41 stations, 10 also using the volumetric method. The exceedingly high sulphur pollution in London calls for investigation and remedy.

ULTRA-VIOLET RADIATION BY THE ACETONE AND METHYLENE BLUE METHOD.—The Research Committee are still engaged on the examination of methods for the measurement of ultra-violet radiation with the object of providing a standard one. There are certain objections to the acetone and methylene blue method. Useful information may, however, be obtained by using the figures obtained by it for comparative purposes; thus the Cardiff figure (Cardiff is a city remarkably free from pollution) is about double the next highest figure—for Kingston-upon-Hull. Further useful comparisons can be made from figures for the centre and outskirts of cities. The obstruction of the radiation by smoke could only be effectively examined by comparing radiation received in a city with that which it should receive if no city were there, and the nearest to that is a comparison with surrounding country on the windward side.

MEASUREMENT OF ATMOSPHERIC POLLUTION IN AMERICA.—An investigation extending over 2 years was carried out in 14 of the largest cities of the United States, Owens's automatic filter and jet dust counter, and the Smith Greenburg impinger for weight and composition of suspended matter, being used. The Aitken nuclei counter was also used, and measurement of daylight taken with the Macbeth illuminometer, and of ultra-violet light with the Reutschler meter. No measurements of deposit were taken and the method adopted was to apply the

methods mentioned throughout a week in each city. The results are recorded in Public Health Bulletin No. 224. The average shade number of all the automatic filter records between 8 a.m. and 4 p.m. in the cities tested was, for winter, 1·3, and the corresponding figure in London, Glasgow and other British cities was 2·4; from these results the conclusion was drawn that British cities were nearly twice as heavily polluted as American.

RECORD OF OBSERVATIONS.—The maximum and minimum monthly deposits as metric tons per sq.km. were:—*Tar*: Bradford 37, Dewsbury (Whitley) and Huddersfield 2; *carbonaceous matter other than tar*: Burnley (Parker Lane) 414, Glasgow (Mearnskirk Hospital) 31; *insoluble ash*: Liverpool (Netherfield Road) 1125 (next highest, St. Helens 575), Glasgow (Mearnskirk Hospital) 44; *ash of insoluble matter*: London (Finsbury Park) 570, Wakefield (Clarence Park) 106; *total solids*: Liverpool (Netherfield Road) 2277, (next highest Burnley [Parker Lane] 1545), Glasgow (Mearnskirk Hospital) 309; *rainfall*: Rochdale 101 mm., London (Victoria Park) 49.

The figures for the current year indicate an increase for tar, and both carbonaceous matter other than tar and insoluble ash indicate rather worse atmospheric conditions. The same applies to soluble ash and total solids. For the latter the greatest increase was at London (Finsbury Park), with Dewsbury (Ravensthorpe) next. Stations showing an increase in all components of deposit compared with the "General Average" were Bournville Village and Works, Dewsbury, Halifax, Sheffield (Nether Green), and Southport (Bedford Road Park).

D. G. H.

Annual Report on Alkali, &c., Works for 1936

THE Seventy-third Report of the Chief Inspectors to the Ministry of Health and to the Department of Health for Scotland deals with methods adopted for preventing the emission of offensive gases in a very wide range of industrial processes. Not only are the processes registrable under the Act reviewed, but many instances are cited in which complaint has arisen in connection with processes outside the scope of the Act. The question of smoke emission also receives more attention than it has done in previous Reports.

The Public Health (Smoke Abatement) Act, 1926,* provides, in effect, that a prosecution for the emission of smoke, other than black smoke, shall not succeed when adequate and proper plant for preventing the creation and emission of smoke is provided and efficiently maintained, and also used in a proper and skilful manner. Efficient stoking goes to the root of the second part of this provision.

The subject of smokes and fumes from burning colliery spoilbanks and from coal carbonisation processes is dealt with in this connection, and the Report suggests that greater efforts at abatement could, and ought to, be made.

Smoke arising from pottery kilns, brick works, blast furnaces, rivet works, and boiler plants is also dealt with.

WASTE GASES FROM ELECTRIC POWER STATIONS.—In connection with electric power stations, the following figures supplied by the London Power Co. are interesting as showing the magnitude of the task of purifying the waste gases from a large power station:

Total weight of coal fired: 430,887 tons (about 50 tons per hour).
Average sulphur-content of coal: 0·91 per cent.
Total volume of gas treated: 200 thousand million cb.ft. (dry at N.T.P.).
Average sulphur emission (898 works tests): 0·025 grain (as S) per cb.ft. of dry gas at N.T.P.
Equivalent of sulphur eliminated by gas washing: 3611 tons.

* Reproduced, as from October 1st, 1937, in Sections 101–106 of the Public Health Act, 1936.

ARSENIC WORKS.—The condensation of arsenious oxide has been satisfactory, the average content of gases escaping from the calcination of arsenical ores being 0·038 grain of As_2O_3 per cb.ft., accompanied by a total acidity of 0·65 grain.

The amount of white arsenic and arsenic soot produced in Cornwall is still small, the figure in 1935 being 172 tons as compared with 3207 tons in 1924. In Sweden the output is of the order of 150 tons per day. The price is low and the lodes now worked in Cornwall are poor in arsenic, so that calciners are finding difficulties in disposing of soot with an As_2O_3 content of between 20 and 30 per cent. A partial refinement appears to be the only means of making a material acceptable to the refiners and avoiding the cost of storage or the danger of exposed heaps of arsenical soot.

CEMENT PRODUCTION WORKS.—Cement has been in high demand throughout the year, and the increased production has resulted in a number of complaints about dust emission. Encouraging progress, however, has been made with the problem of preventing the emission of dust, and electrical precipitation is now practised at a number of works and is in course of installation in many others. The dust concentration can readily be reduced by this process to a concentration below 0·5 grain per cb.ft.

The Report also gives the customary statistics relating to salt cake, sulphuric acid, tar and ammonia.

Connecticut Agricultural Experimental Station

FORTIETH REPORT ON FOODS AND TWENTY-EIGHTH REPORT ON DRUGS

THE Department of Analytical Chemistry of the Station, under the direction of Dr. E. M. Bailey, is primarily concerned with analytical work relating to inspection and control of commercial fertilisers, feeding stuffs, foods, drugs and insecticides, but it also does work for other departments of the State, and collaborates with the Association of Official Agricultural Chemists in its studies of analytical methods.

The present Report summarises the work done in 1935, mainly in connection with food and drug control. Among the subjects of interest discussed are the following:

ORANGEADE.—The regulation in Connecticut and other States requires orangeade to contain at least 15 per cent. of orange juice. Considerable quantities of orange base concentrate are now sent to local distributing agencies, such as fountains and dairies, where it is diluted and made into beverages of the orangeade type. Being an orange product, its nutritional virtues due to vitamin *C* are usually stressed in advertising. According to Bessey and King (*J. Biol. Chem.*, **1933**, **103**, 693) the vitamin-content of undiluted orange juice is of the order of about 0·6 mg. per ml. By mere dilution, and assuming that there is no destruction of vitamin *C* in the preparation of the concentrate, orangeade beverage should contain about 0·09 mg. of the vitamin. Some loss or destruction of vitamin is to be expected in the process of converting juice into a commercial concentrate, and there may be a further reduction through over-dilution.

Fresh juice expressed from oranges in the laboratory without special precautions gave results for vitamin *C* agreeing well with those of Bessey and King.

The ash from the concentrates was substantially the same as from fresh juice. Several samples officially submitted gave the following results:

	Ash g. per 100 ml.	Vitamin *C* (ascorbic acid) mg. per ml.	Approximate juice content (basis of ash) Per Cent.
Concentrate I	0·373	0·289	—
Beverage made from I ..	0·048	0·015	12
Orangeade	0·064	0·019	16
Concentrate II	0·409	0·390	—
Beverage made from II ..	0·074	0·045	18
Orange juice, canned ..	—	0·450	—
Orange juice fresh ..	0·41	0·510	—
Do. ..	0·41	0·510	—

A fair interpretation of these figures is that the beverages made from the concentrates contain from 12 to 18 per cent. of orange juice, but that the vitamin *C* content has suffered some impairment. A sample of orangeade submitted by a local health department contained only 0·012 mg. of vitamin *C* per ml.

DETECTION OF TEASEED OIL IN OLIVE OIL.—The following test, devised by Fitelson, differentiates teaseed oil from any of the other food oils in present use. A few drops of the oil in a mixture of acetic anhydride, chloroform and sulphuric acid are allowed to stand for 5 minutes, and then mixed with ethyl ether. Teaseed oil gives a magenta-red colour, the intensity of which is proportional to the amount of that oil present. Negative results are given by maize, cottonseed, arachis, sesame, sunflower, rapeseed, soya bean, poppy, mustard and olive oils. A faint pink colour may be developed in some instances with olive oil, but it cannot be confused with the characteristic colour given by teaseed oil.

The reliability of the Fitelson test has been demonstrated by experience in Government and State food control laboratories over a period of about a year. Since the test was available no official samples have been found to contain teaseed oil, but three unofficial samples of salad oils submitted by purchasers have shown substantial proportions of that oil.

"COLOUR ADDED" ORANGES.—The legend "colour added" now appears quite generally on Florida oranges. Trees that flower out of season produce what is known as "off-bloom" fruit, the colour of which is variable and not typical of the normal varietal production. Although the oranges may be fully matured and of normal palatability, there is a prejudice against them because the non-uniform natural colour creates an impression that the fruit is unripe and unpalatable. The State of Florida has therefore passed legislation permitting artificial colouring and regulating the practice. The Florida Citrus Commission specifies the requirements that must be met before the fruit may be coloured, and the Commissioner of Agriculture is charged with enforcing these requirements. One of the chief requirements is that no fruit that has not attained a satisfactory degree of ripeness shall be coloured. This condition is judged by the ratio of sugar to fruit acid, which must not be less than 8 to 1.

Under the statutes in Connecticut, artificial colouring of foods is not illegal, provided that the colour is harmless, and that the colouring does not conceal damage or inferiority. All the fruits examined had a sugar to acid ratio exceeding 8 to 1. In four unofficial samples the acidity and distribution of sugars were as follows:—Acidity (as anhydrous citric acid), 0·48 to 0·96 g. per 100 ml.; total sugars, 5·87 to 8·46; sugar to acid ratio, 8·9 to 12·2. One "colour added" sample was dyed with a colour which was not one of the permitted yellow shades; it closely resembled Sudan II.

VITAMIN *D* MILK.—Three methods of imparting vitamin *D* potency to milk are recognised by the Milk Regulation Board of Connecticut, namely, (1) direct irradiation of milk; (2) addition of a concentrate of cod-liver oil; (3) production of milk by cows fed with irradiated yeast. These products are sometimes referred to as "irradiated," "fortified" and "metabolised" milks, respectively. In June, 1935, the Milk Regulation Board charged the Dairy and Food Commissioner with the duty of a systematic inspection of these products offered for sale in the State.

Labelling regulations for vitamin *D* milk require only that the process of production be designated on the bottle cap. The regulations recognise the prevailing commercial practice for the three types of products, and the types are judged accordingly. Thus irradiated milk should contain not less than 135 units per quart; cod-liver oil concentrate milk (or milk fortified with activated ergosterol), 400 units; and milk produced by yeast feeding, 430 units. The relative nutritional efficacy of these different types and potencies is not clearly established at this time, but it is sufficient to say that all are effective for the prophylaxis of rickets.

In conducting the tests on market samples of vitamin *D* milk, the milk is fed to white rats which have been maintained, for about three weeks after weaning, on a diet that will cause them to develop a marked condition of rickets. The amount of milk fed varies according to the potency expected and required for the type of milk fed, and the vitamin *D* content or activity of the sample is judged by the degree of healing in the bones of the animals. Under these conditions, satisfactory healing (recalcification of the bones) should be produced by all three types of milk.

VINEGAR.—Section 2456 of the Connecticut General Statutes provides that no vinegar may be sold or offered for sale as cider vinegar if it is not produced wholly from the juice of apples. It also provides that no drug, or any hurtful or foreign substance, or any colouring matter, or any acid, may be added to vinegar; and that no vinegar may have an acidity of less than 4 per cent. by weight of acetic acid. Section 2457 provides that vinegar shall be labelled to show the kind or identity of the product. Regulations provide that natural vinegars may be diluted to legal strength of acidity if the fact of such dilution is declared; and that dilute acetic acid is not vinegar and may not be sold as such; but it may be sold under its correct name for food purposes if it is free from harmful impurities.

A vinegar may be made from any alcoholic liquid which is subjected to the action of the acetic-acid-forming organism, which organism is present in the so-called "mother-of-vinegar." Vinegars are named according to the source of the alcohol from which acetic acid is produced. Cider and wine vinegars are derived from the alcoholic and subsequent acetous fermentations of apple and grape juices, respectively. Malt or beer vinegar is made by similar fermentations of malted barley or other malted cereals. Similarly, glucose vinegar is derived from glucose solutions, and sugar vinegars from solutions of sugar refiners' syrup or molasses. Vinegar may also be made from distilled alcohol, and such is known as spirit vinegar, or distilled vinegar.

As distilled vinegar should contain little or no formic acid, and commercial acetic acid contains considerable amounts, a formic acid determination has been used to show the presence of diluted commercial acetic in vinegars. Formic acid in excess of 10 mg. per 100 ml. is enough to cause suspicion, at least, if the article is sold as cider vinegar. Cider vinegar itself may apparently yield some formic acid by the procedure employed. This determination is obviously of no value as a diagnostic measure when commercial acetic acid of the purer grade is used.

As the transformation of alcohol into acetic acid is likely to be incomplete in the process of making distilled vinegar, a determination of alcohol is helpful in indicating the source of acetic acid in suspected samples.

Pratolongo (*Ann. Chim. Applic.*, 1925, **15**, 72) suggested the determination of oxygen value and iodine value as a means of distinguishing between dilute

acetic acid and distilled vinegar. Schmidt (*Z. Unters. Lebensm.*, 1935, **69**, 472; Abst., ANALYST, 1935, **60**, 705) pointed out the necessity of removing caramel before applying these tests. It was found, however, that the iodine values were not sufficiently distinctive to be of service. For example, distilled vinegar without caramel colour gave an iodine value of 23, and with caramel added a value of 24 was obtained. Dilute acetic acid without caramel gave an iodine value of 19, and with caramel a value of 23.

The oxygen value is somewhat more indicative, provided the values are low; if they approach those given by distilled vinegar, the conclusions are again uncertain. The oxygen value was determined as follows:

> Take a quantity of vinegar sufficient to yield a solution of 3 per cent. acidity when diluted to 100 ml. To such quantity add 1 g. of "Norit," dilute to a volume of 100 ml. shake for 2 minutes and filter. Take 50 ml. of the filtrate, add 2 ml. of 1:1 H_2SO_4, heat just to boiling and titrate with $N/10$ $KMnO_4$ until the pink colour developed lasts for half a minute. The volume in ml. of $N/10$ $KMnO_4$ consumed, corrected for a blank determination made with distilled water carried through the same procedure, is the oxygen value of the vinegar.

The end-point is fugitive, except with uncoloured dilute acetic acid, and cannot be determined with the same degree of accuracy as in inorganic permanganate determinations.

Trials of distilled vinegar and dilute acetic acid showed values of about 5 for the former and zero for the latter. The method cannot be applied to cider vinegar because the end-point cannot be read to even approximate accuracy owing to the large amount of manganese dioxide formed. Cider vinegars give oxygen values well over 100.

In the examination of commercial vinegars, in only one instance did the oxygen value clearly point to the use of dilute commercial acetic acid. In several other instances the inspection evidence pointed in the same direction, but the oxygen values did not support that conclusion.

RUBBING ALCOHOL.—Alcohol for massage purposes should be grain alcohol rendered unfit for beverage purposes by the addition of suitable chemicals. Acetone, boric acid, diethylphthalate and zinc phenolsulphonate are commonly used as denaturants. Section 2674 of the Connecticut General Statutes makes the use of methyl (wood) alcohol in this and other toilet preparations unlawful.

Twenty-two samples were submitted by the Dairy and Food Commissioner. None of them contained methyl alcohol and no denaturants other than those mentioned were detected. The preparations were declared to contain not less than 70 per cent. of alcohol, and that amount was met or exceeded in all except three of the samples.

FLUID EXTRACT OF ERGOT.—The U.S.P. procedure requires assays of ergot preparations to be made by the cockscomb method. As the Station laboratory has no facilities for carrying out such a technique, a number of samples were examined by the chemical method discussed in *J. Assoc. Official Agr. Chem.*, 1933, **16**, 387; 1934, **17**, 453. The standard used was ergotoxine ethanesulphonate obtained through courtesy of the U.S.P. XI Revision Committee. This chemical procedure is supposed to give results comparable with those obtained by the cockscomb method. The U.S.P. XI standard is not less than 0·5 mg. of ergot alkaloids per ml. of fluid extract.

Eight samples of fluid extract, representing as many manufacturers, were tested, and the results ranged from 0·00 to 0·38 mg. per ml. Three preparations assayed 0·37 to 0·38; three others, 0·11 to 0·22; and two showed 0·005 and 0·00, respectively.

Since the official method of assay was not used, the products were not judged as to their compliance with U.S.P. requirements.

FRUITS OF WILD PLANTS.—Four samples of the fruits of wild plants, upon which birds and other forms of wild life feed, were examined, and the following results were obtained:

	Moisture		Ash		Protein*		Fibre		*N*-free extract		Crude fat (ether extrac	
	A	B	A	B	A	B	A	B	A	B	A	B
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent
High bush cranberry ..	47·32	5·9	1·70	3·03	2·66	4·75	6·32	11·28	37·07	66·16	4·93	8·8(
Japanese barberry ..	27·58	4·80	2·27	2·98	11·03	14·50	6·13	8·06	44·93	59·06	8·06	10·6(
Red-berried nightshade	11·21	4·50	4·76	5·12	15·98	17·19	18·19	19·57	36·54	39·29	13·32	14·3:
Ibota privet	24·14	2·83	2·73	3·50	8·49	10·88	9·13	11·70	44·66	57·19	10·85	13·9(

A. As received. B. Air-dry material. * $N \times 6{\cdot}25$.

FOOD ADVERTISING.—In 1929 the American Medical Association established a Committee on Foods, "for the purpose of preventing or discouraging unwarranted incorrect or false advertising claims in the promotion of food products, and thus protecting the public and the medical profession against deception." The name of this Committee was subsequently changed to "Council on Foods." The Council works in harmony with federal agencies delegated to administer the Federal food law on misbranding, etc., and its scope is essentially in the field of collateral advertising involving "health" and nutritional claims. The work of this Council has been welcomed both by food manufacturers and advertising agencies, and many advertising programmes have been revised and brought into accord with its rules and regulations.

The Council has no authority to impose its decisions, but it grants manufacturers or distributors of food products authority to use its seal when all its requirements have been complied with. This seal is not a guarantee or recommendation of a product, but signifies that the product, its label and all advertising matter relating thereto have been considered by the Council and nothing contrary to its rules has been discovered.

A few of the conclusions adopted by the Council are as follows:

Good food advertising.—Proper food advertising should use the common name of the food concerned, or in the case of a fanciful trade name should identify the ingredients in the order of their decreasing proportions in the product. Such practice prevents deception. Any statement of the physical, chemical, nutritional or physiological properties and values of the food should be truthful and expressed in simple common terms. Proper advertising is free from false implications.

Food advertising claims with scientific or technical significance.—Statements or claims in food advertising with technical, scientific, nutritional, physiological or health significance shall be carefully phrased so as to be in complete accord with established knowledge and authoritative opinion, and shall be free from misleading or incorrect popular implications or interpretations.

Sleep-inducing claims for specific foods.—Sleep-inducing claims are not permissible for specific food beverages, because of their misleading character implying the possession of unique sleep-inducing properties by the specific individual foods, and because they lead to grossly deceptive advertising practices. No objection is taken to statements averring the relaxation value of hot drinks at bedtime for inducing sleep and accompanied by recommendation of the particular food drink for this purpose.

Mastication not an aid to health of teeth and gums.—Claims that the mastication of specific foods "keeps the teeth and gums clean and healthy," and equivalent statements, are meaningless, misleading and deceptive by implication and are not permissible.

Use of term "sterile."—The terms "sterile," "sterilised" and "sterilisation" shall be used in food advertising in their correct significance only. Foods processed to be free from pathogenic organisms or to keep sound and wholesome are not necessarily sterile, *i.e.* free from viable micro-organisms.

Tonic claims.—The term "tonic," or its inflected forms, have vague and misleading meanings or implications in food advertising and are not permissible.

Gelatin not an aid to the digestibility of milk.—There is no satisfactory evidence that gelatin increases the digestibility of milk or milk products. Such claims are not permissible.

Vitamin E claims.—Statements or claims referring to vitamin *E* in advertising to the public imply a need for special sources of the vitamin that is not warranted by present knowledge. Neither claims for vitamin *E* nor mention of the vitamin shall appear on food labels or in advertising.

Blood-building claims in advertising.—Anaemia is a condition in which the blood is deficient in haemoglobin. It may be due to an inadequate diet, but pathological conditions are frequently involved. Blood-building claims should be excluded from food advertising.

Acidosis claims.—The terms "acidosis," "acidity" and "acid" are frequently used in advertising, to play on vague fears of the public. Acidosis is a medical name for a morbid condition in the reserve supply of fixed alkali in the blood and body fluids. The term "acidosis" is so little understood by laymen that its use is misleading.

Whole wheat and Graham foods.—The terms "whole wheat," "entire wheat" and "Graham" as applied to flour and to bread are synonymous. In harmony with this understanding, these terms shall be used as food names, or as parts of food names, only when the sole cereal and farinaceous ingredient is whole wheat. Their use as names for foods with other composition is misinformative and misleading. Descriptive food names shall correctly and properly identify the nature of the foods.

"Resistance" claims in food advertising.—"Resistance" depends on many factors other than diet or any one dietary essential. Insufficiency of a dietary essential may eventually break down health; but more than is necessary of one or more of these essentials for adequate body reserves does not lead to a "super-resistance." "Resistance" produced by adequate nutrition is not to be confused with immunity resulting from antibodies in the body fluids produced by the body cells in their defensive reaction against pathogenic organisms and their toxins. Food advertising should conform to this established knowledge.

Medical Research Council

THE USE OF THE DEVELOPING EGG IN VIRUS RESEARCH*

THIS report deals with a method which has proved of much value in the study of human and animal diseases due to filter-passing viruses—a method which may come to have still greater importance in research work and in practical applications to prevention and treatment. The chorio-allantoic membrane of the developing chick was first used by Rous and Murphy in 1911 for studies of fowl tumours, and since then has been applied to tumours of other sources. In 1931 Goodpasture and Woodruff showed that this egg membrane was very suitable for the growth and study of a number of viruses. The technique was modified in 1933 by Burnet, who introduced the device of transferring the natural air-space from the butt end to the side of the egg, thus providing a relatively large area of susceptible membrane over which the virus can be spread. The success of the method is due to the chorio-allantoic membrane possessing the essential attributes of a living body, and to the fact that the resistance of the embryo is imperfectly developed, so that viruses to which chickens and fowls are immune multiply freely.

The list of viruses which can infect this membrane is so long and varied that it comes almost as a surprise to find that certain viruses cannot grow upon it. The technique is described in detail, the eggs must be fertile, and they must be given a preliminary incubation, usually of 12 days, before inoculation is made. A triangular window is cut in the shell on the side of the egg, the shell membrane is pierced with a needle, lifted from the chorio-allantoic membrane so that a small slit can be made in the former without damage to the latter. After inoculation a rim of paraffin-vaseline is built up round the triangle, a warm sterile coverslip is placed over the opening, the egg is re-incubated, usually for three days, and then examined.

Comparison of the effects of different viruses show remarkable specificity; some viruses, such as vaccinia and psittacosis, produce lesions mainly on the membrane, others (*e.g.* fowl plague and vesicular stomatitis) kill the embryo before characteristic foci on the membrane develop, others, again, such as louping ill and the influenza viruses, produce both local lesions and general effects on the embryo, whilst the viruses of foot and mouth disease, poliomyelitis and rabies have no effect at all.

* Medical Research Council Special Report Series, No. 220. By F. M. Burnet.

By means of the developing egg (1) quantities of bacteria-free virus-containing material can be obtained for experimental work or for immunisation; (2) with suitable viruses it is a satisfactory experimental animal for virus titrations in the case of lethal viruses by determination of the minimum quantity causing death of the embryo and therefore containing at least one virus particle, or in the case of pock-producing non-lethal viruses by noting the number of specific foci produced by a known volume; (3) the study of the virus inactivating properties of immune sera can be made upon it, the inoculation of virus + immune serum being without effect. (4) In certain instances the method can be used as a diagnostic agent, as in differentiating foot and mouth disease and vesicular stomatitis; and (5) by the facility it offers for the propagation of such host-specific viruses as human influenza and mouse ectromelia it should be of value in investigating any infectious disease of unknown aetiology.

It is of interest to note that with the influenza virus the membrane lesions, almost unrecognisable at first, increased in size with egg-passage, and that from the 50th passage titration by pock counting could be made; after the 52nd passage there were indications of harmful effects on the embryos which died between the 4th or 5th day. The time of death was progressively earlier with further passage, and embryos have been found dead in 44 hours at the 76th passage. The virulence of the egg passage strain for ferrets fell so far as to produce a slight rise in temperature only in two out of five as the only manifesting symptoms, yet this virus immunised the ferrets solidly against virulent virus. This loss of virulence with retention of immunising power suggests the possibility of producing on the same lines an innocuous virus which may be used to confer immunity on human beings by intra-nasal administration. D. R. W.

British Standards Institution

BRITISH STANDARD SPECIFICATION FOR DENSITY BOTTLES

A SPECIFICATION for Density Bottles (B.S.S. No. 733—1937)* has been issued by the British Standards Institution, and is intended to complete the series of specifications relating to methods of determining the density of a liquid. The specification for Density Hydrometers was recently issued (*cf.* ANALYST, 1937, 129).

The capacity of a density bottle, standard at 20° C., is defined by the volume of water of temperature 20° C., expressed in millilitres, required to fill the bottle at 20° C.

Part II of the present specification gives precise details for determining the weight of liquid required to fill the bottle at 20° C., and thence the weight of liquid occupying unit volume at 20° C. From this the density—mass per unit volume—of the liquid at 20° C. in grams, per millilitre, is obtained by adding a small buoyancy correction. Directions are also given for obtaining the density at a temperature (t° C.) other than 20° C.

* Copies of these Specifications may be obtained from the Publications Department, British Standards Institution, 28, Victoria Street, London, S.W.1, price 2s. 2d.

BRITISH STANDARD SIZES FOR PAPER

(WRITINGS AND PRINTINGS, WRAPPINGS AND CASINGS, AND TRIMMED BOARDS)

B.S.I. SPECIFICATION, No. 730—1937.* (*Approved by the Chemical Divisional Council, and published under the authority of the General Council, April 12th,* 1937.)

The list of British Standard sizes for paper given in this specification has been prepared at the request of the Australian Standards Association with a view to its adoption ultimately as a British Empire Standard list of sizes. The Committee appointed prepared a list of sizes based very largely on the work carried out several years ago by the Federation of Master Printers and Allied Trades of the United Kingdom of Great Britain and Ireland, and this list was given a very wide circulation, both in the Dominions and in this country, for one year.

In considering the comments received, the Committee has ruled out any proposal which involved more than one size for one name, as being contrary to the interests of industry. The British Standard list of sizes now issued covers the sizes used in this country and the Overseas Dominions, with the exception of India. With regard to Canada, they lean towards the sizes common in the United States. As the Indian sizes differ somewhat, the Indian authorities have been invited to go into the matter in detail with the object of a review in two years' time, when it is hoped it may be possible to obtain agreement to the recognition of the list as a British Empire Standard. In the meantime the Committee is recommending that Australia, South Africa and New Zealand issue the list as their own local standard during these two years.

Appendix A tabulates certain sizes in which official and other publications have been issued for a great number of years. They can only be obtained if a sufficiently large order justifies their being made.

Appendix B deals with the possibility of issuing recommendations with regard to the standardisation of listing and invoicing weight and stating substance. As various sections of industry differ in their views on these points, no specific recommendations are made, but it is suggested that, for writing and printing papers, the following system may eventually prove suitable for adoption. It is used at present by H.M. Stationery Office, and has the approval of the Federation of Master Printers. Consideration of the system is invited with a view to its adoption in two years' time.

(1) *Listing and invoicing weight.*—That the method of listing and invoicing paper in terms of weight, in pounds, should be per 1000 sheets for paper and per 100 boards.

(2) *Packing.*—That paper should be packed in 500 sheets and boards in 100's.

(3) *Indication of substance.*—That the substance of paper and boards should be indicated in terms of grams per square metre, and that the substance as well as the weight should be shown on all packages, the weight of 1000 sheets being exclusive of wrapper and string.

NOTE.—The substance of a paper has in the past been stated in terms of weight of a ream of 500 sheets (or other quantity, *e.g.* 472, 480, 504, 508, 512 or 516 sheets) of the size concerned, the stated weights of paper similar in substance varying directly in relation to the area of the sheets and the number of sheets in the ream.

In order to have a simple method of making comparisons with regard to substance, no matter what the size of the paper or the number of sheets in the package, a system of symbols has been adopted and is in use by H.M. Stationery Office, by means of which immediate comparisons can be made. In terms of grams per square metre (a system similar to that in use in some Continental countries) a simple method is afforded for the convenience of both the supplier and the user. This is preferable to a system of substance numbers based upon the weight of a package or other quantity of each size of paper. The weight of 1000 sheets must be in accordance with that of the specified substance expressed as grams per square metre of the paper, excluding wrapper and string. For instance, in the case of demy, $17\frac{1}{2} \times 22\frac{1}{2}$ in.—100 grams per square metre = 56 lb. per 1000 sheets.

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* Copies of these Specifications may be obtained from the Publications Department, British Standards Institution, 28, Victoria Street, London, S.W.1, price 2s. 2d.

International Union of Chemistry

THE CLASSIFICATION OF QUALITATIVE ANALYTICAL REACTIONS

THE Committee* appointed by the International Union of Chemistry for the study of new analytical reagents, which made a critical examination of existing data on the sensitivity (limit of identification) and selectivity of tests of identity, at its meeting in Paris, May, 1937, has decided to distinguish between *specific* and *selective* reactions (and reagents) and recommends this convention for general use. Reactions (and reagents), which under the experimental conditions employed are indicative of one substance (or ion) only, are designated as *specific*, whilst those reactions (and reagents) which are characteristic of a comparatively small number of substances are classified as *selective*. From this it follows that it is permissible to describe reactions (or reagents) as having varying degrees of selectivity; on the other hand, a reaction (or reagent) can be only specific or not specific.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS

Food and Drugs

Detection and Estimation of Raw Milk in Pasteurised Milk by the Orla-Jensen Method. F. Stoppel. (*Z. Unters. Lebensm.*, 1937, **73**, 327–329.)—The "creamometric" method of Orla-Jensen (*Z. Unters. Lebensm.*, 1932, **63**, 300; Abst., ANALYST, 1932, **57**, 383) is modified to enable it to indicate the presence of small amounts of raw milk in pasteurised milk. The amount of sample taken is increased six times and is diluted to four times its original volume, instead of twice, and the time allowed for separation of the cream is increased to 20 hours. The dimensions of the apparatus are correspondingly modified. The procedure is as follows:—The milk (30 ml.) is heated for 5 minutes at 50° C. in a 100-ml. flask and then quickly cooled. The flask is completely filled with water and connected by means of a piece of rubber tubing, about 5 cm. long, with the lower end of a Hoyberg butyrometer tube also filled with water. The connection should be perfectly water-tight, but the inclusion of a small bubble of air (about 1 ml.) facilitates mixing. The apparatus is inverted several times until the contents (120 ml.) are thoroughly mixed. The apparatus is then allowed to stand at a temperature of 12 to 15° C., for 20 hours, after which the depth of the cream layer in the butyrometer is read. The factor C of Orla-Jensen (*loc. cit.*) is calculated by multiplying the depth of the cream layer by 4 (the dilution), and dividing by the fat-content of the undiluted milk. The factor A of Orla-Jensen, being of less importance for the purpose of this determination, is not determined. When amounts of 5, 10, 15, 20 and 30 per cent. of raw milk were added to a pasteurised milk of fat-content 3·0 and original C-value 3·3, the C-value was altered to 8·0, 27·0, 33·3 and 53·0, respectively. The average values of the factor C for additions of 0, 5, 10, 15, 20 and 30 per cent. of raw milk (the ranges being given in

* W. Böttger (Leipzig), F. Feigl (Vienna), A. S. Komarovsky (Odessa), C. J. Van Nieuwenburg (Delft), N. Strafford (Manchester).

brackets) are:—2·8 (2·0 to 4·0), 6·0 (4·0 to 9·3), 16·4 (3·9 to 22·7), 28·8 (22·7 to 34·0), 41·1 (32·0 to 47·0) and 55·9 (47·4 to 64·0). The advantages of the method are its convenience, the absence of chemical operations, and the absence of the personal error associated with methods depending upon colorimetric or turbidimetric determinations. A. O. J.

Bromine in Argentine Flours and Wheats. J. Viggiano and E. F. H. Türk. (*Anal. Assoc. Quim. Argentina*, 1936, **24**, 131–134.)—The method used for determining bromine in flours and wheats was as follows:—Ten g. of the finely-ground sample are mixed in a platinum or nickel crucible with water and 5 ml. of 10 per cent. sodium carbonate solution, and the mixture is heated over a small flame until carbonised. The residue is ashed at dull red heat in a muffle furnace; it is important to obtain a carbon-free ash, as otherwise negative results are obtained with small quantities of bromine. The residue is taken up in 10 ml. of boiling water and filtered. Five ml. of the filtrate are acidified with conc. sulphuric acid (6 drops) and the Denigès–Chelle reaction is applied by adding 4 drops of conc. hydrochloric acid, 4 drops of a 10 per cent. potassium chromate solution, and 1 ml. of conc. sulphuric acid and mixing. The liquid is cooled in water for at least 5 minutes, treated with 1 ml. of sulphuric magenta reagent (prepared by adding 10 ml. of a freshly-made 0·1 per cent. solution of basic fuchsin to 100 ml. of dilute (1 : 20) sulphuric acid) and 1 ml. of chloroform (washed with water to remove traces of alcohol), and shaken vigorously for 1 minute. A violet colour in the chloroform layer indicates the presence of bromine. The bromine is estimated by comparing this colour with a series of standards prepared by the above-described method from a potassium bromide solution, 1 ml. of which is equivalent to 0·0125 mg. of bromine (0·0186 g. of potassium bromide per litre) (Chelle and Vitte, *Ann. Falsif.*, 1936, **29**, 98; Abst., ANALYST, 1936, **61**, 343). A blank reaction is carried out. The following experimental results are given:

Region from which the wheat was obtained	Bromine		
	Whole wheat mg. per kg.	Flour mg. per kg.	Bran mg. per kg.
Basavilbaso	6	4	6
Necochea	2·5	2·5	2
Córdoba	6	5	6
Tandil	4	4	4
Lincoln	4·5	2·5	4
Concordia	6	4·5	7
Tres Arroyos ..	2·5	3·5	2·5
San Francisco ..	4	4·5	4
Agustina, F.C.P. ..	2	2	2
Chacabuco, F.C.P. ..	2	2	2·5
Average ..	3·95	3·45	4·00

E. M. P.

Extraction and Preliminary Examination of Various Coloured Compounds in Red Wines. A. Dangoumau and G. Debordes. (*Bull. Soc. Chim.*, 1937, **4**, 910–911.)—Fifty ml. of red wine were extracted continuously with

ether for 8 days, during which time the original liquid diminished in volume to about 3 ml. of a deep red liquid soluble in water. Ether did not extract the colour from this liquid, whilst alcohol removed part of it. The alcoholic and aqueous fractions were examined spectrographically, the spectra being identical with that of the original wine. After long standing, the extract formed two layers, the ethereal layer being bright yellow. The experiment was repeated, 150 ml. of the wine being extracted with ether. After distillation of the solvent there was left a brick-yellow substance soluble in ether. Petroleum spirit did not extract colouring matters from small quantities of wine, but removed a yellow substance from large quantities. E. M. P.

Identification of Wine Vinegar. A. Schmidt. (*Z. Unters. Lebensm.*, 1937, **73**, 441–447.)—As described previously (*Z. Unters. Lebensm.*, 1935, **69**, 472; Abst., ANALYST, 1935, **60**, 705) the "oxidation value" of wine vinegar is the number of ml. of $N/10$ potassium permanganate solution required to impart a red colour persisting for 2 minutes to 50 ml. of vinegar acidified with sulphuric acid. For wine the value is 50 or more, whilst for wine vinegar of acidity 9·2 to 9·4 per cent. the value is about 30, and for wine vinegar of 3 per cent. strength it varies from 8 to 12. The iodine value (Schmidt, *loc. cit.*) is determined by adding excess of $N/100$ iodine solution to 25 ml. of vinegar which has been made alkaline, allowing the mixture to stand for 15 minutes, acidifying with hydrochloric acid, and titrating the excess of iodine with $N/100$ sodium thiosulphate solution. With concentrated wine vinegar about 400 ml. of $N/100$ iodine solution must be used, since the iodine value is extraordinarily high, and for wine vinegar of acidity 3 per cent. 150 ml. are required to provide the necessary 25 per cent. excess of iodine solution. The iodine value of wine is 150 to 250, that of concentrated wine vinegar about 300, and that of the weaker wine vinegar 90 to 100. No other form of vinegar has so high a value. Since the iodine value of wine vinegar is about double that of the wine from which it is prepared, it follows that substances absorbing iodine are formed during the acetic fermentation. The author has previously stated that the difference between the iodine values of wine and spirit vinegars is small. This statement, based upon the examination of a few unauthenticated commercial samples, is now found to be erroneous and, by the investigation of a number of fully authenticated samples of wine vinegar, the iodine value is shown to be a very important distinguishing character. The determination must not be carried out by the method of Pratolongo (*Ann. Falsif.*, 1933, **37**), which involves distillation, because not all the substances reacting with iodine are volatile. Acetylmethylcarbinol, $CH_3COCH(OH)CH_3$, present in wine vinegar, is formed during acetic fermentation by the condensation of two molecules of acetaldehyde brought about by the enzyme carboligase. It may be detected by conversion into diacetyl which, in turn, may be converted into nickel dimethylglyoxime, but the simplest method for its detection and determination is a modification of that of Arbenz and Pritzker (*Mitt. Lebensmittelunters.*, 1931, **22**, 354) depending upon its power of reducing cold Fehling's solution. The cuprous oxide so formed is transferred to a narrow centrifuge tube, in which it is washed by centrifuging with water, alcohol and ether, dried at 30 to 40° C., and weighed.

The smallest amount found in wine vinegar of 3 per cent. acidity was about 60 mg. per litre; in wine vinegar of 9 per cent. acidity it reached 190 mg. per litre. In a mixture of equal parts of wine and spirit vinegars the amount found was 38 mg. per litre and, since acetylmethylcarbinol does not occur in spirit vinegar, the same result would have been obtained if the diluent had been water. The minimum values suggested for wine vinegar of strength corresponding with 3 per cent. of acetic acid are:—oxidation value, 8 ml. of $N/10$ potassium permanganate solution; iodine value, 90 ml. of $N/100$ iodine solution; acetylmethylcarbinol-content, 60 mg. per litre. The values found for samples of wine vinegar of strength corresponding with 9 per cent. of acetic acid were:—oxidation value, 30 to over 40; iodine value, 240 to 300; acetylmethylcarbinol-content, 140 to 193. The acetylmethylcarbinol-content is particularly useful for the detection of the adulteration of wine vinegar with spirit vinegar. The glycerol-content and the extract of wine vinegar vary considerably, and the ester value does not serve as a useful criterion.

A. O. J.

Detection of Oils derived from the *Cruciferae* in Edible Oils. J. Grossfeld. (*Z. Unters. Lebensm.*, 1937, **73**, 409–426.)—Methods depending upon the separation of erucic acid (Holde and Marcusson, Abst., ANALYST, 1910, **35**, 401; Thomas and Mattikow, *J. Amer. Chem. Soc.*, 1926, **48**, 968; Täufel and Bauschinger, *Z. angew. Chem.*, 1928, **41**, 157; Kimura, Abst., ANALYST, 1930, **55**, 645; Bertram, *Z. Unters. Lebensm.*, 1928, **55**, 179) have the disadvantage that the separation is tedious and difficult. Tortelli and Fortini (Abst., ANALYST, 1910, **35**, 401) used the iodine value of the solid fatty acids which give lead salts insoluble in ether. Kreis and Roth (Abst., ANALYST, 1913, **38**, 434) used fractional precipitation of the fatty acids as lead salts. The resemblance of erucic acid to iso-oleic acid in its behaviour suggested that a modification of the method of Grossfeld and Peter (*Z. Unters. Lebensm.*, 1934, **68**, 348; Abst., ANALYST, 1935, **60** 105) for the detection of hardened fats could be applied to the detection of rape oil in other edible oils. The modification consists in the addition, to the oil, of lauric acid, which has the effect of promoting crystallisation, bringing down the erucic acid by adsorption, and compensating for the differences in vegetable oils caused by variations in the content of individual saturated and unsaturated acids. The "erucic acid value" is determined as follows:—About 500 mg. of the oil and 0·6 ml. (500 mg.) of melted lauric acid are saponified beneath a reflux condenser for 10 minutes with 10 ml. of alcoholic potassium hydroxide solution (40 ml. of potassium hydroxide solution of sp.gr. 1·5 with 40 ml. of water made up to 1 litre with 96 per cent. alcohol). To the cooled soap solution are added 50 ml. of lead acetate solution (5 g. of crystalline lead acetate and 5 ml. of 96 per cent. acetic acid made up to 1 litre with 80 per cent. by vol. alcohol), 2·5 ml. of 95 per cent. acetic acid and 10 ml. of water. The mixture is heated beneath a reflux condenser until the lead salts have dissolved, then cooled, and allowed to stand at 20° C. in a stoppered flask, with occasional shaking, for 2 or 3 days. The deposit is filtered off in a sintered glass crucible (Schott und Gen. 10G/3), and washed with 10 to 15 ml. of 70 per cent. alcohol. The crucible is placed in a continuous extractor and extracted with a mixture of

equal parts of 96 per cent. alcohol and 96 per cent. acetic acid. The warm solution of the lead salts is rinsed into a 400-ml. flask with 10 ml. of the alcohol and acetic acid mixture, and the iodine absorption is determined by the method of Margosches, Hinner and Friedmann (*Z. angew. Chem.*, 1924, **37**, 334). For this purpose, 20 ml. of alcoholic 0·2 *N* iodine solution are added, the mixture is well shaken and diluted with 200 ml. of water and, within two hours of the dilution, the excess of iodine is titrated with *N*/10 sodium thiosulphate solution. A blank determination is made upon 30 ml. of the alcohol and acetic acid mixture. The number of ml. of *N*/10 iodine absorbed is the "erucic acid value." By applying the method to known mixtures of olive oil and erucic acid it was found that the relation between the weight of erucic acid in mg. (y) and the erucic acid value (x) is given by the equation $y = 52{\cdot}1x - 0{\cdot}9x^2 - 217{\cdot}2$. Since x is 4·52 when y is 0, titrations of less than 5 ml. are ignored. By comparison of the actual with the calculated values it is found that the erucic acid is determined with an accuracy of ± 11 mg., corresponding with 2·2 per cent. of rape oil. The erucic acid value of pure rape oil is about 12, corresponding with 54 per cent. of erucic acid. A. O. J.

Characterisation of the Acetyl Group in some Medicaments by means of "Lanthanum Blue." A. D. del Boca and A. L. Remezzano. (*Quim. e Ind.*, 1937, **14**, 195–197.)—The formation of a blue adsorption compound of iodine on basic lanthanum acetate is used as a test for the acetyl group. To 1 ml. of the liquid under examination is added 1 ml. of 5 per cent. lanthanum nitrate solution, 1 ml. of *N*/50 iodine solution, and *N* ammonia until the liquid is pale yellow. In the presence of an acetate a blue colour appears in the cold, and there forms a gelatinous precipitate; with small quantities of acetate the colour is violet and appears on warming. The test is not affected by nitrates, chlorides, bromides, or iodides, but phosphates and sulphates must be removed. Oxalates and formates are destroyed during the preliminary distillation (see below), while other organic acids can be separated by distillation. Propionic acid behaves in the same way as acetic acid, and butyric and valerianic acids invalidate the test. The reaction has been applied to heroin, aconitine, aspirin, tannigen, acetanilide, antifebrin and salophen, the following methods being recommended:

Heroin and Aconitine.—A quantity of 0·01 to 0·02 g. of the alkaloid is distilled from a 50-ml. flask with 2 to 3 ml. of 50 per cent. sulphuric acid; half the quantity of liquid is distilled over. Four or 5 drops of the distillate are added to a 5 per cent. lanthanum nitrate solution, and the mixture is treated with *N*/50 iodine solution and *N* ammonia. With small quantities of acetate a ring test should be made, the ammonia being run in down the side of the tube.

Aspirin and Tannigen.—The hydrolysis is carried out with ferric chloride instead of sulphuric acid, the technique being as follows:—A quantity of 0·01 to 0·02 g. of the material is distilled gently with 2 or 3 ml. of 30 per cent. ferric chloride solution until about 2 ml. of distillate have been obtained; the test is applied to this.

Antifebrin, Exalgin, Phenacetin and Salophen.—The procedure described for heroin and aconitine was used; the first three gave positive results, but salophen gave a negative result. E. M. P.

Fluorine-content of Gelatin. L. S. Stuart, D. Dahle and R. W. Frey. (*J. Amer. Leather Trades Chem.*, 1937, 205–210.)—The fluorine-content of samples of commercial gelatin of German, American and British origin was found to range from 4 to 10 p.p.m. The fluorine-content of gelatin made from calfskin cured with salt to which fluorides had been added varied according to the manner in which the skin was washed prior to liming. With thorough washing in running water these gelatins had about the same fluorine-content as gelatin cured with salt alone or as commercial gelatin. Skins washed by still soaking contained more fluorine. It was concluded that an increased fluorine-content of gelatin made from calfskin cured with salt plus fluorides may result from the use of commercial salt containing impurities capable of forming difficultly soluble fluorides, and also from lime containing fluorine used for the purpose of de-hairing. The method recommended for fluorine determination involves separation by a single distillation by Willard and Winter's method (*Ind. Eng. Chem., Anal. Ed.*, **1933, 5, 7**; Abst., ANALYST, 1933, **58**, 242), followed by its determination in the distillate by the peroxide and titanium method (Wichman and Dahle, *J. Assoc. Off. Agr. Chem.*, **1933, 16**, 612; Abst., ANALYST, 1934, **59**, 132). S. G. C.

Commercial Thyme. C. E. Sage. (*Perf. and Essent. Oil Record*, 1937, **28**, 127–128.)—In order to elucidate the very different aroma, which has been more pronounced in recent years, between French and English thyme (*Thymus vulgaris*), seed found in a consignment of imported thyme leaf was germinated, and it was confirmed that the resulting plants, grown in England, were *T. vulgaris*. It is suggested that the difference in scent between foreign and English-grown thyme is analogous to that found in Surrey peppermint grown in this country and in America. The difference would appear to be due to climate. Thyme of Spanish origin may show considerable differences in scent, but this is usually due to the fact that different species often grow side by side, and these contain varying amounts of carvacrol, thymol and citral. Analyses of 15 samples of commercial shipments of dried thyme leaves showed the following variations:—total ash, 6·64 to 13·82 per cent.; acid-insoluble ash, 0·83 to 5·80; essential oil, 0·64 to 1·84 per cent., and $n_D^{20°}$ of the oil 1·491 to 1·512. Traces of arsenic (not usually above 1/100th of a grain per lb.) may be present in dried thyme, and for culinary purposes it is important to free the herb from grit. D. G. H.

Chemical Examination of *Clerodendron infortunatum*. Part I. H. N. Banerjee. (*J. Indian Chem. Soc.*, 1937, **14**, 51–57.)—*Clerodendron infortunatum*, the Indian Bhant or Bhat (N.O. *Verbenaceae*) is a shrub common throughout the warm regions of India from Gurhwal and Assam to Ceylon. The young twigs, ground to a paste and made into pills, are used for the destruction of intestinal worms. A sample of the dried leaf had the following composition:—ash, 8·04; protein, 21·12; crude fibre, 14·84; free reducing sugars, 3·00; and total sugars (after inversion), 17·05 per cent. The air-dried, finely powdered leaves were treated with Prollius fluid, but no alkaloids were found. Extracts of the dried leaf were made with various solvents, and petroleum spirit was found to dissolve 3·85 per cent., giving a deep greenish-yellow, very bitter solution. On concentration, crystals were formed, and these, when re-crystallised from boiling

50 per cent. alcohol, yielded long needles melting at 161 to 162° C., and having a very bitter taste. On steam-distillation of the green pasty residue from the petroleum spirit extract a volatile oil with a strong odour of the drug was obtained. By repeated extractions with 60 per cent. alcohol a further crop of crystals of the bitter substance, to which the name *clerodin* was given, was obtained, the total yield being 0·12 per cent. of the air-dried leaf. After complete removal of the clerodin, and by repeated extraction of the pasty mass with 80 per cent. alcohol, crystals of a sterol were obtained. The dark residue was decolorised in ethereal solution by means of charcoal and fuller's earth, and saponified, the soap was dissolved in water, and the free fatty acids liberated and purified. The "liquid" acids (80 per cent.) consisted of oleic 74·9, and linolenic 25·1 per cent., and the "solid" acids (20 per cent.) of stearic, 48·8, and lignoceric acid, 51·2 per cent. Carotene and xanthophyll were separated from the unsaponifiable matter. The sterols comprised 1·20 per cent. of the oil, and hydrocarbons appeared to be almost absent from the residual unsaponifiable matter, which seemed to consist mainly of aliphatic alcohols. Varying amounts of clerodin were found in different parts of the plant; young leaves and twigs collected before rains contained 0·12 per cent.; just after rains 0·55 per cent.; in old leaves there was 0·05 per cent., and in stems and roots only traces were found. Clerodin had no haemolytic action on human red blood corpuscles, nor had it any bactericidal properties when tested against *B. coli*. The toxicity to earthworms was tested by Sollman's method (*J. Pharm. Exp. Therap.*, 1918, **12**, 129), and the clerodin killed them in aqueous solution within half-an-hour. The toxicity of drugs to earthworms is taken to be more or less parallel to their toxicity to parasitic worms, according to Straub and Tradelenberg, and drugs toxic to earthworms are regarded as possessing possibilities as anthelmintics. Small fish and mosquito larvae were killed in 30 minutes and 2 hours, respectively (Fink and Haller's method, *J. Econ. Entom.*, 1936, **29**, 595), so that clerodin is to be regarded as toxic to the lower forms of life. It was quite harmless to a rabbit fed with 0·1 g. dissolved in 10 ml. of olive oil. Clerodin is soluble in hydrochloric acid, olive and castor oils and glycerol, and slightly so in paraffin oil and vaseline. D. G. H.

Histology of *Podophyllum*. T. E. Wallis and S. Goldberg. (*Quart. J. Pharm.*, 1937, **10**, 40–51.)—This is a detailed account of the histology of *Podophyllum peltatum*, and is to be followed by a similar account of *P. emodi*. Measurements are given for the extreme range, as well as for the average values, of the dimensions of the important cells in each part of the drug. The important features to be sought for in the powder of American podophyllum (*P. peltatum*) are the brown epidermal cells, which are mostly elongated rectangular prisms, and usually 4 to 10 times as long as they are wide. In addition, a small number of similar cells, which are approximately isodiametric, are present, and all of these contain a dark red-brown material containing tannin. Cluster crystals of calcium oxalate are also present, and many of these exceed 60μ in diameter. Starch grains, ranging from 2 to 30μ in diameter and having an average value of 15μ, should also be seen. S. G. S.

Biochemical

Reactions of Amino and Imino Acids with Formaldehyde. M. Levy and D. E. Silberman. (*J. Biol. Chem.*, 1937, **118**, 723–734.)—Further data are given for the reaction of formaldehyde with amino and imino acids. (See also *J. Biol. Chem.*, 1932, **99**, 767; 1934, **105**, 157; 1935, **109**, 365.) The method consisted in the determination of the variation in the apparent dissociation constants of the amino and imino groups with the formaldehyde concentration. The substances reported on are *dl*-alanine, *dl*-valine, *l*-aspartic acid, *l*-tryptophane, *dl*-sarcosine and *l*-hydroxyproline. The results indicate that amino acids may react with one or two molecules of formaldehyde, whereas imino acids can react only with one. The reaction of asparagine with formaldehyde is interesting in that a pyrimidine derivative is formed, but the rate of the reaction is very slow. As a result of this difference between asparagine and other amino acids it is concluded that the usual conception of methylene amino formation is incorrect, and other possibilities are discussed. S. G. S.

Determination of Lactic Acid in the Presence of Methylglyoxal. E. Bauer and F. Ziegler. (*Hoppe-Seyler's Z. phys. Chem.*, 1937, **247**, 1–5.)—In the determination of lactic acid, trouble from interfering substances such as methylglyoxal is frequently encountered. Several workers have suggested different methods for the elimination of these substances, but no method has been really satisfactory. The authors claim that oxidation with hydrogen peroxide in an acid medium prior to the distillation removes the methylglyoxal without affecting the lactic acid. The modified apparatus and manganese sulphate and sulphuric acid mixture of Fürth and Charnass was used with tenfold excess of hydrogen peroxide. Protein in the solution under examination interferes with the method and requires removal with sodium tungstate solution. Five ml. of $N/5$ phosphate buffer and 10 ml. of 10 per cent. sulphuric acid are added to the necessary amount of the solution under examination in a 50-ml. flask, and the whole is treated with 5 ml. of a 10 per cent. solution of sodium tungstate. The volume is adjusted to 50 ml., and the precipitate is removed by centrifuging or by filtration. Forty ml. of the filtrate (4/5 of the original) are pipetted into the distillation flask, and to this is added 10 ml. of the manganese sulphate and sulphuric acid mixture, 10 ml. of water and 0·2 ml. of perhydrol. Broken porcelain is added, and the whole is heated and kept gently boiling for 6 minutes. To remove excess of hydrogen peroxide potassium permanganate solution is added until a permanent pink colour is obtained, and the usual distillation is then carried out. In the absence of protein the test solution is oxidised without previous treatment with sodium tungstate. S. G. S.

Properties of Ricin. S. Inoue. (*J. Soc. Chem. Ind. Japan*, 1937, **40**, 122–123B.)—Ricin was prepared by separating the oil from the flour of *Ricinus communis* L. and extracting the residue with a 10 per cent. solution of sodium chloride at 25° to 30° C. Globulin was then removed by dialysis for 4 days, and the filtered solution was saturated with ammonium sulphate; this caused a bright brown substance to separate, and an aqueous solution of this was dialysed in running

water until no more sulphate ions were removed. The solution was evaporated under a reduced pressure at 40° C., a yield of 2·5 per cent. of ricin protein being obtained. This was shown to be an ampholytic hydrophilic colloid soluble in water or dilute sodium chloride solution, solutions in water being coagulated by heat or by addition of alcohol. It gave positive reactions with the biuret, Millon, xantho-protein, ninhydrin, lead sulphide, Adamkiewicz, glyoxylic acid and diazo reagents for proteins, and a normal sample contained 16·81, 4·63 and 8·66 per cent. of total nitrogen and nitrogen coagulated by heat and tannin, respectively. The iso-electric point was determined by measuring the degree of turbidity produced on adding alcohol to solutions of ricin buffered at *p*H 4·5 to 9·0, and it was found to be 5·4 to 5·6. Examination under the ultra-microscope indicated that the mechanism of the coagulation of blood corpuscles by ricin is associated with the coagulation of ricin itself. The rate of coagulation was measured by adding 0·1 to 1·0 ml. of ricin to 1 ml. of blood at *p*H values ranging from 4·6 to 10·0, and it was shown that coagulation occurs only between *p*H 5·6 and 5·8 and between 8·9 and 9·1 It is therefore concluded that the so-called toxicity of ricin is mainly a colloidal phenomenon, and that determinations of such toxicity are valueless unless reference is made to the *p*H value. J. G.

Estimation of Total Vitamin *C* in Foodstuffs. P. N. Sen-Gupta and B. C. Guha. (*J. Indian Chem. Soc.*, 1937, **14**, 95–102.)—The following methods have been investigated: (1) treatment with trichloroacetic acid, (2) leaving in contact with trichloroacetic or hydrochloric acid, (3) heating in carbon dioxide or nitrogen for different periods, (4) treatment with hydrogen sulphide in the cold, and (5) treatment with hydrogen sulphide in the hot condition. The last method gave the highest value for ascorbic acid, and appears to give the total amount present, comprising the free vitamin, the ascorbic acid which is released by heating and the reversibly oxidised ascorbic acid. The possibility of this treatment releasing some non-specific reducing substance is guarded against by the addition of formaldehyde to the dye. This method is carried out by cutting 10 g. of the material into small pieces, and adding it to 50 ml. of water. Hydrogen sulphide is passed into this solution for 30 to 60 minutes, and, while the gas is still bubbling through the liquid, the whole is heated for 15 minutes on a boiling water-bath under a reflux condenser. The hydrogen sulphide is then removed by means of a current of carbon dioxide or nitrogen, and the suspension is treated with 2·5 ml. of a 20 per cent. solution of trichloroacetic acid. The mixture is filtered or centrifuged, and the filtrate is made up to 100 ml. This solution is used for the titration of 0·5 ml. of *M*/10 2:6-dichlorophenolindophenol solution to which 1 ml. of *M* formaldehyde and 1 ml. of glacial acetic acid have been added. The titration should be completed within one minute. S. G. S.

Vitamin *C* in Vegetables. Critical Investigation of the Tillmans Method for the Determination of Ascorbic Acid. G. L. Mack and D. K. Tressler. (*J. Biol. Chem.*, 1937, **118**, 735–742.)—The extraction of ascorbic acid from vegetables has been carried out by substituting a strongly ionised acid for the more usual acetic or trichloroacetic acid of the Tillmans method. It is claimed that with the lowered *p*H value, oxidation does not take place, and if, in addition,

metaphosphoric acid is added, the inactivating effect of copper is eliminated. Sulphuric acid of strengths ranging from 5 to 15 per cent. is advocated, and it is also probable that hydrochloric acid may be equally successful. The use of these mineral acids eliminates the hydrogen sulphide treatment for all but exploratory work, and this greatly simplifies the procedure. Substances other than dehydroascorbic acid may be reduced by prolonged hydrogen sulphide treatment, and the strongly ionised acid prevents the interfering material from reacting with the titration reagent. The apparent increase in ascorbic acid on heating an aqueous extract, such as that from cabbage, is ascribed to the decomposition of the dehydroascorbic acid. S. G. S.

Estimation of Vitamin B_1. A. Schultz, L. Atkin and C. N. Frey. (*J. Amer. Chem. Soc.*, 1937, **59**, 948–949.)—Determinations were made of the volume of carbon dioxide liberated in 3 hours by the fermentation of dextrose in the presence of various known amounts of vitamin B_1. For each test, 1 g. of yeast and 100 ml. of an aqueous solution containing synthetic salt mixture, buffer, and 3 g. of Merck's dextrose were used. The temperature was 30° C., and the oscillations were 100 per minute. Results with Merck's natural crystalline vitamin B were:

Merck's natural crystalline vitamin B_1 mg.	Ml. of gas in 3 hours
None	185
0·001	215
0·005	305
0·010	350
0·040	395
0·100	405

Determinations of vitamin B_1 based on these results and on the rat-growth test agreed well. Synthetic vitamin B_1 (Merck's vitamin "Betabion") gave results almost identical with those for the natural product. E. B. D.

Toxicological and Forensic

Some Forensic Aspects of Dermatitis. H. E. Cox. (*Medico-Legal Review*, 1937, **5**, 123–133.)—Study is made of the record of about 5000 cases of dermatitis, arising from furs or textiles, the results being considered in relation to idiosyncrasy and "reasonably fit" as mentioned in the Sale of Goods Act. Sensitisation by means of organic compounds is discussed in relation to cosmetics, hair-dyes and perfumes. The incidence of dermatitis due to dyed furs is about 1 in 10,000, and to dyed textile fabrics about 1 in a million. (See also *Chem. and Ind.*, 1937, **56**, 568.)

Bacteriological

Gastro-enteritis associated with *Proteus vulgaris*. J. D. A. Gray. (*Brit. Med. J.*, May, 1937, 916–917.)—An outbreak of food poisoning affecting at least 18 persons occurred in Avonmouth. All had eaten cockles sold by an

itinerant vendor, and only one person who had eaten any of the cockles escaped illness. The incubation period was 3 to 6 hours. Only two of those affected were seriously ill; these took 7 to 10 days to recover, whilst the rest had practically recovered within 24 to 48 hours. The cockles were prepared under very unhygienic conditions; during part of the process of preparation they were kept in salt and water in a galvanised iron bath, which was also used for the family ablutions. Unfortunately, none of the suspected batch of cockles and no vomits were available for bacteriological examination; faeces, however, were examined, and no enteric, dysentery or food-poisoning bacilli were found, but two cases (2 and 3) showed *Proteus vulgaris*. The blood of cases 2 and 3 did not agglutinate any of the Salmonella or dysentery bacilli, but agglutinated several strains of *Proteus vulgaris* to a titre, the one up to, and the other considerably over, the normal limit recognised for the Weil Felix reaction in the diagnosis of typhus fever, and strains isolated from cases 2 and 3 to a titre of over 1–125. The same titre of agglutination was also shown by a strain of this bacillus that was isolated from the rinsings of the bath. It is suggested that this evidence is sufficient to assign the cause of the outbreak to *Proteus vulgaris* Cases of food poisoning proved to be due to *Proteus vulgaris* are rare. D. R. W.

Rapid Detection of *B. tuberculosis* in Milk. M. L. C. Maitland. (*Lancet*, May 29, 1937, 1297.)—Reference is made to the observation of Torrence (1927), followed up by Matthews (1931), Davies (1933) and Cowan and Maddocks (1935), that tubercle bacilli in milk are usually associated with a particular kind of cell, the endothelial cell, which generally occurs in characteristic groups, and that tubercle bacilli are found more quickly, more easily and more efficiently by the examination of films for these cell groups with the 2/3 in. objective and subsequently for tubercle bacilli within such groups. The best conditions for the examination of milk on these lines were studied experimentally, the technique finally adopted is described, and the results thus obtained with the milk of some 950 cows by this technique are recorded. The author found that it was advantageous (1) to centrifuge the milk at a fairly high speed, 2500 r.p.m., for three minutes only; (2) to make films from the surface of the deposit by removal with a loop bent at right angles to the wire; (3) to spread the film of deposit with a slide and not with a loop, the cell groups tending thereby to accumulate round the edges of the film; (4) to wash the films with ether-alcohol before staining; and (5) to examine samples from each udder of the cow separately. It is claimed that as many as 40 films can be examined microscopically in one hour (presumably by one person).

The milk of all the cows of 36 farms, the bulk milk of which had previously been shown to contain tubercle bacilli by guinea-pig inoculation, was examined by this method. The results were as follows:—(*a*) In 11 farms, no tubercle bacilli were found in any sample (confirmed by guinea-pig inoculation in 6 farms); there was a history of the disposal of one or more cows since the original sample was taken. (*b*) In 2 farms, cell groups were found, but no tubercle bacilli (tubercle bacilli were found in a second sample from 1 cow). (*c*) In 21 farms, 1 cow, and in 2 farms, 2 cows, gave milk showing tubercle bacilli (confirmed biologically).

In a second series, the milk of all the cows of 26 farms not previously tested was examined. Two farms each showed 2 cows with tubercle bacilli in their milk (confirmed biologically), and the milk of the remainder of the cows of these 2 farms showed no tubercle bacilli biologically. Bulk samples of milk from 16 of the remaining farms showed no tubercle bacilli biologically; the remaining 8 farms were not checked biologically. D. R. W.

Organic

Detection of the -SH Group by means of Organometallic Compounds. H. Gilman and J. F. Nelson. (*J. Amer. Chem. Soc.*, 1937, **59**, 935–937.)—Bismuth triethyl and lead tetraethyl can be used for the detection of the –SH group. They do not react with the hydrogen in –NH and –C ≡ CH groups nor with simple –OH groups (although some strong carboxylic acids undergo generally limited reactions), and there is no interference by azo or nitro groups. Bismuth triethyl was prepared as follows:—To magnesium ethyl bromide solution prepared from 1·4 moles of ethyl bromide in 400 ml. of ether, 200 g. of an ethereal solution containing 0·4 mole of bismuth chloride were added in an atmosphere of nitrogen. (Bismuth triethyl is inflammable in air.) After heating until the reaction was complete, most of the ether was removed on the water-bath by fractionation through an efficient column. The remaining ether and bismuth triethyl were distilled at 4 mm. pressure from an oil-bath, which was heated gradually to a final temperature of 170° C. This distillate was collected in a modified Claisen flask (250 ml.), which was cooled by a mixture of ether and solid carbon dioxide. After distillation of the ether from the distillate, the bismuth triethyl was collected at about 123° C. (150 mm. pressure). *Method of Analysis.*—The usual Zerewitinoff apparatus and method were used, all the work being carried out in an atmosphere of nitrogen. Five ml. of a 25 per cent. (by volume) solution of bismuth triethyl in *n*-butyl ether were run into the side-bulb through a bent pipette with a stopcock. The main bulb, full of nitrogen, was attached to the gas burette, immersed for a few minutes in the water-bath at 25° C., and the system was closed to the atmosphere. The bulb was heated for a definite time on a boiling water-bath, cooled quickly, and then placed again in the water-bath at 25°C. Temperature, volume and pressure readings were made 15 minutes after heating was discontinued. Results (after deduction of blank tests) for numerous compounds, with results for similar experiments with purified lead tetraethyl, are tabulated for various times. They are given as fractions of the reactive hydrogen which reacted in these times. Except for its inflammability, bismuth triethyl is preferable to lead tetraethyl. The latter is highly toxic; bismuth triethyl gives high values for –SH groups and is less interfered with by –OH groups. It is suggested that these organo-metallic compounds may be useful in establishing the existence of thioenolisation in compounds such as thioacetic acid. E. B. D.

Polarimetric Determination of Water in Acetic Acid. G. Toennies and M. Elliott. (*J. Amer. Chem. Soc.*, 1937, **59**, 902–906.)—The amount of water in acetic acid can be determined polarimetrically by allowing it to react with a known amount of acetic anhydride in the presence of a small amount of perchloric

acid and determining the excess of anhydride by the decrease in optical rotation caused by its reaction with *d*-camphoric acid to form *d*-camphoric anhydride. The perchloric acid acts as a powerful catalyst for both reactions. *Method.*—About 0·0065 g.-mol. of *d*-camphoric acid is weighed in a 25-ml. graduated flask, a definite amount of standardised acetic anhydride is added, and the flask is made up to the mark with the solution to be tested, in which a small amount of perchloric acid of known water-content is dissolved. The amount of acetic anhydride should be such that after reaction with the expected amount of water 3·25 m.ml. of acetic anhydride remains for reaction with the camphoric acid. The amount of perchloric acid present is 0·000125 g.-mol. The amount of acetic acid used is determined by weighing. Polarimetric readings of the solution are taken in a 2-dm. tube until the optical rotation becomes stationary. The specific rotation of the *d*-camphoric acid used is determined in 0·25 *M* solution in glacial acetic acid; that of the anhydride is $[\alpha]_{Hg}$—0·8° (wave length 546·1$m\mu$). The probable experimental error is calculated, and the method is considered accurate to at least ±1 per cent. for water concentrations of 0·1 to 1 per cent. Determinations of 0·1 and 0·6 per cent. of water were made within limits of ±0·001 and ±0·008 per cent., respectively.

E. B. D.

Acidimetric Determination of Glycerol and Erythritol by means of Periodates. M. L. Malaprade. (*Bull. Soc. Chim.*, 1937, **4**, 906–910.)—In the author's original method (*Bull. Soc. Chim.*, 1934, **1**, 850), which consisted in oxidising glycerol to formic acid and formaldehyde with sodium periodate, followed by titration of the acid in the presence of methyl red, a preliminary approximate determination had to be effected, and the final determination was made in the presence of only a very small excess of sodium periodate, which is slightly acid to methyl red. This method has now been modified by the addition, before the titration, of conc. potassium nitrate solution, which precipitates the periodate as the sparingly soluble potassium periodate. The method can be still further improved by using solid potassium periodate as the oxidising agent. The procedures are as follows:

(A) *With sodium periodate.*—The liquid containing the glycerol is rendered neutral to methyl red by the addition of strong alkali or acid (hydrochloric acid, which reduces periodic acid, must not be used). An excess of a sodium periodate solution is added, and the mixture is allowed to stand for 20 minutes and then treated with conc. potassium nitrate solution; the precipitation of potassium periodate indicates the presence of an excess of periodate. The acid is then titrated with alkali hydroxide solution until the indicator becomes straw-yellow. The quantity of glycerol is calculated from the relation

$$1\ C_3H_8O_3 \rightarrow 1\ HCOOH \rightarrow 1\ NaOH.$$

(B) *With potassium periodate.*—An excess of solid potassium periodate is added to the neutralised solution containing the glycerol, the mixture is shaken for 20 minutes, and the formic acid is titrated as described above. The method is valid in the presence of salts of strong acids and alkalis which do not reduce or precipitate periodic acid, of substances (such as ethyl alcohol) which do not react with periodic acid in the cold, and of substances (such as glycol) which react with

periodic acid without the production of acids. Very weak bases and acids weaker than formic acid must not be present. The experimental results quoted show that the method is accurate. It can also be applied to erythritol, 1 g.-mol. of which gives 2 g.-mol. of formic acid. E. M. P.

Depot Fat of the Ceylon Lizard (*Varanus Salvator*, Laur.). T. P. Hilditch and H. Paul. (*Biochem. J.*, 1937, **31**, 227–228.)—The abdominal fat of a very young kabaragoya or Ceylon lizard (*Varanus salvator*) has been examined. This carnivorous animal enters water readily and feeds on fish, snakes, birds, eggs, carrion, etc. The golden-yellow fat was almost completely liquid at 20° C., and had saponification equivalent 283·9, iodine value 70·8, acid value 4·5, and unsaponifiable matter 1·6 per cent. The fatty acids (54·6 g.) from the hydrolysed fat were separated by the lead salt and alcohol method. The "solid" and "liquid" acids were separately converted into the methyl esters and fractionally distilled in a vacuum. Expressed as per cent. on the total component acids, the acids consisted of myristic, 4·2; palmitic, 29·3; stearic, 9·8; C_{16} unsaturated, 12·3 (mean unsaturation —2·0H); C_{18} unsaturated, 39·6 (mean unsaturation —2·7H); C_{20} unsaturated 4·8 (mean unsaturation —5·5H). The characteristics of lizard fatty acids, as intermediate between those of land and aquatic animals, is confirmed. The presence of 12 per cent. of palmitoleic acid and of small amounts of C_{20} unsaturated acids shows the resemblance to the "aquatic" type of fat, whilst a high content of saturated acids is what is found in the fat of land animals. D. G. H.

Seed Wax of *Simmondsia Californica*. T. G. Green, T. P. Hilditch and W. J. Stainsby. (*J. Chem. Soc.*, 1936, 1750–1755.)—*Simmondsia californica*, Nutt., N.O. *Buxaceae*, is an evergreen shrub indigenous to southern California and southern Arizona, and the nature of the liquid oil obtained from the seeds has already been found to be exceptional (Greene and Foster, *Bot. Gazette*, 1933, **94**, 826). Four hundred g. of mature seeds were used for the present investigation, and 48 per cent. of a golden-yellow oil were obtained on extraction with petroleum spirit. This yielded approximately equal weights of fatty acids and fatty alcohols. Glycerol was absent. The mixed acids were recovered from the insoluble and soluble lead salts, converted into the methyl esters and fractionally distilled in a vacuum. From the fractionation data obtained it was evident that the acids yielding insoluble lead salts were not saturated fatty acids, but differed little from the main bulk of those acids whose lead salts had remained in solution in the alcohol, and that the acids from the soluble lead salts were for the most part similar to the former, except that not more than 6 to 7 per cent. of the total acids had methyl esters of a mean equivalent of 292·0 and iodine value 71·3, indicating the probable presence of oleic and palmitic acids. The main component acid was found to be an eicosenoic acid, identified as $\Delta^{11:12}$-eicosenoic acid. Small quantities of a higher acid, possibly docosenoic, were also present.

The alcohols were separated by fractional distillation in a vacuum into three main fractions, none of which appeared to be wholly composed of an individual compound. A portion of the second fraction was hydrogenated, and some of the saturated alcohols were recrystallised. Another portion of the hydrogenated

alcohols was oxidised with chromic acid in acetic acid solution. A portion of the third fraction was oxidised with permanganate in acetone. Definite evidence of the presence of $\Delta^{13:14}$-docosenol was obtained, and eicosenol (probably $\Delta^{11:12}$-eicosenol) is considered to be the other main alcohol component. So far as is known, no other fatty material from seed endosperm or embryo possesses a similar composition. It may be noted that the seed of *Simmondsia californica* itself has certain morphological abnormalities. D. G. H.

Iodine Value of Derivatives of Cinnamic Acid. A. Lespagnol and J. Bruneel. (*J. Pharm. Chim.*, 1937, **25**, 454–457.)—The (Hübl) iodine values found and calculated, respectively, were as follows:—cinnamic acid (recrystallised several times), 27·7 to 29·4, 171; commercial ethyl cinnamate dissolved in chloroform, 24·8 and 25·0, 144; benzyl cinnamate (recrystallised), 4·5 to 6·7, 106; cinnamyl cinnamate (m.p. 41·5° C.), 83·8 to 87·0, 184; cinnamyl alcohol (recrystallised), 182 and 187, 191. The periods of contact varied from 2 to 12 hours. Care is therefore necessary in interpreting the iodine value of drugs containing cinnamic acid or its derivatives (*cf.* Beaugeard, *Bull. Sci. Pharmacol.*, **1934**, **41**, 210), and the method of Vohlmar and Samdahl (*Bull. Soc. Chim.*, **1935**, **2**, 826), which is based on the fixation of bromine atoms by double linkages, is therefore preferred. J. G.

Use of Coloured Powders for the Detection of Poison Gases used in Warfare. H. L. Ligtenberg. (*Chem. Weekblad*, 1937, **34**, 321.)—J. Thomann (see *Protar*, March, 1937, p. 81) and A. P. J. Hoogeveen (*Chemie en Luchtbescherming*, 1937) have suggested the use of a powder containing Sudan red for dusting over areas suspected of contamination by mustard gas. A blood-red colour indicates a positive reaction, and this method is preferable to the use of paper impregnated with the reagent, because contact between the gas and the dye is facilitated physically, and because the contrast in colours before and after reaction is more marked (*cf.* Ligtenberg, ANALYST, 1937, **62**, 326). The method, however, is not specific (*cf.* Ligtenberg, *loc. cit.*), and the following modification is recommended:—One part of Sudan red is ground well with 1000 parts of ground chalk, and this powder is then mixed with 3000 parts of non-purified sea-sand. The sand is not essential, but it enables the dye to be distributed more easily and over a larger area. If a positive reaction results, another powder containing 1 part of ferric chloride and 7 parts of ground chalk is dusted over the first, when a green colour is obtained within 1 minute if mustard gas or brom-benzyl cyanide or phenylcarbylamine chloride (which are used to mask it) is present. Carbon dioxide, fatty oils, chloropicrin and diphosgene produce a rust-coloured stain, which may turn green after a prolonged period; benzene and lubricating oils, which may occur on the roads in large cities, do not react. The ferric chloride powder is hygroscopic and should be stored in a suitable container, and it should be used on surfaces (*e.g.* paving stones) which are not too porous, as a dry porous stone can prolong the period of reaction by 30 minutes. J. G.

Inorganic

Method of Qualitative Analysis without the use of Hydrogen Sulphide. M. B. Rane and K. Kondaiah. (*J. Indian Chem. Soc.*, 1937, **14**, 46–50.)—The scheme is based on the separation of the basic constituents into five groups. The mixture is dissolved as far as possible in hydrochloric acid, the liquid is boiled with an excess of nitric acid, and evaporated to dryness, and the residue is digested with nitric acid. The insoluble portion may contain silver, antimony, tin and any insoluble matter (Group I). To the solution, after filtration, is added ammonium sulphate solution, and the liquid is boiled; the precipitate, which is filtered off, may contain barium, strontium, calcium and lead (Group II). A portion of the filtrate is tested for phosphate by means of ammonium molybdate; the rest of the solution is heated, and a liberal excess of ammonia is added, followed by ammonium phosphate until no further precipitation occurs; the precipitate may contain hydroxides, phosphates or arsenates of iron, aluminium, chromium, bismuth, manganese, calcium and magnesium (Group III). The liquid, after filtration, is boiled with an excess of sodium hydroxide, giving a precipitate which may contain cobalt, nickel, copper, cadmium and mercury (Group IV). The liquid may contain zinc and arsenic (Group V). The methods suggested for the detection of the constituents of the groups follow customary lines. Alkali metals are tested for in the original mixture after removal of other bases by "barium hydroxide and sulphuric acid treatment." S. G. C.

New Method for the Determination of Cadmium and its Separation from Zinc. C. Mahr and H. Ohle. (*Z. anal. Chem.*, 1937, **109**, 1–5.)—Cadmium solutions containing thiourea, when treated with ammonium tetrathiocyanato-diammine-chromiate (Reinecke's salt), yield a complex precipitate of the composition $[Cd(CSN_2H_4)_2][Cr(CNS)_4(NH_3)_2]_2$, containing 12·47 per cent. of cadmium. The dried precipitate may be weighed, or its chromium-content determined volumetrically after conversion into chromate. A single precipitation separates cadmium from arsenic, antimony, nickel, cobalt, iron, manganese, chromium, aluminium, alkaline earths and alkalis, as well as from 1000 parts of zinc. The acidity of the cadmium solution should be *N* (or less if other metals preponderate), and its thiourea-content 1 per cent.; it is treated with a filtered solution of Reinecke's salt, containing 1 per cent. of thiourea, in moderate excess. After ½ to 1 hour's cooling in ice-water with occasional agitation, the liquid is filtered through a porous glass-crucible, and the pale-red, finely-crystalline precipitate is washed with ice-cold 1 per cent. thiourea solution, which is finally displaced by 3 to 4 washes with ice-cold absolute alcohol. The precipitate is dried at 110° to 120° C., and weighed.

For the volumetric determination of cadmium, the precipitate is collected on a filter-paper, washed with 1 per cent. thiourea solution, and gently ignited (with the filter-paper) in a nickel crucible. The residue is fused with sodium peroxide, the mass is dissolved in water, and the solution is boiled for 15 minutes for the destruction of hydrogen peroxide. The chromate is titrated in the acidified liquor with iodide and thiosulphate in the usual manner. W. R. S.

9-Methyl-2-3-7-trioxy-6-fluorone as Reagent for the Detection of Antimony. R. Duckert. (*Helv. chim. Acta*, 1937, **20**, 362–367.)—The reagent is used in saturated alcoholic solution acidified with hydrochloric or sulphuric acid. It yields a red precipitate with antimony in acid tartrate solution, preferably at *p*H 4. The reaction is stated to be highly sensitive and to be given equally well by ter- and quinquevalent antimony. Some other ions, in particular, cerium and germanium, give similar colours; iron gives a dark-violet colour. The test is being investigated, and further details are to be published shortly. A method of preparing the reagent is outlined. S. G. C.

Atomic Weights of some Radiogenic Leads. G. P. Baxter, J. H. Faull, Jr., and F. D. Tuemmler. (*J. Amer. Chem. Soc.*, 1937, **59**, 702–705.)—The following results have been obtained in the determination of the atomic weights of certain radiogenic leads:

Cerussite, Wallace, Idaho, U.S.A. (Common) ..	207·21
Samarskite, Glastonbury, Conn., U.S.A.	206·34
Pitchblende, Beaverlodge Lake, N.W.T., Canada ..	206·08
Pitchblende, Katanga, Africa	
Black, insoluble portion	206·04
Yellow, hydrochloric-acid-soluble portion ..	206·05

It is pointed out that these values, together with the percentages of important components, point to the presence of several per cent. of common lead in all three radioactive minerals.

Colorimetric Determination of Ferric Iron with 7-Iodo-8-hydroxyquinoline-5-sulphonic Acid. J. H. Yoe and R. T. Hall. (*J. Amer. Chem. Soc.*, 1937, **59**, 872–879.)—The properties of 7-iodo-8-hydroxyquinoline-5-sulphonic acid (known as "ferron"), which was suggested by Yoe (*J. Amer. Chem. Soc.*, **1932, 54**, 4139; Abst. ANALYST, 1933, **58**, 54) as a reagent for the colorimetric determination of ferric iron, are examined in detail. The solubility of ferron in various solvents, its physical and chemical properties, the nature of the colour with ferric iron, the influence of various ions, sensitivity, conformity to the Lambert–Beer law, and the effects of *p*H, of ageing, and of temperature were investigated. The maximum solubility (0·7200 g./100 ml. of solution) was obtained in a water-acetone (1:1 by vol.) mixture. From the results the following are considered to be the optimum experimental conditions:—Sufficient reagent should be added to give 3 mols. of ferron per atom of ferric iron. For Nessler-tube work, using 50-ml. (tall form) tubes, a fixed amount of reagent is added over a whole range of iron concentration. For iron concentrations of less than 1 p.p.m., a solution free from any interfering ions is prepared, the acidity is adjusted to *p*H 2·5 by means of hydrochloric acid and potassium phthalate buffer, and 0·25 ml. of the ferron reagent is added. The colour develops at once. The solution is diluted to the mark and mixed thoroughly. For iron concentrations of 1·0 to 2·0 and above 2·0 p.p.m., 1 ml. and 2 ml. respectively of reagent are added; concentrations greater than 4·0 p.p.m. should not be used. The maximum sensitivity is from 1 to 2 p.p.m. of ferric iron. The sample solution must always be tested with Congo red or methyl orange paper and, if necessary, the *p*H must be approximately adjusted before the buffer is added.

Iron has been determined in glass sand and glass, in rocks, and in alloys by this method, the weight taken being one which gives 1 to 2 p.p.m. of iron. (A rough preliminary analysis is made if necessary.) In glass sand and glass the iron is determined as follows:—A sample is weighed after drying at 110° C. It is fused with sodium carbonate in a platinum crucible, cooled, and treated by the perchloric acid method (Hillebrand and Lundell, *Applied Inorganic Analysis*) for the dehydration of silicic acid. The silica is filtered and washed with dilute hydrochloric acid and then with water. To the filtrate in a porcelain dish, ammonium hydroxide is added until Congo red paper just shows acidity. After sufficient evaporation on a steam bath, the iron is determined colorimetrically as described above, 10 ml. of the buffer solution being used. If too much aluminium is present, it must be removed before the determination of the iron (or, a corresponding amount of aluminium salt may be added to the standard). Cupric ions interfere with this test when only 0·2 p.p.m. are present. Stannous, stannic, and titanium ions and dichromate and nitrite ions must not be present in concentrations of more than a few tenths of a part per million. For precise work they should be removed entirely. Fluoride must be removed, because it causes partial bleaching, and relatively large amounts of phosphate retard, but do not prevent, the reaction. Cobalt, nickel, and chromium should be present only in limited amount; aluminium ion may be present up to a concentration 5 times that of the ferric ion. Over the range of concentrations examined, the colour follows the Lambert–Beer law. Ageing and temperature (15° to 40° C.) do not affect the colour. (*Cf.* Yoe, *loc. cit.*, and Clark and Sielong, *Ind. Eng. Chem., Anal. Ed.*, 1936, **8**, 256–7; Abst., ANALYST, 1936, **61**, 632.) E. B. D.

Detection of Periodate. R. Fabre and T. Tomesco. (*J. Pharm. Chim.*, 1937, **25**, 241–244.)—A solution of zinc acetate produces a precipitate in an acetic acid solution of periodate, but none in iodate solution under the same conditions. The authors use a reagent containing 2 per cent. of zinc acetate in 3 per cent. acetic acid. The reaction detects 0·001 g. of periodate ion in 1 ml. at room temperature; in hot solutions the sensitiveness is 10 times greater. The composition ascribed to the precipitate agrees with the formula $Zn_5(IO_6)_2$. W. R. S.

Analysis of Refractories. E. Azzarello and F. Abramo. (*International Association for Testing Materials, London Congress, Group B, Paper No.* 59, *May*, 1937.)—A modified procedure is proposed to overcome difficulties in the usual method due to the accumulation of salts in the solution incidental to the successive precipitations of sesquioxides, calcium and magnesium. Silica is first separated and determined as usual. The solution containing the other constituents is divided into two parts. In one part, zirconium, aluminium and titanium are determined by the method of Grewe (*Arch. Eisenhüttenwesen*, 1934, **7**, 505). In the other part, iron, manganese, titanium, zirconium, calcium and magnesium are precipitated together as follows:—The solution is approximately neutralised with sodium carbonate, diluted to 200 ml., and slowly poured while hot, with vigorous shaking, into 25 to 35 ml. of hot sodium hydroxide solution (sp.gr. 1·332). Fifteen ml. of hydrogen peroxide (3 per cent.) are added, the solution is boiled for 10 minutes, 1 g. of sodium carbonate is added, and the liquid is diluted to 300 ml. with boiling

water, kept hot for 2 to 3 hours, and then left for 16 to 18 hours. The precipitate is filtered off on a sintered glass filter and washed with 1 per cent. sodium carbonate solution, the filtrate is rejected, and the precipitate is dissolved in hydrochloric acid. The solution is diluted to 100 to 150 ml., and the iron, etc., is precipitated with ammonia and hydrogen peroxide, re-precipitation being advisable if the precipitate is voluminous; the combined filtrates are reserved. The precipitate is dissolved in dilute sulphuric acid, and the solution is divided into two portions. To one portion (diluted to 100 ml.), 2·5 g. of ferrous sulphate are added, and then hot sodium bicarbonate solution (8 per cent.) until a persistent turbidity is produced; a further 4 ml. of the bicarbonate solution are added, and the liquid is boiled for 1 minute and then filtered with the aid of suction. The precipitate, which contains the zirconium and titanium, and in which these elements may be determined if desired, is washed with hot water. The turbid filtrate, acidified with sulphuric acid, is concentrated by evaporation, and manganese is determined by the bismuthate method. In the second portion of the solution, iron is determined by any convenient method. The reserved ammoniacal filtrate, mentioned above (100 ml.), to which has been added 2·5 g. of ammonium acetate, is heated to boiling, and the magnesium is precipitated by the addition of a moderate excess of 5 per cent. alcoholic solution of 8-hydroxyquinoline. After standing for half-an-hour the precipitate is filtered off on a sintered glass filter and washed with a hot dilute ammoniacal solution of ammonium acetate, followed by alcohol containing 0·7 per cent. of ammonia (sp.gr. 0·9); the magnesium is determined gravimetrically or volumetrically as usual with this precipitate. The filtrate is evaporated to 100 ml., and calcium is precipitated as oxalate from the boiling liquid by the addition of 20 ml. of a boiling solution of ammonium oxalate; calcium is determined as usual. Alkali metals are determined separately in the refractory by the Lawrence Smith method. S. G. C.

Microchemical

Volumetric Determination of Small Amounts of Water by means of Cinnamoyl Chloride. C. J. van Nieuwenburg. (*Mikrochemica Acta*, 1937, **1**, 71–74.)—On investigating a number of acid halides for use as substitutes for naphthyloxychlorophosphine in the determination of less than 5 mg. of water, cinnamoyl chloride was found to be most satisfactory; it is much less expensive, and does not show the irregularities often encountered with the phosphine. It reacts readily with water, and is practically non-volatile by itself. When used with a spiral absorption tube of 7 mm. internal diameter it gave results concordant within 0·1 mg. The results were 0·02 to 0·09 mg. too high, which the authors attribute to incomplete drying of the air and to the quartz tube used. These points are being further investigated. J. W. M.

Micro-determination of Glycerol in Fats and Phosphatides. G. Blix. (*Mikrochemica Acta*, 1937, **1**, 75–77.)—The method is based on a methoxyl determination according to Vieböck and Brecher (*Ber.*, 1930, **63**, 3207, and Friedrich, *Die Praxis der quantitativen organischen Mikroanalyse*, Leipzig, 1933). The apparatus is modified from the Pregl methoxyl apparatus. The distilling portion

is attached by a ground-glass joint to the distilling tube, and the washing device has a glass tap underneath for emptying, instead of a cork; there is also a ground-glass stopper to close a side-tube on the washing device for cleaning purposes. The delivery tube is also closed above by a ground-glass stopper. The lipid material is dissolved in pure benzene, and a definite volume (0·3 to 3 ml.) is pipetted into the distillation flask. The benzene is removed by gentle evaporation in a slight current of air, in the presence of 2 ml. of hydriodic acid (sp.gr.=1·70) and a few pieces of red phosphorus. The washing device is charged with 0·3 ml. of 5 per cent. sodium thiosulphate solution, and the receiver with 3 ml. of 10 per cent. sodium acetate in glacial acetic acid, and 2 to 3 drops of bromine. A stream of purified nitrogen is passed through slowly at such a rate that there is always one bubble in the receiver, and the distillation flask is heated for 3½ hours in a glycerin bath at 120° to 125° C. The lipids are hydrolysed and the isopropyl iodide formed from the glycerol is collected in the receiver, the contents of which are titrated according to the method of Vieböck and Brecher. Extremely good results are recorded. J. W. M.

Micro-detection of Nitrate. F. Werr. (*Z. anal. Chem.*, 1937, **109**, 81–91.)—The method is based upon the ready nitration of xylenol ($CH_3 : CH_3 : OH = 1:3:4$) by nitrates in presence of sulphuric acid, the volatility of nitroxylenol, and its property of forming a coloured alkali salt. The procedure is almost as simple as the baryta-water test for carbon dioxide. The reagents required are sulphuric acid (75 per cent. by vol), 0·5 *N* sodium hydroxide solution, and xylenol (A.R.). The apparatus consists of a 150-ml. flask fitted with a perforated rubber stopper carrying a doubly-bent glass tube, the vertically-descending member of which is water-cooled and dips into 2 ml. of 0·5 *N* sodium hydroxide contained in a test-tube.

The material (0·1 g. of solid, or a solution containing this amount of dissolved substance) is treated with sufficient distilled water to bring the volume to 100 ml., the solution being filtered if necessary. Preliminary tests should be made for hydrogen peroxide or per-acids; if present, they are destroyed by boiling the alkaline solution. One ml. of the prepared solution is introduced into the flask containing 5 ml. of 75 per cent. sulphuric acid and one drop of xylenol, and the flask is closed with a stopper and left in the cold for 10 to 15 minutes, with occasional shaking. Water (20 ml.) and a few glass beads are added, and the stopper with outlet-tube inserted. The distillation is conducted so as to yield about 10 ml. of distillate in 5 minutes. After cooling, the alkaline liquid in the test-tube is viewed against a white background; a barely perceptible yellow colour reveals 0·001 mg. or less of nitrogen as nitrate. The depth of colour increases with the nitrate-content, large amounts giving a reddish-yellow to red colour. A blank test should be made with the reagents, and the apparatus should be flushed with steam before and after use. Nitrites give a positive reaction and should be destroyed before the test is made. The sensitiveness is 0·001 mg. of nitrogen at a concentration of 1:1,000,000. The method is considered specific and more sensitive than the usual reactions, while it is not so hypersensitive as the diphenylamine test.

When, as described above, the test is carried out on 1 mg. of the sample, the

presence of even a high proportion of chlorides and of most other salts, in the sample, has no considerable effect; but if a larger quantity, *e.g.* 100 mg., were used, a high proportion of sulphides, halides, oxyhalides, dichromates, etc., might cause serious errors. W. R. S.

Micro-determination of Iodine by a Catalytic Method. E. B. Sandell and I. M. Kolthoff. (*Microchemica Acta*, 1937, **1**, 9–25.)—A procedure is described for the determination of quantities of iodide of the order of 0·05 to 3γ in 1 ml. of solution or in a suitable amount of solid sample (sodium chloride). The method is based upon the strong catalytic effect of iodide on the reaction between ceric sulphate and an excess of arsenious acid in dilute sulphuric acid (1·5–2 *N*). The effect of the following factors on the velocity of the reaction was studied: the iodine concentration, the temperature, and the presence of foreign substances. The velocity increases with increased iodine concentration up to 1γ per ml., and also increases with rise in temperature (from 4 minutes at 2·5° C. to 1 minute at 25° C.) under the following conditions:—The potassium iodide solution of known concentration is measured from a micro-burette into a 2 × 7 cm. micro-beaker, and is followed by 2 ml. of 0·1 *N* sodium arsenite solution, 1 ml. of 6 *N* sulphuric acid, 0·1 ml. of 0·001 *M* *o*-phenanthroline ferrous sulphate (as indicator), and water to bring the total volume to 4 ml. The mixture is poured quickly into another micro-beaker containing 1·0 ml. of 0·1 *N* ceric ammonium sulphate solution and sufficient sulphuric acid to make the acidity 1·5 *N*. A stop-watch is started at the instant of mixing. The mixed solution is quickly transferred back to the original beaker, immersed in a water-bath at known temperature and shaken gently. The first appearance of a pink tinge is taken as marking the completion of the reaction. The presence of foreign salts leads to a considerable change in reaction velocity. With an unknown solution this difficulty can be overcome by adding a suitable known amount of iodine and comparing the catalytic activity of the solution so obtained with that of the original solution. It is best to make the volumes of both solutions the same for the comparison. In the absence of chloride the linear relationship between catalytic activity and iodine concentration fails for low concentrations. Therefore, if chloride is not present in the sample, it is necessary to add about 10 mg. of sodium chloride. The concentration of iodine in the reaction mixture should generally be less than 1:2,000,000, for amounts of iodine (as iodide) from 0·05 to 1γ; in the presence of 5 to 15 mg. of sodium chloride the mean error is about 10 per cent. J. W. M.

Summary of Microchemical References in 1936. P. Haas. (*Mikrochemica Acta*, 1937, **1**, 106–119.)—Thirteen pages of references are arranged in alphabetical order of the authors under the following subjects: inorganic (preparative and analytical), physical and physical methods, organic (preparative and analytical), biochemistry, medical chemistry, pharmacy, toxicology and forensic chemistry, applied chemistry (technical, mineralogical, agricultural, foodstuffs, etc.), and apparatus. J. W. M.

Physical Methods, Apparatus, etc.

Inflammability of Cork and Wood Dusts. W. Kühn. (*Chem.-Ztg.*, 1937, **61**, 406–408.)—Dusts are classified into 3 categories, *viz.* (1) those which both ignite and transmit combustion readily; (2) those which ignite readily, but do not readily transmit combustion; (3) those which are stable in both respects. Cork and wood dusts belong to the first category, and their behaviour is governed by the following factors:—*Sp.gr.*—Average values are: cork, 0·24 and wood (pine and fir) 0·34, so that the former will float more readily. *Particle-size.*—Ignition temperatures for samples of cork dust completely passing a 200-mesh sieve, almost completely passing it, and passing to the extent of 50 per cent. are: 975° C. (flame spreads rapidly), 620° C. (0·6 per cent. unburnt) and 630° C. (flame spreads rapidly), respectively. Corresponding data for samples of wood dust which was too flocculent to be sieved, which could be sieved with difficulty, and which was sieved to the extent of 20 per cent. are: 985° C. (light smoke), 610° C. (3·2 per cent. unburnt), and 635° C., respectively. Unsieved cork developed on ignition an explosion-pressure of 0·52 atm., and sieved wood 0·9 atm. Sieve tests have also shown that the (average ?) size of the particles of cork dust is 7·7 per cent. less than that of wood dust, and that the former dust is more uniform in size. No traces of wood were found in the bronchial secretion 14 hours after exposure to an atmosphere containing wood dust, whilst with cork dust traces were still to be found after 48 hours; the former dust promotes sneezing more readily than the latter, probably on account of the larger size and coarser structure of the wood-dust particles. *Water-content.*—As this increases, the ignition-temperature rises, but smoke-formation is not necessarily affected. The raw materials and air-dry dust may contain the following percentages of water, respectively:—Wood, 15 to 40 (mean 28), 7 to 12 (mean 9); cork, 4 to 18 (mean 8), 2 to 7 (mean 6). In moist air, however, wood dust will absorb five to six times its weight of moisture, and this lowers further its tendency to ignite. *Particle structure.*—This depends on the type of disintegrator used. Particles of wood dust vary considerably in size and shape and may be fibrous, angular or rod-shaped and produce aggregates of these forms. Cork dust particles, however, are usually round plates with serrated edges, and are associated with very small particles, some of which are 0·08μ in diameter. Unlike wood particles, they do not "ball," but are always mobile. *Ease of oxidation.*—This is primarily a function of the size, structure and shape of the particles, since it depends on the surface exposed. The heat generated by the disintegration process is also important, and temperatures of 70° C. for wood and 98° C. for cork may be reached; it has also been stated that pyrophoric carbon may be produced in this process. To summarise, the evidence suggests that cork dust is more liable to ignition than wood dust. J. G.

Examination of Essential Oils by Measurement of the Ultra-violet Absorption. D. Van Os and K. Dykstra. (*J. Pharm. Chim.*, 1937, **25**, 437–454.)—Lambert–Beer absorption-curves, obtained by the usual method, are given for the range 200 to 400 $m\mu$. A Scheibl rotating sector-disc was used to control the intensity of the comparison spectrum, and the source of radiation was a

spark-discharge passing between two tungsten electrodes. The solvent used was alcohol purified by distillation over iodine, followed by decolorisation by means of zinc and several distillations over freshly-burnt lime. The wave lengths (in $m\mu$) of max. absorption of the various oils and their constituents were as follows:—*Oleum Anisi*, 260 (anethole, 260, min. 237; methyl chavicol, 225·5 to 285, min. 245·5 and 282·5). It was therefore possible to use the method to calculate the amount of anethole present (87 and 89 per cent. in the two samples tested), and also to detect adulterants of oil of anise (which lower the anethole content) and conversely, oil of anise in other oils. *Oleum aurantii*, 320 (residue after distillation 319, min. 274). The presence of methyl anthranilate is not apparent from the absorption curves, and *d*-limonene and *n*-decyl aldehyde have little influence on them; if ethyl phthalate is present the shape of the curve is altered considerably. *Oleum Bergamottae*, 311, min. 278. These values (and another min. at 238) were also obtained from the residue after distillation. The effects of linalyl acetate, linalol, limonene (*cf.* supra) and nerol are of less importance. All adulterants modify the shape of the curve, especially if they are aromatic in character. *Oleum Cajuputi* gives curves with no characteristic features. Cineole is very transparent to ultra-violet light, and *d*-terpineol gave a max. at 231$m\mu$. *Oleum Cari* showed the features of *d*-carvone rather than of *d*-limonene (*cf. supra*), namely, max. at 318 and 225, and min. at 282$m\mu$. Adulteration with aromatic ethers produces other maxima, whilst adulteration involving removal of carvone decreases the absorption. *Oleum Caryophylli* gave curves having the characteristics of eugenol, the proportion of which (86 and 89 per cent.) may therefore be calculated; they had max. at 282 and 229, and min. at 253. Acet-eugenol had a max. at 272, whilst caryophyllene absorbed continuously. Most adulterants decrease the absorption. *Oleum Chenopodii.*—Max. at 273, 264 and 258, and a min. at 235, were attributed to cymene, ascaridole being without effect, as shown by its continuous absorption curve. Adulterants (*e.g.* mixtures of anethole, cineole and menthol and ethyl benzoate and salicylate) modify the curve considerably.

J. G.

Reviews

THE BIOCHEMISTRY OF THE LIPIDS. By H. R. BULL, Ph.D. Pp. ix + 169. New York: John Wiley & Sons, Inc.; London: Chapman & Hall, Ltd. 1937. Price 13s. 6d. net.

This is a textbook intended for graduate students, and the author has endeavoured to give an account of the present knowledge of the biochemistry of fats in less than 160 pages. He is to be congratulated on having gone a very long way towards success; he has written an "up-to-date, readable" book, which covers the ground and keeps, on the whole, due perspective which adopts the fresh points of view developed in this field in recent years, and breaks away from the older, stereotyped treatment of the subject that has persisted too long in most textbooks on the subject.

If the reviewer could only leave it at that, it would be well. But Dr. Bull has gone so far in showing how a comparatively short book of this scope can be written

as to cause regret that it has not been done still better. The chief criticism one would offer is that, in so brief a volume, every page is valuable, and that the matter in not a few pages might have been omitted, or at least drastically curtailed. A few instances in support of this contention are: The sulphated and sulphonated detergents (pp. 7 and 8) have no connection with biochemistry, unless it be to point the analogy between their surface and interfacial properties and those of the natural phosphatides (an analogy to which no reference is made); moreover, there are at least two mis-statements in this section. The table on p. 9, the structural formulae on p. 20 and p. 34, and in some other places, and a number of other bulky tables of physical data not wholly necessary for the purpose of the book might have been omitted or abbreviated, and space thereby set free for fuller treatment of other topics.

The plan of the book is good, but some of the chapters are disproportioned; that on "fats and oils" might well have been expanded, perhaps at the expense of those on "sterols" and on "alcohols, waxes and hydrocarbons." It is impossible to deal adequately with sterol and related chemistry in the course of the book under review, and it would have been better to abandon the attempt to do so. Waxes and hydrocarbons, on the other hand, seem to get more space than their bearing on the main theme warrants. The chapters on "phospholipids" and emulsions are excellent, compact, and modern, and it is by comparison with these that the rest of the book appears capable of improvement. The book is marred somewhat by minor errors; *e.g.* clupanodonic acid is given as $C_{18}H_{28}O_2$ (p. 5), whilst references to authors quoted in the text are given or omitted somewhat haphazardly and the literature references, where given, are sometimes incomplete.

Discussion of current hypotheses is, of course, a matter in which each is largely entitled to his own opinion. Thus, while Dr. Bull (p. 11) says that any theory of the formation *in vivo* of fatty acids must account for the occurrence of only even numbers of carbon atoms in the molecule and for the general absence of short-chain acids, the reviewer feels that such a theory must, even more, explain the overwhelming abundance in nature of oleic acid, with the ethenoid bond dividing the carbon chain into two groups containing 9 carbon atoms; and where (p. 152) he says that it "appears as if the mechanism for fat synthesis had partly failed" in the case of milk-fats, the writer would suggest that the mechanism in question is probably more, instead of less, elaborate than usual. Again, in the course of an exceedingly fair and full survey of the reviewer's recent contributions in this field, Dr. Bull seems (p. 92, par. 2) to miss the point of the oleic-stearic glyceride relationships which have been established in ox, sheep and pig depôt fats. On pp. 95–97, in discussing changes in fats during seed ripening, a reference should certainly have been made to the important work of Ivanov and of Eyre on linseed.

The book is prefaced by an account of the various formal classifications of fats or "lipids" which have been proposed from time to time. It appears uncertain how far such arbitrary classifications serve any useful purpose beyond that of presenting to the student a simplified or bird's-eye view of the field under discussion. Even so, the classification of the unit-substances—the fatty acids, alcohols, etc.—by Bloor as "derived lipids" seems singularly incongruous. Do architects or builders classify bricks and mortar as "derived houses"?

T. P. HILDITCH

SOIL CONDITIONS AND PLANT GROWTH. By Sir E. JOHN RUSSELL. 7th Ed. Pp. viii + 655. London: Longmans, Green & Co., Ltd. Price 21s. net.

A scientific work which has reached a seventh edition in 27 years must be considered as a "best seller." The success of this volume is due not only to the importance of the subject, but also in a large measure to the readable manner in which the facts relating to soil problems are presented. The author has the advantage of a long experience at an institution which, for nearly a century, has been known as a centre of soil research.

The soil in relation to the earth itself is but as a thin film of tarnish and altered slag on a sphere of iron alloy, its thickness being less than a millionth of the radius of the globe, and its mass less than a ten millionth of the earth's mass. Yet on this thin film, with its gaseous envelope, the whole life of the earth depends. Prof. V. M. Goldschmidt, in a lecture recently given to the Chemical Society, has endeavoured to show why the distribution of elements on the earth's crust is so different from what, on considerations of density, it must be in the interior and is mainly silicious rather than ferruginous. Sir John Russell devotes his six hundred odd pages to a discussion of the thin disintegrated upper layer of the crust, which has become what we know as soil, and its relation to the growing plant.

The practical study of the soil and the plant has occupied mankind from prehistoric times, and a mass of empirical data has been accumulated which scientists have sought to explain and augment. The mere fact of plant growth is amazing; so little is put in the soil, so much taken out, year after year, until the soil shows signs of exhaustion. Whence comes all the substance of the plant? Theodore de Saussure, son of the inventor of the hair hygrometer, first showed that plants both breathed like animals and, unlike animals, were able to decompose the carbon dioxide of the air and to build up from it the greater part of their substance, whilst the part that entered through the roots was small but essential, supplying nitrogen which was not assimilated direct from the air and ash constituents "qui peuvent contribuer à former, comme dans les animaux, leur parties solides ou osseuses," and that the absorption from the roots was selective. It was some time before this view was generally accepted; in fact, many of those who contributed most to a correct knowledge of the chemistry of the soil showed great acuity of vision in some directions, but a remarkable blindness in others. Progress was hampered by the limitations of the chemistry of the period. It is, however, remarkable to note how new ideas on chemistry and new methods of investigation have been applied to the study of the soil, as well as the great contribution made by sciences other than chemistry to the study of soil problems. Consider on the one hand a mass composed of particles of all sizes, from colloid dimensions to large stones, in all states of aggregation from powder to large clods, made up of complex silicates, quartz, carbonates, with dead organic matter, continually being changed by weathering and the varied activities of a vast mixed population of bacteria, protozoa, fungi, algae, molluscs, insects and worms, the numbers of the various groups of the smaller organisms varying greatly even from hour to hour, the mass having a varying water-content, free, adsorbed and combined, and being under some conditions so hard that it cannot be dug, and at others thixotropic, its properties profoundly modified

by changes of pH, contributing its offering to the plant in a very dilute solution around and between its particles, and, on the other hand, the plant itself with its delicate system of root hairs. To gain anything like a clear notion of soil conditions and plant growth it becomes obvious that the best endeavours of a group of workers in several sciences, actively collaborating are necessary, and it is not surprising that divergent views are held on many points which on first examination appeared amenable to a simple explanation.

This book gives a good conspectus of the many aspects of the subject in what must be considered a very concise form, with full references to the original sources of information. The reviewer has noticed only one ambiguous or ungrammatical sentence. Referring (on p. 147) to plants as having "synthesised complex organic substances containing much stored up energy," the author says, "After death these substances are added to the soil and so impart the mineral matter stores of energy which *they* (the italics are the reviewer's) had not previously possessed"; and one misprint (p. 269) where a discussion of podzolisation is headed "The Process of Polarization." Even Homer nods!

Of the many contributions made by the author to agricultural science, this authoritative survey of a rapidly growing subject is perhaps one of the most valuable.

J. H. COSTE

CANNING PRACTICE AND CONTROL. By OSMAN JONES, F.I.C., and T. W. JONES, B.Sc. Pp. xii + 266. London: Chapman & Hall, Ltd. 1937. Price 25s. net.

The authors' Preface states that "their aim has been to put together a book of practical value that would include those items of information the canner needs if he is to put on the market a properly processed foodstuff. . . . The definition of canned food that has been adopted is that of foodstuff hermetically sealed and processed in a metal container." It is stated further that "much of purely academic interest, particularly in regard to the chemistry of foodstuffs in general, has been deliberately omitted." The object has been not to compile a treatise, but rather a bench book.

These statements and others in the Preface naturally limit criticism. Since this is the first book on Canning to be published in this country, its announcement was awaited with great interest, and two classes of readers looked forward to receiving it—the expert canner and the young technologist starting his career in a canning factory. The expert will be disappointed at such scanty treatment of the subject where there was room for extended discussion; the beginner will welcome the book for its outline of the main problems involved in canning.

The importance of the laboratory in the food factory is again and again emphasised, and it is very evident that the chemist must have a sufficient knowledge of food bacteriology. Indeed, the bacteriological bias is very marked throughout the work—and rightly so.

Fifteen chapters cover a wide range of topics, from the design and equipment of the cannery and its laboratory (including water supply, cannery waste and hygiene) to the raw materials, the can itself, and the general micro-biology involved. A useful chapter deals with the important subject of food values and vitamins.

There is a chapter on the preparation and use of culture media, another on the staining of specimens for microscopical examination, and one on cultural notes of the principal food-spoiling organisms (excellently illustrated).

The authors enjoin the reader all too frequently to consult the original literature. Thus, corrosion of the container is dismissed in twenty-four lines. It is not modern practice (as stated on p. 30) to stamp out can ends and then in another press to effect corrugations; the whole is effected in one operation. Again, in contradistinction to the statement on p. 40, lids are date coded *before* closing the cans. Although Chapter VI details certain tests for tinplate, no mention is made of the now routine mechanical tests, such as the Erichsen test or the Jenkins bend test, which assume considerable importance in high-speed can-making plants. The gelatin-ferricyanide test for porosity of tinplate is described on p. 90, but not the best method, which is to employ Ferraco paper, this securing a rapid and permanent record of the condition of the tinplate surface.

The experienced analyst will find several points on which to join issue with the authors, who have simply detailed the more elementary routine methods.

The bacteriological treatment is adequate for ordinary routine purposes, and it is here that the book really merits its claim to be a bench book. Of course, special problems involving special bacteriological investigations arise in all food factories. Then long experience or a special training is called for. The reviewer notes with pleasure the authors' outright dismissal of "ptomaine poisoning."

Paper, printing and binding leave nothing to be desired. The illustrations are exceptionally fine. Expansion of the text in the next edition to include adequate treatment of the fundamentals of the chemistry and engineering of canning could make this work the standard in its field. WILLIAM CLAYTON

SOAP: ITS COMPOSITION, MANUFACTURE AND PROPERTIES. By W. H. SIMMONS, B.Sc. 4th Ed. Pp. 140. London: Sir Isaac Pitman & Sons, Ltd. 1936. Price 3s.

The appearance of a new edition of this popular book will be welcomed by the large number of persons who have direct and indirect contact with the industry. For a book of such a modest size a clear picture is given, with most of the branches, including glycerin recovery, well portrayed in their correct proportions; thus, brief details are included of many technical points, and warnings are given to the inexperienced of many of the pitfalls that may be encountered.

The technical information is well related to the commercial description of the products, but it is felt that the section on soap powders has not been brought quite up to date. This revised edition, however, includes references to many advances that have taken place recently in the soap industry, such as those which relate to improvements in manufacture, in the form of the soap products, in medicated soaps and in such associated materials as silicates and phosphates, and makes brief reference, under "soap substitutes," to sulphonated compounds. L. V. COCKS