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New Apparatus for Determining the Temperature of Crystallisation of Cocoa Butter

(*Read at the Meeting, May* 2, 1934)

By S. A. ASHMORE, B.Sc., A.I.C.

The work here described was the outcome of a repetition of experiments by Bywaters and his collaborators (Analyst, 1927, **52**, 324), based upon the characteristic difference in behaviour of Borneo tallow and cocoa butter when allowed to cool without stirring. The temperature at which separation of solid fat occurs is a constant for each fat, and an apparatus has been devised whereby this temperature can be determined with precision and rapidity on as little as two grms. of fat. The Tyndall effect has been utilised by projecting a beam of light through a small tube containing the molten fat suitably housed in a darkened chamber; as soon as any separation of solid particles of fat takes place, a scattering of light occurs, and the tube containing the fat appears luminous against the darkened background. The temperature at which this is first seen is noted, and has been termed the crystallisation temperature; for cocoa butter it has proved to be a valuable criterion of purity. When applied to the examination of fats extracted from chocolate confectionery, of both home and foreign manufacture, its indications have been particularly useful in detecting Borneo tallow and certain other adulterants, the presence of which would not be indicated by the iodine value. The apparatus may have uses in other directions, and for this reason the present note has been communicated.

A diagram of the apparatus is shown in Fig. 1. It consists of a glass water-jacketed tube fitted with a cork through which pass a thermometer, and (to convey the light from a lamp to the centre of the apparatus) a piece of colourless glass

rod bent as shown in the diagram. The end of the thermometer is surrounded by a small glass tube ($1\frac{3}{4}'' \times \frac{3}{8}''$), closed at one end. This contains the molten fat, and is attached to the thermometer by means of a rubber sleeve, so that the thermometer bulb is kept central. This small tube rests on a seating cut in another cork, which is also bored to take the glass rod; in this way the tube sits symmetrically over the end of the glass rod. With the exception of the tip, the glass rod inside the inner jacket is first covered with a layer of polished tin-foil, and over this is tightly secured a layer of black paper; this enables sufficient transmission of light through the rod to take place by internal reflection. The illumination is provided by a 60-watt "daylight" lamp suitably screened from the eyes; by adjusting the lamp against the outside end of the glass rod, a beam of light is led, by internal reflection, along the glass rod and projected upwards through the molten fat. The outside of the water-jacket is covered with a layer of black paper, and in this, a small observation window (indicated by the dotted rectangle in the diagram) is cut, so that the appearance of the fat on cooling can be noted.

In practice, when the fat is well above its crystallisation temperature, the appearance is as shown pictorially at 1 in Fig. 2. Apart from the thermometer bulb, an illuminated ring of light at the meniscus of the fat is all that can be seen in the darkened chamber. When the temperature has fallen to the point at which crystallisation is beginning, the solid particles of separated fat cause a scattering of the light, and the sides of the small tube can be seen faintly outlined, as at 2 (Fig. 2). This temperature, at which the mass is just uniformly turbid, is taken as the crystallisation temperature; the appearance, as indicated at 3 (Fig. 2), shows the tube strongly outlined by the light of the now numerous particles of separated fat. Condition 3, Fig. 2, occurs at a temperature 0·2 to 0·4° C. lower than that at which condition 2, Fig. 2, is noted. It will thus be seen that the onset of crystallisation is well defined, and independent observers have had no difficulty in agreeing within about 0·2° C. on the temperature of crystallisation indicated by the appearance at 2, Fig. 2.

It is, of course, essential that the fat should be perfectly clear and bright in the molten condition, and for the small amounts of fat extracted from chocolate, a filtering device working on the syringe principle, and having a plug of filter paper in the nozzle as filtering medium, has been adopted. The filtered fat is kept in a water-bath at 40° C. for several minutes to ensure that it is in a thoroughly molten condition, and while at this temperature it is introduced into the inner jacket, the air of which is kept at a steady temperature of 18° C. by the circulation of water through the outer jacket. The determination of the point at which crystallisation first begins is facilitated by fitting a small black cardboard hood over the observation window, and by suspending a large sheet of black paper behind the apparatus to give a darkened background.

In order to establish the range of crystallisation temperature for genuine extracted cocoa butter, a quantity of the fat was extracted from a number of cocoa nibs previously handpicked free from shell and germ. The fat was extracted in the usual way with petroleum spirit, and, after removal of the solvent, was filtered. The following results were obtained:

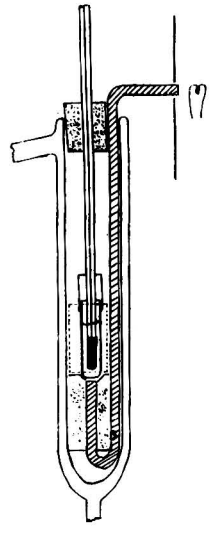
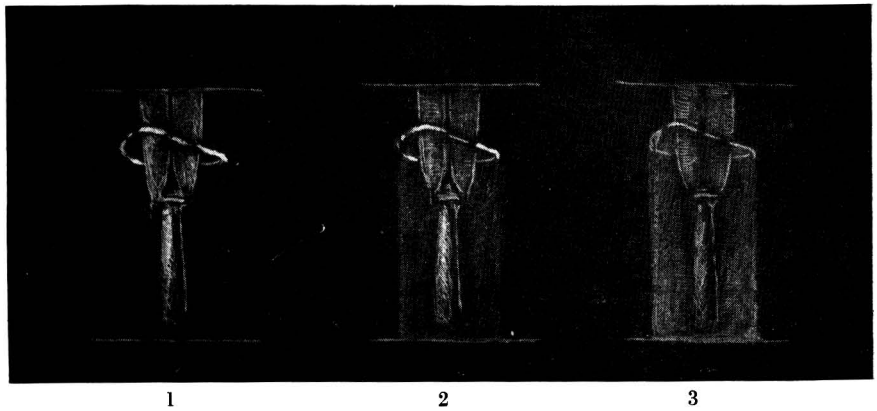


Fig. 1



1 2 3

Fig. 2.
Determination of Crystallisation Temperature

Source of cocoa butter	Crystallisation temperature °C.
Accra nibs	20·0
Arriba ,,	20·7
Carácas ,,	20·3
Trinidad ,,	20·8
Java ,,	20·3

Shell butter causes a slight lowering of the crystallisation temperature.

Examination of mixtures of Borneo tallow and cocoa butter gave the following results:

Borneo tallow Per Cent.	Cocoa butter Per Cent.	Crystallisation temperature	
		Observer 1 °C.	Observer 2 °C.
0	100	20·2	—
20	80	22·0	21·8
40	60	24·0	24·0
50	50	24·8	24·8
60	40	27·0	—
70	30	28·5	—
80	20	29·5	29·4
100	0	31·2	31·4

I wish to acknowledge the help received from Mr. A. A. Russell and Mr. V. H. Field, and to thank the Government Chemist, Sir Robert Robertson, for permission to publish this note.

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Detection of Diamines in Leather

BY WILFRED MATHER, A.M.C.T., F.I.C., AND WILLIAM J. SHANKS, A.M.C.T.

(*Read at the Meeting, April* 4, 1934)

THE detection of diamines was a part of the work covered by Cox (ANALYST, 1929, **54**, 694) in his examination of dyed furs. Forster and Soyka (*J. Soc. Dyers & Col.*, 1931, **47**, 99) investigated the fate of the dyestuff intermediates in the dyeing of furs, and extended the reactions for the detection of the diamines.

Immediately afterwards, analysts used these reactions, especially Cox's tests, for the detection of diamines and amino-phenols in dyed and finished leathers, and sometimes reports were issued showing the presence of *p*- and *m*-phenylene-

diamines, even in undyed leathers. When the published tests for the detection of diamines in leather were used, attention was not given to the fact that the tannins present in the leather were capable of interfering seriously with the tests and, in some of the tests, of confusing or even simulating the reactions produced by diamines.

Callan and Strafford (ANALYST, 1931, **56**, 625) studied the detection of diamines in the presence of tannins, and made a further extension of Cox's tests. They added diamines to the extractives made by submitting the leather to 48 hours' extraction with 1 per cent. acetic acid.

Humphreys and Phillips (ANALYST, 1932, **57**, 290) advanced the work by adding known quantities of diamines to leathers and extracting the diamines with acid solutions. These acid extractives were freed from tannin by re-extracting them with benzene, the tannins being soluble in the benzene, whilst the diamine hydrochlorides were left in the aqueous acid portion. A conclusion drawn by Humphreys and Phillips was that leathers containing meta-diamines did not show the test for those diamines in the acetic acid extracts, but did so in hydrochloric acid extracts. We find, however, that 1 per cent. acetic acid does extract diamines from leather in two hours in the cold.

In Callan and Strafford's work the diamines were added to the leather extract, so that the tests were not carried out on leathers actually containing diamines. Up to the present, therefore, we consider that there is no published method capable of quickly detecting diamines in leathers containing tannin, and we are pleased to be able to publish tests which we have devised and used for some considerable time. Our first aim was to find a method of getting rid of the interfering and masking effects of the tannins in the acid extract of the leather, and precipitation of the tannins with lead acetate solution was found to be an effective means of doing this. This operation, and the subsequent removal of the excess of lead acetate from the filtrate by means of ammonium sulphate, does not interfere with the diamines originally extracted from the leather.

With the exception of the indamine reaction, which was not found completely successful in the presence of excess of tannin, all the tests for diamines previously advanced give rise to red, brown, or yellow colour reactions, and seem to show results differing in degree rather than kind.

We therefore endeavoured to devise tests which, while readily giving sharp colour changes, would also be capable of detecting small quantities of diamines. After producing satisfactory tests capable of giving fair results in the presence of tannin, we evolved the method whereby the influence of tannin could be overcome by means simpler than extraction with benzene.

METHOD.—The details of the method used in our laboratories for some considerable time are as follows:—Three grms. of leather are cut up into strips and extracted in a beaker for 2 hours with 30 ml. of $N/10$ hydrochloric acid in the cold. The extract, which is usually coloured, is then decanted, and 10 ml. are treated with excess of lead acetate solution to precipitate the tannin. The lead tannate is filtered off, and the filtrate is tested with a drop of lead acetate solution to establish complete removal of the tannin. The excess of lead acetate in the

filtrate is removed by addition of ammonium sulphate solution, the lead sulphate is filtered off, and the filtrate is made up to 20 ml.

This treatment of the leather extract does not interfere with any diamine which may be present, as can be shown by adding a small quantity of diamine to a tannin solution before proceeding to precipitate the tannins with lead acetate solution.

DETECTION OF META-DIAMINES.—Four ml. are made slightly alkaline with ammonia, 1 drop of a 0·1 per cent. solution of dimethyl-*p*-phenylene-diamine hydrochloride is added, and the tube is well shaken. Four drops of 0·1 per cent. potassium dichromate solution are added, and the tube is again well shaken, after which the solution is acidified with acetic acid and well shaken, and any colour change is noted (*a*). The solution is next warmed on the water-bath to 50° C., and the change in colour is noted (*b*); afterwards the liquid is boiled for two or three minutes, and the colour change is noted (*c*). If metaphenylene-diamine or metatoluylene-diamine is present, the observations are:—(*a*) slowly turns blue; (*b*) pronounced blue; (*c*) deep red. Paraphenylene-diamine, dimethyl-paraphenylene-diamine, para-amino-phenol, and methyl-para-amino-phenol do not give these reactions.

In a treated tannin solution this test is capable of detecting 0·0004 per cent. of metaphenylene-diamine or 0·0002 per cent. of metatoluylene-diamine.

DETECTION OF PARAPHENYLENE-DIAMINE.—*A*. Four ml. of the lead-treated leather extract are treated as for the detection of meta-diamines, 4 drops of 0·1 per cent. solution of dimethylaniline hydrochloride being used, instead of the dimethyl-*p*-phenylene-diamine solution.

If paraphenylene-diamine is present in the leather extractive, the colour changes are:—(*a*) greenish; (*b*) green; (*c*) green.

If dimethylparaphenylene-diamine is present, the colour changes are:—(*a*) pink changing to purple; (*b*) blue changing to green; (*c*) green.

Metaphenylene-diamine, metatoluylene-diamine, para-amino-phenol, and methyl-para-amino-phenol do not give these reactions.

This reaction is capable of detecting 0·0001 per cent. of paraphenylene-diamine in a treated tannin solution.

B. Four ml. of the lead-treated leather extract are treated as in *A* (detection of paraphenylene-diamine), 4 drops of 0·1 per cent. solution of aniline hydrochloride being used, instead of the dimethylaniline hydrochloride. If paraphenylene-diamine is present in the leather extract, the colour changes are:—(*a*) green; (*b*) blue; (*c*) red.

If dimethylparaphenylene-diamine is present, the colour changes are:—(*a*) pink changing to purple; (*b*) blue changing to green; (*c*) green.

Metaphenylene-diamine, metatoluylene-diamine, para-amino-phenol, and methyl-para-amino-phenol do not give these reactions.

This reaction is capable of detecting 0·0002 per cent. of paraphenylene-diamine in a treated tannin solution.

The following table summarises the results obtained in the tests:—

Reagent	Temp.	Leather containing no added diamine	Leather containing 0·05 per cent. meta-phenylene-diamine	Leather containing 0·05 per cent. para-phenylene-diamine	Leather containing 0·05 per cent. meta-toluylene-diamine	Leather containing 0·05 per cent. dimethyl-para-phenylene-diamine	Leather containing 0·05 per cent. para-amino-phenol	Leather containing 0·05 per cent. methyl-para-amino-phenol
Dimethyl-para-phenylene-diamine (0·1 per cent. solution)	Normal	Orange-red	BLUE	Orange-red	BLUE	Orange-red	Orange-red	Orange-red
	50° C.	Light red	DEEPER BLUE	Light red	DEEPER BLUE	Light red	Light red	Light red
	Boiled	Yellow-brown	DEEP RED	Yellow-brown	DEEP RED	Yellow-brown	Yellow-brown	Yellow-brown
Dimethylaniline (0·1 per cent. solution)	Normal	Yellow	Yellow	YELLOW-GREEN	Yellow	YELLOW	Yellow	Yellow
	50° C.	do.	do.	GREEN	do.	YELLOW-GREEN	Yellow-orange	Orange
	Boiled	do.	do.	DEEPER GREEN	do.	GREEN	Orange	do.
Aniline (0·1 per cent. solution)	Normal	Yellow	Yellow	GREEN-BLUE	Yellow	YELLOW	Yellow	Yellow
	50° C.	do.	do.	BLUE	do.	GREEN	do.	do.
	Boiled	do.	do.	ORANGE-RED	do.	ORANGE-RED	do.	do.
Ortho-toluidine (0·1 per cent. solution)	Normal	Yellow	Yellow	BLUE	Yellow	YELLOW	Yellow	Yellow
	50° C.	do.	do.	BLUE	do.	GREEN	do.	do.
	Boiled	do.	do.	RED	do.	ORANGE-RED	do.	do.
Para-phenylene-diamine (0·1 per cent. solution)	Normal	Yellow	PURPLE	Yellow	PURPLE	Orange	Yellow	Yellow
	50° C.	do.	PURPLE	do.	RED-PURPLE	do.	do.	Orange-yellow
	Boiled	Brown-yellow	RED	Brown-yellow	RED-PURPLE	do.	Orange-yellow	do.
Meta-toluylene-diamine (0·1 per cent. solution)	Normal	Yellow	Yellow	PURPLE	Yellow	GREEN	Yellow	Yellow
	50° C.	do.	do.	PURPLE	do.	BLUE	Orange-yellow	Orange-yellow
	Boiled	Orange-yellow	Orange-yellow	RED-PURPLE	Orange-yellow	ORANGE-RED	Orange	Orange

The reactions shown in this Table are the same for both undyed and dyed vegetable-tanned leather.

CHROME-TANNED LEATHERS.—When diamines are present in chrome-tanned leather they can be detected by the same tests as those used for vegetable-tanned leather. The diamines are extracted with $N/10$ hydrochloric acid, and, inasmuch as there are usually no free vegetable tannins present, the tests can be carried out on the hydrochloric acid solution directly, that is, without treatment with lead acetate, etc.

To illustrate this, the following types of chrome-tanned leathers were tested:— *A*. Full-chrome lambskin, for glove leather, undyed; *B*. full-chrome lambskin, for glove leather, dyed; *C*. semi-chrome Persian, for glove leather, undyed; *D*.

semi-chrome Persian, for glove leather, dyed; *E.* semi-chrome sheep, for clothing, dyed black and finished with black pigment.

In each instance the leather was damped on each side with a solution of diamines and made to take up 0·02 per cent. of the diamine. The samples were then completely dried overnight, and on the following day they were extracted with *N*/10 hydrochloric acid and tested as described.

In every instance diamines were detected and identified by means of the colour reactions given in the table.

SUMMARY.—Tests have been worked out whereby para- and meta-diamines can be detected in dyed and finished leathers when present in extremely small quantities. Paraphenylene-diamine can be detected when present to the extent of 0·005 per cent. in the leather; metaphenylene-diamine when present to the extent of 0·017 per cent.; and metatoluylene-diamine to the extent of 0·01 per cent. These will be seen to be small quantities when it is realised that a hat leather weighs 10 to 12 grms., and has a surface area of 50 square inches on each side.

Furthermore, the tests are capable of showing that the para- and meta-diamines are extracted from leather in the cold by 2 hours' extraction with *N*/10 hydrochloric acid or 1 per cent. acetic acid solution, and that the interfering influences of the tannins present in the leather can be removed by simple means.

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DISCUSSION

Dr. H. E. COX said that the paper was a valuable contribution to a difficult subject; he must, however, disclaim any responsibility for the mis-use of the tests in his paper to which the authors had referred. It seemed to him an elementary precaution in dealing with a supposed dyed leather to determine whether or not it really was dyed, and if it was, with what class of dyestuff. He thought that, in general, it was better to extract the diamine in a fairly pure state as suggested by Humphreys and Phillips, rather than to use precipitation methods. The reactions shown by the authors appeared to be variations of the well-known indamine test, and the indamines, on heating, produced safranine or analogous substances which were, no doubt, responsible for the red colours shown on boiling.

Mr. C. E. SAGE mentioned that different parts of skins had to be examined. He had to deal with fur, and with the leather. On the skins used for making suede shoes it was not only the dye used by the manufacturer of the leather, but also the substances used in the various preparations for renovating or cleaning them which might be a source of danger to the wearers.

A Rapid Qualitative Test for Ethylene Glycol and its Application in the Presence of Glycerol

By A. W. MIDDLETON, B.Sc., A.I.C.

(*Read at the Meeting, April* 4, 1934)

During the last few years the use of the higher fatty acid esters of ethylene glycol and of glycerol as soap substitutes and emulsifying agents has been developed in America and on the Continent. The analyst confronted with the investigation of a product possibly containing esters of one, or both, of these alcohols has no ready means of identifying the glycol in the presence of glycerol. Such a test would enable him to place more reliance on the interpretation of his glycerol result; the usual chemical determination of glycerol depends on the determination of hydroxyl groups either by acetylation or by oxidation. Three molecules of a glycol ester differ from two molecules of a corresponding glycerol ester only in that they contain two hydrogen atoms more, and this slight difference might not be detected in the ordinary course of commercial analysis. To differentiate between the esters or mixtures of them it would be necessary to isolate them or their alcohols, in a pure state—a tedious and expensive undertaking. Hence an attempt was made to find a rapid test for ethylene glycol suitable for application in the presence of glycerol.

There is scant literature on the detection of glycol. B. Müller (*Chem.-Ztg.*, **1920, 44**, 513), who was not concerned with their differentiation, applied the methods of determining glycerol to glycol. He found that the acetin and dichromate methods, and the method of weighing the carbon dioxide formed on oxidation with a mixture of sulphuric acid and chromium trioxide, give as good results with glycol as with glycerol. Fleury and Marque (*J. Pharm. Chim.*, 1920, **10**, 241) based a method of determination on the oxidising action of alkaline mercuric iodide and potassium iodide on the hydroxyl groups in glycol, glycerol, and other polyols. The reaction is not strictly quantitative, but it was found useful for the determination of glycol, mannitol, inositol, and dulcitol. Wolff (*Chem.-Ztg.*, **1917, 41**, 608) studied Denigès' tests for glycerol, in which he oxidised the alcohol with alkaline bromine, removed the excess of bromine, and then added solutions of various phenolic substances which give coloured bodies with the aldehydic decomposition products from the glycerol. He found that technical glycol and glycerol gave similar colours, and that these colour-tests had no differential value. He suggested the determination of the refractive index of the alcohols as a rough means of deciding the extent of admixture. Thomas and Micsa (*Chem. Zentr.*, **136**, [i], 1925) used R-acid as a reagent for polyhydroxylic alcohols. This reagent, after oxidation of the alcohols by bromine water, ultimately gives with glycol a blue colour surrounded by a yellow ring, and with glycerol a greenish-blue colour surrounded by a yellow ring. Other polyhydroxylic alcohols and aldehydic bodies give intensely coloured products. Alber (*Mikrochem.*, 1929, **7**, 21) reviewed the microchemical reactions for glycol, glycerol, and mannitol, and found that a

modified Denigès' test involving the use of β-naphthol and "layering" with sulphuric acid, as described by Fischer and Tafel (*Ber.*, 1887, **20**, 3384), was the only useful method of differentiation. Glycol gives a reddish-violet and glycerol a green colour when the pure alcohols are used. Wolff (*loc. cit.*), who used technical glycol, found the β-naphthol test not so satisfactory.

None of these methods for the determination or detection of glycol is practicable in the presence of glycerol. With those that give a coloured solution as their end-point, the difference between glycol and glycerol is slight, and does not permit of their detection when mixed together. The methods of Fleury and Marque, and of Müller are dependent on the determination of the hydroxyl groups in the molecule, and are hardly suitable for differentiation between the various polyhydroxylic alcohols.

From an examination of the reactions of these alcohols (Beilstein; Richter; J. M. Lawrie, *Glycerol and the Glycols*, Chem. Catalog. Co.), oxidation appears to be a suitable basis for a rapid qualitative test. Moderate oxidising agents convert glycerol into glycerose, whilst more energetic oxidation causes complete decomposition into carbon dioxide and water. With glycol, moderate oxidising agents give oxalic acid. Chief among these is nitric acid, giving aldehydic bodies from glycerol, oxalic acid from glycol, and yet only slowly oxidising oxalic acid itself to carbon dioxide and water. As oxalic acid is readily detected, a test was tried which was based on the oxidation of the free alcohols by nitric acid, followed by the examination of the decomposition products for oxalic acid.

It was decided to use a 10 per cent. aqueous solution of the alcohol in all experiments. This would prevent too much loss of alcohols by evaporation during the concentration of their solution after saponification of the esters and removal of the fatty acid portion (*i.e.* by the method necessary to obtain the alcohol in the ordinary course of analysis). After a few preliminary results the following was adopted as an initial procedure:

Two ml. of nitric acid (sp.gr. 1·42) were added to 10 ml. of the solution, and the mixture was evaporated to approximately 1 ml. in a small evaporating basin. Five ml. of 20 per cent. w/v ammonium chloride solution were then added, and the dish was allowed to cool. The liquid was made slightly alkaline by adding, 1 ml. at a time, 10 per cent. sodium hydroxide solution, until there was a slight smell of ammonia, after which the solution was boiled until neutral, *i.e.* until no odour of ammonia could be detected. One ml. of 10 per cent. w/v barium chloride solution was next added and, after about 15 minutes (during which time the liquid was cooled in a bath of cold water), the precipitate was filtered off on a No. 40 Whatman filter paper and washed with about 5 ml. of water, *i.e.* until the filter paper was nearly stain-free. The precipitate was then rinsed into a boiling-tube by means of 5 ml. of water, 7·5 ml. of 2 *N* sulphuric acid were added, and the mixture was heated just to boiling-point. The resulting oxalic acid solution was cooled and filtered from the barium sulphate through a Whatman No. 40 paper. The filtrate was treated with 0·6 *N* potassium permanganate solution until no more was decolorised *in the cold*; usually there was no decolorisation, and if decolorisation occurred it was regarded as being due to decomposition products other than oxalic acid. The solution was then boiled,

and any further decolorisation of the potassium permanganate was regarded as showing the presence of oxalic acid, and hence the initial presence of glycol.

The experimental results relate chiefly to variations of the procedure in order to obtain the optimum conditions for the test. Experiments were then made to determine the rough limits of sensitivity of the test. When a large proportion of glycerol was present and the solution was boiled for a considerable time with the alkali, it gave a brown colour, but this brown colour could be readily removed by washing the subsequently precipitated barium salt. It was found that 1 grm. of glycerol gives no oxalate precipitate, whilst 0·1 grm. of glycol will give a positive result; yet, in the case of a mixture, this amount of glycol cannot be detected, and at least 25 per cent. of the alcohols present must be glycol in order to obtain a definite reaction for that substance.

Experiments were then made in which the different points of the procedure were varied singly. The results, all on an aqueous solution of 0·2 grm. of glycol in 10 ml. of water, showed that the procedure could not be improved; that the use of too much nitric acid interfered by precipitating barium nitrate with the oxalate; and that the solubility of barium oxalate necessitates that the bulk of the solution should be kept as low as possible at the neutralisation stage.

Other experiments definitely established the interfering effect of glycerol. It was found that in its presence quite considerable amounts of glycol could not be detected, while blanks showed that the test was still quite sensitive to glycol in the absence of glycerol.

Attempts were made to rectify this effect of the glycerol, but they were not successful. The glycerol used was free from the heavy metals most likely to catalyse the oxidation of the oxalic acid by nitric acid. Alteration of the speed of evaporation of the nitric acid and alcohol mixture, from which the glycol might boil off in the presence of glycerol, did not seem to give any improvement; neither did increasing the concentration of the sodium hydroxide from 10 to 20 per cent., with a consequent reduction in the bulk of the liquid from which the barium oxalate was precipitated. Fuming nitric acid (sp.gr. 1·5) was of no use, as it gave positive results with glycerol in the absence of glycol. Evaporation of the oxidising liquid to 0·5 ml. gave better results in some cases, but the results were unreliable.

Thus the test developed can be used for the detection of glycol alone and for mixtures of glycol with glycerol containing up to 75 per cent. of the latter. It is sensitive to 0·1 grm. of glycol in 10 ml. of aqueous solution, and will detect as little as 0·3 grm. of glycol in the presence of 0·7 grm. of glycerol in 10 ml. of aqueous solution.

At the suggestion of Dr. H. E. Cox, attempts were made to adapt the test to the detection and determination of trimethylene glycol in the presence of glycerol, but the results were very unsatisfactory; the method is not applicable to trimethylene glycol.

I wish to thank Dr. A. M. Ward for his interest and helpful criticism.

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A Rapid Test of Thickness of Tin Coatings on Steel*

By S. G. CLARKE, B.Sc., Ph.D., A.I.C.

The methods at present available for the determination of thickness or weight per unit area of tin coatings on steel involve chemical analysis, the test specimen being dissolved in acid, and the tin present in the solution in the stannous condition being titrated with a standard oxidising solution. These methods are capable of yielding accurate results, but their usefulness for inspection purposes is limited. Thus, whereas chemical analysis records the total tin present on an article, it is frequently desired to determine the thickness of tin on a particular portion of the surface such as, for example, one side only of a tinned sheet. Further, with specimens other than thin sheet, dissolving the specimen for chemical analysis may occupy considerable time, and there is the possibility of interfering matter being dissolved from the basis metal; alternatively, where the basis metal is not completely dissolved, there may be uncertainty as to the completeness with which the tin coating is dissolved. Owing to the readiness with which bivalent tin is oxidised by the air, it is desirable to use an apparatus in which the dissolved tin can be kept in an inert atmosphere until the titration is completed.[1]

It appeared that a method in which the tin coating could be selectively dissolved off and determined by loss in weight of the specimen might offer advantages. A method of this type is already available for electro-deposited cadmium coatings on steel,[2] in which cold hydrochloric acid containing antimony chloride is employed for dissolving off the cadmium without appreciable attack of the steel. It has now been found that tin also dissolves readily in this solution, rendering possible an extension of this stripping method to tin coatings on steel.

Description of Method.—*Solution required.*—Twenty grms. of antimony trioxide (Sb_2O_3) dissolved in 1 litre of cold hydrochloric acid (sp.gr. 1·146).

Procedure.—(*a*) *Determination of tin on total area.*—The article to be tested, after being freed from grease by treatment with a grease solvent and weighed, is immersed in the solution. When the evolution of gas has ceased, the article is allowed to remain in the solution for a further period of 1 minute. It is then withdrawn, washed without delay in water, mopped with a wet soft cloth or cotton-wool swab to remove black powdery deposit, dried and weighed. The area from which the tin has been removed is measured.

(*b*) *Determination of tin on selected area.*—The degreased and weighed specimen is coated with Necol varnish† applied by means of a brush (the selected area being left uncoated), and allowed to dry for 15 minutes. The tin coating is then removed from this area by immersion as in (*a*). After being washed with water, the Necol film is removed by rubbing with a cotton-wool swab saturated with acetone, in which the film is readily soluble; the article is finally washed with acetone, allowed

* Communication from the Research Department, Woolwich.
† A cellulose varnish supplied by Imperial Chemical Industries, Ltd.

to dry, and weighed. This method is suitable for determining the amount of tin on one face of tinplate sheet or as an alternative to cutting up articles which are not too large for weighing.

Calculation of results.—The thickness of coating may be calculated with the aid of the following formulae:

$$T\ (\text{inch}) = \frac{W\ (\text{grm.})}{A\ (\text{sq.inch}) \times 118} \text{ or } T\ (\text{cm.}) = \frac{W\ (\text{grm.})}{A\ (\text{sq.cm.}) \times 7{\cdot}2}$$

T = thickness; W = weight of coating; A = area of coating.

It has been found that the following corrections should be applied to the calculated thickness:

(*a*) hot-dipped coatings: 4×10^{-6} inch (1×10^{-5} cm.) to be deducted.

(*b*) electro-deposited coatings: 1×10^{-6} inch ($2{\cdot}5 \times 10^{-6}$ cm.) to be deducted.

The weight of tin found may be converted into the customary trade units[3] from the relation 0·1 grm. per 100 sq.cm. of surface = 14·5 ounces per base box, the appropriate correction to be deducted being 1 ounce per base box for a hot-dipped coating. One lb. of tin per base box is approximately equivalent to a thickness of 6×10^{-5} inch ($1{\cdot}5 \times 10^{-4}$ cm.). With the hot-dipped coatings, the result obtained is inclusive of the tin present in the alloy layer.

Notes.—For routine testing of tinned sheet steel, a test-specimen measuring 10×5 cm. is a convenient size with which to work. The time occupied in stripping varies with the thickness of the tin layer; 0·0001 inch thickness of unalloyed tin requires approximately 50 seconds; the alloy layer on ordinary tinplate requires approximately 45 seconds. With hot-dipped coatings, the correction given above applies to ordinary commercial coatings having a tin-iron alloy layer corresponding with 3 ounces of tin per base box; a greater correction is necessary for coatings having a higher alloy-content, as referred to later.

INVESTIGATION OF METHOD.—Investigation was carried out to establish the method and to determine its accuracy and reliability.

(i) *Electro-deposited Coatings.*—Pieces of sheet steel (C, 0·08; Si, 0·01; Mn, 0·40; S, 0·035; P, 0·025), $10 \times 5 \times 0{\cdot}04$ cm., having tin coatings of thickness ranging from 0·0001 to 0·0005 inch deposited from a sodium stannate solution, were tested by the method described. The weight of steel base was found to have decreased, as a result, by 0·002 to 0·003 grm., an amount which would introduce an error of only about 1×10^{-6} inch (0·3 ounce per base box) in the thickness calculated from loss in weight. After the stripping process no tin could be detected on the specimens by a method sensitive to 0·0005 grm.[4]; a very thin film of antimony, weighing 0·0008 grm. on 100 sq.cm., was, however, found by the iodide colorimetric method.[5]

During the period of immersion hydrogen was steadily evolved, accompanied by the separation of spongy antimony, for a time which was roughly proportional to the thickness of the deposit, and varied from 40 to 60 seconds for 0·0001 inch of thickness with specimens initially completely coated. As in the case of the cadmium coatings previously studied,[2] the time of gas evolution for a unit thickness of deposit was somewhat lower when one-half of the specimen was originally not

tinned. The cessation of the evolution of gas was abrupt, ranging over about 5 seconds from the period of steady evolution.

(ii) *Hot-dipped Coatings.*—Two grades of commercial tinplate sheet, nominally $1\frac{1}{2}$ and $3\frac{1}{2}$ lb. of tin per base box, were employed. Since some variation in thickness of the tin coating on different areas was anticipated, strips were cut along the length of the sheets (excluding the edges), and these strips were further cut into numbered pieces, giving test specimens measuring 10 × 5 cm. The tin coating on alternate specimens was determined by chemical analysis, and the intermediate specimens were tested by the stripping method. Both methods revealed an increase of 4 to 6 ounces per base box in the tin coating from one end to the other of the sheets. The results obtained by the stripping method, without the application of any correction, were uniformly slightly higher than those obtained by chemical analysis, the average differences of closely agreeing results amounting to 1·09 and 1·04 ounce per base box for the $1\frac{1}{2}$ and $3\frac{1}{2}$ lb. grades, respectively.

In the course of stripping in the hydrochloric acid and antimony chloride solution, the rate of hydrogen evolution remained fairly uniform for a time which was approximately proportional to the thickness of the unalloyed tin layer, as found with electro-deposited coatings; the evolution of gas then slowed down considerably for about 45 seconds before ceasing, and this slow period appeared to correspond with attack of the alloy layer. The period of gas evolution was not sufficiently definite to afford a satisfactory indication of the thickness of coating.

Effect on the tin-iron alloy layer.—Specimens, after stripping and removal of the loose black deposit by swabbing, were smooth, and showed more or less faint dark striations, which followed in outline the pronounced markings of the alloy layer, as revealed after the removal of unalloyed tin from tinplate by the plumbite stripping method.[6] Chemical analysis showed that the amount of tin in the alloy layer was 3·2 ounces per base box for both grades of tinplate tested, and that the amount of tin remaining after stripping in the hydrochloric acid and antimony chloride solution was 0·3 ounce per base box. The bulk of the tin-iron alloy had, therefore, been dissolved, whilst a very thin layer of an alloy present at the iron surface remained unattacked.

Further tests on the alloy layer were carried out on weighed steel pieces coated with known amounts of tin by electro-deposition and subsequently heated under uniform conditions, which had the effect of producing, beneath the tin coating, an alloy layer found to contain 0·038 grm. of tin per 100 sq.cm. (5·5 ounce per base box) without loss of any of the tin coating. On stripping these specimens in the hydrochloric acid and antimony chloride solution, practically the whole of this alloy layer dissolved off, only 0·003 grm. of tin per 100 sq.cm. (0·38 ounce per base box) remaining, as found by chemical analysis. After making allowance for this remaining tin and also for the normal degree of attack on the basis steel in the stripping process as given above, the loss in weight of the steel base showed that 0·0117 grm. of iron, which was previously combined in the alloy, had simultaneously dissolved. This weight of iron is slightly greater than that required by the formula $FeSn_2$ (0·0088 grm.), suggesting that, possibly, an alloy richer in iron was present.

The above results showed that, on stripping a hot-dipped tin coating in the hydrochloric acid and antimony chloride solution, the tin and the iron in the alloy layer are almost completely dissolved, and an empirical correction for the iron, amounting to one-third of the alloyed tin, is necessary. With ordinary commercial tinplate, the alloyed tin appears to be fairly constant within narrow limits at about 3 oz. per base box,[6] for which a correction of 1 oz. per base box, as found in the present work, is appropriate.

(iii) *Effect of the Stripping Solution on Ferrous Basis Metals.*—Mild steel (C, 0·08), high carbon steel (C, 0·82), phosphoric grey cast iron, stainless steel and austenitic nickel-chromium steel were tested by immersion in the stripping solution for 5 minutes. With the exception of cast iron, all the materials remained unchanged in appearance, and the weight loss was less than 0·002 grm. for 10 sq. inch of surface exposed. Cast iron was slowly attacked, with evolution of hydrogen, losing 0·008 grm. for 10 sq. inch of surface.

(iv) *Stripping a Selected Area of Tinplate.*—A single coating of Necol varnish, applied locally with a brush and allowed to dry at room temperature for 15 minutes before immersion, showed no tendency to become detached during the stripping operation. It did not appear essential for the coating to be quite dry to the touch before proceeding with the stripping process. The weight of tin coating on sheet steel found by stripping each face separately was in good agreement with the known total weight. Areas of exposed steel on a partially tinned surface do not require varnishing.

SUMMARY.—A method is described in which the thickness or weight per unit area of tin coatings on steel may be determined by loss in weight of a specimen on stripping the coating in a solution of antimony chloride in hydrochloric acid. The coating dissolves rapidly, a thickness of 0·0001 inch of tin requiring about 1 minute, and the attack on the basis steel is negligible. The method is applicable to both electro-deposited and hot-dipped tin coatings. With hot-dipped coatings, the underlying layer of tin-iron alloy is almost completely dissolved, and a correction for the iron constituent amounting to one-third of the alloyed tin is necessary. Since the alloyed tin on commercial tinplate appears approximately constant at 3 ounce per base box ($1{\cdot}1 \times 10^{-5}$ inch), the correction to be deducted is 1 ounce per base box (4×10^{-6} inch). The tin coating may be protected from the action of the stripping solution by a coating of Necol varnish, thus permitting the determination of thickness on a selected area.

The author is indebted to Mr. J. C. Prytherch, B.Sc., for carrying out some further confirmatory tests.

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The Micro-Determination of the Molecular Weight of Volatile Liquid Compounds

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(*Read at the Meeting of the North of England Section, April* 14, 1934)

THE microscopical method originally devised by Barger,[1] and more recently modified by Rast,[2] is probably the earliest method of determining molecular weight on only a few mgrms. of solute. No liquid solutes of low boiling-point were examined by Barger, and it appears from an examination of the original paper that the method is not suitable for such liquids.

Various forms of apparatus have been described[3–6] for the micro-determination of molecular weight by ebullioscopic methods, but no simple micro-apparatus appears to be available for the ebullioscopic determination of the molecular weight of volatile liquids in particular.

The simple cryoscopic method of Rast[2], in which the substance is dissolved in camphor, presents difficulties when applied to the particular case of volatile liquids, on account of the possible loss of liquid by evaporation. Vapour-density methods appear to be superior to all others for the examination of volatile substances.

Two micro-methods for the determination of vapour-density have been described by Bratton and Lochte,[7] and by Niederl and Saschek,[8] respectively. The micro-vapour density apparatus here described gives reasonably accurate results, and is less complicated than those referred to above.

The apparatus is a modification of the macro-form devised by P. Blackmann, and described by him in a number of papers.[9] It consists essentially of a stoppered glass vessel *A* (Fig. 1), a fine bore capillary tube *B* (sealed at one end and containing a short thread of mercury) (Fig. 2), and a small stoppered weighing bottle *C* (Fig. 3).

Before using the apparatus it is necessary to determine the capacity of the vessel *A* when it contains the tube *B* and the bottle *C* (unstoppered). This is done by weighing: (i) when filled with air; (ii) when completely filled with distilled water. The capillary tube *B* is most conveniently prepared in the following manner:

A piece of thermometer stem is very thoroughly cleaned and dried. One end of the tube is sealed in the Bunsen flame, and the open end is immediately dipped into a little pure dry mercury; when the mercury has risen in the capillary tube to a height of about 0·6 cm. the tube is withdrawn and allowed to cool to room temperature.

The determination of molecular weight is carried out in the following manner:—The capillary tube *B* is clamped in a vertical position, and the length (L) of the air-thread enclosed by the mercury is measured by reading the divisions marked on the tube. The temperature (t_1) is recorded at the same time.

The capillary is now introduced into the vessel A, together with the weighed liquid contained in the stoppered bottle (C). The apparatus is set in a vertical position, and the stopper D is brought into position by means of the thread attached to it.

The length (L_c) of the air-thread is now measured as before. If the stopper (D) has been manipulated with sufficient care, the value (L) does not usually differ from L_c.

It is absolutely essential that the closed apparatus shall be air-tight during the next stage of the determination. The joint between the stopper D and the edge of the neck of the vessel A is therefore covered with a small amount of a concentrated solution of celluloid in amyl acetate.

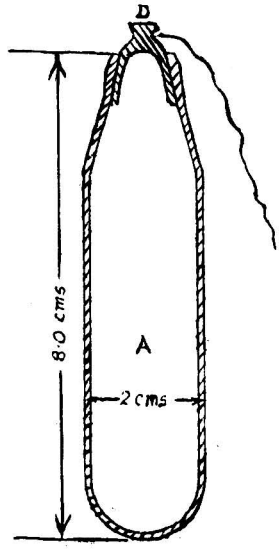

Fig. 1.

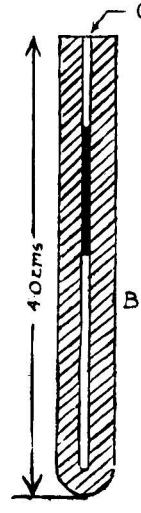

Fig. 2.

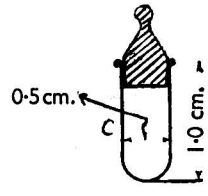

Fig 3.

When this solution has evaporated to dryness, the apparatus is heated in a suitable oven to a temperature (t_2) about 40° C. above the b.pt. of the particular liquid under examination, and the length (k) of the air-thread is measured at short intervals during a period of about 5 minutes. The minimum value is recorded as the correct length.

The vapour-density is calculated from the formula

$$d = \frac{31068wkL_c\,(273 + t_1)}{PLV\,(L_c - k)}$$

where d represents the density relative to hydrogen; V, the volume of vessel A determined as already described; w, the weight of substance taken in grms.; and P, the atmospheric pressure in mm. of mercury. The molecular weight is obtained by multiplying the value of "d" by two.

The derivation of the formula is given by Blackmann in the references at the end of this paper.

The following table contains some of the results obtained for the molecular weight of acetone. (d = 29·0.) (Mol. weight = 58·0.)

V Ml.	P Mm.	t_1 °C.	W Mgrm.	L Divs.	L_c Divs.	k Divs.	d
13·93	768·8	22·5	8·84	12	12	9·5	28·8
,,	,,	21·0	7·14	,,	,,	9·8	27·12
,,	,,	23·0	13·10	,,	,,	8·8	30·90
,,	,,	23·0	9·15	,,	,,	9·3	27·10
,,	,,	23·5	10·40	,,	,,	9·1	28·10
,,	,,	23·5	8·80	,,	,,	9·6	30·30

I wish to express my thanks to the Directors of Imperial Chemical Industries, Ltd., for permission to publish this work, which was carried out in the Research Laboratory of their subsidiary Company, Imperial Chemical Industries (Alkali), Ltd., Northwich.

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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 APPLICATION OF FILTERED ULTRA-VIOLET RAYS IN THE INVESTIGATION OF POISONING CASES

VARIOUS Continental workers[1] have carried out researches on the use of filtered ultra-violet light in the medico-legal detection of poisons.

Among these, Danckwortt and Pfau[2] express the opinion that inorganic substances, and certain alkaloids, especially morphine, lend themselves to detection by filtered ultra-violet light.

Examination of inorganic substances is carried out by direct exposure, while with solutions Goppelsroeder's method of capillary analysis, as modified by Ernst and Jentschitsch,[3] is used.

In the writer's experience the only fluorescent substance of medico-legal importance in the inorganic group is calomel, which, when exposed to the rays, exhibits a brilliant salmon-pink luminescence. As regards alkaloids in solution the fluorescence shown is in no way specific or characteristic.

Since many different substances give the same colour zones, no useful purpose is served by the employment of filtered ultra-violet light, more particularly when

the range of delicate analytical tests at present available permits of accurate results. Furthermore, the presence of impurities, by giving rise to adventitious fluorescence, may completely mask any results obtained.

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THE DETERMINATION OF TARTARIC ACID IN CIDER

To 100 ml. of cider, containing about 5 per cent. of alcohol (by vol.), are added 2 ml. of glacial acetic acid, 0·5 ml. of potassium acetate solution (20 per cent.), and 15 grms. of pure potassium chloride in the form of powder. When solution is complete, 15 ml. of 95 per cent. (by vol.) alcohol are added, the mixture is stirred thoroughly, and the beaker is covered and allowed to stand overnight. Its contents are then filtered through a Gooch crucible charged with paper pulp, and the residue is washed three times with 20 ml.; in all, of wash liquor (15 grms. potassium chloride in 100 ml. of water, and 20 ml. of 95 per cent. alcohol). The pulp and precipitate are returned to the beaker by means of hot water, and the liquid is titrated at boiling-point with 0·1 *N* sodium hydroxide solution (phenolphthalein as indicator). Each 1 ml. of alkali ≡ 0·015 grm. of tartaric acid.

This method, which is substantially the German 1921 wine method, has met with considerable criticism. With a few slight modifications, however, it may be made a useful routine test for cider.

(i) A solubility correction must be added to the titration figure. This is usually given as 1·5 ml. of 0·1 *N* sodium hydroxide solution, but I find it to vary from 0·7 ml. at 15° C., to 1·15 ml. at 20° C. and to 1·6 ml., at 25° C. These are averages.

(ii) National mark ciders may not contain more than 1 grm. per litre of tartaric acid. Here precipitation of potassium hydrogen tartrate may be very slow and incomplete. Hence, 0·2 grm. of pure potassium hydrogen tartrate should be added before the alcohol, and subtraction from the titration figure must be made, besides addition of the solubility correction. Examples:

	A Per Cent.	B Per Cent.
With potassium hydrogen tartrate	0·057	0·065
Without potassium hydrogen tartrate	0·024	0·025

(iii) Ciders may contain from 2 to 3 grms. per litre of tartaric acid. The potassium hydrogen tartrate will begin to settle out even before the alcohol is added. The 0·5 ml. of potassium acetate is inadequate, but 2 ml. have been found sufficient in all cases examined. Thus:

	C			D	
	Potassium acetate solution	Tartaric acid Per Cent.		Potassium acetate solution	Tartaric acid Per Cent.
	0·5 ml.	0·292		2 ml.	0·312
	1·5 ml.	0·312		3 ml.	0·308
	3 ml.	0·309	(Saponified)	2 ml.	0·321
(Saponified)	2 ml.	0·318			

(iv) Variations in alcohol-content of the cider.

Alcohol Per cent. (by vol.)	Solubility corr. at 20° C. Ml. of 0·1 *N* NaOH
0	1·35
5	1·05
10	0·9
15	0·8

The error due to this cause is insignificant. These conclusions are exemplified by the following determinations of tartaric acid in two ciders:

	Potassium acetate Ml.	Potassium hydrogen tartrate Grm.	Tartaric acid Grm.	Sodium hydroxide solution Ml.	Tartaric acid found Per Cent.
5 per cent. alcohol	0·5	0·2	—	9·5	(corr. 1·1 at 18·5°C.)
Cider E	0·5	0·2	—	13·7	0·063
,, ...	2	—	0·2	16·2	0·260
,, F	0·5	0·2	—	15·05	0·083
,, ...	2	—	0·2	17·7	0·282
Standardisation ..		0·2		10·6	

(v) *Influence of colloids.*—Whilst malic, lactic and citric acids, tannin, sugar and potassium phosphate do not interfere with the accuracy of this test, additions of pectin in the proportions present in cider cause the results to be low.

Pectin, per cent.	0	0·05	0·1	0·2	0·3
Tartaric acid found	0·197	0·190	0·187	0·186	0·185

When weighed quantities of tartaric acid are added to cider the results are generally slightly low. The deficiency is independent of the amount of tartaric acid present, and from the average of my tests amounts to 0·0075 grm. Some allowance for this can be made by increasing the solubility allowance by 0·5 ml.

Tartaric acid, freshly added to cider, can be fairly accurately accounted for, but not when it has been added for some time. Bacterial destruction takes place, perhaps esterification occurs, and precipitation of potassium hydrogen tartrate, and calcium tartrate has been encountered.

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A NEW CONSTANT FOR FIXED OILS—HYPOCHLOROUS ACID VALUE

THE substitution which occurs in the ordinary methods of determining the degree of unsaturation of an oil is to a large extent eliminated by using hypochlorous acid as the reagent. The method consists in saponifying the oil neutralising the solution (bromthymol blue as indicator), and then determining the amount of hypochlorous acid absorbed by the sodium salts of the fatty acids. It is not necessary to remove completely the alcohol used in the saponification.

Five ml. of sodium hypochlorite solution (prepared from bleaching powder solution and sodium carbonate, containing slight excess of sodium carbonate and brought approximately to the strength, 5 ml. ≡ 20 to 25 ml. of $N/10$ thiosulphate solution) and excess of potassium iodide solution are put into a conical flask and acidified with sulphuric acid (dil.). The liberated iodine is titrated with thiosulphate solution, and the test is repeated with standard sulphuric acid solution; the

volume of the latter required just to liberate the full amount of iodine from the solution is thus determined. From the reactions—

$$(a)\quad Na_2CO_3 + H_2SO_4 = Na_2SO_4 + H_2O + CO_2$$
$$(b)\quad 2NaOCl + H_2SO_4 = Na_2SO_4 + 2HOCl$$
$$(c)\quad 2HOCl + 4KI = 2KOH + 2KCl + 2I_2$$
$$(d)\quad 2KOH + H_2SO_4 = K_2SO_4 + 2H_2O$$

the amounts of sulphuric acid required, respectively, to neutralise the sodium carbonate of the hypochlorite solution, to liberate hypochlorous acid and to neutralise the potassium hydroxide in equation (*c*) are determined. From 0·12 to 0·125 grm. of oil is saponified in the usual way with 25 ml. of approx. *N*/5 alcoholic potassium hydroxide solution. The excess of the alkali is exactly neutralised with standard hydrochloric acid, bromothymol blue being used as indicator, and the alcohol is then evaporated on the water-bath until a pasty mass remains. The soap is dissolved in water, and the solution is transferred to a 750-ml. flask and diluted to 600 ml. To this 5 to 8 ml. of sodium hypochlorite solution are added, followed by just enough sulphuric acid of known strength to neutralise the free sodium carbonate and to liberate hypochlorous acid from the hypochlorite. The flask is quickly closed by means of a rubber stopper fitted with a dropping funnel containing potassium iodide solution, well shaken, and kept in a dark and cool place for 5 to 15 minutes. The potassium iodide solution in the funnel is then blown in and the flask well shaken, after which the dropping funnel is washed and taken out. The solution is now acidified with excess of dilute sulphuric acid, and the liberated iodine is titrated with standard thiosulphate solution. A blank test is made at the same time, and the difference between the thiosulphate figures gives the amount of hypochlorous acid absorbed (1 ml. N/10 thiosulphate ≡ 0·002625 grm. of HOCl).

The following results are typical of those obtained:

Oil or fat	Iodine value = HOCl value	Time of absorption	Hypochlorous acid absorbed Per Cent.
Arachis	87·6	18·1	15 minutes	18·0
Buffalo ghee	35·4	7·3	do.	7·8
Coconut	9	1·9	do.	1·9
Olive	88	18·2	do.	17·6
Mustard	108	22·3	do.	22·3
Sesame	110	22·7	do.	22·1
.. ..	—	—	1 hour	22·6
Linseed	185	38·2	15 minutes	35·1
.. ..			2 hours	35·3
Fish (*Clupea ilisha*) ..	88	18·2	15 minutes	17·8

The results obtained in these and analogous experiments have shown that the absorption is complete in 5 to 15 minutes, and that the time factor has no material influence.

We wish to express to Dr. H. K. Sen our thanks for his advice throughout the work.

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NOTE ON THE EXAMINATION OF DRIED GREEN MINT

VERY considerable quantities of mint (*Mentha viridis*, Linn.) are grown for culinary use, apart from the crops raised for distillation of the essential oil.

Although the herb has no food value, it is consumed, in the aggregate, in quite notable amounts, and although a few shipments from abroad have been found adulterated with other leaves, it is not usual for a herb, which grows near the soil and needs careful cultivation to keep it clear of weeds, to be found mixed with the leaves of a tree growing to a considerable height.

The identification of adulterants is not a difficult matter for anyone with a knowledge of plant histology, but there are other factors which determine the quality of mint, and of these the following are of significance:—(i) Absence of an undue proportion of stem; (ii) freedom from old brown leaf; (iii) absence of earthy impurity and sand; (iv) presence of a due proportion of volatile oil.

ANALYSES OF DRIED MINT

Total ash Per Cent.	Alkalinity of water-soluble ash, as K_2O Per Cent.	Ash insoluble in hydrochloric acid Per Cent.	Volatile oil by volume Per Cent.
12·15	1·94	1·84	1·1
10·78	1·02	0·63	
11·75	1·87	0·99	1·4
11·03	1·58	0·65	
10·95	1·97	1·10	
10·43	1·35	1·20	
9·7	1·44	0·85	1·41 (English)
10·76	1·21	1·2	
11·31	1·61	0·82	0·92 (French)
9·81	1·23	0·82	
10·29	1·42	0·78	0·84
10·78	2·37	10 per cent. stalk	
12·15	2·04	8 per cent. stalk	
10·39	2·35	— (Belgian)	
9·37	1·67	0·43 (*Ailanthus* present).	

The herb grown in this country is sold, in season, in the fresh green state, but for out-of-season requirements it is dried and rubbed through sieves. If the herb is cut before it has attained the flowering stage the bulk of the stem is too large to pass through a number 10 sieve, and, consequently, the requirement that dried rubbed mint shall contain not more than 2 per cent. of stems above 3 mm. in diameter, is a reasonable one, but it does not prevent the inclusion of a quantity of almost wire-like small stems if the crop had been left too late before harvesting.

The presence of brown leaf means diminished flavouring qualities.

Earthy impurities, and more particularly sand, should be strictly limited. The presence of both is usually indicative of harvesting in wet weather, which splashes soil on to the leaves, and some limit ought to be recognised for such impurities. The "ash" is a good indication of the cleanliness of the sifted herb, and the amounts which are soluble in water and the proportion insoluble in hydrochloric acid are a useful guide to quality and genuineness.

The amount of essential oil in the fresh herb is variable, and the process of drying introduces an unknown loss, but the flavour of a well-dried "mint" depends on the proportion of oil it contains.

The analyses of a number of typical samples, representing many hundreds of bales, have afforded me the following figures, which should be some guide to the quality of the supplies marketed. The greater number were of foreign

origin, but it is hoped to increase the home-grown crop, and, if possible, to improve the flavour by more satisfactory methods of drying.

The examination of the mint for arsenic showed its presence in a few samples in proportions up to one-thirtieth of one grain per pound. This may be attributable to the contamination with earthy matter which contained it, or possibly to the use of arsenical sprays in the vicinity of the growing herb.

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LOSSES IN MEAT ON COOKING

WE have read with great interest, and not a little astonishment, the note on the above subject on page 405 of the June issue of the ANALYST. This Laboratory has been associated with the investigation of chilled beef and frozen beef from the Argentine and Australia for over forty years—in fact, since the early days of commercial shipments of chilled meat from the Argentine. We have done numerous cooking experiments, but we have never obtained such divergent results between the losses on cooking of fresh English beef and chilled beef. With frozen beef one would expect much greater losses, owing to exudation of the contents of the broken cells—a phenomenon which can readily be observed under the microscope. In the case of chilled beef, stored during transit at temperatures round about 30° F., the cells are not broken or burst, and there would appear to be no reason why such chilled beef should show excessive loss of weight on cooking.

We understand that Mr. Arnaud's results have been based upon cooking pieces of steak of about 1 lb. weight. In our own experiments, carried out recently, we were unable to confirm his results upon such small samples, and we also extended the experiments to include joints of various sizes.

In order to place the reliability of the samples beyond question, Lt.-Colonel Dunlop Young, O.B.E., the lately retired Chief Meat Inspector at Smithfield, kindly consented to purchase the meat in the ordinary way of retail trade from a butcher in the City, and to hand the meat to us.

By courtesy of the Gas Light & Coke Company we were provided with two carefully equalised gas cookers (New World No. 3 with Regulo Control), and on each occasion an English joint was cooked in one oven, and an Argentine chilled joint, of approximately equal weight was cooked in the other. A member of the staff of the Gas Company assisted with the cooking and acted as a neutral observer of the whole of the experiments.

Each joint was carefully weighed just before it was placed in the oven, and weighed again after it had been cooked and had cooled, and the dripping and gravy were each carefully weighed. The digestion experiments were carried out upon 10 grms. of the minced lean, in 100 ml. of distilled water containing 1 ml. of 20 per cent. hydrochloric acid and 0·5 grm. of pepsin. This mixture was placed in an incubator at 37·5° C. for four hours, and was then filtered, after which the undigested fibrin was dried and weighed. The moisture was determined in the original lean meat after cooking, and the undigested fibrin was calculated on the dried lean. The mineral matter was determined by ashing the wet lean in platinum crucibles.

These figures, which have been obtained as the result of unbiassed investigation by three independent workers, show that the loss of weight on cooking English beef and Argentine chilled beef is approximately identical. Moreover, the digestibility, mineral-content and nett loss of weight on cooking are also so nearly identical as to make it quite safe to say that meat of either kind is equally economical, quite apart from price.

LOSS OF WEIGHT ON COOKING ENGLISH JOINTS AND CHILLED ARGENTINE JOINTS AND THE RESPECTIVE DIGESTIBILITY AND MINERAL-CONTENT OF THE COOKED MEATS

Nature of joint	Weight of joint ozs.	Weight of joint after cooking ozs.	Gross loss of weight ozs.	Per-centage loss of weight (gross)	Weight of dripping ozs.	Weight of gravy ozs.	Weight of joint + dripping + gravy ozs.	Nett loss of weight ozs.	Per-centage loss of weight (nett)	Moisture in lean (dried at 105° C.) Per Cent.	Mineral matter in wet lean Per Cent.	Un-digested matter (calc. on dry lean) Per Cent.
Scotch sirloin	78·75	61	17·75	22·5	8·25	0·2	69·45	9·3	11·7	63·25	1·0	20·7
Argentine sirloin ..	78	59	19	24·3	9·25	0·4	68·65	9·36	11·8	67·5	1·1	15·0
English wing end ..	183	126	57	31·1	29·75	1·25	157	26	14·2	58·4	1·0	17·8
Argentine wing end ..	182·25	120	62·25	34·0	32·0	2·0	154	28·25	15·4	59·2	1·05	13·5
Grilled English steak ..	15·5	13·5	2	13·0	—	—	—	2	13·0	65·9	1·00	20·7
Grilled Argentine steak..	17·0	15·0	2	11·7	—	—	—	2	11·7	63·7	1·27	21.6
English top side.. ..	80·0	55·25	24·75	30·9	9·6	1·3	66·25	13·75	17·2	66·1	1·20	16·3
Argentine top side ..	81·5	56·75	24·75	30·3	8·25	1·36	66·06	15·44	19·5	66·6	1·22	11·5
English wing rib ..	161	110	51	31·6	26·68	1·06	137·75	23·25	14·3	61·5	1·02	23·5
Argentine wing rib ..	178	122	56	31·4	29·5	1·19	152·69	25·31	13·4	64·7	0·97	36·0
English fried rump steak	17	14·75	2·25	13·2	—	—	—	2·25	13·2	65·5	1·07	22·1
Argentine fried rump steak	18·75	16·0	2·75	14·4	—	—	—	2·75	14·4	65·8	1·10	19·8
English top side.. ..	80	56·75	23·25	29·4	8·06	1·18	66	14	17·9	67·0	1·20	16·3
Argentine top side ..	80·5	53·5	27	33·7	10·5	0·5	64·5	16	20·0	63·7	1·12	18·2

These results may be summarised as follows:

	Argentine chilled beef Per Cent.	English beef Per Cent.
Average gross loss of weight	25·7	24·5
Average nett loss of weight (taking account of dripping and gravy)	15·1	14·6
Mineral matter in wet lean after cooking	1·12	1·07
Undigestible matter (calculated on dried lean)	19·3	19·6

The results obtained by Mr. Sciver have been submitted to me by the Editor.

The four pieces of meat enumerated in my experiments already published (*loc. cit.*) were supplied to me by a butcher holding a high position in his trade. Each piece of meat was labelled, and I still possess the labels. In answer to an enquiry addressed to him, the butcher concerned replied:—"There can be no question that the piece of steak despatched to you . . . with label attached was chilled Argentine, as described. Unfortunately, I kept no record of any particular brand, but it must have been 'X' or 'Y,' as my purchase book shows.

"You may be sure that I was very particular with regard to the despatch of the samples; in fact, I cut and packed them myself."

A further sample of meat upon which similar experiments were made was supplied by another butcher, and he also assures me that the meat supplied was Argentine chilled.

The results published were obtained on meat cooked and weighed by me both before and after cooking.

F. W. F. ARNAUD

SESSIONS HOUSE
MAIDSTONE, KENT

Notes from the Reports of Public Analysts

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

CITY OF BIRMINGHAM

REPORT OF THE CITY ANALYST FOR THE FIRST QUARTER, 1934

OF the 1481 samples submitted by the Food and Drug Inspectors during the first quarter of the year, 1424 were taken informally. Fifty-four samples were adulterated, and two were incorrectly labelled.

ASPIRIN TABLETS.—One, out of five samples, was of very inferior quality, and contained 2 per cent. of free salicylic acid, corresponding with 2·6 per cent. on the aspirin used; the B.P. limit for salicylic acid is 0·05 per cent., so that over fifty times too much was present. In addition, over 6 per cent. of French chalk was contained in the tablets—an unusually large quantity. Most tablets contain no insoluble mineral matter, and it seems quite unnecessary to incorporate any such material in tablets of this kind. No further action could be taken, as the tablets in question were old stock and the packers had gone out of business.

DRIED MINT.—Two samples were found to be adulterated with about 25 per cent., and 35 per cent., respectively, of *Ailanthus* leaves. There have been, during the last twelve months, a number of prosecutions in this country, both of retail vendors and of wholesale firms, for the sale of such adulterated mint (*cf.* ANALYST, 1934, 535), and it has been shown that the adulteration has been carried out before importation and that a number of reputable wholesale firms have been deceived. The firms concerned with the supply of the two samples mentioned were communicated with, and both have recovered all the adulterated mint supplied to the shops in question and have done everything in their power to collect as much as possible from their retail customers all over the country.

TEA "WITHOUT TANNIN."—One sample was stated on the label to contain all the essential goodness of tea without any injurious tannin. The analysis showed that 13·9 per cent. of tannin was present.

The label on another sample (China tea) described it as practically free from tannin, a quite incorrect statement, since 8·6 per cent.—a normal amount for China tea—was found to be present. Both the firms concerned were cautioned, and agreed to discontinue the use of the incorrect descriptions.

H. H. BAGNALL

CITY OF LEEDS

ANNUAL REPORT OF THE CITY ANALYST FOR 1933

THE total number of samples analysed in 1933 was 3696, of which 1985 were food and drugs, and 69 were fertilisers and feeding stuffs. Of the food and drug samples, 186 were adulterated.

FORM OF MILK CERTIFICATE.—During the hearing of a case the Stipendiary Magistrate asked me if it would not be better to base the percentage of added water on the freezing-point rather than on the non-fatty solids, and on being told that such a course had already been adopted by one County Council who were making use of Section 4(1) of the Milk and Dairies (Amendment) Act, 1922, His Worship said that due consideration might be given to this matter. The outcome of this suggestion has been a re-drafting of the form of certificate, following a consultation with the Town Clerk, who decided, however, not to proceed under the Milk and Dairies (Amendment Act), 1922, which forbids, amongst other things, the addition of water to milk intended for sale, and the sale of any milk to which any such addition has been made, but to make continued use of the Food and Drugs (Adulteration) Act, 1928. In a case where a milk contains less than the 8·5 per cent. of non-fatty solids required by the Sale of Milk Regulations, 1901, and has a freezing-point higher (that is, nearer 0° C.) than −0·530° C., the minimum amount of water added to the milk is now calculated from the freezing-point.

This was done in the successful prosecution of two retailers in partnership, for selling milk containing at least 26 per cent. of added water. The matter was carried a step further when a farmer appeared on February 7th for selling 2 samples of milk which the freezing-point showed to contain at least 3·8 per cent. and 4·2 per cent. of added water, respectively.* Before use was made of the freezing-point, it was the practice to prosecute only in respect of milks containing a minimum of 5 per cent. of added water (even though satisfactory appeal-to-cow samples had been obtained within 48 hours) in order to allow for the remote possibility of an appreciable day-to-day variation in the composition of the milk.

SAGO.—All 4 samples submitted proved to be tapioca. As a result, a letter was sent by the Medical Officer of Health to the Leeds and District Grocers and Provision Dealers Association, and this letter was read at a meeting of the latter on February 28th, 1934, following which some prominence was given to the matter in the local press, and since then one firm has displayed in its window the various forms of tapioca on sale. These are Flake, Seed Pearl, Medium, and Bullet. Sago is usually of the same size as pearl tapioca, but is brown.

BISMUTHATED MAGNESIA TABLETS.—One sample was bought and found to be 6·8 per cent. deficient in the amount of bismuth carbonate declared, and 34·2 per cent. deficient in magnesium carbonate. There was also present 9·3 per cent. of talc, which was undeclared on the label. The retailers were communicated with respecting these matters.

C. H. MANLEY

* The first sample contained 3·15 per cent. of fat and 8·10 per cent. of solids-not-fat, and the second 3·70 per cent. of fat and 7·90 per cent. of solids-not-fats. The figures for Δ were 510 and 508, respectively. An appeal-to-cow sample gave: fat, 3·70; solids-not-fats, 8·70 per cent.; Δ, 547.—C. H. M.

Department of Scientific and Industrial Research

FUEL RESEARCH

ANALYSIS OF COMMERCIAL GRADES OF COAL. PART II*

THIS report marks the completion of a systematic examination of the commercial grades of coal produced and marketed in the South Yorkshire, Nottinghamshire and Derbyshire coalfield. It deals with the output of 17 collieries producing 130 grades of coal, which range from those yielding good metallurgical coke to non-coking coals. They include the well-known Yorkshire "hards," first-class locomotive fuel, as well as general steam-raising, gas and household coal.

The sampling was carried out at the pit-head—at the conveyor belts or at chutes delivering the coal to wagons or store. The period of sampling and the number and size of the increments were decided after a consideration of the hourly fluctuations of the coal. The gross samples were taken by hand, but the reduction to laboratory size was carried out in a mechanical crushing and sampling equipment mounted on a commercial motor van and operated from the engine by auxiliary shaft and driving gear.

The work done on these samples and the presentation of the results follow exactly the lines of a previous report (Survey Paper No. 20) dealing with 39 collieries in the same area. Each colliery is dealt with separately, the grades of coal samples from it are listed, and the sizes are given. For each grade is given a proximate analysis, the contents of sulphur, nitrogen and chloride, and the calorific value. In addition, the percentage of phosphorus is given for grades that are suitable for coke manufacture, and, in certain cases, the results of ultimate analyses and of laboratory carbonisation assays at 600° C.

The sampling of commercial grades of coal at the pit-head is a natural corollary to the examination of the seams as they occur below ground, which is being undertaken under the Fuel Research Board in the Physical and Chemical Survey of our National Coal Resources. A study of the seams shows us the material as it is or might be mined or marketed, where to look for this or that quality, how best to conserve our supplies of those coals suited for special purposes, and how to develop workings to best advantage. Commercial sampling shows us the material as it is actually handed over to the consumer, sometimes as the carefully prepared product of a single seam, sometimes as a blend of several seams.

In the South Yorkshire area the Parkgate, Barnsley and Silkstone, three of the chief seams, have already been examined as they occur in the field, and this part of the work is continuing. In most cases in the present report one or more of these seams forms part of the commercial output examined, and it is interesting to note the close agreement between the results obtained and those given by samples of the seams cut underground.

* Fuel Research Survey Paper No. 31. Yorkshire, Nottinghamshire and Derbyshire Coalfield. Part II. H.M. Stationery Office, 1934. Price 9d. net.

Government of Madras

ANNUAL REPORT OF THE CHEMICAL EXAMINER FOR THE YEAR 1933

In his Annual Report Lt.-Col. Clive Newcomb states that the work of the department shows a marked increase over the preceding year (1518 cases with 7129 articles, as against 1437 cases with 6136 articles in 1932). The rise is due entirely to the amount of medico-legal work, the general analyses having diminished from 198 articles in 1932 to 170 in 1933. It is probable that the increase is due to greater care in the investigation of crime rather than to an increase in crime.

Human Poisoning Cases.—During the year there were 372 cases, with 2116 articles, in which 450 persons were affected and 277 died. Poison was found in 173 cases, opium heading the list with 28 cases, and oleander coming next with 27 cases. Arsenic was found in 22 cases, and mercury in 17, and there were three cases of poisoning by datura or other mydriatic alkaloidal poison.

Dried Snake Venom as Poison.—In a case of attempted suicide to avoid a charge of murder, a man was admitted to the hospital semi-conscious and in a state of collapse. It was stated that he had injected into himself some poison through a small wound on the left thigh. White arsenic (9/10 grain) was found in the viscera. One of the articles received in this case was a packet containing yellowish translucent scales. The powder had the usual appearance and chemical properties of dried snake venom, and was definitely proved to be snake venom, by its fatal effects when injected into a guinea pig, and by the absence of fatal effects when injected along with anti-venin serum. The man had therefore probably taken arsenic by the mouth, and, as an additional precaution, injected snake venom into the wound in his thigh, in a resolute attempt to commit suicide.

Arsenic in Love Potions.—Love potions are not unusual articles to be sent for examination and frequently contain dangerous poisons. One examined this year contained 3½ grains of white arsenic—a dose which would very likely prove fatal. It would be questionable if these so-called love potions are not sometimes given on account of love for some person other than the recipient. Where a wife has given poison to a husband it forms a useful defence, if need be, to aver that the poison was given as a love potion, as in the following case.

Barium Poisoning.—A man was given some powder in soup by his wife and subsequently developed symptoms of acute gastro-intestinal irritation. The next day, although vomiting had ceased, he had severe pain in the abdomen and numbness and pains in his limbs. Gradually his speech became incoherent, and he became unconscious and died on the third day. The powder said to have been given was found to be a mixture of barium carbonate and barium sulphide, and the viscera were all found to contain barium. In some of the bed linen soiled by excretions we were unable to find any barium. The wife was acquitted chiefly on two grounds: (1) the judge held that proof of poisoning by barium was incomplete, in that the doctor who did the post-mortem had not stated that the rectum was congested, and this is said in some books to be a sign to be found in fatal cases of barium poisoning, and in that we had not found barium on the bed linen; and (ii) the woman gave a defence that she had only given the powder as a love potion.

Copper Sulphate Poisoning.—Although copper sulphate is not a very deadly poison on account of the violent vomiting it produces, it sometimes happens that, owing to collapse, vomiting is insufficient to eliminate the poison, and the victim dies. In one such case copper was found in all the viscera analysed, and the amounts found are of interest, as this is not a very common form of fatal poisoning.

A woman died suddenly. The stomach contained some bluish-white material, and the lungs, liver and kidneys were all congested. The stomach contained

copper equivalent to about 32 grains of crystalline copper sulphate, and the stomach-contents contained about $10\frac{1}{3}$ grains. In the small intestines the amount of copper was equivalent to about 18 grains of the sulphate, in the liver it was equivalent to about 2/5 grain, and in one kidney the amount was equivalent to about 3/5 grain.

Madar Juice Poisoning.—From time to time cases of fatal poisoning from drinking the milky juice of the plant *Calotropis gigantea* are investigated in the laboratory. The symptoms of madar juice poisoning are not very clear, as in most cases little information is available as to their nature. It would appear to be not only a gastro-intestinal irritant, but also a cerebro-spinal poison.

In one case a woman drank the juice at about 7 a.m. The symptoms began at 7.30 with vomitings and purgings followed by convulsions in which she rolled on the ground. She collapsed and died at about 8 a.m. The post-mortem signs were bloody discharges in the nostrils and mouth, the lungs were congested, and the heart was empty. Trachea was injected, liver, spleen and kidney were congested. The stomach was congested and contained about 2 oz. of chyme-like fluid. The small intestines were congested, the brain and the membrane were also congested. The reactions of madar juice were given by the stomach and its contents in this case.

In another case of post-mortem there was no discharge from the nose and mouth. The lungs were congested, the pericardium was congested, the heart was empty and congested and there was congestion of the trachea, of the stomach, liver, spleen, small intestines and brain. A four-months-old foetus was found in the uterus. The reactions of madar juice were obtained from the stomach and contents in this case.

In addition to being taken by the mouth, madar juice is often used as an abortifacient, and, when so used, is inserted into the uterus on a small tampon.

Further investigation of the composition of madar juice has been made. One hundred ml. of the juice yielded 15·12 grms. of total solids, and 1·75 grm. of ash. The crystalline extract obtained from an alcoholic extract of the juice after saponification and extraction with ether (ANALYST, 1934, 37) has been examined. After repeated crystallisation it yielded a white crystalline substance containing: Carbon, 80·5; hydrogen, 11·53, and oxygen, 7·93 per cent., corresponding with the formula, $C_{27}H_{46}O_2$; mol. weight (average of several determinations) 405; iodine value (pyridine sulphate dibromide method), 63·2.

In routine analysis of articles for madar juice the following system of examination can be used:—To the alcoholic extract of the suspected substance alcoholic potash is added until the liquid is alkaline, and the whole is heated on a water-bath to drive off the alcohol. The residue is taken up with water and extracted with ether. The ethereal extract is tested as mentioned in the last annual report (*loc. cit.*). Presence of madar is further confirmed by distilling the above-mentioned aqueous residue after extraction with ether, with alcohol and 50 per cent. sulphuric acid. The characteristic smell of madar esters may be recognised in the distillate.

Poisoning with Marking Nut.—A man was accused of pouring some blistering liquid on to a woman, and stained portions of cloth were sent for examination. The stains were extracted with alcohol, the alcoholic extract was treated with lead acetate to precipitate proteins and organic acid and filtered, lead was removed from the filtrate by means of ammonium oxalate, and the filtrate from the lead precipitate was diluted with water and extracted with petroleum spirit. The extract, when evaporated, left a thick brownish liquid, becoming bluish-green on addition of potassium hydroxyde (a chemical reaction of marking nut). A little of the liquid, when rubbed on the skin of the hand, produced, after two days, very severe irritation and blistering, the blisters tending to spread along the margin until the whole of the back of the hand was swollen and blistered. There was intense itching and oozing of serum, and the back of the hand was stained black

for some weeks where the juice had been applied. The delayed action of marking nut juice or of the extract from it is very striking, and makes it a very dangerous poison (*cf.* ANALYST, **1932, 57, 796**).

Areca Nut Poisoning.—The investigation on areca nuts has been continued, with a view to throwing some light on cases of poisoning by areca nut occasionally reported. Air-dried areca nuts contained 11·0 per cent. of moisture, and yielded 11 per cent. of fat on extraction with ether. The fat melted at 49° C., had n_D^{51}, 1·450, saponification value, 203, and iodine value, 26·5. It had no toxic action on frogs. The nuts yielded 0·8 per cent. of ash. The alkaloids are difficult to extract, as they are only sparingly soluble in the common organic solvents, and the difficulty is enhanced by the fact that the small portion of the alkaloidal fraction extractable with ether volatilises at the temperature of the steam-oven. A few mgrms. of an alkaloidal fraction were obtained by extraction of an ammoniacal aqueous extract of the defatted sample with ether, followed by spontaneous evaporation of the ether. The residue gave precipitates with phosphotungstic acid and Mayer's reagent. A portion injected into a frog produced paralytic symptoms, but the frog ultimately recovered. The investigation is still in progress (*cf.* ANALYST, **1932, 57, 391**).

EXAMINATION OF BOMBS.—Forty-two cases (with 236 articles) in which bombs were concerned were investigated, and there were also 7 cases (with 24 articles) in which explosives other than explosives legitimately imported had been used.

Most of the exhibits were bombs or the remnants from the explosion of bombs of the kind usual in South India, *viz.* with a mixture of potassium chlorate and arsenic sulphide as the explosive agent. Such a mixture is safe to handle when wet, but, when dry, explodes very violently on percussion or friction. Experiments showed that the mixture was safe to handle while wet with alcohol, but after evaporation of the alcohol the experimental bomb exploded very violently when thrown on to a hard gravel path.

In a series of connected cases bombs of a different type were used. In these, gunpowder formed the explosive, and was set off by the action of sulphuric acid on a mixture of sugar and potassium chlorate. A brass vessel with a screw top, known as a *kooja*, was used as the container, and the whole bomb was a very dangerous affair, perhaps more dangerous to the man who was throwing it than to the persons at whom it was thrown, since the setting-off arrangements were very primitive, and the bombs were liable to go off (and *two* of them did) when they were not intended to do so.

Among the articles captured by the police in these cases were various materials for making these bombs. In addition to ordinary gunpowder, there was a mixture of potassium chlorate and nitrate as the oxidising agent, and dried coffee grounds instead of charcoal. Although not so good as a properly-made gunpowder, this mixture was quite a powerful explosive when set off in a confined space.

The danger of allowing the usual chlorate and arsenic sulphide mixture to dry was shown by an explosion of some exhibits which were being sent back to the local court after having been shown in the appeal court.

IDENTIFICATION OF BULLETS AND CARTRIDGE CASES.—Experiments have been made to find out with what degree of accuracy it is possible to identify bullets and cartridge cases.

In one such case a man had been shot, and a 380-lead bullet was recovered from his body at the post-mortem examination. On the man suspected of shooting him a 380-revolver was found. The question was—had this bullet been fired from this revolver? We fired about 24 rounds out of the revolver into water so that they should not be deformed, and proceeded to examine them. In diameter, the number of lands or grooves, and the width and pitch of the lands, all the

experimental bullets corresponded with the suspected bullet. A closer examination of the bullets under the microscope showed that on all of them were certain small scratches, too small to be seen with the naked eye, but sufficiently well marked or characteristically placed with reference to one or other of the lands to make one fairly certain that all the bullets had been fired from the same revolver. A comparison of these scratches under a comparison microscope settled the question, as the scratches in many of the lands exactly corresponded when seen together under this instrument, and no scratch which could be found on all the experimental bullets was absent on the suspected bullet. The comparison microscope is an instrument by which two bullets each placed under a separate microscope can be viewed simultaneously as if placed side by side. (*Cf.* Stirling, ANALYST, **1933**, **58**, 684.)

As a test of the accuracy of this method the police fired 24 bullets out of four revolvers, all of the same make and pattern, numbered the bullets haphazard, kept the key as to which had been fired out of which revolver, and sent the bullets to us. We found no difficulty in sorting out the bullets into four lots, and in every case we were correct.

In one case the question was asked—had a certain small hole in a blood-stained cloth been made by a bullet or by other means. The part of the cloth round the hole was soaked with blood, and this obscured any of the markings which we have previously shown (Annual Report for 1931: ANALYST, 1932, **57**, 718) to be characteristic of bullet holes. Analysis of a portion of the cloth, cut from the edges of the hole, showed the presence of lead, and analysis of other equally blood-stained parts of the cloth did not show any lead, so that there was good evidence that the hole in question had been caused by a leaden object having been forcibly pushed through the cloth, and with little doubt this object was a bullet.

IDENTIFICATION OF TYPESCRIPT.—The work reported in the annual report for 1932 (ANALYST, 1934, 39) on the identification of typescript with the typewriter on which it has been produced has also been continued, with the assistance of the police, and the accuracy of the method described has been put to a test as severe as we could make it. By the kindness of one of the firms who sell typewriters, 24 specimens of typescript were obtained from 12 machines, all brand new, all with the same sort of ribbons on the same paper, and all typed by the same typist, who was an expert at typing. When examined either with the naked eye or under a microscope they all looked perfect and identical, except six in which we found slight variations in one of the letters after prolonged examination. In the remaining 18 no differences could be seen, and in all of them the alignment of all the letters was, to ordinary observation, perfect. By microscopic measurement, however, of the alignment of about 200 of the nine commonest letters (E T A O I N S R H) in each of the 18, we were able to sort out 9 pairs, and a reference to the key, which had been kept locked up, showed that all the pairs were correct. The method is very tedious, but, given the time and patience and a sufficiency of the specimen of typescript, appears to be very reliable.

New U.S. Pharmacopoeial Standards for Cod Liver Oil*

THE new U.S.P.X.† (1934) Standards for vitamins *A* and *D* and Vitamin Assays have just been published. These have been developed through a series of conferences with the vitamin experts of the United States and an extensive study of the general standard by the Sub-Committee on Organic Chemicals. The vitamin standards and vitamin assays represent the studies and conclusions of the U.S. Pharmacopoeial Vitamin Advisory Board. The "release" of this announcement, as an interim revision, has been authorised by the U.S.P. Committee of Revision and U.S.P. Board of Trustees, the standards to become official on January 1, 1935.

The U.S.P. Vitamin Advisory Board also recently announced the "release" of "Reference Cod-liver Oils" of known vitamin *A* or vitamin *D* potency, expressed in U.S.P.X. (1934) Units. These "Reference Oils" are to be used as standards in vitamin *A* or *D* assays for determining the potency of Cod-liver Oil, Cod-liver Oil Concentrates, irradiated ergosterol or other irradiated products, or products containing vitamin *A* or vitamin *D*, whether medicines or foods. This Reference Oil is obtainable in 30-ml. containers and is standardised for either its vitamin *A* or vitamin *D* potency. The price per package of either the vitamin *A* or vitamin *D* standard is $2.50.

THE NEW U.S.P.X. COD-LIVER OIL STANDARDS

Minimum standard for Vitamin A *for U.S.P. Cod-liver Oil.*—The minimum Vitamin *A* standard for U.S.P. Cod-liver Oil shall be not less than 600 U.S.P. (1934) Vitamin *A* Units or its equivalent, 600 International Vitamin *A* Units.

Minimum standard for Vitamin D *for U.S.P. Cod-liver Oil.*—The minimum Vitamin *D* standard for U.S.P. Cod-liver Oil shall be not less than 85 U.S.P. (1934) Vitamin *D* Units or its equivalent, 85 International Vitamin *D* Units.

One "United States Pharmacopoeia Unit of Vitamin *A*" is equal in growth-promoting and anti-ophthalmic activities for the rat, to one International Unit of Vitamin *A*, as defined and adopted by the Conference of Vitamin Standards of the Permanent Commission on Biological Standardisation of the League of Nations in June of 1931; one "United States Pharmacopoeia Unit of Vitamin *D*" is equal, in anti-rachitic potency for the rat, to one International Unit of Vitamin *D* as defined and adopted by the Conference of Vitamin Standards of the Permanent Commission on Biological Standardisation of the League of Nations in June of 1931.

The Relation of the New U.S.P. Cod-liver Oil Vitamin Units to Other Units now in Use.—One of the valuable contributions made by the U.S.P. Vitamin Advisory Board in its study of assay reports submitted by many laboratories, when determining the potency of the U.S.P. Reference Cod-liver Oil, was an approximate relationship between the new U.S.P.X (1934) Units for Vitamins *A* and *D* (it should be remembered that these units are identical with the International Units) and other Vitamin Units now referred to on labels and in the literature.

These conversion factors have been published by the Vitamin Board with the hope that they may help to clarify the existing confusion due to the use of so many unofficial units, and with the hope that hereafter in the U.S.A. all vitamin *A* and *D* values will be expressed in the new U.S.P. Units.

* To become official January 1, 1935.

† U.S.P.X. came into force in January, 1926; the U.S.P.X., 1934, is a revision, not a new edition.—ED.

These conversion factors should not be considered as having any official recognition. They are probably not absolutely exact and are given for information only.

CONVERSION FACTORS FOR VITAMIN *A* AND *D* UNITS

Vitamin A *Units*

One U.S.P.X Vitamin A *Unit*, 1 *"Sherman Unit"* or 1 *A.D.M.A. Vitamin* A *Unit* are each approximately the equivalent of 1·4 International Vitamin *A* Units or 1·4 U.S.P.X (1934) Vitamin *A* Units.

The new U.S.P. minimum standard of 600 *U.S.P.X* (1934) *Vitamin* A *Units* per grm. of Oil is approximately the equivalent of

420 U.S.P.X (1926) VITAMIN *A* UNITS

Vitamin D *Units*

One Steenbock Unit of Vitamin D is approximately the equivalent of 2·7 International or U.S.P.X (1934) Units.

One International Vitamin D *Unit or* 1 *U.S.P.X* (1934) *Vitamin* D *Unit* is approximately the equivalent of

3·25 A.D.M.A. VITAMIN *D* UNITS

One International Vitamin D *Unit* or 1 *U.S.P.X* (1934) *Vitamin* D *Unit* is approximately the equivalent of

1·66 OSLO VITAMIN *D* UNITS

The new U.S.P. minimum standard of 85 *U.S.P.X.* (1934) *Vitamin* D *Units* per grm. of Oil is approximately the equivalent of

31·5 STEENBOCK UNITS
276 A.D.M.A. UNITS
142 OSLO UNITS.

Copies of the U.S.P.X (1934) interim revision of Cod-liver Oil Tests or the Reference Cod-liver Oil may be obtained by addressing the Chairman of the Committee of Revision.

E. FULLERTON COOK

FORTY-THIRD STREET AND WOODLAND AVENUE
PHILADELPHIA, PA.

International Atomic Weights, 1934*

	Symbol.	At. No.	At. wt.		Symbol.	At. No.	At. wt.
Aluminium ..	Al	13	26·97	Neodymium ..	Nd	60	144·27
Antimony ..	Sb	51	121·76	Neon	Ne	10	20·183
Argon	A	18	39·944	Nickel	Ni	28	58·69
Arsenic ..	As	33	74·91	Niobium	Nb		
Barium ..	Ba	56	137·36	(Columbium)	(Cb)	41	93·3
Beryllium ..	Be	4	9·02	Nitrogen ..	N	7	14·008
Bismuth ..	Bi	83	209·00	Osmium ..	Os	76	191·5
Boron	B	5	10·82	Oxygen ..	O	8	16·0000
Bromine ..	Br	35	79·916	Palladium ..	Pd	46	106·7
Cadmium ..	Cd	48	112·41	Phosphorus ..	P	15	31·02
Caesium ..	Cs	55	132·91	Platinum ..	Pt	78	195·23
Calcium ..	Ca	20	40·08	Potassium ..	K	19	39·096
Carbon ..	C	6	12·00	Praseodymium	Pr	59	140·92
Cerium ..	Ce	58	140·13	Radium ..	Ra	88	225·97
Chlorine ..	Cl	17	35·457	Radon ..	Rn	86	222
Chromium ..	Cr	24	52·01	Rhenium ..	Re	75	186·31
Cobalt ..	Co	27	58·94	Rhodium ..	Rh	45	102·91
Copper ..	Cu	29	63·57	Rubidium ..	Rb	37	85·44
Dysprosium ..	Dy	66	162·46	Ruthenium ..	Ru	44	101·7
Erbium ..	Er	68	167·64	Samarium ..	Sm	62	150·43
Europium ..	Eu	63	152·0	Scandium ..	Sc	21	45·10
Fluorine ..	F	9	19·00	Selenium ..	Se	34	78·96
Gadolinium ..	Gd	64	157·3	Silicon ..	Si	14	28·06
Gallium ..	Ga	31	69·72	Silver	Ag	47	107·880
Germanium ..	Ge	32	72·60	Sodium ..	Na	11	22·997
Gold	Au	79	197·2	Strontium ..	Sr	38	87·63
Hafnium ..	Hf	72	178·6	Sulphur ..	S	16	32·06
Helium ..	He	2	4·002	Tantalum ..	Ta	73	181·4
Holmium ..	Ho	67	163·5	Tellurium ..	Te	52	127·61
Hydrogen ..	H	1	1·0078	Terbium ..	Tb	65	159·2
Indium ..	In	49	114·76	Thallium ..	Tl	81	204·39
Iodine	I	53	126·92	Thorium ..	Th	90	232·12
Iridium ..	Ir	77	193·1	Thulium ..	Tm	69	169·4
Iron	Fe	26	55·84	Tin	Sn	50	118·70
Krypton ..	Kr	36	83·7	Titanium ..	Ti	22	47·90
Lanthanum ..	La	57	138·92	Tungsten ..	W	74	184·0
Lead	Pb	82	207·22	Uranium ..	U	92	238·14
Lithium ..	Li	3	6·940	Vanadium ..	V	23	50·95
Lutecium ..	Lu	71	175·0	Xenon ..	Xe	54	131·3
Magnesium ..	Mg	12	24·32	Ytterbium ..	Yb	70	173·04
Manganese ..	Mn	25	54·93	Yttrium ..	Y	39	88·92
Mercury ..	Hg	80	200·61	Zinc	Zn	30	65·38
Molybdenum ..	Mo	42	96·0	Zirconium ..	Zr	40	91·22

* Reproduced by permission of the Chemical Society. Copies of this table printed on cards can be obtained on application to the Assistant Secretary, Chemical Society. Price 2d. each. (*cf.* ANALYST, 1934, 414).

Medical Research Council*

THE CHEMISTRY OF FLESH FOODS AND THEIR LOSSES ON COOKING

SINCE practically all the existing analytical figures for flesh foods refer to uncooked foods, the Medical Research Council made grants to the authors of this report for systematic analyses of the cooked foods, and the scope of the work was gradually extended into a thorough investigation of the composition of flesh foods.

Part I (pp. 43) gives full details of the scheme of analysis evolved, including the determination of total nitrogen, non-protein nitrogen, extractive nitrogen, fat, carbohydrate, chlorides, total and inorganic phosphorus, sodium, potassium, calcium, magnesium, and iron. The system is applicable to any *animal* material.

Micro-analytical methods have been used, even when large amounts of material were available and large samples were originally taken, and the technique throughout is described in detail.

Errors of sampling have been shown to be relatively small, and fortuitous analytical errors to be equally small; hence, analytical work has not been duplicated, except for total nitrogen, when small samples of heterogeneous material have had to be taken.

Representative samples of meat were obtained by cutting up the material with a stainless-steel knife and thoroughly mixing the fragments. Fat and lean were analysed separately. Fish flesh was broken up with a fork until sufficiently homogeneous for sampling without mincing or grinding. Separate analyses were made of gravies and drippings.

Tables (pp. 48–65) are given showing the composition of all the fish and meat foods examined; all were in the cooked state, except in the comparatively few instances where the foods are eaten raw. The various cuts are arranged alphabetically under the name of the meat, not of the animal. Eggs are included among animal organs.

Formulae have been worked out for calculating how much of any constituent could be obtained from 100 grms. of the food as served, or from 100 grms. of the raw food. Thus, if a piece of meat weighed R grms. when raw, and C grms. when cooked, E grms. were edible, and the edible portion contained x grms. of some constituent, then the amount of this constituent in 100 grms. of edible portion will be

$$\frac{100x}{E} = A$$

and the amount that can be obtained from 100 grms. of the food as served will be

$$\frac{100x}{C} = B.$$

The multiplying factor (F_1) to convert A (the figure given in the tables) into B, will be

$$\frac{B}{A} = \left(\frac{100x}{C}\right) \div \left(\frac{100x}{E}\right) = \frac{E}{C}$$

i.e. F_1 is the ratio of the weight of the edible portion to the weight of the cooked meat. In the same way it may be shown that F_2 is the ratio of the weight of the edible portion to the raw weight. It is also possible with the aid of the factors F_1 and F_2 to calculate the amount of the various loss which may take place in the cooking and serving of the food.

FACTOR FOR PROTEIN.—It has repeatedly been shown that the factor 6·25 is not the correct one, and it would therefore be better if this factor were given up

* By R. A. McCance and H. L. Shipp. pp. 146. London: H.M. Stationery Office. Special Report Series. No. 187. Price 2/6 net.

and only the figure for protein nitrogen were used. In the present tables, however, both the total and the protein nitrogen have been determined, and the latter has been multiplied by 6·25 for the benefit of those who expect to see a figure for protein.

PURINE NITROGEN.—The present results tend to confirm such previous figures as are available, but these constituents have not previously been determined in many of the materials now examined, and the results are interesting.

	Purine nitrogen per 100 grm. of edible substances Grm.	Purine nitrogen per 100 grms. of total nitrogen Grms.
Beef (general average)	0·081	1·70
Mutton ,, ,, ...	0·063	1·57
Pork ,, ,, ...	0·069	1·58
Birds ,, ,, ...	0·094	1·97
Brains	0·033	1·60
Hearts	0·166	2·80
Livers and kidneys (average) ..	0·140	3·00
Sweetbreads	0·426	11·50
Flat fish (miscellaneous)	0·059	1·85
Gadoids	0·069	2·06
Other demersal fish	0·069	2·00
Whiting	0·092	2·94
Cod's roe (hard)	0·120	3·30
Herring (no roes)	0·150	4·00
Sprats (smoked and fresh, average) ..	0·180	4·60
Sardines	0·234	6·80
Whitebait	0·323	10·30
Herring roe (soft)	0·484	11·90

INORGANIC SALTS.—The percentage of soluble salts in meat and flesh is not greatly changed by cooking; boiling may lower the percentage a little, roasting and frying raise it.

The sodium varied, as a rule, between 50 and 120 mgrms. per 100 grms. of cooked material, and was slightly higher in fish than in meat. There appears to be more sodium present than is required to account for the chlorine as NaCl. This is attributed to the facts that: (i) some of the sodium in serum and intercellular fluids is present as bicarbonate; (ii) muscle fibres adsorb sodium ions loosely on to their external surfaces.

The figures for potassium range, as a rule, between 200 and 400 mgrms. per 100 grms.

LOSSES ON COOKING.—Part II deals with the methods of studying the cooking of meat and its accompanying losses. Many of the results are expressed in graphical form. Experiments described show that there is no evidence that a pellicle forms on the outside of a joint when cooking begins at a high temperature. Unless, therefore, the procedure can be supported on grounds of palatability or digestibility, there seems to be no reason why an oven should be raised to a high temperature before the joint is inserted, or why the water should be boiling before the meat is put in. Both would appear, indeed, to be a waste of heat. When fully cooked, beef has been found to lose the same amount of weight and to show the same loss of water and salts. Egg proteins do not shrink when heated to 100° C. The extent of the shrinkage of beef, fish, kidney and liver proteins is but little affected by raising the temperature from 80° to 100° C., but is slightly increased by raising the temperature from 100° to 120° C. Brain does not shrink below 80° C., but does so at higher temperatures. Fish muscle tends to lose weight if soaked in aqueous solutions below about p_H 5·6. Acids and alkalis inhibit heat

shrinkage of muscle proteins. In fish shrinkage tends to be greatest between p_H 4·0 and 4·5 (where loss of weight on soaking is greatest); in meat maximum shrinkage occurs at p_H 6. It is suggested that the predominating protein in fish has a more acid iso-electric point than that of beef.

Shrinkage on heating muscle proteins is more rapid and extensive 40 hours after death than it is immediately after the animal has been killed. After steam-heating, the percentage loss of water always agrees with that of the soluble salts. Fifty per cent. of the water and salts in beef may thus be lost, and a still higher percentage in kidneys. The percentage loss of water always exceeds the percentage loss of weight, but the relationship between the losses depends upon the loss of the other constituents. On lowering the p_H of the cooking medium, meat and fish tend to lose more of their cations and less of their anions; on raising the p_H the reverse takes place. The expressed juices of fish are more alkaline than those of beef. In roasting, the loss of water is nearly all due to evaporation, the loss of salts (which are left on the surface of the meat) being small. The loss of salts can be increased by reducing evaporation. Frying in deep fat leads to such rapid evaporation of water from the surface that the loss of salts is reduced to a minimum. The loss of protein is more complicated than that of salts, because there are several sources of loss. The loss is slightly greater on boiling than on steaming, and is greater at 120° C. than at 100° C. Kidneys lose a higher percentage of their proteins than other organs, and losses up to 20 per cent. have been observed. The loss of fat on cooking is due to liquefaction by heat; shrinkage of the proteins has little influence on the loss of fat.

The pamphlet concludes with a bibliography occupying 6 pages.

The National Physical Laboratory

METROLOGY DEPARTMENT

TESTS ON VOLUMETRIC GLASSWARE*

No fundamental changes have been made from the preceding edition, but several points have been dealt with in more detail, as, for example, the testing of burette taps and permissible schemes of subdivision of scales on graduated glassware.

The contents of the pamphlet are as follows:—Tests undertaken. Transmission and insurance of apparatus.

Regulations Governing Class A Tests.†—Laboratory marks and certificates—Units of volume—General conditions and methods of test—Flasks—Pipettes—Burettes—Graduated pipettes—Graduated cylinders—Density—Bottles and pyknometers—Vessels for gas analysis—Standard volumetric glassware for testing tar and its products—Other apparatus.

Regulations Governing Class B Tests.‡—Laboratory marks and certificates—Units of volume—General conditions and methods of test—Flasks—Pipettes—Burettes—Graduated pipettes—Graduated cylinders—Vessels for gas analysis—Standard volumetric ware for testing tar and its products—Vessels for milk testing—Other apparatus.

Printed forms for use as advice notes, to be sent in respect of each consignment, may be obtained on application to the Director. The use of these forms instead of a firm's ordinary advice notes is optional, but is recommended.

* New Edition. Pp. 28. April, 1934. Copies may be obtained free of charge on application to The Director, National Physical Laboratory, Teddington, Middlesex.

† The examination of vessels intended to possess the highest accuracy.

‡ The examination of vessels intended to possess only commercial accuracy.

Copies of a separate pamphlet, entitled "Tests on Volumetric Glass Ware used in Dairy Chemistry," may be obtained on application to the Director.

The present pamphlet concludes with a list of verification fees and other charges, and with schemes of sub-division and numbering for intervals of 1 ml., 2 ml. and 5 ml. or decimal multiples or sub-multiples thereof.

Full particulars relating to certificates, reports and statements, insurance and custody of instruments, terms of payment, etc., will be found in the "General Regulations," issued separately and obtainable upon application to the Director.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS

Food and Drugs Analysis

Detection of Gelatin in Cultured Buttermilk and Cottage Cheese. G. A. Richardson and N. P. Tarassuk. (*J. Assoc. Off. Agric. Chem.*, 1934, **17**, 314–319.)—The Stokes test (ANALYST, 1897, **22**, 320), which is the official method of the Association, gives erroneous results with sour milk or cream, cultured buttermilk and cottage cheese, owing presumably to the presence of products resulting from the effect of heat, acidification or proteolysis on the proteins; the various modifications suggested (see Mendelsohn, *id.*, 1930, **55**, 567) are not entirely satisfactory. Addition of picric acid produces a precipitate (which is particularly voluminous with cottage cheese made from rennet) even in the presence of gelatin, and this is attributed to derived proteins which mercuric nitrate does not remove. In applying the test to cultured buttermilk, therefore, the sample is stirred thoroughly at 25° C., 10 ml. are shaken with 10 ml. of mercuric nitrate solution, and the mixture is filtered after it has been allowed to stand for 5 minutes (No. 1 filtrate). The reagent, which must be free from mercurous ions, is prepared by dissolving mercury in twice its weight of nitric acid and diluting the solution to 25 times its volume with water. An aliquot part of filtrate No. 1 is mixed with half its volume of a saturated solution of picric acid, and the type of precipitate, if any, is noted; the remainder of the filtrate is cooled in ice-water and shaken well with half its volume of a 20 per cent. solution of trichloroacetic acid, left for 16 hours at 8° to 10° C., with occasional shaking, and the precipitate is again noted. The mixture is filtered, and the filtrate is warmed at 50° to 55° C. for at least 5 minutes, and is observed at this temperature after addition of half its volume of a warm solution of picric acid saturated at 50° C. With cottage cheese, 5 grms. are extracted with 10 ml. of water at 50° to 60° C., and the extract is treated with 5 ml. of mercuric nitrate reagent as before; the resulting precipitate is then removed after 5 minutes, and the filtrate is treated with 5 c.c. of the mercuric nitrate solution, after which the above procedure (addition of picric acid, etc.) is followed. Tables illustrate the observations and conclusions at each stage, but it is advisable to carry out control experiments simultaneously. It is evident that cultured butter-milk and cottage cheese (especially when made with rennet) contain derived proteins which are not removed completely by mercuric nitrate, but which

with picric acid give a flocculent precipitate with a clean serum, as distinct from the turbidity due to gelatin picrate. Under the conditions described these, but not gelatin, are removed by trichloroacetic acid as a flocculent precipitate with a clear serum, so that subsequent addition of picric acid clearly distinguishes those samples which contain gelatin; 0·1 to 0·25 per cent. produces a cloudy solution, and greater amounts a yellow precipitate. Trichloroacetic acid also causes coagulation of derived proteins, which may then be removed by filtration and the picric acid test applied to the clear serum. The alternative method of Jacobs and Jaffe (*Ind. Eng. Chem., Anal. Ed.*, 1932, **4**, 418) gives excellent results with fresh samples for which rennet has not been used, but otherwise requires modifying by the introduction of precipitation with trichloroacetic acid. J. G.

Detection of Soya Bean Flour in Manufactured Foods. C. H. La Wall and J. W. E. Harrisson. (*J. Assoc. Off. Agric. Chem.*, 1934, **17**, 329–334.)—Soya bean flour (2 to 15 per cent. of the wheat flour used, or 2 to 10 per cent. of the finished food) has been stated to improve the flavour, food value, appearance, yield, lasting value, binding- shortening- and egg-requirements of bakery and sausage products. Tests are as follows:—(i) A strip of red litmus paper is partly immersed in a mixture of 0·5 grm. of sample with 5 ml. of a 2 per cent. solution of urea, contained in a stoppered tube, which is heated at 40° C. for 3 hours. Liberation of ammonia by the urease in soya bean flour produces a blue colour. (ii) Bromphenol blue also gives a blue colour at the end of the heating period. (iii) For quantitative work (*cf. Chem. Weekblad*, 1916, **13**, 254) a mixture of 150 mgrms. of the sample with 10 ml. of a 1 per cent. solution of urea is warmed in a stoppered test-tube for 30 minutes at 25° C., and is then titrated with 0·1 *N* sulphuric acid, with methyl orange as indicator. (iv) For work on the micro-scale (*e.g.* with pea-nut butter), the oil is extracted with ether, and about 2 grms. of the residue are mixed in a small porcelain crucible with a 2 per cent. solution of urea. The crucible is at once covered with a slide, on the underside of which hangs a drop of 10 per cent. hydrochloric acid, and is placed overnight in a warm place; any crystals of ammonium chloride are identified in the usual way. (v) A stiff paste of the flour with 5 per cent. potassium hydroxide solution appears yellow in the presence of soya bean flour. (vi) The supernatant liquid and residue obtained on shaking 1 grm. of the ground sample with 10 ml. of petroleum spirit are coloured yellow if soya flour is present; 5 per cent. gives a yellow spotted residue. (vii) A paste of wheat flour with concentrated hydrochloric acid develops a lavender colour much more rapidly if soya flour is present. (viii) Microscopical features are the characteristic hour-glass shaped cells of the sub-epidermal layer of the bean and the yellow colour of the aleurone-containing cells after treatment with a solution of iodine. Test (i) gave negative results for all of the cereals and legumes tested which did not contain soya flour (10 out of 24), and (ii) confirmed these results except with soup beans, where the reaction was faintly positive. Test (iii) gave titration figures of 0·2 or less, 0·75 or less, 19·20 to 23·00, 3·10 to 10·40, and 33·10 ml. of 0·1 *N* acid for cereals, legumes (excepting soya and jack beans), whole soya beans, soya flours, and jack beans, respectively; 2 and 5 per cent. of soya flour in semolina and in unsmoked sausage, respectively, were

detectable by (i), but unsatisfactory results were obtained with cakes prepared with chemical leavening agents. With Test (iv) a few crystals of ammonium chloride were obtained even in control experiments, and these were attributed to spontaneous decomposition of the urea solution. J. G.

Presence of Methyl Alcohol in Alcohols from Wine, including Marc Wine and Fruit Wine. M. Flanzy. (*Compt. rend.*, 1934, **198**, 2020–2022.)—By the method previously described (ANALYST, 1934, 193), determinations have been made of the proportions of methyl alcohol in a number of potable spirits; the results are expressed as mgrms. of methyl alcohol per litre of total alcohol. For wine spirit and weak wine spirit (trois-six), the values are always less than 1000; here and in other cases also, the trois-six show slightly the higher figures. Marc spirit and weak marc spirit give 1000 to 2000; weak piquette (second cuvé) spirit, usually about 2000, but in one instance almost 4000; cognac, 300 to 400 for Hennessy, Martell and Boulestin brands, 500 to 800 for "self" samples, and 1500 for a piquette spirit; armagnac, 500 to 800; kirsch, mirabelle, and quetsch, 1200 to 1600; calvados, 300 to 1200 for distillery products, and 750 to 1600 for propriétaire products; perry spirit, 660 to 750; cider marc spirit, about 6000. For one and the same kind of fruit, the nature of the soil, the climate and the vintage have no marked influence on the proportion of methyl alcohol found. T. H. P.

New Pentabromoacetone Process for determining Citric Acid in Wine. P. Berg and G. Schulze. (*Z. Unters. Lebensm.*, 1934, **67**, 605–613.)—The oxidation of citric acid to acetonedicarboxylic acid and the determination of this as pentabromoacetone gives highly accurate results with pure aqueous solutions of the acid, provided that certain conditions are observed, but the presence of other organic compounds, as in wine, introduces disturbing factors (*cf.* Kogan, *Z. anal. Chem.*, 1930, **80**, 112; Täufel and Mayr, *ibid.*, 1933, **93**, 1; Bartels, ANALYST, Abstr., 1933, **58**, 164). The authors have modified the process in the following directions: (i) The additions of permanganate, potassium bromide, and sulphuric acid are adjusted to the extract-content of the wine. (ii) The oxidation and bromination are carried out in presence of ammonium sulphate, which assists the formation and separation of the pentabromoacetone, even when the content of citric acid is low. (iii) The optimum temperature of this reaction is established as 15° C. and the optimum time as 1 hour; no attempt must be made to hasten the reaction by mechanical means. (iv) Precautions in the addition of the ferrous ammonium sulphate for removing the manganese oxide precipitate are indicated. (v) Slight modifications are made in the conditions of titration given by Kometiani (ANALYST, 1932, **57**, 192). Detailed instructions are given for the procedure to be followed with wines of different characters.

The permissible additions of citric acid to wine or must in various European countries are stated. No such addition is allowed in Germany—where treatment with potassium ferrocyanide is legal—and the German wines examined contained not more than 0·2 grm. of citric acid per litre. In most other countries from 0·5 to 1 grm. of the acid may be added per litre, although in Greece no limit is fixed. White Bordeaux often contains more than 2 grms. of the acid (in one instance 3·7 grms. were found) per litre. T. H. P.

Simple Method for the Purification of Citrus Pectin. H. R. Nanji and J. J. Chinoy. (*Biochem. J.*, 1934, **28**, 456–462.)—Pectin which has been derived from *Citrus* fruits and contains 90–95 per cent. of pectin, can be further purified by treatment with bromine or iodine solutions at temperatures between 20° and 40° C. To 50 ml. of pectin solution (0·5 per cent.) in water, 3 ml. of $N/10$ iodine or 5 ml. of $N/10$ bromine are added, and the mixture is allowed to stand for about 12 hours at 20° C. or 2 hours at 40° C. The excess of halide is then removed by extraction with ether or by aeration at 40° C., after which the pectin is precipitated with 95 per cent. alcohol, filtered off and dried at 100° C. for 20 hours. Chlorine water, under similar conditions, causes the breakdown of the pectin, and dilute hydrogen peroxide has no action. The halogens have little action on pectic acid or sodium pectate. The yields of furfuraldehyde and carbon dioxide from the purified products agree with the accepted figures for a purified pectin, and the methoxyl-content approaches the theoretical value for a fully methoxylated pectic acid. S. G. S.

Component Fatty Acids and Glycerides of the Milk-Fat of Indian Camels. D. R. Dhingra. (*Biochem. J.*, 1934, **28**, 73–78.)—The component fatty acids and glycerides of the milk-fat of the camel have been studied in detail in order to compare them with those of the milk-fats of the cow, buffalo, sheep and goat. In north-western parts of India milk from camels is utilised as an article of diet by some tribes, but the fat is rarely extracted from the milk and used as a substitute for "ghee." It is shown that the fatty acids of the milk-fat of the camel differ from those of cow, buffalo, goat and sheep milk-fats in a lower content of fatty acids volatile in steam. Camel milk-fat can, to a great extent, be distinguished from the milk-fats of other animals by its lower Kirschner and Reichert–Meissl values. The amount of the lower fatty acids is in decreasing order in the milk-fats of the sheep, goat, cow, buffalo and camel. The content of the fully-saturated glycerides in camel milk-fat is a function of mean unsaturation, like that of the milk-fats of other animals. The component fatty acids of the fully-saturated glycerides of camel milk-fat are in proportions somewhat different from those in the milk-fats hitherto studied. The composition of the fatty acids of the mixed saturated-unsaturated glycerides of camel milk-fat is little different from that of most cow and buffalo butters, but more so from that of goat and sheep milk-fats; the latter show a higher percentage of the butyric-lauric group of acids and a correspondingly lower proportion of unsaturated acids. From the point of view of general properties and of the component glycerides present, there seems no reason why camel milk-fat should not be used as a substitute for cow, buffalo, *etc.*, milk-fats. The general characteristics of the milk-fat were as follows:—Acid value, 0·2; saponification equivalent, 259; iodine value, 40·8; Reichert–Meissl value, 16·4; Polenske value, 1·6; Kirschner value, 14·3; solidification point, 35·3° C.; n_D^{40}, 1·4555. The component acids of the fully-saturated glycerides were: Butyric, 3·4; caproic, 1·0; caprylic, 0·1; capric, 2·1; lauric, 2·2; myristic, 18·4; palmitic, 55·0; and stearic acid, 17·8 per cent. P. H. P.

Influence of Salts used in Curing on the Oxidation and Yellowing of Lard. C. H. Lea. (*J. Soc. Chem. Ind.*, 1934, **53**, 182–184T.)—To investigate the effect of salts used in curing on the rate of oxidation of lard, the freshly-rendered leaf fat was pipetted into flat-bottomed crystallising dishes, and allowed to solidify, and the required salt solution was added so that the depths of the layers were 1·5 and 3·5 mm., respectively. A synthetic bacon pickle was used containing in 100-ml., sodium chloride 26·0 grms., potassium nitrate 3·5 grms., and potassium nitrite 0·03 grm. Seven dishes of one kind were placed in each of a number of glass-stoppered jars (to prevent loss by evaporation) and stored in the dark at 20° C. To examine the fat, the aqueous layer was poured off, the fat was washed with water and dissolved in ether; the solution was washed 3 times with water, and dried over sodium sulphate, and the solvent was removed *in vacuo* at 50° C. Results showed that the salts used in pickles for curing bacon have a very pronounced accelerating effect on the oxidation of pork fat. A rancid flavour usually appears in the fat at peroxide-oxygen values of the order 8 to 10 ml. per grm. It was found that after the induction period the fat under water oxidised more rapidly than that under the salt solution, possibly owing to the reversal of the catalytic effect of the salts, or to the reduced solubility and rate of diffusion of oxygen in the concentrated solution. The nitrite appeared to have little influence when added to the chloride and nitrate in quantity up to 0·15 per cent., but had a slight accelerating effect at 1 per cent. D. G. H.

Fractional Extraction of Soya-Bean Oil and the Drying Properties of the Fractions. T. Yamada. (*J. Soc. Chem. Ind. Japan, English Abstr.*, 1934, **37**, 190–192B.)—Soya-bean oil has been fractionated as follows:—100 grms. of the oil were dissolved in an equal quantity of acetone, and the solution was chilled at −20° C. On standing for a short time at this temperature a butter-like mass separated out. The clear liquid was decanted. The residual oil was dissolved in 100 ml. of acetone, and the chilling and decanting processes were repeated. Ten fractions were thus obtained with iodine values (Wijs) ranging from 152 to 100, the original oil having an iodine value of 137. The drying powers of the fractions, tested by measuring the gain in weight of the oils spread on glass plates and exposed to the air over a period of about 50 days, were in proportion to the degree of unsaturation, a maximum in the weight increment-time curve for oil of iodine value 145 and for the original oil occurring at about 27 days and 42 days, respectively. S. G. C.

Adulteration of Aloes. E. Léger. (*J. Pharm. Chim.*, 1934, **126**, 533–535.)—A well-prepared sample of powdered aloes is entirely soluble in officinal (French Codex) ammonia diluted with nine times its volume of water, and a use of this fact will disclose adulteration. The tests for aloes given in the French Codex are sensitive with samples adulterated to the extent of 50 per cent.; Klunge's reaction, however (red colour given by iso-barbaloin with copper sulphate in presence of sodium chloride and alcohol), is positive in the sample containing 10 per cent. of Barbadoes or *Curaçao* aloes. Treatment with ammonia should be followed by treatment with benzene, which dissolves rosin but scarcely any aloes. Klunge's reaction will distinguish the Cape aloes type from aloes from Antilles,

Curaçao, Barbadoes, etc. If the quality and purity of the sample are to be determined, no test will take the place of the determination of the chlorinated aloines. This determination will detect adulteration of the more expensive Curaçao type (containing 31 to 34 per cent. of the aloines) with the cheaper Cape type containing 17 to 20 per cent. D. G. H.

Colour Reaction of Mono-carboxylic Chrysanthemic Acid (from the Hydrolysis of Pyrethrin I). M. Audiffren. (*J. Pharm. Chim.*, 1934, **126**, 535–536.)—Monocarboxylic chrysanthemic acid in aqueous concentration of about 1 in 1000 to 1 in 10,000 gives no reaction with Denigès' reagent, but in a sulphuric acid medium, a reaction occurs, sensitive to 1 part in 100,000 of the acid. One ml. of the Denigès' reagent is added to 1 ml. of the dilute chrysanthemic acid solution, followed by 0·4 to 0·5 ml. of concentrated sulphuric acid, poured in rapidly so that the acid sinks to the bottom of the tube. After a few seconds the liquid is mixed, and, after a momentary contact, a pink or red colour develops, changing to violet and then green. After 24 hours a yellow precipitate is formed. If the sulphuric acid is added, drop by drop, the reaction is less sensitive. A positive reaction was obtained with the first fractions of the distillate from the urines of patients treated by pyrethrin employed as an anthelmintic, and it appears that pyrethrin I is rapidly eliminated in the urine, giving, among other products of decomposition, monocarboxylic chrysanthemic acid. D. G. H.

Detection of Very Small Quantities of Nicotine. A. Wenusch. (*Z. Unters. Lebensm.*, 1934, **67**, 601–605.)—Various reactions which have been suggested for the detection of nicotine are unsuitable for this purpose, as they are reactions of impurities or decomposition products of the nicotine. Several new reactions now described admit of the identification of nicotine in very small quantities. A liquid material is acidified slightly with dilute sulphuric acid and concentrated on a water-bath. The residue, or a solid product to be tested, is made strongly alkaline with cold concentrated sodium hydroxide solution and either repeatedly and vigorously shaken with petroleum spirit or extracted in a Soxhlet apparatus. The petroleum spirit extract is shaken with about 5 ml. of dilute sulphuric acid (1:20), which takes up any nicotine as sulphate. If a drop of the acid solution gives no precipitate or opalescence with silicotungstic acid solution, nicotine is absent. In the opposite case, the 5 ml. of acid are treated with 5 ml. of concentrated potassium hydroxide solution and distilled from a ceresine bath (up to 170° C.). The distillate is tested as follows to identify the nicotine.

(1) Two ml. are boiled for some minutes with a few crystals of 2 : 4-dinitrochlorobenzene, cooled and treated with alcoholic 1 per cent. potassium hydroxide in excess; a violet colour indicates the presence of a pyridine nucleus. (2) This is confirmed by treating a few drops of the distillate on a porcelain plate with Selmi's reagent (potassium platinum iodide), which gives a black precipitate (in presence of 1:10000 of nicotine, after long standing). The remainder of the distillate is made strongly alkaline with concentrated sodium (not potassium) hydroxide and thoroughly shaken with several portions of ether, and the ethereal extract is treated with an ethereal picric acid solution until precipitation is complete (absence of precipitate shows absence of nicotine). The precipitated dipicrate is

collected on a small weighed filter, washed with ether, dried and weighed, and then dissolved in hot water (3 ml. per 0·01 grm. of picrate); the solution, filtered while hot, is left to crystallise and the melting-point of the crystals (about 218° C.) is determined. The nicotine may be further characterised by treating a little of the dipicrate with sodium hydroxide and silver acetate and testing the resulting nicotyrin (i) with a solution of *p*-dimethylaminobenzaldehyde in concentrated phosphoric acid (violet colour), (ii) with vanillin and hydrochloric acid (red colour), and (iii) with quinone in phosphoric acid solution (violet colour). T. H. P.

Biochemical

Biological Value of Proteins. V. The Comparative Biological Value of the Proteins of Whole Wheat, Whole Maize and Maize Gluten, measured by the Growth of Young Rats. M. A. Boas-Fixsen, J. C. D. Hutchinson, and H. M. Jackson. (*Biochem. J.*, 1934, **28**, 592–601.)—The biological value of a protein is measured by the gain in weight per grm. of protein ingested. On this basis the proteins of whole wheat and whole maize in diets containing 9 to 10 per cent. of protein have values of the same order (1·30 to 1·36). These may increase to 1·4 to 1·5 after the food has been cooked. Maize gluten is inferior to the proteins from the whole grain, probably owing to a lower proportion of zein. Results indicate that if the value is

$$\frac{\text{Weight increase in grms.}}{\text{Protein ingested in grms.} - 10}$$

the ratios are constant and independent of the food. This gives 1·85 for wheat and 1·73 for maize at the 9 to 10 per cent. level, with no difference between raw and cooked food. The 10-grm. quantity is regarded as the "maintenance requirement," as distinct from growth needs. S. G. S.

Fat Metabolism in Fishes. III. Selective Formation of Fat-Deposits. J. A. Lovern. (*Biochem. J.*, 1934, **28**, 394–402.)—The examination of fats from various parts of the body of certain fishes reveals the fact that for each one examined, there is a considerable variation in the composition of fatty acids, indicating a highly selective formation of fat-deposits. This paper deals with the liver and peritoneal fats of the conger eel and the fats from depots and organs of the dolphin and porpoise. Fats from the porpoise and dolphin are of a type similar to those from the sperm whale, which is a zoological related species. For these animals the organ-fats differ from the depot-fats, which contain large quantities of iso-valeric acid. The blubber of a porpoise foetus also contains iso-valeric acid, together with a large amount of highly unsaturated acids. The heart-fat of a porpoise contains acid more unsaturated than those from the liver, which is contrary to the theory that the latter organ acts as a desaturating agent. S. G. S.

Isolation of Crystalline Stercobilin. C. J. Watson. (*J. Biol. Chem.*, 1934, **105**, 469–472.)—A simpler and more efficient method than that previously described (*Z. physiol. Chem.*, 1932, **204**, 57, **208**, 101; 1933, **221**, 145; and *Proc. Soc.*

Exp. Biol. and Med., 1933, **30**, 1207) has been worked out for the isolation of stercobilin from human faeces. The faeces are ground, mixed with glacial acetic acid to a semi-liquid consistence, and ether is added in sufficient quantity to give a clear supernatant fluid after mixing. The ether is decanted, and the extraction is repeated 15 to 20 times until the extract is of a relatively pale colour, glacial acetic acid being added after every second or third extraction. The greater part of the ether is removed by vacuum distillation, and the acetic acid solution is poured into 6 to 8 volumes of 1 per cent. hydrochloric acid, and left to stand overnight to effect more complete precipitation. The filtrate is covered with a considerable amount of ether, and sufficient sodium acetate is added to make the solution negative to Congo red. After shaking, the extraction is repeated 5 times with smaller amounts of ether, which removes the major portion of the copromesobiliviolin and any porphyrins. The residual aqueous solution is repeatedly extracted with small amounts of chloroform, and this is next extracted many times with water. The filtered aqueous extract is rather strongly acidified with 25 per cent. hydrochloric acid, and the stercobilin may then be obtained as hydrochloride by extraction with chloroform. This solution is superficially dried over anhydrous sodium sulphate, filtered, concentrated, and poured into 15 to 20 volumes of dry petroleum spirit, when there is an almost quantitative precipitation of the hydrochloride, which is re-dissolved in chloroform and re-precipitated. The free substance, possessed of both basic and acidic properties, may be obtained by dissolving the hydrochloride in a small amount of 0·1 *N* sodium hydroxide solution, and this solution is mixed with an equal amount of chloroform, acidified in a separating funnel with a few drops of acetic acid, and shaken. The free stercobilin passes slowly into the chloroform, and the solution, which is dried by filtration through chloroform-moistened paper, is concentrated so that crystallisation takes place. The free substance is much less soluble in hot chloroform than the hydrochloride, and the crystals are of more reddish appearance. The stercobilin thus isolated appears to be identical in every way with that obtained by the original method. From the total amount of faeces for an 8-day period (920 grms.) from a typical case of congenital haemolytic jaundice, 2·2 grms. of crystalline stercobilin hydrochloride were obtained. It seems probable that the hydrochloride crystallises with chloroform of crystallisation, which is lost partly on standing, and more completely on heating; this would account for the colour-changes from the light yellow of the fresh crystals to the yellow-orange of crystals exposed for a short time to the air. D. G. H.

Methyl Alcohol in Foliage Leaves. Relation between the Alcohol and Chlorophyll. M. Flanzy. (*Compt. rend.*, 1934, **198**, 2118–2120.)—The proportion of methyl alcohol in samples of green, and partly and entirely etiolated leaves was determined in the distillate obtained after boiling 1 kilo. of material in water, cooling and distilling. The distillate was suitably concentrated and purified, and the alcohol was determined by the micro method previously described (ANALYST, 1934, 193). Methyl alcohol was found in all the samples examined, and a larger proportion was present in the green leaves than in etiolated leaves of a given plant, whilst in the same species the greater the proportion of

chlorophyll the larger was the amount of the alcohol found. On 1 kilo. of the dry material the quantity varied from 895 mgrms. of alcohol for etiolated cabbage leaves to 3426 for green chicory leaves, 7174 for dark green nettle leaves, and 4718 for light green nettle leaves. There appears to be a definite methyl alcohol figure for each species, and it remains to be determined whether the figure is a simple function of the chlorophyll activity or of the quantity of chlorophyll present as well. Since there is a relation between the proportion of chlorophyll and the amounts of certain metals present, it may follow that there is a connection between the methyl alcohol-content and the presence of these metals. D. G. H.

Separation of Cysteine from Ascorbic Acid by means of Mercuric Acetate. A. Emmerie. (*Biochem. J.*, 1934, **28**, 268–269.)—A method is described by which cysteine, a source of error in the determination of ascorbic acid by means of 2.6-dichlorophenolindophenol solution, is removed quantitatively with mercuric acetate solution. Van Eekelen, Emmerie, Josephy and Wolff (*Nature*, 1933, **132**, 315) stated that cysteine is precipitated with mercuric acetate, whereas ascorbic acid is reversibly oxidised and can be quantitatively regenerated by means of hydrogen sulphide (see Tillmans, Hirsch and Dick, *Z. Unters. Lebensm.*, 1932, **63**, 267; ANALYST, 1932, **57**, 397). The ascorbic acid content of solutions of pure ascorbic acid, lemon juice and orange juice has been quantitatively recovered by the mercuric acetate precipitation. The reduction with hydrogen sulphide must be effected in a slightly acid medium, because otherwise, especially in alkaline solution, reducing sulphur compounds may be formed. It is important to mention that mercuric acetate solution removes many impurities; it also removes ergothioneine in acid medium. For the precipitation, a 20 per cent. solution of mercuric acetate in water is prepared and filtered after one day, as some hydrolysis takes place. To the solution to be tested (which must be slightly acid, excess of acid being removed by calcium carbonate and filtration) the mercuric acetate solution is added, drop by drop, in a graduated centrifuge-tube until the precipitation is finished. Care is taken to avoid a large excess of mercuric acetate. After centrifuging, the solution is treated with hydrogen sulphide, filtered, and left standing overnight, when the hydrogen sulphide is removed with nitrogen (controlled by lead acetate paper), and the titration is then carried out. *Example.*—To 20 ml. of a dilute orange juice solution (titration value 0·5 ml. indicator per ml. solution) 80 mgrms. of crystalline cysteine hydrochloride were added (4 mgrms. per ml.). This solution of ascorbic acid and cysteine decolorised, after 1 minute, about 0·9 ml. of indicator per ml. solution (the indicator being added, drop by drop, to the solution). From this solution 18 ml. (after calcium carbonate treatment) required 3 ml. of mercuric acetate solution, and the procedure given above was followed. Then 1 ml. solution required 0·43 ml. indicator, calculated $\frac{18}{21} \times 0{\cdot}5 = 0{\cdot}43$ ml. The sodium nitroprusside test was completely negative.

P. H. P.

Separation of Carotenes by Adsorption. H. H. Strain. (*J. Biol. Chem.*, 1934, **105**, 523–535.)—Separation of carotenes was found to be most successful by

passing a solution of the carotene mixture over columns composed of a finely-divided adsorbent (Tswett columns). Magnesium oxide, prepared from the hydroxide and sold as Micron Brand magnesium oxide No. 2641 was found to be the most satisfactory adsorbent tried. This oxide exhibits a very high resolving power for different carotenes, so that each separated as a single and distinct zone on the column, and the carotenes were easily removable by elution with petroleum spirit containing ethanol; the magnesia may be used again after drying at 110–150° C. α- and β-carotenes, separated by this method, were identical with similar components separated from carrot-root and palm-oil by other methods, and with untreated carrot-root carotene of similar rotation. A small quantity of γ-carotene was also always isolated by adsorption on magnesium oxide, and smaller quantities of other coloured substances were found associated with it, but in no case was there any indication of a component of carrot-root carotene less readily adsorbed than α-carotene, by any of the adsorbents tried. Palm-oil, however, contained a small amount of a yellow carotenoid which was only slightly adsorbed on magnesium oxide. The preparation of the Tswett columns and the procedure with magnesium oxide as adsorbent are described in detail. Lead oxide, charcoal and siliceous earth with dichloromethane, and with petroleum spirit and dichloromethane, fibrous alumina, barium oxide, calcium oxide, calcium hydroxide, charcoal (norit), fullers' earth, lead hydroxide, basic lead carbonate, lead sulphide, mercuric chloride, and mercurous chloride, were all tried but were less successful than magnesium oxide as adsorbents. Magnesium oxide may also be used for separating carotenes from other pigments, such as lycopene and the chlorophylls and xanthophylls, and for the isolation of carotenes from natural products without any previous crystallisation. "Hydralo" was found excellent as an adsorbent for separating carotenes from lycopene, and from many naturally occurring waxes. D. G. H.

Stability of Carotene in Olive-Oil. R. G. Turner. (*J. Biol. Chem.*, 1934, **105**, 443–454.)—A 0·2 per cent. solution of carotene in olive-oil containing hydroquinone (0·1 per cent.) as stabiliser loses approximately one-half of its vitamin-*A* activity in a year. The action of light or of temperature up to 37·5° C. has little influence on this loss, but at 100° C. the destruction is hastened. Low temperatures retard the conversion of carotene to the achroo-form, and at 10° C. the loss of colour is no more rapid in absence than in presence of stabilisers. Quinhydrone is as effective as hydroquinone in preventing loss of colour. Without stabiliser, an olive-oil solution of carotene loses its colour completely in 6 to 9 months, and an ethyl-laurate solution in 3 to 4 months, at room temperature. When a stabiliser is present, both solutions tend to retain their red pigmentation at 6 to 7 red Lovibond units for 12 to 17 months, the vitamin activity having approximately one-half of its original value at this time. Apparently the loss of potency of carotene in highly concentrated solutions is not due solely to conversion of the active to the inactive form, slow precipitation until a saturation point is reached, being probably a contributory cause. T. H. P.

Determination of Carotene in Butter-Fat. H. M. Barnett. (*J. Biol. Chem.*, 1934, **105**, 259–267.)—The colours of solutions of carotene in petroleum spirit bear no constant relation to those of solutions in various edible oils. Butter-fat decolorised with activated carbon is useless as a solvent for carotene, which undergoes rapid decomposition in the fat. Carotene may, however, be determined spectro-photometrically if refined coconut-oil is used as a solvent and diluent. For the test experiments use was made of carotene purified by repeated crystallisation from hot petroleum spirit, 25 mgrms. of the product being dissolved in 50 grms. of the coconut-oil to make a stock solution. Curves are given showing the percentage transmission at wave-lengths varying from 455 to 500 $m\mu$ for concentrations of 0·5, 1·5 and 2·5 mgrms. of carotene per kgrm. of butter-fat in a 3-cm. cell. Application of these curves to determine the carotene in the fats of five butters shows good agreement between the concentrations indicated at ten different wave-lengths within the above region. Moreover, the method seems applicable also to dilute solutions of carotene in oils such as Wesson oil or oleomargarine, provided, of course, that carotene is the only pigment of consequence present.

In determining the carotene-content of butter-fat, it is suggested that the sample be diluted with refined coconut-oil so as to bring the carotene-content between 0·75 and 2 mgrms. per kilo., and that determinations be made at the various wave-lengths indicated, coconut-oil being used as a blank. An average of the results thus obtained should be more accurate than readings taken at only one or two wave-lengths. Moreover, by the use of a wider spectral region, any addition of foreign colouring matters may become apparent. The colorimetric method of determining carotene described by Palmer (*Carotinoids and Related Pigments*, Amer. Chem. Soc. Monograph Series, New York, 1932) gives inaccurate results, since the colour-intensity of carotene in oils is several times that in petroleum spirit. The values obtained by this method, when multiplied by 0·28, give results in good agreement with those of the above spectro-photometric method. The colorimetric method is hence useful if no spectro-photometer is available.

T. H. P.

Modified Spectrophotometric Method for the Assay of Carotene and Vitamin *A* in Butter. A. E. Gillam. (*Biochem. J.*, 1934, **28**, 79–83.)—Values for the carotene, xanthophyll and vitamin *A* contents of 70 English butters have been obtained spectrophotometrically. Mean values are recorded. It is shown that, on the average, the ratio of carotene to xanthophyll in butter is 14 : 1 by weight. It is further pointed out that if 94 per cent. of the light-absorption of butter-unsaponifiable matter at 455–460$m\mu$ is taken as being due to carotene, the results on individual samples are sufficiently close to those actually determined after removal of the xanthophyll to justify the approximation by the economy effected in time and material. The spectrophotometric method previously developed by Gillam, Heilbron, Morton, Bishop and Drummond (*Biochem. J.*, 1933, **27**, 878; ANALYST, 1933, **58**, 630) for the determination of carotene and vitamin *A* in butter has been improved for routine purposes. Full details of the modified method are given. To calculate the percentage of carotene in the butter, use in made of the fact that $E^{1\%}_{1\,cm.}$ 463$m\mu$ (in chloroform) = 1900 for pure carotene.

P. H. P.

New Colour Test for the Determination of Vitamin *A*. E. Rosenthal and J. Erdélyi. (*Biochem. J.*, 1934, **28**, 41–44.)—By a simple modification of the antimony trichloride reaction described by Carr and Price (*Biochem. J.*, 1926, **20**, 297) the authors have devised a reaction which is characteristic of vitamin *A*, and which allows its exact determination in cod-liver oil. When the oil containing vitamin *A* is heated together with catechol and antimony trichloride, both dissolved in chloroform, the blue colour first produced soon turns into a violet-red colour. The following reagents are necessary:—(i) Alcohol-free, absolute chloroform. (ii) A freshly-prepared 0·5 per cent. solution of catechol in chloroform. (iii) A cold saturated solution of antimony trichloride in chloroform. The specimen of oil to be tested is first diluted with chloroform. To 1 ml. of this solution 1 ml. of the catechol solution and 3 ml. of the antimony trichloride solution are added. The test-tube containing the mixture is immediately transferred to a water-bath at 60° C. for 1 to 2 minutes. The blue colour first produced changes during this heating into an intense violet-red hue. In the cold this colour is not produced, as the change then takes the same course as without catechol. Subsequent heating of the solution which has undergone the change in the cold does not lead to the production of the violet-red colour; therefore, heating must take place immediately after the mixing of the reagents. For quantitative determinations, the solution, immediately after it has turned violet-red, is compared in a colorimeter with a 0·01 per cent. solution of potassium permanganate. From 0·1 to 0·5 ml. of cod-liver oil should be used, diluted with chloroform to 1 ml. Spectroscopic examination of the red fluid shows four characteristic absorption bands: (i) A broad band at the boundary of green and yellow, having indistinct limits; peak intensity at 552$m\mu$; (ii) a narrower distinct band between green and blue; peak intensity at 476$m\mu$; (iii) a dim band in the blue; and (iv) a dim band in the violet. Vitamin *A* solutions inactivated by irradiation do not give the reaction. The following carotenoids which were examined do not give the reaction: Carotene, lycopin, zeaxanthin, capsanthin and physalin. The same reaction carried out with ergosterol shows the colour change in an inverse succession, the solution first becoming pink and changing slowly to blue. The reaction with catechol, as described, is an advance in two respects: (i) it makes possible differentiation of vitamin *A* from carotenoid substances; (ii) the violet-red colour, similar to that of a dilute solution of potassium permanganate, is more stable than the blue colour obtained according to the Carr and Price method. P. H. P.

Some Factors which affect the Assay of Vitamin *A* by the Spectrographic Method. R. J. MacWalter. (*Biochem. J.*, 1934, **28**, 472–475.)—Measurements of vitamin *A*-content, on the basis of absorption at 328$m\mu$, involve the assumption that irrelevant absorption at this wave-length bears a constant relation to the relevant absorption for all oils, or is negligible in comparison with it. Aeration experiments show that this is not the case, and suggest that the ageing of an oil by oxidation may increase the irrelevant and decrease the relevant absorption. The measurements should be made on the unsaponifiable fraction in order to avoid errors arising from previous oxidation. The vitamin-content, as measured by the absorption at 328$m\mu$, is regarded of value only with oils having a potency

exceeding 10 to 20 blue units (C.P.). For less potent oils, the "non-vitamin *A*" absorption is a considerable fraction of the total absorption. S. G. S.

The Formaldehyde-Azo-Test for Vitamin B_1. H. W. Kennersley and R. A. Peters. (*Biochem. J.*, 1934, **28**, 667–670.)—The test for vitamin B_1, by means of diazotised sulphanilic acid (the Pauly reaction), has been found to require a definite p_H value, and also to be more stable in the presence of formaldehyde. The reagent is prepared by dissolving 5·76 grms. of sodium bicarbonate in 100 ml. of water and adding 100 ml. of *N* sodium hydroxide solution. For the test, 0·5 ml. of diazotised sulphanilic acid [To 1·5 ml. of sulphanilic acid solution (4·5 grms. + 45 ml. of 37 per cent. hydrochloric acid (sp.gr. = 1·19) dissolved in 500 ml. of water), 1·5 ml. of sodium nitrite solution (25 grms. of 90 per cent. sodium nitrite in 500 ml. of water) is added and the mixture left in ice for 5 minutes. Six ml. of sodium nitrite solution are then added, and the mixture is again placed in ice for 5 minutes, after which it is diluted to 50 ml. and kept in ice 15 minutes before use. (See also Koessler and Hanke, *J. Biol. Chem.*, 1919, **39**, 505.)] is added to 1·25 ml. of the reagent in a small test-tube. After one minute, 1 drop (0·3 ml.) of 40 per cent. formaldehyde is added, and then the vitamin B_1 in 0·1–0·3 ml. of solution of acidity greater than p_H 4·0. A pink colour develops slowly, and increases in intensity for 30 to 60 minutes. After this time it is almost constant, although the pink becomes more pronounced up to 24 hours, owing to the fading of the yellow. Standards may be made with different concentrations of the vitamin, from 2 to 7 pigeon-doses, and kept for 14 days. The limit of sensitivity is 1 vitamin B_1 unit (1 pigeon-dose or 1·5–2·0γ of crystalline vitamin). With 1 unit, the colour is ill-defined. Even in the purest specimen yet obtained the colour is tinged with yellow. The colour from 10γ approximates to that from a mixture of Sørensen's acid and alkaline phosphates (2 + 8) of p_H 5·0 at 15° C., containing in 103 ml., 100 ml. of phosphate mixture and 3 ml. of 0·02 per cent. phenol red. A simple comparator is preferred to a colorimeter, but the use of the tintometer has not been properly explored. The test can be used as a preliminary one for extracts of seeds, roots, etc., and also for fractionation of the vitamin, as it agrees closely with the bird-test. S. G. S.

Bacteriological

Individual Resistance of Micro-organisms, especially Yeasts, to Ultra-violet Rays. J. Beauverie. (*Compt. rend.*, 1934, **198**, 2017–2019.)—The value of cellophane in experiments on the effect of ultra-violet rays on micro-organisms is emphasised. This material is traversed by rays of wave-length down to 250$m\mu$, and thus transmits abiotic rays. Moreover, preparations made on it may be observed directly under the microscope, even with an immersion lens. In the experiments described, yeast from a young culture on wort-gelatin was mixed with water to a thin emulsion, portions of which were spread on sheets of cellophane. These were examined under the microscope to ensure that only single layers of cells were present. One preparation was kept in a moist atmosphere as a control, and the others were exposed for 30 seconds, 3 minutes or 5 minutes,

at a distance of 30 cm., to the radiation of a 400-watt mercury vapour lamp (15,000 volts). The first effect of the exposure is to inhibit the multiplying power of the cells. This is followed by alteration in their structure, which shows itself in more or less deformed vacuoles and increasingly granulated cytoplasm, and is, to some extent, reversible, although it may continue until the cell is killed.

T. H. P.

***Byssochlamys Fulva* and its Effect on the Tissues of Processed Fruit. M. Olliver and T. Rendle.** (*J. Soc. Chem. Ind.*, 1934, **53**, 166–172T.)—An unusual feature observed during an investigation on the softening of sound fruit, which had been processed under normal conditions and stored for several weeks, was disintegration without obvious gas-production, abnormal odour or flavour, or colour-change, the general effect being merely that of over-processing; this was found to be due to a new species of fungus, *Byssochlamys fulva* (*cf.* Olliver and Smith, *J. Botany*, 1933, **72**, 196). The most striking characteristics of young cultures of this fungus, which resists normal processing conditions and grows well in a reduced supply of oxygen, are very long tangled chains of ovate to elongate conidia which arise from sterigmata with swollen bases. Ten-day cultures produce masses of asci, which appear as spheres in the mycelial felt and develop eight ascospores each, and these are very resistant to physical and chemical changes; there is no peridium or hyphal web. *B. fulva* is cultivated best on a medium prepared by steaming 100 grms. of potato mash with 800 ml. of tap water for 30 minutes; the filtrate from this mixture is diluted with an equal volume of water, and to this are added, agar, 1·5 per cent.; sucrose, 10·0; magnesium sulphate, 0·1; dipotassium hydrogen phosphate, 0·1; sodium chloride, 0·1; ammonium sulphate, 0·2; and calcium carbonate, 0·2 per cent. The medium is autoclaved for 30 minutes at 15 lbs., and is then filtered and sterilised. The optimum growth-temperatures are between 30 and 37° C., and the mycelium may be floccose, filmy, leathery or waxy according to the conditions, and is usually filmy or waxy on fruit, although it bears no relationship in quantity to the degree of disintegration produced. *B. fulva* also grows on Czapek-Dox agar, on Henrici's peptone-sucrose, and (without producing liquefaction) on gelatin; it inverts and degrades sucrose, the maximum concentration tolerated being 60 to 65 per cent. Experiments indicate that the softening action on fruit is due to destruction of the cementing pectinous substances rather than of the cellulose, and this is accelerated by addition of inorganic salts and, particularly, of 1 per cent. of peptone; this may explain the wide variations in effect observed in practice and with different fruits (*cf.* Raistrick and Smith, *Biochem. J.*, 1933, **27**, 1814). Results are erratic with fruit juices, probably on account of submergence of the spores, and, although *B. fulva* is not parasitic, so far as fruit on the plant is concerned, it is capable of rapid growth on damaged surfaces. The source of *B. fulva* has now been traced to the fields and orchards, where strawberries, damsons and plums seem most liable to attack (37, 50 and 71 per cent., respectively, of those examined were infected). It is suggested that *B. fulva* is present on fruit only by reason of atmospheric distribution of the conidia or asci, or by direct contamination from the soil, and that processed fruit provides the ideal medium for its growth; it has been found in 16 different districts

in England, but is apparently unknown abroad. Cold storage at 10° to 20° F. for several months, and treatment with ammonia (2·5 ml. per litre) and with acetaldehyde (1 part in 200) inhibits growth without killing; mature ascospores in certain fruit syrups survived temperatures of 86° to 88° C. for 30 minutes, and exposure to live steam for 3·5 minutes was required before the fungus was killed. The fungus tolerates concentrations of sulphur dioxide (in the presence of sucrose and peptone) up to 450 parts per million, and is comparatively little affected by changes in p_H value of 2·0 to 7·0 (optimum growth at 3·0) and, in the case of old cultures, by immersion in 100 per cent. alcohol. J. G.

Toxicological and Forensic

Sericite in Foundry Dust. C. S. Hurlbut and D. S. Beyer. (*J. Ind. Hyg.*, 1934, 169–176.)—A study of the dusts of two foundries carrying out similar operations showed that the dust in one was extremely high in sericite, whilst the other had only a moderate amount. This difference was traced to the moulding sands used. A high incidence of silicosis claims occurred in the foundry in which the dust was high in sericite, in spite of lower dust-counts and more favourable employment conditions in that foundry. No silicosis claims were made in the foundry having little sericite. The findings in these respects were in harmony with those of Jones. A sericite-rich sand may contribute a large percentage of sericite particles to the respirable dust in a foundry, even though this sand is only a small portion of the total. Sericite particles, because of their shape, will remain suspended in the air longer than quartz particles of the same size. Further studies should be made to determine the sericite-content of foundry sands, particularly those used where silicosis claims have arisen. Meanwhile, preference may well be given to the use of foundry sands having little or no sericite. An experiment confirmed the previous deduction, and showed that the original high percentage of sericite, relative to quartz and other constituents, increased with time. After 75 minutes of settling in still air, nearly 100 per cent. of the material remaining in suspension was sericite.

Agricultural

Determination of Aluminium in Pasture-Grasses, etc. F. B. Shorland. (*Trans. Roy. Soc. New Zealand*, 1934, **64**, 35–49.)—The method employed was essentially that used by Lampitt and Sylvester for the determination of aluminium in foodstuffs (ANALYST, 1932, **57**, 418), involving colorimetric determination of the red aluminium lake of aurintricarboxylic acid with the aid of the Lovibond tintometer; it was found to be accurate to within about 5 per cent. Careful cleaning of the sample grasses prior to analysis was found to be necessary in order to remove dust and soil particles, which frequently contain appreciable amounts of aluminium, and lack of attention to this detail is considered to have resulted in previous investigators over-estimating the aluminium content of these materials. The method of cleaning adopted was thoroughly to brush the selected material to remove loosely adhering matter; a weighed sample was then washed in several successive

changes of distilled water until the washings showed no appreciable amount of suspended matter. The destruction of the organic matter, either by ashing or by wet oxidation with nitric and sulphuric acids, is stated to leave from 35 to 65 per cent. of the total alumina in the insoluble residue; no details for the recovery of this part of the alumina are given. The distribution of alumina in bamboo, pampas-grass, toetoe (*Arundo conspicua*), rye grass, red and white clovers was studied, the amounts of alumina found varying from 0·0025 per cent. in bamboo stems to 0·114 per cent. in rye-grass roots. The alumina was shown to be concentrated chiefly in the roots, but the leaves contained more than the stems. The figures obtained on carefully cleaned pastures show that, in general, the alumina-content is below 0·025 per cent., and values quoted in excess of this are considered to indicate contamination of the sample by dust, etc. S. G. C.

Determination of Naphthalene in Insecticides. W. L. Miller. (*J. Assoc. Off. Agric. Chem.*, 1934, **17**, 308–313.)—The following methods have a higher degree of accuracy than existing procedures and, with the exception of that for louse powder (which is recommended as a check or for use in the presence of volatile solvents), are considerably shorter:—*In Mineral Oils.*—Cresols, phenols or alkaloidal bases are removed by extraction in succession with 10 per cent. sodium hydroxide solution, dilute acid and water, and 10 ml. of the residual sample are extracted twice with 20 ml. of a mixture of 90 ml. of acetone and 10 ml. of water. The extracts are transferred to the heavier of two Babcock cream bottles which balance to within 3 grms., 10 ml. of water are added, the mixture is shaken, and 3 minutes later the oil which rises is returned to the separating funnel; the extraction process is then repeated, this extract being transferred to the other bottle. Both bottles are then centrifuged at 1200 r.p.m. for 10 minutes, the top layers are removed, and the combined residues are diluted to 100 ml. with the acetone solution. Of this, 50 ml. are pipetted, with stirring, into 250 ml. of a saturated solution of picric acid, and after 5 minutes the precipitate is collected on a Gooch crucible containing filter paper and a thick layer of asbestos, and washed with a 0·2 per cent. solution of picric acid. The contents of the crucible are then boiled for 5 minutes with 200 ml. of water and an excess of 0·1 *N* sodium hydroxide solution, the mixture being then cooled and titrated with 0·1 *N* hydrochloric acid to phenolphthalein (x ml.). If 0·05 ml. is deducted for the picric acid retained on the filter and a further 0·05 ml. for each 20 ml. of alkali required, then $0{\cdot}012806x$ gives the naphthalene-content.

In Low-boiling Solvents.—A quantity of sample containing not more than 5 grms. of naphthalene and 7 grms. of *p*-dichlorobenzene is diluted to 100 ml. with chloroform, and 10 ml. of the mixture are stirred with 1 grm. of picric acid and 10 ml. of chloroform. The solvent is removed on the water-bath below its b.pt., and the residue is dissolved in 15 ml. of warm alcohol to which 100 ml. of water are added. The mixture is cooled and filtered, and the residue is titrated as described above.

In Wood Chips and Essential Oils.—A sample containing not more than 1 grm. of naphthalene is extracted for 10 minutes with 40 and 20 ml. of hot alcohol in succession, and half of the combined and diluted extracts (50 ml.) is mixed with

200 ml. of a cold saturated solution of picric acid, the mixture being then filtered and titrated as described above.

In the Presence of p-*Dichlorobenzene, Camphor and Inorganic Fillers.*—An aliquot portion of a solution of the sample in acetic acid (equivalent to 0·10 to 0·15 grm. of naphthalene) is added to a mixture of 80 ml. of water, 40 ml. of concentrated sulphuric acid and sufficient acetic acid to provide a total of 70 ml. of this substance. The mixture is warmed to 70° C., and 0·5 *N* potassium permanganate solution is added until the rose colour no longer rapidly fades to orange. The temperature is then raised to 90° to 91° C., and the addition is continued at such a rate that the two colours just merge, the reading (x ml.) being taken as soon as the rose colour persists for 6 seconds; then the percentage of naphthalene = $0{\cdot}5 \times 0{\cdot}7115x$/(weight of sample). The reaction is controlled oxidation to phthalic acid, and camphor interferes, since it also is oxidised under these conditions.

Louse Powders, etc.—A 500-ml. Kjeldahl flask is provided with an inlet connected with a steam-generator, and reaching to the bottom, and an outlet of 7 mm. tubing projecting a short distance into the flask, and connected with a condenser composed of 20-mm. tubing. This delivers into a cooled 1000-ml. Erlenmeyer flask provided with an air-vent and containing sufficient water to cover the end of the condenser. The sample (equivalent to at least 2 grms. of naphthalene) is weighed into the flask, 100 ml. of water and a little sodium hydroxide solution are added, and the naphthalene is removed by slow steam-distillation for 1 hour. The distillate and rinsings are filtered after acidification with sulphuric acid, the naphthalene being then washed with a little water and dissolved in at least 50 ml. of hot acetic acid. An aliquot portion of the resulting solution is treated as described in the preceding method. One per cent. of essential oil (which, however, is rarely present) gives an error in the direct titration of 0·5 to 1·5 per cent. Satisfactory results were obtained for 20 samples of the above types containing 1·91 to 73·42 per cent. of naphthalene; refined and commercial naphthalenes gave values of 100·08 and 99·47 per cent., respectively. J. G.

Colorimetric Method for the Determination of Rotenone. C. R. Gross and C. M. Smith. (*J. Assoc. Off. Agric. Chem.*, 1934, **17**, 336–339.)—Since the Jones gravimetric method (*Ind. Eng. Chem., Anal. Ed.*, 1933, **5**, 23) is inapplicable to small quantities, and the Durham colour reaction (*id.*, 1933, **5**, 75) is not sufficiently permanent for quantitative work, the following procedure is suggested: —A solution of the sample in acetone (other solvents, including methyl ethyl ketone, are unsatisfactory), corresponding with 0·05 to 0·3 mgrm. of rotenone per ml., is prepared, and 2 ml. are mixed with 2 ml. of a fresh, clear and colourless 10 per cent. solution of potassium hydroxide in 95 per cent. ethyl alcohol in a test-tube, which is then immersed in a bath at 20° C. for exactly 2 minutes. Six ml. of a mixture containing equal volumes of nitric acid and a 0·025 per cent. solution of sodium nitrite are then added, and the tube is quickly stoppered and cooled to 20° C. by immersion in the bath for at least 30 seconds; the resulting red colour is matched after 15 minutes, but before 50 minutes, against that produced from a standard at the same time and under the same conditions. After several hours a

vigorous reaction occurs which may cause the contents of the tube to boil over. Dihydrorotenone and deguelin give the reaction, but toxicarol, tephrosin, *iso*-tephrosin, nicotine, pyrethrum extract, or the primary decomposition-products of rotenone and deguelin (dehydrorotenone, rotenone and dehydrodeguelin) do not interfere. High results (by 50 to 100 per cent. compared with the gravimetric method) obtained with derris or tuba roots were attributed mainly to deguelin, although the possibility of the presence of other unidentified constituents also exists. A mixture of 1 per cent. of rotenone in diatomaceous earth gave six results ranging from 0·94 to 0·98 per cent., and 7 samples of commercial rotenone containing 86 per cent. and 1 containing 65 per cent. of rotenone (according to the polarimetric and gravimetric methods) were shown to contain 85 to 90 and 82 per cent., respectively. Fruit or leaves should be washed with a jet of acetone. Analyses made several weeks after spraying showed the presence of 7 to 30 mgrms. per fruit and 1 to 5 mgrms. per leaf for rotenone, and rather higher results for a derris spray. J. G.

Organic Analysis

Oxidation by means of Organic Per-Acids. J. Böeseken. (*Chem. Weekblad*, 1934, **31**, 166–170.)—The literature of the applications of oxidation by organic per-acids to investigations in organic chemistry is reviewed critically, and numerous examples are cited. Per-acetic acid is most commonly employed, and is usually prepared by the action of strong hydrogen peroxide on acetic anhydride in the presence of a little concentrated sulphuric acid, which is added as a catalyst towards the end of the reaction; if the proportion of hydrogen peroxide is not allowed to exceed 80 per cent., and the temperature is kept below that at which the flask can be held in the hand, a yield of 30 to 50 per cent., free from acetyl peroxide is obtained by subsequent distillation (*cf.* W. C. Smit, *Rec. Trav. Chim. Pays-Bas*, 1930, **49**, 685). Strong solutions of per-acetic acid in glacial acetic acid are not spontaneously explosive so long as catalytic impurities are absent and the temperature does not exceed 30° to 40° C. On one occasion a violent explosion was traced to the presence of a little acetyl peroxide in the distillation flask, and the potassium iodide and starch test should therefore be applied to ensure its absence; acetyl peroxide hydrolyses slowly, with the formation of acetic and per-acetic acids. On the other hand, per-benzoic acid (m.pt. 41° to 43° C.), which is best prepared by the action of sodium ethoxide on dibenzoyl peroxide (*cf.* Baeyer and Villiger, *Ber.*, 1900, **33**, 1569), may be handled safely if dissolved in ether or in chloroform, although such solutions are less suitable for storage than solutions of per-acetic acid in glacial acetic acid. J. G.

Identification of Amino Acids by means of 3 : 5-Dinitro-Benzoyl Chloride. B. C. Saunders. (*Biochem. J.*, 1934, **28**, 580–586.)—3 : 5-Dinitro-benzoyl chloride is recommended as a reagent for the rapid identification of many amino-acids. In alkaline solution the condensation is almost instantaneous, giving a good yield of crystalline derivative. The normal proportions are 1 mol. of amino acid to 1 mol. of the acid chloride in the presence of 2 mols. of sodium

hydroxide, but different acids require slightly different treatments. Some of the derivatives are insoluble in alkali, others require precipitation by dilute acetic or hydrochloric acid. The acidic amino acids are less reactive to this reagent, and this has been made the basis of a method of separating glycine and several other amino acids from aspartic acid. Glycine can be separated from glutamic acid by this method, for, although both give derivatives, that from glycine is formed preferentially. Tyrosine does not react in alkaline solution, neither does oxidised glutathione. Thiolacetic acid gives an S-3 : 5-dinitrobenzoyl thiolacetic acid, and although cysteine gives an S-derivative, it cannot be used for identification. The method is an improvement on the pyridine one of Wertheim (*J. Amer. Chem. Soc.*, 1929, **51**, 3661), as no fusing is necessary. S. G. S.

Condensations of Furan Compounds. III. Condensation Products of Furfural with Acetone in Acid and Alkaline Media, and a New Method of Determining Small Quantities of Furfural. W. W. Tschelinzeff and E. K. Nikitin. (*Bull. Soc. Chim.*, 1934, **1**, 184–189.)—In either an acid or an alkaline medium, furfural condenses with acetone, giving difurylidene-acetone or, if the acetone is in excess, furylidene-acetone. Under the action of sulphuric acid, these products condense, giving, for the former a reddish-violet, and for the latter an orange-red compound. By means of the first of these, acetone may be determined (*ibid.*, 1932, **51**, 875), and the second is now used for determining furfural.

To detect furfural, a glass rod, moistened with a drop of acetone containing 1 mgrm. of concentrated hydrochloric acid, is placed in the liquid to be tested or in the vapour therefrom; a bright orange-red colour indicates furfural. This reaction is shown in presence of other substances, but aliphatic aldehydes react with acetone before the furfural does, so that excess of acetone must be ensured. As little as 0·001 per cent. of furfural in solution is detectable in this way.

For the determination, 1 ml. of the solution, containing not more than about 0·1 per cent. of furfural, is mixed with 0·1 ml. of acetone (purified by formation of the bisulphite compound, hydrolysis and distillation, this series of operations being carried out three times, and the final product dried over anhydrous sodium sulphate) and 5 ml. of 50 per cent. sulphuric acid. The mixture is heated in a water-bath at 50° C. for 30 minutes, and the colour developed compared in a colorimeter with that obtained similarly from 1 ml. of 0·1 per cent. furfural solution. The intensity of the coloration is accurately proportional to the concentration of the furfural. The condensation of the furfural and acetone may be effected by means of sodium hydroxide, at 20° C., the liquid being acidified prior to the colour comparison. In this case the intensity of the resulting coloration is proportional to the square of the concentration of the furfural, the condensation in the alkaline medium evidently following a course different from that in the acid medium. T. H. P.

Observations on the Action of Chlorine on Lignified Tissues. F. M. Wood. (*Biochem. J.*, 1934, **28**, 52–61.)—During an investigation of the origin of a pink colour produced in lignified tissues when the chloramine reaction is employed, differential tests for coniferin and vanillin were made, since it was thought possible that the colour might be due to a compound of vanillin or coniferin, or their

derivatives, with chlorine. Other possibilities were that it might be due to a compound of lignin with chlorine or to the nature of the lignin present. Experimental work is described in which it is shown that the colour is not due to lignone chloride or to the products of chlorination removable by sodium sulphite, that it is closely associated with the presence of iodine and also of lignin, but that the nature of the lignin itself is not concerned, so that the latter association is either very loose or the substance is "encrusted" on the lignin. It is shown that vanillin is present when the colour is produced, and that there is more vanillin in older than younger tissues, in autumn than spring wood, in cortical than in stelar sclerenchyma, and that it is naturally present in the cell-wall. Hemicelluloses and coniferyl alcohol are not responsible for the observed effect. Under conditions similar to those used with plant sections experiments were made with vanillin, chlorovanillin, vanillic acid, vanillic acid treated with chlorine, and amines or chloramines, in which filter-paper, "Tarantulle" and wood-pulp were used upon which to "encrust" the compounds concerned. Indications were obtained that a vanillal derivative was formed which reacted with iodine to produce a substance coloured similarly to that obtained in plant tissues. The conclusion drawn is that vanillin, in loose association with some form of lignocellulose, is acted upon by chlorine, producing a chlorovanillin, which with the chloramines produced from protein within the cell, forms a vanillal derivative forming a pink substance in the presence of iodine. Should the chlorine not affect the vanillin to any marked degree, probably vanillin itself may react with the chloramines from the cell-contents and still produce the colour. It is possible that both series of reactions take place simultaneously. The possibility that vanillin is an excretory product, produced when the function of the tissues concerned has become chiefly that of mechanical support, is discussed. This theory is upheld by the fact that vanillin predominates in older tissues and in autumn wood. P. H. P.

Tetrachlorophthalimide as a Reagent in Qualitative Organic Analysis. C. F. H. Allen and R. V. V. Nichols. (*J. Amer. Chem. Soc.*, 1934, **56**, 1409.)—The potassium derivative of tetrachlorophthalimide reacts with many halogen compounds to give substituted tetrachlorophthalimides, which are useful for purposes of identification. A list of the melting-points of some 24 derivatives which have been prepared is given in the paper, and an improved method of preparation of the reagent is described. S. G. C.

Inorganic Analysis

Cadmium Sulphate as a Basis for Acidimetry. S. E. Q. Ashley and G. A. Hulett. (*J. Amer. Chem. Soc.*, 1934, **56**, 1275–1278.)—The clear, well-developed crystals of $3CdSO_4.8H_2O$, which are obtained by crystallisation of cadmium sulphate from aqueous solutions, are claimed to be a satisfactory standard material for standardisation purposes. A weighed crystal, or quantity of crystals, is dissolved in water, and the solution is electrolysed over a mercury cathode for a time sufficient to cause the deposition of the whole of the cadmium. The solution, which then contains the stoichiometric amount of free sulphuric acid, is removed from the electrolytic cell (which is finally washed out with water),

and is used for the standardisation of alkali in the usual way. The results obtained with alkali solution standardised independently had a mean deviation from the theoretical of 3·5 parts per 10,000. S. G. C.

Selenium as Indicator in the Bromate Titration of Arsenic. L. Szebellédy and K. Schick. (*Z. anal. Chem.*, 1934, **97**, 186–191.)—Colloidal selenium can be used as a reversible oxidation-reduction indicator. Under the conditions given below, which must be carefully adhered to, it gives good results in the bromate titration of arsenious acid. The arsenite solution is treated with 5 grms. of sodium bromide, and diluted to 35 ml. Strong hydrochloric acid (25 ml.) is added, and the solution is warmed to 55° or 60° C. After addition of 1 ml. of a molar solution of selenious acid (which produces a dark red colour) the solution is titrated with 0·1 *N* bromate solution, added drop by drop, until the last of the pink cloudiness is removed with a single drop of bromate solution. W. R. S.

Volumetric Determination of Tin with Potassium Iodate. J. B. Ramsey and J. G. Blann. (*J. Amer. Chem. Soc.*, 1934, **56**, 815–818.)—The reduction of tin to the stannous condition is effected by precipitation on zinc and solution of the spongy tin in hydrochloric acid in an atmosphere of carbon dioxide, 0·1 grm. of nickel or cobalt chloride being added to hasten solution of the sponge. The solution is then cooled in water, while carbon dioxide is passed through the flask, and diluted with 100 ml. of 0·2 *N* sodium bicarbonate solution (freed from air by boiling, and cooled in a current of carbon dioxide). The acidity of the resulting solution should be approximately normal. It is titrated under carbon dioxide with potassium iodate solution until a faint yellow colour proves liberation of iodine. The sides of the flask are at once washed down, and the excess of iodine is determined with thiosulphate and starch solution. The results by this method are claimed to be more accurate than those obtained by the usual iodine titration, which the authors show to give slightly low results. W. R. S.

Butylphenylarsonic Acid as a Reagent for the Gravimetric Determination of Iron. K. A. Craig and G. C. Chandlee. (*J. Amer. Chem. Soc.*, 1934, **56**, 1278–1279.)—The ferric iron solution (200 ml.), which may contain hydrochloric, nitric or sulphuric acid, is adjusted in acidity to 0·4 *N*, heated nearly to boiling, and 0·75 grm. of the reagent, dissolved in 100 ml. of hot water (80° to 90° C.), is added with stirring, yielding a white flocculent precipitate of the iron compound. The liquid is kept hot for 20 to 30 minutes and allowed to cool; the precipitate is filtered off, washed first with warm 0·02 *N* hydrochloric acid, and then with warm water, ashed, and ignited to ferric oxide. The accuracy was found to be within 4·5 parts per 1000 in tests with solutions of iron salts alone and in the presence of 0·1 to 0·2 grm. of dissolved nickel, cobalt, manganese, copper or cadmium oxide, and also 0·05 to 0·1 grm. of the dissolved oxides of potassium, aluminium, magnesium, calcium, beryllium, vanadium, lanthanum, erbium, and neodymium. In the presence of chromium as dichromate, reprecipitation was necessary after dissolving the first formed precipitate in acid, whilst with thallium present, the precipitate required to be washed with a saturated solution of the reagent in

1 per cent. hydrochloric acid, in order to remove hydrolysed thallium salt. The precipitation is prevented by substances which form complex compounds with iron—for example, fluoride or tartrate. The following interfere: Zirconium, tin, thorium, uranium, and cerium. Satisfactory results were obtained in the direct determination of iron in Monel metal and German silver. S. G. C.

Separation of Iron, Aluminium, and Chromium from Bivalent Metals by means of Ammonium Benzoate. I. M. Kolthoff, V. A. Stenger, and B. Moskovitz. (*J. Amer. Chem. Soc.*, 1934, **56**, 812–815.)—The reagent is a solution of 100 grms. of ammonium benzoate in 1 litre of water. The chloride solution of the metals (100 ml.), containing iron as ferric salt, is cautiously neutralised with dilute ammonia until the precipitate re-dissolves very slowly, and treated with 1 ml. of glacial acetic acid and ammonium chloride, if necessary, to the total amount of 1 grm.; a measured volume of the reagent (20 ml. per 0·065 grm. Al or 0·125 grm. Fe or Cr) is slowly stirred in. The liquid is stirred and heated to gentle boiling (5 minutes if chromium is absent, 20 if it predominates). The precipitate is collected and washed with a solution containing 10 grms. of ammonium benzoate and 20 ml. of glacial acetic acid per litre. For exact work the filtrate and washings should be evaporated to about 50 ml. for the recovery of any non-precipitated iron or chromium. The co-precipitation of the bivalent metals is much less pronounced than in the ammonia method, a single precipitation being usually sufficient in ordinary work. Phosphoric acid is partly co-precipitated.

W. R. S.

Determination of Zinc in Steel. H. A. Bright. (*Bureau of Stds. J. Research*, 1934, **12**, 383–389.)—Three methods have been investigated. (*a*) *Ammonia Precipitation Method.*—The solution of a 5-grm. sample in dilute sulphuric acid, oxidised with ammonium persulphate, is poured slowly, with stirring, into a considerable excess of dilute ammonia solution containing about 4 grms. of dissolved ammonium sulphate, and the solution, containing the zinc, is filtered, the precipitate being washed with ammoniacal ammonium sulphate solution (2 per cent.). The filtrate is evaporated to about 150 ml., neutralised to methyl orange indicator with 1 : 1 sulphuric acid, and 7·5 ml. of this acid are added in excess. Any copper or molybdenum present is precipitated from the solution with hydrogen sulphide, and the sulphides are filtered off and washed. The filtrate is boiled to expel hydrogen sulphide, and cooled; the solution is neutralised with ammonia, and sulphuric acid is added to give an acidity of 0·01 *N*. The zinc is then precipitated with hydrogen sulphide in the usual way, and the precipitate is finally ignited to zinc oxide. The method gave good results with 0·005 to 0·30 per cent. of zinc. (*b*) *Ether Extraction Method.*—The iron in a solution of 25 grms. of the steel is removed by the usual method of extraction with ether; the hydrochloric acid in the aqueous solution is removed by evaporation with sulphuric acid; the residue, after dilution, is poured into ammonia, and the determination is completed as in (*a*). This method was found to be the most satisfactory for amounts of zinc of the order of 0·001 per cent. (*c*) *Direct Precipitation with Hydrogen Sulphide.*—A 15-grm. sample is dissolved, so far as possible, in 96 ml. of 1 : 5 sulphuric acid, and the solution is filtered. The filtrate is diluted to 450 ml.,

and 5 grms. of ammonium sulphate and 60 ml. of citrate buffer solution (20 grms. of citric acid and 450 grms. of sodium citrate dissolved in 1 litre) are added, yielding a solution of p_H 2·5. The solution is saturated with hydrogen sulphide and kept overnight. The precipitate is filtered off, and dissolved in hot 1 : 9 sulphuric acid; the solution is boiled to remove hydrogen sulphide, and any copper, etc., is precipitated with hydrogen sulphide after adjustment of the acidity to about 2·5 per cent. of sulphuric acid. The precipitate is filtered off, and the zinc in the filtrate is determined as in (*a*). The method gave satisfactory results when 0·003 per cent. or more of zinc was present. The three methods were tested on solutions of various steels, in some cases known to be spectroscopically free from zinc, to which known amounts of zinc had been added. The results of analyses of 4 representative materials showed that the zinc-content of steels and irons is very small—of the order of less than 0·0005 per cent. S. G. C.

Gravimetric Determination of Thallium as Cobaltinitrite. S. Nisihuku (*J. Soc. Chem. Ind. Japan, English Abstr.*, 1934, **37**, 180B.)—Thallium may be precipitated as thallium cobaltinitrite $Tl_3Co(NO_2)_6$ by addition to the solution of sufficient of cobaltinitrite reagent [a mixture of equal volumes of a solution of (*a*) 28·6 grms. of cobalt nitrate and 50 ml. of 50 per cent. formic acid in 500 ml., and of (*b*) 180 grms. of sodium nitrite in 500 ml.], both the test solution and the reagent being previously warmed to about 40° C. The compound, which is formed as a scarlet crystalline precipitate containing 64·67 per cent. of thallium, is filtered off after 1 hour, washed with cold water, and dried at 120° C. The method is stated to have been applied to the determination of thallium and its separation from other metals, but no further details are given. S. G. C.

Determination of Iodide by Photometric Titration. S. Hirano. (*J. Soc. Chem. Ind. Japan, English Abstr.*, 1934, **37**, 177–178B.)—When an acid solution of an iodide is titrated with iodate solution, the reaction (1) occurs so long as iodide is present, the result being that the solution becomes progressively darker in colour owing to the iodine being liberated. On continuing the titration, the

$$(1)\quad IO_3^- + 5I^- + 6H^+ = 3I_2 + 3H_2O.$$
$$(2)\quad IO_3^- + 2I_2 + 6H^+ = 5I^- + 3H_2O$$

reaction (2) occurs, and the colour becomes progressively paler. Hence, measurement of the colour tone of the solution affords a means of determining the iodide. The author measured by means of a photo-electric cell the light transmitted through the solution during the titration of 0·01 *N* potassium iodide solution with 0·01 *N* potassium iodate solution, and found a sharp minimum corresponding with the end-point of reaction (1); it was concluded that the method is suitable for determining small amounts of iodide. S. G. C.

Microchemical

Detection of Sucrose in Lactose. M. Wagenaar. (*Pharm. Weekblad*, 1934, **71**, 281–284.)—The test depends on the fact (*id.*, 1933, **70**, 1030) that a solution of α-naphthol in glycerin gives, in the presence of sulphuric acid, a blue-violet or an orange condensation-colour in the presence of a ketose or aldose group, respectively. A suspension of 5 mgrms. of the sample in a drop of reagent is

therefore mixed on a microscope slide, by means of a platinum wire, with a drop of concentrated sulphuric acid; a blue-violet colour after 10 minutes, particularly at the edge of the drop, indicates the presence of a ketose compound. If the result at this stage is negative, it is advisable to hold the slide over a boiling water-bath for about 30 seconds, and to compare the resulting colour with that obtained with a specimen of pure lactose after similar treatment. The reaction is specific for fructose or sucrose, and will detect 1 per cent. or more of the latter; it may be applied to the residue after evaporation of the solution obtained by extracting the sample (according to the Dutch Pharmacopoeia) with dilute alcohol, which dissolves the sucrose in preference to the lactose. J. G.

Micro-titrimetric Determination of Nicotine in Tobacco. J. Bodnár and v. L. Nagy. (*Z. Unters. Lebensm.*, 1934, **67**, 598–601.)—The method of determining nicotine in fermented tobacco described by Bodnár, Straub and v. Nagy (*Biochem. Z.*, 1928, **195**, 103; 1929, **206**, 410; 1930, **227**, 452) has been found satisfactory by a number of analysts, but others (*e.g.* Dafert and Bollbecher, ANALYST, 1932, **57**, 389) consider that it gives inaccurate results. This method is carried out as follows:—One grm. (0·5 grm. for a high nicotine-content) of the powdered tobacco is mixed in a 50-ml. stoppered cylinder with 1 ml. (0·5 ml.) of 20 per cent. sodium hydroxide solution by means of a small glass rod, which is afterwards dropped into the cylinder. After addition of 20 ml. of a 1 : 1 ether and petroleum spirit mixture, the cylinder is vigorously shaken and left until the tobacco has settled. Ten ml. of the perfectly clear liquid are transferred to a 100-ml. Erlenmeyer flask, which is then heated on a steam-bath until only 1 to 1·5 ml. of a dark greenish-brown, viscous liquid remains (1 to 2 minutes). The flask is at once removed from the bath, 10 ml. of water and 1 or 2 drops of saturated alcoholic methyl red solution are added to the residue, and the liquid is titrated with 0·01 *N* hydrochloric acid until the greenish-yellow colour changes, fairly sharply, to red: 1 ml. of 0·01 *N* acid ≡ 1·620 mgrm. of nicotine.

Determinations of the nicotine-contents of a number of fermented and unfermented tobaccos have been made by this method, and also by that of Pfyl and Schmitt (ANALYST, 1927, **52**, 728). The two methods gave results in good agreement, and the maximum difference between four separate results for a single sample of tobacco was the same for the two methods. The micro-method requires practice, and the first results of even a skilled analyst may be subject to various errors, the sources of which are indicated. T. H. P.

Microchemical and Microscopical Identification of Santonin. M. Wagenaar. (*Pharm. Weekblad*, 1934, **71**, 260–264.)—Santonin is usually purified by sublimation (*cf.* Van Zijp, *id.*, 1927, **64**, 278; 1931, **68**, 526), and may then contain artemisin (oxy-santonin), many of the properties of which are similar to those of santonin. The sublimed santonin is soluble in lime-water and forms the calcium salt of santoninic acid; the original crystals, however, are unsuitable for identification under the microscope, and it is advisable to dissolve them in a drop of a mixture of acetone and water, from which santonin crystallises on warming. A preferable method is to use a drop of pyridine (in which santonin is very soluble), a drop of water being then added, when characteristic, thin,

sharply-defined rectangular crystals separate, which may be identified under the microscope, particularly if polarised light is used; this procedure serves to detect 1 mgrm. of santonin. Solutions of sublimed santonin in warm strong sulphuric acid, when cooled and diluted with water, deposit hour-glass shaped plates and similar effects result on diluting solutions in warm glycerin. Van Zijp's reaction (*loc. cit.*) is best carried out on a solution of santonin in sulphuric acid, to a drop of which is added, in succession, a drop of water and a drop of a solution of iodine in potassium iodide solution. The resulting precipitate is re-dissolved in acetone, and, on evaporation, well-formed dichroic square crystals result, which are usually yellowish-green to colourless, but are sometimes dark-brown to red, in which case they are produced as rectangles; the latter have been attributed to the periodide of artemisin. Lindo's test (in which a purple colour is produced with solutions in sulphuric acid by the action of ferric chloride) is essentially a test for the lactone group, and may also be carried out with a solution of ferric ammonium alum. The pale red colour produced with an alcoholic solution of potassium hydroxide is not well-suited to microscopical work, but it may be improved and rendered sensitive to 2 mgrms. of santonin if this alkali is replaced by freshly-ignited lime, and glycerin is used as solvent. J. G.

Collected References. Arsenic, Antimony, Tin, and Bismuth. K. Heller. (*Mikrochem.*, 1934, **14**, 369–406.)—References since 1926 are collected for the four elements. In the qualitative section a brief critical summary of the methods is given, together with details of the sensitivities. The references in this section comprise:—General separation 21, arsenic 52, antimony 19, tin 24, bismuth 25. In the quantitative section the methods include spectrographic, gravimetric, volumetric, colorimetric, and electrical methods, as well as application to different materials, such as foodstuffs, gases, and animal tissues. The references in this section comprise:—General 5, arsenic 52, antimony 14, tin 16, bismuth 33. In all, 260 references are given. J. W. M.

New "Spot" Test for Beryllium. A. S. Komarowsky and N. S. Poluektoff. (*Mikrochem.*, 1934, **14**, 315–317.)—A new reagent made by coupling *p*-nitroaniline and orcinol reacts with beryllium; magnesium does not interfere with the test, except when present in relatively large amounts. *Preparation of Reagent.*—A hydrochloric acid solution of 1·38 grm. of *p*-nitroaniline is cooled to 0° C. and diazotised with a cooled concentrated aqueous solution of 0·85 grm. of potassium nitrite, and the resulting solution of *p*-nitrobenzyldiazonium chloride is mixed at 0° C. with a sodium hydroxide solution containing 1·42 grm. of orcinol. On acidifying the mixture the dyestuff is precipitated; it is separated, washed with dilute acid, and water, and dried. A 0·025 per cent. solution in *N* sodium hydroxide is used. *Test.*—A drop of the reagent is placed on a filter-paper, and followed by a drop of the test solution in a capillary, the point of which is allowed to touch the middle of the fleck. A further drop of reagent is added, and in the presence of beryllium a brilliant orange-red colour appears. The smallest amount recognisable is 0·2γ of beryllium in 0·04 ml. of the test solution; this corresponds with a *concentration limit* of 1 : 200,000. Under the same conditions magnesium gives a yellow-brown precipitate, but this masks the beryllium colour only when

the magnesium is present in considerable excess; a preliminary separation must then be made. Calcium, strontium, barium and aluminium do not interfere, neither do the rare-earth metals lanthanum, praseodymium, neodymium, cerium, zirconium and polonium. Zinc gives the same colour, but it is discharged by moistening the fleck with a drop of a 25 per cent. solution of potassium cyanide, so that 0·6γ of beryllium may thus be detected in the presence of 715 times the amount of zinc. Nickel, cobalt, copper, cadmium, and silver, which give coloured hydroxides, interfere, but the interference is prevented by complex cyanide formation. J. W. M.

Physical Methods, Apparatus, etc.

Capillary Index of Certain Vegetable Oils. H. Marcelet. (*Compt. rend.*, 1934, **198**, 2073–2074.)—The volumes of the drops of water and of a sodium hydroxide solution of known strength delivered from a burette into a benzene solution of the fatty acid under examination are measured, and the ratio of the difference of these two numbers to the volume of a drop of pure water is the capillary index or emulsifying power for the fatty acid. (Dubrisay and Picard, *Compt. rend.*, 1923, **177**, 589; 1924, **178**, 205; *Dubrisay, Compt. rend.*, 1925, **181**, 1060, 1142; 1932, **194**, 1076.) This procedure has been applied to various vegetable oils by measuring the volume of 6 drops of water, and subsequently of 6 drops of 0·0016 *N* sodium hydroxide solution in a solution of 1 part of the oil in 100 parts of benzene, the drops being allowed to fall at the rate of 3 per minute. The capillary indices found for certain oils were as follows:—Virgin olive oil, 61 to 65; bright olive, 63 to 69; refined olive, 27 to 33; olive pulp oil, 41 to 48; maize oil, 15 to 19; soya bean oil, 13; sesame oil, 58; and arachis oil, 24. The figures for crude and refined olive oil were confirmed by examining a large number of specimens of various origins representing 421 tons of oil, and the average figures were 66·9 for crude, and 31·4 for refined oil. D. G. H.

Electro-Magnetic Method of Measuring Specific Gravity. L. R. Bishop. (*J. Inst. Brewing*, 1934, **40**, 92–94.)—The principle of the apparatus is similar to that employed by Lamb and Lee (*J. Amer. Chem. Soc.*, 1913, **35**, 1666), and its purpose is to provide a check on (and possibly to replace) the specific-gravity bottle method. The liquid under investigation is contained in one of a pair of double-walled vertical tubes, which are used alternately and are filled and emptied at the bottom by means of a suction pump, the liquid being recovered at the end of the measurement in a bottle inserted between the pump and the tube. The tubes, which contain thermometers, are in the form of an inverted cone at the bottom, a solenoid being wound on this portion, so that a current from an accumulator flowing through the wire exerts an electro-magnetic attraction on a hollow soft-iron float in the liquid and causes it to sink. If the current is then gradually reduced until the float just begins to rise again, its strength (A) at this point is a measure of the sp.gr. of the liquid. Three small projections on the base of the float prevent it from sticking to the side of the tube, and allow the liquid to drain away, and self-centring of the float with respect to the magnetic field is ensured by the shape of the base of the tube; this shape also produces a rapid acceleration

of the rate of rise of the float, since, when the current is just too weak to hold the float down, the latter rises into a weaker magnetic field. The electrical circuit is described and shown in diagram. The apparatus was tested with 96 worts, covering a wide range of specific gravities and temperatures, and the equations found for one tube were:

$$S = 1018{\cdot}375 + 0{\cdot}0339A - 0{\cdot}2038T \pm 0{\cdot}124, \text{ and}$$
$$S_1 = 1021{\cdot}406 + 0{\cdot}0348A \pm 0{\cdot}119;$$

where S is the sp.gr. of the wort (60° F./60° F.), that of water at 60° F. being taken as 1000·000; and S_1 is the apparent sp.gr. at the temperature T (in °C.). Corrections for temperature are given in a table (which applies only to worts), and are calculated from the change in sp.gr. of water with temperature and the results of Day and Amos (*id.*, 1914, **20**, 196). When allowance was made for these, standard errors were obtained for the respective tubes corresponding with 0·4 and 0·5 lb. of extract (10 per cent. worts). The relation between A and S may also be shown graphically. J. G.

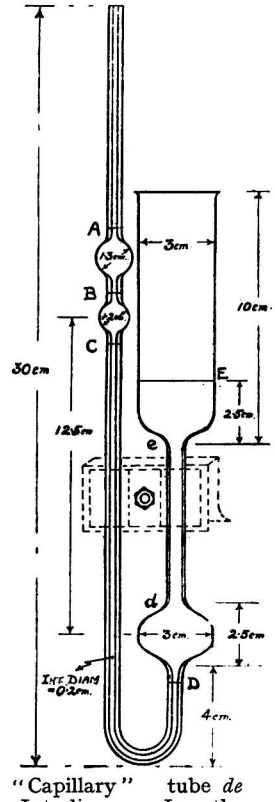
Measurement of the Strength of Gelatin Gels. L. H. Lampitt and M. E. G. Norris. (*J. Soc. Chem. Ind.*, 1934, **53**, 179–182T.)—The apparatus devised for measuring gel strength was constructed from a "decimal balance" designed to weigh a load of 10 kilos. The large stirrup and balance pan were replaced by a smaller stirrup and weight pan, and the centre of the pan support was drilled to carry the standard plunger, which consisted of a rod terminating in a hemisphere, 4·5 mm. in radius. A small pan for weights was suspended from the knife-edge originally provided for the weight-pan. The pointer and scale of the balance were retained, since these provided a means of following the plunger movement, which was thereby magnified 10·7 times. A platform on which to place the gels, adjustable in height by means of a screw-thread cut in the base, was mounted beneath the plunger. An aluminium container with a gate control, from which fine, dry, silver sand could be run into a cup on the scale-pan above the plunger, was attached by means of a bar to the central column of the balance. The rate of sand-flow was controlled by the size of the aperture in the gate, which was such that 2·85 grms. of sand per second could pass when the container was full. The rate of flow rèmained reasonably constant up to 60 seconds, but after this it was reduced, owing to the alteration in the level of the sand remaining in the container. For making a measurement, the balance is adjusted to a 0·1 grm. weight in the pan, until the pointer rests at zero, and the gel is placed on the platform so that the plunger will engage the centre of the surface. The load is applied by opening the sand-control gate, which is shut off when the pointer reaches the 5th scale-division, corresponding with a depression of 4·5 mm. The plunger is lifted, the gel is removed, and the weight required to produce the depression is measured by placing weights on the pan until the pointer balances at zero. The result is multiplied by 10. From the apparent gel-strength the initial load of 1 grm. is subtracted and also the weight required to displace the beam from zero to division 5, the latter constant figure being determined experimentally. The gels are prepared by pipetting 100 ml. of water and putting 17 grms. of gelatin (ground, or in small pieces) in 125-ml. wide-necked bottles with ground glass

stoppers, with internal dimensions of 5 cm. in diameter, and 7 cm. from base to shoulder. The bottles are rotated until the gelatin is wet and are then left for 15 to 30 minutes. They are then placed, shoulder deep, in a cold water-bath, and the temperature of the water raised electrically to 67° to 68° C. A control bottle with a thermometer in a similar gel is used with each batch of gels, and at 65° C. the heat is shut off, the bottles are left for 10 minutes, and then removed, and their contents are stirred so as not to produce froth. The bottles are left to cool until the temperature of the control is 30° C., when the stoppers are removed and wiped dry from condensation water, the contents of the bottles are again stirred, the stopper replaced, the bottles are put in a specially constructed water-thermostat maintained at 21° C. ± 0·05° C., for 16 hours, and the strength of the gels is then measured. Results can be satisfactorily reproduced by this method. A comparison of flat and hemispherical plungers was in favour of the latter, since by its use a large depression may be produced without permanent strain, and the degree of accuracy measured may thus be increased. If the ageing period be extended beyond 16 hours, a correction may be made. Between 17° and 23° C. the relation between gel-strength and temperature was found to be practically linear. It is very important to keep the temperature of the gel constant during the ageing period. D. G. H.

An Ostwald Viscometer for Tar. A. R. Lee. (*J. Soc. Chem. Ind.*, 1934, **53**, 69–70T.)—The disadvantage of the Hutchinson and Redwood types of viscometer is the fact that they do not give absolute results; the falling-sphere method is unsuitable for tar on account of the difficulty in locating the sphere in an opaque medium, and the large amount of sample required; and the rotating-cylinder apparatus does not ensure the accurate and uniform temperature control necessary for road tars (for which a change of 0·1° and 5° C. may produce changes of viscosity of 5 and 300 per cent., respectively). The apparatus described gives reproducible results with only 20 ml. of an opaque liquid over a wide range of temperatures, with a timing error of 1 : 1000 even with very viscous tars. It may be completely immersed in a thermostat; readings may be repeated on the same sample for any desired temperature-range without dismantling or cleaning; there is no drainage error; the density of the tar may be neglected if the instrument is suitably calibrated; and the relation between the pressure and the rate of flow may be measured by the use of a supplementary pressure (*vide infra*). The tube *de* in the figure varies from 2 to 6 mm. in length, and serves for viscosities of from a few poises up to 10^6 poises, but the ratio of length to radius must always be the same, so that the rate of shear is constant for the same liquid in the different instruments. A three-way tap above A connects either with atmospheric pressure or with suction, and suction is used to fill the tube accurately with mercury between the marks A and D; the apparatus is then connected with atmospheric pressure, and immersed in hot water, and the tar is poured into the right-hand limb until it reaches D, any air being expelled by the rise and fall of the mercury produced by manipulating the three-way cock. A rubber bung with a glass tube is then placed in the wide limb, and the whole apparatus is allowed to assume the temperature of a thermostat (controlled to within 0·002° C.), the mercury being then sucked up to A, and allowed

to fall again under atmospheric pressure, the time (t seconds) for it to pass from B to C being noted. No correction for expansion of the mercury is required at higher temperatures, because the bulb AB allows the mercury to be well within the bulb Dd before timing begins. The apparatus is calibrated by means of a transparent liquid (*e.g.* syrup), which may be used in the falling-sphere apparatus, or by extrapolation from the values obtained with a smaller viscometer of known constant, the two instruments being filled at the same time and from the same supply, and allowance being made for differences in density (the density of tar varies from 1·14 to 1·26, and may be taken for this purpose as 1·20 for any tar). The viscosity of any tar is given by $\eta = (At - B/t)$, the constants A and B being determined from tests with liquids of widely different viscosities, or with the same liquid at two different temperatures; the error for the abbreviated expression $\eta = Kt$ is less than 1 per cent. Although the design of the apparatus ensures that the driving-pressure is proportional to the difference between the densities of the mercury and the liquid being examined, this pressure may be measured directly by measuring the respective *vacua* required to maintain the level of the mercury at B and at C, and if this method is used for the liquids employed for the calibration and for the test, there is no need to know their densities. The effect of driving-pressure on the rate of flow may be studied by using an auxiliary vessel to produce either a vacuum or an additional pressure. Thus, when an external pressure varying from −6·4 to +17·1 cm. of mercury was applied to a syrup known to obey Poiseuille's law, the product of the mean driving-pressure and t varied only between 972 and 998. The time of flow may be controlled by altering the size of the bulb BC; viscosities of 50 and 10^5 poises may correspond with about 15 seconds and 10 to 15 minutes, respectively. The instrument is emptied by bringing the cool tar to the bend below D and inverting the apparatus; it is cleaned by immersion in a hot solution of soap.

J. G.



"Capillary" Int. diam., cm.	tube *de* Length. cm.
0·2	2·5
0·3	3·75
0·4	5
0·5	6·25
0·6	7·5

Reviews

TAUTOMERISM. J. W. BAKER, D.Sc., Ph.D., A.R.C.S., F.I.C. Pp. viii + 332. London: George Routledge & Sons, Ltd. 1934. Price 25s. net.

Of the various subjects usually dealt with as branches of "organic" chemistry, tautomerism is undoubtedly one of the most interesting, and the appearance of a monograph on the subject is very welcome. The wrapper of the book informs us that the work is "a detailed and broad survey of the whole of this field in the light of modern theoretical conceptions," a statement which is virtually repeated in the preface, though qualified by the statement that "although there is a fair measure of agreement amongst chemists with regard to the fundamentals of electronic theory, there is naturally some divergence of opinion in relation to detail. Hence, in attempting to deal with theories which are in such a fluid state of development, it has been considered desirable to adopt a single-minded viewpoint in order to maintain a continuity of argument throughout this monograph."

The above quotation should be kept in mind, for otherwise the reader with a limited knowledge of the subject might receive the impression that practically all the important work had been carried out in the last few years, and so give less credit to earlier workers than they deserve.

Besides the preface, the book contains fourteen chapters and an appendix consisting of the paper read before the Chemical Society on the occasion of the general discussion on May 12th, 1933. The book finishes with subject- and author-indexes which appear to be accurate; the reviewer has found no mistakes in them, except the omission of one acute accent and one *umlaut* sign.

Chapter I is an historical introduction to the subject, and the author sees early glimmerings of the tautomeric idea in Kekulé's dynamic conception of the benzene nucleus and the equilibrium of the two di-*iso*butylenes described by Butlerov in 1877. Erlenmeyer's unsuccessful attempt to isolate vinyl alcohol and the existence of two isomeric methyl-isatins by Baeyer lead to the tautomeric theory of Laar (1885). Mention is made of the oscillation theory of Baly and Desch (isorropesis, 1904), and of the experiments of Lowry and Desch (1909) on the relationship between absorption spectra and isomeric change, which rendered the theory of isorropesis untenable. Meanwhile, Laar's original conception needed modification on account of the work of L. Claisen, W. Wislicenus and L. Knorr. Claisen obtained (1896) two forms of acetyldibenzoylmethane of different melting-points, but both of the same molecular weight—one soluble in sodium carbonate solution and giving a coloured iron salt, the other non-acidic and indifferent to ferric chloride. Enol and keto forms were found to be distinct individuals, and later work has shown that both forms of a tautomeric compound may be isolated in many cases, provided suitable conditions are chosen.

The correlation of tautomerism with reversible additive reactions is treated in Chapter II following the lines laid down by Miss Usherwood (Mrs. Ingold) in 1923, whilst Chapter III deals with the modern theory of tautomeric change. Since the subject is regarded from an electronic standpoint, the names of J. J. Thomson, G. N. Lewis, Langmuir, Robinson, Lowry, and Ingold are found in

connection with the electronic characteristics of groups. We are thus led to the consideration of atomic linkage, whether by electrovalency or co-valency, and the concept of the inductive effect naturally follows. The standpoint taken by the author is well illustrated by a quotation (p. 33): "Examples of tautomeric changes known to these early workers were almost exclusively those which involved the migration of a hydrogen atom, and the modern ionic mechanism simply involves an extension of the views outlined above to all types of tautomeric systems, no matter whether the mobile group is a positive hydrogen ion or some other cation (*e.g.* a metal), or whether it is an anion. On this view tautomeric change involves the separation of either a cation or an anion, leaving an electromeric ion (anion or cation respectively), so that the eliminated ion has two possible points of recombination." It is such electromeric ions ("common ions") which chiefly engage the attention of the author, and the location of the charge on such ions is examined at length in the succeeding chapters. These deal with mobility and equilibrium of cationotropic systems (one could wish for a fuller description of experimental methods, both here and throughout the book); dyad, triad and pentad systems, ring-chain tautomerism, valency tautomerism, anionotropy, transannular tautomerism and pinacolic electron-displacement.

It will be seen that the work deals with several subjects outside the narrower conception of tautomerism, *e.g.* the "pinacolic electron-displacement" of Ingold and Shoppee, or the theory of Clar as to the cause of colour in the case of *lin*-benzanthracene and *lin*-dibenzanthracene. The book, which is essentially an exposition of the views held by one school as to the cause (and course) of electron displacement in the molecules of carbon compounds, could have been rendered even more complete had the subject of substitution in the aromatic series been considered at length. The reviewer fully admits that this would be outside the purview of a work on tautomerism.

Reference should be made to the Appendix by Professor Ingold, where the "Significance of Tautomerism and of the Reactions of Aromatic Compounds in the Electronic Theory of Organic Reactions" is dealt with. The development of the idea of mesomeric effect and applications to the optical activity of the nitro-anion (Kuhn) and retention of optical activity during the bromination of a ketone (Leuchs and Watke) are especially interesting to some chemists.

Whether the reader agrees with the author in all points or not, he will readily admit the thoroughness with which Dr. Baker puts forward the views of the school he represents. No objection can be taken to this; one is disarmed by the statement in the preface. At the same time one could wish that a fresh edition would handle the experimental side in rather greater detail and assign rather more space relatively to compounds which contain other elements besides carbon, hydrogen and oxygen in the molecule. J. T. HEWITT

QUALITATIVE CHEMISCHE ANALYSE NACH DEM SCHWEFELNATRIUMGANG. By the late GEORG VORTMANN and ROBERT LIEBER. Pp. viii+184. Vienna: Emil Haim & Co. Price 7.50 RM., bound 9.00 RM.

Since 1898 the sodium-sulphide method of qualitative analysis has been in use in the Technical College (Technische Hochschule) in Vienna, in place of the

usual hydrogen-sulphide procedure. The original procedure, developed by Dr. Vortmann, has been considerably modified, and the group-divisions described in the present book are:

I. Sulphides, carbonates and hydroxides insoluble in sodium sulphide solution:
   A. Sulphides insoluble in dilute hydrochloric acid (1 : 20).
   B. Sulphides, carbonates and hydroxides soluble in dilute hydrochloric acid (1 : 20).

II. Sulphides soluble in sodium sulphide.

Tests for alkali metals are made on a separate sample. The method is stated to be cleaner, cheaper, and more rapid; also the analysis is carried out without the necessity for the student to leave his bench, a number of organic compounds may be present without interfering, and tungsten, molybdenum and vanadium can be more easily detected, than in the hydrogen-sulphide procedure.

In this book the procedure described includes tests for a number of less common elements, such as cerium, thorium, zirconium, titanium, beryllium, gold, platinum, tungsten, molybdenum and vanadium.

The tests for cations occupy 100 pages and those for anions 20 pages. They include a number of the modern "spot" tests and tests involving the use of organic reagents. The book is clearly written, and will undoubtedly be of service not only to those who wish to use the sodium-sulphide procedure, but also to all interested in inorganic analysis.

The book might have been improved by a better index, and by a few tabular representations of the separations involved.

JANET W. MATTHEWS

LE ACQUE MINERALI D'ITALIA. QUADERNO PRIMO: LAZIO. Commissione Permanente per le Acque Minerali d'Italia. Pp. viii+239. Rome. 1933.

In abundance and variety of natural waters, Italy probably occupies the foremost place among the countries of Europe. Many of these waters exhibit curative properties, and were famed on this account even in very early days. Later, many of the finest establishments at which the waters were taken either suffered neglect or were destroyed by the barbaric invader. Despite occasional protests, little effort was made to recondition the wells until after the constitution of the Kingdom of Italy, when, following chemical, clinical, and pharmacological investigations, the activity of certain of the spas was revived. This revival has gradually progressed, and is being so stimulated and encouraged under the present régime that Italians no longer need go abroad to take a cure.

The present volume, issued under the authority of a permanent commission presided over by Professor Nicola Parravano, is apparently the first of a number projected, and deals with the waters of Latium. With the help of a number of maps, the geology of the district is described, and, for each of the waters, the chemical, physico-chemical, radio-active, and microbiological properties are given in detail. A separate bibliography is supplied for each of the waters. The make-up of the book is pleasing, and a number of excellent reproductions of pencil sketches of different wells are included. No price is stated.

THE DETECTION OF CRIME: AN INTRODUCTION TO SOME METHODS OF SCIENTIFIC AID IN CRIMINAL INVESTIGATION. By W. M. ELSE and J. M. GARROW. Pp. xxi+195. London: The Office of *The Police Journal*, 69, Great Russell Street. 1934. Price 6s. net.

In this country the scientific investigation of crime is based on a principle different from that adopted on the Continent. With the exception of finger-print investigation, the development of which has been brought to a high state of perfection, the police authorities here rely, in the main, upon scientific assistance from outside, and police laboratories of the type established in many cities abroad are as yet unknown. Both systems have their advantages and their drawbacks, and a combination of both would probably give the best results. If the external system is to be efficient, it is essential that the superior police officers in each area should be well acquainted with the possibilities of scientific examination, and be in a position in every case to call upon the help of the right specialist without delay. Stress is laid upon this point by Dr. Roche Lynch in his foreword to this useful little handbook. The aim of the authors, who hold official positions in the Derby Constabulary and have a wide experience of the difficulties of criminal investigation, has been to give an outline of the principles of the methods used by the outside expert, and thus prevent the loss of valuable evidence through ignorance of what can be done in a given case.

After a preliminary chapter on the training of the faculties of observation and deduction, the authors proceed to the discussion of the various subjects that might require scientific examination and interpretation. There is a chapter on how to search for blood-stains, seminal stains, dusts and soils, and the information on blood-stains is supplemented in a later chapter by a description of the significance of blood-tests, including the determination of blood-groups. Other chapters in this part of the book deal with the identification of hairs and fibres, the examination of handwriting, typescript, and charred papers, the use of dyes and stains in detection methods, the identification of firearms and projectiles, the differentiation of motor tyres (illustrated by a series of plates), preservation of excreta, etc., in toxicological cases, together with points of procedure for the guidance of the policeman.

The second part of the book, headed "Technique," contains seven chapters, dealing with microscopy, photomicrography, and microchemical tests, and is entirely practical. The descriptions are simple and clear, and the authors are obviously giving the results of their own experience. It might, perhaps, be mentioned that they and Superintendent Evans were associated with Professor Brose in working out the fluorescence method of photographing finger-prints described in this journal (ANALYST, 1934, 25). Among the numerous useful sections is one on the structure of the feathers of different species of birds, which is illustrated by plates.

This part of the book is far beyond the scope of the average policeman, and it rather suggests that the Continental system of the police laboratory may not be so far distant after all. Regarded as a whole, the book will be found of value not only by the police, for whom primarily it has been written, but also by the

wider public who keep in touch with the scientific investigation of crime; and the authors may be congratulated upon having produced a work that is scientifically sound and full of interest.

At the end of each section are given the names and addresses of specialists who undertake the kind of work described. This is information required by the police, although it would be out of place in a handbook not having such a specific purpose in view.

EDITOR

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