FEBRUARY, 1928. Vol. LIII., No. 623.

THE ANALYST

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

An Ordinary Meeting of the Society was held at the Institute of Chemistry on Wednesday, January 11th, the President, Mr. E. Richards Bolton, F.I.C., being in the chair.

Certificates were read for the first time in favour of Andrew R. Buchanan and Arthur Gordon Francis, B.Sc., F.I.C.

Certificates were read for the second time in favour of George Reginald Barnes, B.Sc., Cecil Abell Bassett, B.Sc., A.I.C., Ethel Irene Beeching, M.Sc., A.I.C., Harold Pease Buttrick, A.I.C., Cecil Owen Harvey, B.Sc., A.R.C.S., A.I.C., Harold Vivian Horton, B.Sc., A.I.C., Thomas Howard, M.Sc., A.I.C., Harold McKee Langton, M.A., B.Sc., A.I.C., William Alfred Nottage Markwell, Walter George Messenger, B.Sc., A.I.C., Edward John Newby, B.Sc., Horace Samuel Rooke, M.Sc., A.I.C., S. Sera, Claude Trevine Symons, B.A., F.I.C., David Rees Thomas, M.B., Ch.B., and Wilfred A. Whitley.

The following were elected Members of the Society:—Alfred Harry Bateman, B.Sc., A.I.C., Arthur Owen Blackhurst, M.D., William Clayton, D.Sc., F.I.C., Charles William Cornwell, B.Sc., A.I.C., and Thomas Riley, A.I.C.

The following papers were read and discussed:—"The Determination of small quantities of Benzoic and Cinnamic Acids, with some notes on the Colorimetric Determination of Salicylic Acid," by J. R. Nicholls, B.Sc., F.I.C.; "Report of the Preservatives Determination Committee of the Chemists of the Manufacturing Confectioners' Alliance and of the Food Manufacturers' Federation," by L. E. Campbell, M.Sc., Ph.D., F.I.C.; "Notes on the Effect of other Reducing Substances on the Determination of Sulphur Dioxide," by J. W. Black, B.Sc., A.I.C., and B. W. J. Warren, F.I.C.; "(i) Rapid Determination of Sulphites by Alkaline Liberation, or Extraction, and Titration. (ii) Barium Sulphate Losses in Gravimetric Estimations," by H. R. Jensen, M.Sc., F.I.C.; "Determination of Sulphur Dioxide in Sausages," by Osman Jones, F.I.C.; "(i) Note on the Oxidation of

Sulphites by Air. (ii) Note on the Titration of Dilute Sulphite Solutions with Standard Iodine Solutions," by H. M. Mason, M.Sc., F.I.C., and G. Walsh; and "Determination of Sulphur Dioxide in Fatty Substances," by A. W. Knapp, B.Sc., F.I.C., and R. J. Phillips, F.I.C.

Jubilee of the Institute of Chemistry

THE PRESIDENT represented the Society at the dinner given by the Institute of Chemistry to celebrate its Jubilee, and presented the following address:—

THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS
TO
THE INSTITUTE OF CHEMISTRY.

WE, the President, Council and Members of the Society of Public Analysts and other Analytical Chemists, wish to offer our warmest congratulations to the INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND on attaining its Jubilee.

Throughout the fifty years that have passed since it was founded the Institute has never failed to show an active interest both in our Society and in its individual Members, the majority of whom are actually members of the Institute, and the friendly ties which from the first have united the two bodies have grown closer with increasing co-operation in mutual activities.

The position which the Institute has gained in the public esteem is a proof that the value of its great work in raising the status of the profession of Chemistry has become generally recognised and appreciated, and we feel confident that the next half century will see its influence still more widely extended.

That the Institute may continue to prosper and enjoy the loyal support of all Chemists is the cordial wish of this Society.

Seal
of the
Society

Signed on behalf of the Society of Public Analysts
and other Analytical Chemists.
E. RICHARDS BOLTON,
President.

LONDON, *December 15th, 1927.*

Death.

WE greatly regret to record the death of Charles James Waterfall, F.I.C., who joined the Society in 1903.

Oil Bromide Films and their use in Determining the Halogen Absorption of Oils.

BY HAROLD TOMS, M.Sc., A.I.C.

(*Work done under the Analytical Investigation Scheme.*)

(*Read at the Meeting, December 7th,* 1927.)

IT has already been shown that the "insoluble bromide" of linseed oil (ANALYST, 1924, **49**, 77; *Annual Reports*, 1924, 158) is identical with that obtained from other vegetable drying oils (ANALYST, 1926, **51**, 387). It seemed desirable, therefore, to establish, as far as possible, the constitution of this compound, for it is very probable that the unsaturated compound from which it is derived is very largely responsible for the drying properties of these oils.

The first part of this paper is an account of the efforts to establish the constitution of this substance, and the second is an account of an accurate micro-method for determining the unsaturation value of oils. This method, which appears to be of general applicability, was evolved to overcome certain difficulties that arose in the course of the work mentioned above. Incidentally, new light has been thrown on the peculiar behaviour of tung oil.

PART I.

THE CONSTITUTION OF THE INSOLUBLE BROMIDE.—Analysis shows that the insoluble bromide obtainable from most of the drying oils is probably dilinolenolinolin-bromide—a conclusion supported by the independent work of Eibner and Schmidinger (*Chem. Umschau*, 1923, **30**, 293). Hydrolysis ought, therefore, to give two molecules of hexabromostearic acid and one molecule of tetrabromostearic acid. Although the first acid has been found, the latter for a long time escaped detection; the probable reason for this has only recently been discovered. After many failures it was decided to debrominate the bromoglyceride and to attempt the isolation of the linolic acid by hydrolysis of the unsaturated product.

The debromination proved extremely difficult. This is somewhat remarkable in view of the ease with which α- and β-linolenic acids are obtained by the debromination of hexabromstearic acid (*cf.* Erdmann and Bedford, *Ber.*, 1909, **42**, 1324). The usual methods were tried and failed. Even the remarkable method involving the use of sodium iodide in ethyl aceto-acetate solution, which was used with success by Grün (*Z. angew. Chem.*, 1916, **29**, 37) for debrominating $\alpha\alpha$-dilinolin bromide, failed in this case. Metallic sodium and metallic calcium in alcohol removed the bromine, but produced a solid hydrogenated fat.

Partial success was ultimately obtained by means of zinc dust and 95 per cent. alcohol. The reaction is very slow indeed; only after 80 hours of continuous boiling was any product obtained, and even then the yield was prohibitively small.

Attempts at synthesis have been entirely abortive, for it was found that the acid chloride or bromide of hexabromostearic acid cannot be obtained, and the three methods available for preparing glycerides of known structure, *viz.* that of Fischer (*Ber.*, 1920, **53** (B), 1589), that of Bergmann (*Ber.*, 1921, **54** (B), 936) and that of the present author (*J. Chem. Soc.*, Trans., 1921, **119**, 1035–1040), all require acid chlorides or bromides of reasonable purity. It was found that phosphorus chlorides and phosphorus bromides under various conditions carbonise the bromo acids, whilst thionyl chloride gave only a small yield of the acid chloride, which, since it could not be separated from the unchanged acid, was useless for the delicate, selective esterification methods mentioned above.

EXPERIMENTAL.

THE DEBROMINATION OF DILINOLENO-LINOLIN BROMIDE.—The bromoglyceride (5 grms.) was suspended, together with zinc dust (10 grms.), in 95 per cent. alcohol (100 c.c.), and boiled continuously for 80 hours. After cooling, the mixture was filtered with the aid of the pump. The residue contained much unchanged substance. The filtrate, which contained a few suspended droplets of oil, was concentrated, poured into water, extracted with ether, and the ethereal extract dried over anhydrous sodium sulphate. On removing the ether a few drops of a deep yellow oil resulted. This oil could not be purified by distillation, as it resinified on heating, even at low pressure. This experiment was repeated several times on separate preparations of dilinoleno-linolin bromide. In each case the resulting compound was a yellow oil, which hardened rapidly in the air, giving a tough and somewhat crystalline film. It had a specific gravity of 0·957 at 10° C., and was soluble in ether, acetone, benzene and chloroform.

Unsaturation Value.—The smallness of the amount available and the consequent need for the conservation of the material suggested the idea of "drying" a small weighed amount of the oil in an atmosphere of bromine, and from the increase in weight determining the unsaturation value. The brominated film thus produced would be still available for crystallisation and subsequent determination of the melting poing. (Bromine content of film = 55·6 per cent.; theory for dilinoleno-linolin bromide=59·4 per cent).

The film was crystallised from tetralin and acetone and gave a small amount of product which, according to the mixed melting-point test, was identical with the original bromoglyceride. In a later experiment a few drops of the oil were brominated in ethereal solution, and the precipitate, after crystallisation, proved to be identical with the original compound. The filtrate from the bromination liquor left, on evaporation, a small amount of a bromine-containing oil which would not crystallise. This behaviour is comparable with that recorded by various investigators for hexabromo- and tetrabromostearic acids.

Hydrolysis of the Oil.—One grm. of oil was hydrolysed with 10 c.c. of 20 per cent. alcoholic potassium hydroxide solution. After two hours the acids were isolated in the usual manner. The mixed acids were dissolved in ether and brominated, and the resulting precipitate was found to be hexabromostearic acid. On evaporating the solvent from the filtrate there resulted a very viscous oil, which up to the present has resisted all attempts to crystallise it. It was purified by dissolving it several times in ether and cooling the solution, whereby further small amounts of hexabromostearic acid were removed. Finally, a liquid product resulted which contained 56 per cent. of bromine* (Stepanow's method), showing that probably it was mainly a liquid form of tetrabromostearic acid. It may be identical with that recently described by Santiago and West (*Phil. J. Sci.*, 1927, **32**, 41-52).

PART II.

GRAVIMETRIC DETERMINATION OF THE BROMINE VALUE.—During the attempts to remove bromine from dilinoleno-linolin bromide it became necessary to determine the unsaturation value of drops of oil far too small in amount to give results by the usual Wijs method. It therefore occurred to me to allow thin weighed films of oil to "dry" in an atmosphere of bromine. From the increase in weight the bromine value can be obtained, and this, by multiplying by the factor—

$$\frac{\text{At. Wt. of Iodine}}{\text{At. Wt. of Bromine}},$$

should give the ordinary iodine value. The apparent success of this method, as indicated in Part I, suggested the possibility of a simple gravimetric method for finding the unsaturated value of minute quantities of oils in general. It was, therefore, tested on a large number of oils, and it was found that not only do the values agree well with those obtained by the usual methods for the majority of unoxidised oils, but also that the appearance of the film is in many cases characteristic of the particular oil.

METHOD AND APPARATUS.—A single drop of oil (0·02 to 0·03 grm.) is spread in a thin film, about 0·2 mm. thick, on a weighed microscope slide, and placed in a wide tube closed at each end with a waxed cork, and containing a boat in which are placed a few drops of bromine. After 20 to 30 minutes the slide is taken out, and the excess of bromine is removed, either by heating to 50–60° C., or by a current of warm air. From the increase in weight of the slide the bromine value can be calculated. For the less unsaturated oils larger amounts are needed, and then it is advisable to use a larger plate, with a similar one for a counterpoise. The method is characterised by extreme simplicity, for it eliminates all errors due to graduated flasks, and burettes; furthermore, it reduces manipulation to a minimum, and hence is capable of great accuracy.

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* Theory for tetrabromostearic acid is 53·3 per cent.

The characteristics of the brominated films are indicated below:

OIL.		CHARACTER OF FILM.
Linseed	..	Hard, gritty film, wrinkled surface, not very glossy, colourless.
Linolenic acid	..	Similar to above.
Boiled linseed	..	Smooth brown glossy film.
Candle-nut	..	Inclined to wrinkle, but soft, sticky and glossy.
Rubber seed	..	Smooth, glossy, very sticky and viscous.
Perilla	..	Wrinkled, hard, no gloss, but tends to run into streaks and lakes rather than to remain even.
Poppy seed	..	Varnish-like, very soft and sticky.
Tung	..	Very high gloss, but soft and sticky, no tendency to wrinkle. Yellowish.
Cotton seed	..	Extremely soft and sticky.
Soya bean	..	Slightly glossy, but soft.
Menhaden	..	Opaque, coarsely wrinkled, dull surface.
Sea elephant	..	Shiny transparent film, soft and sticky.
Whale	..	Transparent film, covered with minute granules.
Castor	..	Transparent droplets.
Apricot kernel	..	Opaque droplets.

EXPERIMENTAL.

(A) DUPLICATES, SHOWING THE DEGREE OF ACCURACY OBTAINABLE.

(i) *Olive Oil :*

(*a*) Oil taken, 0·0344 grm.; bromine absorbed, 0·0185 grm. Bromine value = 53·8.

(*b*) Oil taken, 0·0418 grm.; bromine absorbed, 0·0224 grm. Bromine value = 53·6.

Calculated iodine value = 85·3
Value found by independent investigator = 85·4

(ii) *Menhaden Oil :*

(*a*) Oil = 0·0601 grm.; bromine absorbed, 0·0665* grm. Bromine value = 110·6.

(*b*) Oil taken, 0·0242 grm.; bromine absorbed, 0·0268. Bromine value = 110·6.

(iii) *Tea seed Oil :*

(*a*) Oil taken, 0·0276 grm.; bromine absorbed, 0·0157 grm. Bromine value = 56·9.

(*b*) Oil taken, 0·0170 grm.; bromine absorbed, 0·0096 grm. Bromine value = 56·5.

Calculated iodine value = 90·2.
Experimental ,, ,, = 90·0.

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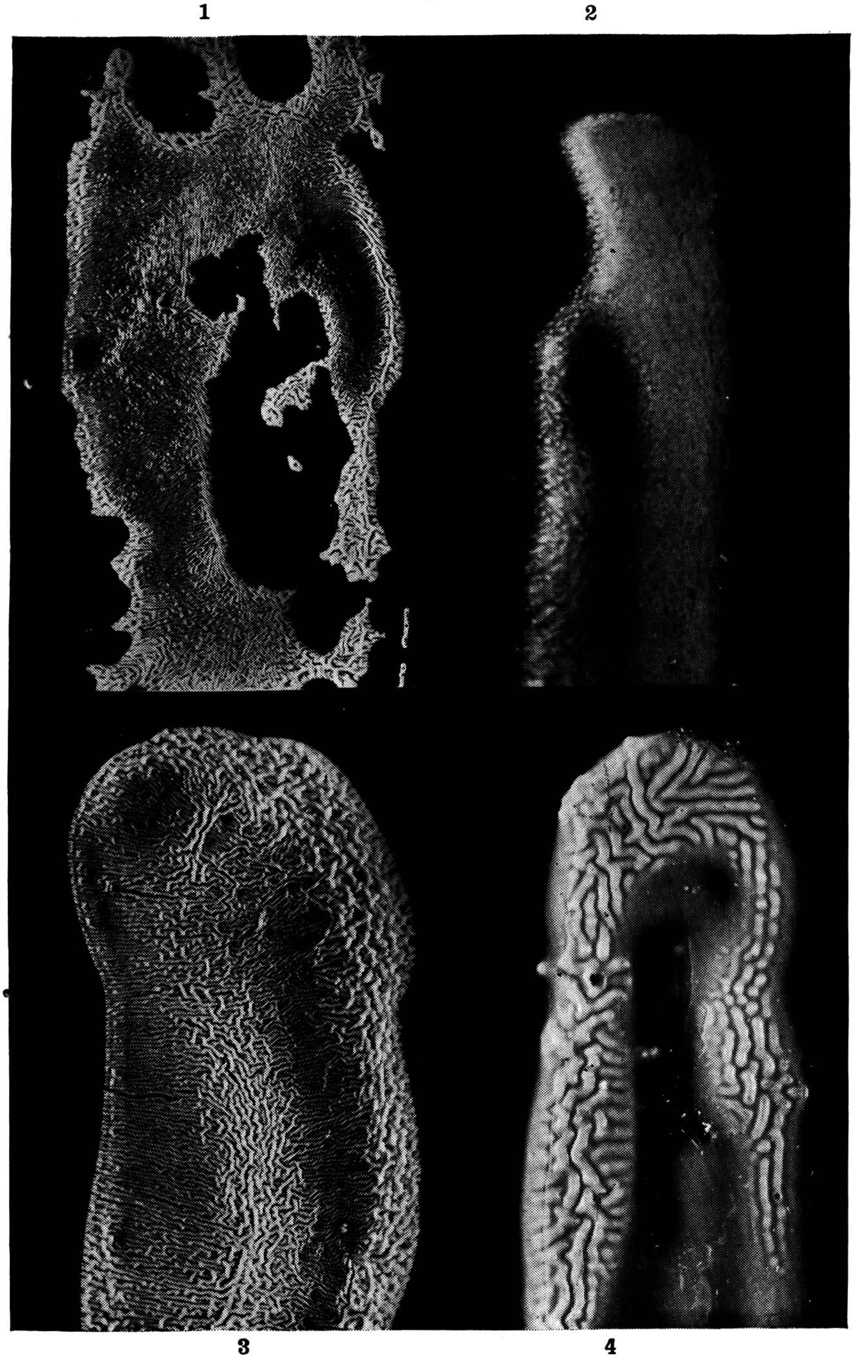
* The film showed no change in weight after 5 days.

OIL BROMIDE FILMS.



1. Perilla Oil. 2. Tung Oil. 3. Linseed Oil.
4. Menhaden Oil.

(*Magnified* $2\frac{1}{2}$ *diameters.*)

(B) RESULTS OBTAINED WITH VARIOUS OILS.

Drying Oils.

Oil.	Bromine value.	Calculated iodine value.	Observed iodine value (Wijs).
Linseed I (Calcutta)	115·2	182·9	182·6
Linseed II (Baltic)	126·2	201·0	202·5
Boiled linseed	76·6	121·6	115·2
Linolenic acid	133·9	212·2	210·0
Perilla	130·0	206·4	205·4
Rubber seed I	88·75	141·2	140·7
Rubber seed II	88·52	140·5	140·3
Candlenut	104·6	166·1	165·6
Tung	142·2	225·5	162·0
Soya and linseed mixture	108·5	172·3	172·6
Poppy seed	88·7	141·0	136·4
Soya bean	82·94	131·7	130·3
Sunflower seed	81·8	129·8	129·2

Semi-drying Oils.

Oil.	Bromine value.	Calculated iodine value.	Observed iodine value (Wijs).
Castor	62·6	99·7	105·2
Colza	63·4	100·6	106·8
Cotton seed	62·4	99·5	101·5
Maize	66·4	105·3	106·2

Non-Drying Oils.

Oil.	Bromine value.	Calculated iodine value.	Observed iodine value (Wijs).
Tea seed I	56·5 56·9	90·2 —	90·0 —
Tea seed II	50·8	80·8	81·0
Apricot kernel	65·5	104·0	101·3
Olive	53·6	85·3	83·9
Arachis I	61·8	98·4	99·7
Arachis II	60·2	95·5	95·8
Coconut (*a*)	5·5	8·7	7·2
(*b*)	4·79	7·62	7·2

Fish and Marine Animal Oils.

Oil.	Bromine value.	Calculated iodine value.	Observed iodine value (Wijs).
Menhaden	110·6	184·0	180·0
Sperm	57·2	90·8	90·5
Dugong	32·4	51·5	52·5
Sea elephant	67·4	107·0	107·3
Penguin	60·4	96·0	89·3
Seal	96·4	153·0	153·1
Sardine	103·8	164·8	121·0
Sea leopard	77·6	123·5	127·1
Weddell seal	82·6	131·2	137·8
Seal	91·8	146·0	150·2

It will be seen from these results that the iodine value calculated from the bromine absorption, as determined by this micro method, agrees closely with the iodine value determined by the Wijs method in the case of most unoxidised oils. Oils containing oxidised acids (*e.g.* boiled linseed oil) give, as was to be expected, a higher absorption with bromine than with the Wijs reagent. Some of the fish and marine animal oils were old specimens in which some decomposition of the glycerides had probably taken place. On the other hand, castor oil, containing ricinoleic acid, gives a lower halogen absorption with bromine than with iodine chloride. This may be a distinguishing characteristic between oleic acid and ricinoleic acid. The high result in the first determination with coconut oil is obviously due to experimental error; in the second determination a much larger amount (0·06 grm.) of the same fat was used on a plate, 6 inches by 2 inches.

THE SPECIAL CASE OF TUNG OIL.—On reference to the first table of results (*vide supra*) it will be seen that in the case of tung oil there is a difference of over 63 units between the calculated and observed iodine values. This difference, being far too great to ascribe to experimental error, rendered it desirable that the matter should be investigated in as many cases as possible.

The results of this investigation are summarised in the table below.

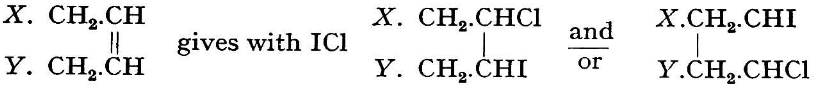
Tung Oil.

Sample.	Wijs value experimental.	Br value, experimental.	Wijs value, calculated.	Ratio of Calc. Wijs value to experimental Wijs value.
I	165·2	139·3	221·3	1·34
II	161·7	137·2	217·9	1·35
		136·6	217·0	1·34
III	169·6	146·3	232·4	1·37
IV	167·8	143·4	227·8	1·36
V	168·3	146·2	232·1	1·37
VI	154·9	128·3	203·8	1·32
VII	168·4	145·6	231·3	1·37

These results are probably due to the peculiar structure of the characteristic acid of tung oil. The unsaturated acids of the ordinary oils contain groupings of double bonds, such as

$CH_2.CH{=}CH.CH_2.CH{=}CH.CH_2$ and $CH_2.CH{=}CH.CH_2.CH{=}CH.CH_2CH{=}CH.CH_2$

i.e. non-conjugated systems, and hence each double bond acts independently of the others. It seems that both bromine vapour and iodine monochloride in solution act in the same way on such structures, that is, they give direct addition products which are completely saturated. In the case of iodine monochloride the product contains iodine and chlorine in amounts proportional to the atomic weights of these elements, thus—



$$\begin{array}{l} X.\ CH_2.CH \\ \qquad\quad \| \\ Y.\ CH_2.CH \end{array} \text{ gives with ICl } \begin{array}{l} X.\ CH_2.CHCl \\ \qquad\qquad | \\ Y.\ CH_2.CHI \end{array} \ \frac{\text{and}}{\text{or}}\ \begin{array}{l} X.CH_2.CHI \\ \qquad\quad | \\ Y.CH_2.CHCl \end{array}$$

α-Elaeostearic acid, which is present to the extent of 60 per cent., or more, in tung oil, has an entirely different structure, thus—

$$C_4H_9.CH{=}CH.CH{=}CH.CH{=}CH.(CH_2)_7.COOH.$$

Such a conjugated structure would normally react according to Thiele's theory—

(i) $—CH{=}CH.CH{=}CH.CH{=}CH— + X_2$
gives $—CHX.CH{=}CH.CH{=}CH.CHX—$

(ii) $—CHX.CH{=}CH.CH{=}CH.CHX— + X_2$
gives $—CHX.CHX.CH{=}CH.CHX.CHX.—$

(iii) $—CHX.CHX.CH{=}CH.CHX.CHX— + X_2$
gives $—CHX.CHX.CHX.CHX.CHX.CHX.—$

and hence the last two atoms to enter the system would experience a definite retardation. Thus the usual method of determining the iodine value by contact with Wijs solution, for two hours, may be insufficient for complete saturation. In the case of pure α-elaeostearic acid (Böeseken, *Rec. Trav. Chim. Pays-Bas*, 1927, **46**; ANALYST, 1928, 54) it was shown recently that the addition of iodine monochloride takes place in three stages; the first is complete in a few minutes, the second in half-an-hour, and the third in six days. Incidentally, it is a matter of common observation that tung oil is the only one which, during a Wijs determination, is characterised by the precipitation of iodine, and it is very probable that in this case the final product is a chloro-compound. If the results obtained on the free acid can be applied to the oil, then the ordinary iodine values correspond to a two-thirds saturation of the glyceride of α-elaeostearic acid. From the results recorded in this paper it appears that bromine vapour acts very differently from iodine monochloride in solution, or even bromine itself in solution, which appears to saturate only two of the three pairs of double bonds (*cf. Ber.*, 1925, **58**, 216; 1916, **59**, 1319), whereas bromine vapour appears to saturate all bonds simultaneously, regardless of whether the system is conjugated or otherwise. If this is so, then the more nearly the composition of any sample of tung oil approaches 100 per cent. of the triglyceride of α-elaeostearic acid, the more nearly will the ratio $\frac{\text{calculated iodine value}}{\text{experimental iodine value}}$ approach the limiting value of 1·5.

Attempts were made to make the vapour of iodine monochloride act on tung oil. At room temperatures a greenish film resulted, giving an increase in weight of 7 to 10 per cent.; at 100° C., a brown flaky film, which did not stick to the glass, resulted. The increase in weight for a single sample varied from 60 to 70 per cent. Obviously, iodine monochloride vapour is useless in this connection.

CALCULATION OF THE PERCENTAGE OF α-ELAEOSTEARIC TRIGLYCERIDE IN TUNG OIL.—In the following calculation it is assumed that tung oil contains two types of substances. Type A consists of those which contain non-conjugated systems of double bonds, and therefore gives the same iodine value regardless of the method used. Type B is the glyceride of α-elaeostearic acid, which gives (i) only two-thirds its normal or theoretical iodine value with Wijs's solution in two hours; but (ii) gives its full value by the bromine vapour method, and hence the full calculated iodine value. The molecular formula for α-elaeostearic triglyceride is $C_{57}H_{92}O_6$, and its molecular weight is 872. The theoretical iodine value after two hours, corresponding to the addition of 12 atoms of iodine, is 174·8 per cent. Let this equal n per cent. Also, the complete iodine value corresponds to the addition of 18 atoms of iodine, and is 262·5 per cent. Let this be m per cent. Further, let the substances of type A have an iodine value of x per cent. under both sets of conditions.

Suppose, in any experiment under condition (i) the mixture (A+B) absorbs p per cent. of iodine, and under condition (ii) it absorbs q per cent. of iodine, then

we have
$$\frac{Ax}{100} + \frac{nB}{100} = \frac{p}{100}(A+B)$$

and
$$\frac{Ax}{100} + \frac{mB}{100} = \frac{q}{100}(A+B)$$

$$\frac{A}{B} = \frac{m-n-(q-p)}{q-p}$$

but
$$\begin{aligned} m-n &= 262{\cdot}5-174{\cdot}8 \\ &= 87{\cdot}7 \end{aligned}$$

$$\frac{A}{B} = \frac{87{\cdot}7-(q-p)}{q-p}$$

hence for any sample A/B can be found.

and percentage of $B = \frac{(q-p).100}{87{\cdot}7}$,

where q is the iodine value of the sample calculated from the bromine value, and p is the iodine value found by the usual Wijs method.

The amount of α-elaeostearic triglyceride, in each of the tung oils used, as calculated from the table on page 74, is:

	Per Cent.		Per Cent.
I	64·0	V	72·8
II	63·2	VI	55·8
III	71·7	VII	71·8
IV	68·5		

SUMMARY AND CONCLUSIONS.—1. The insoluble bromide obtainable from most of the drying oils has, with difficulty, been debrominated and the presence of hexabromostearic acid confirmed, whilst the presence of tetrabromostearic acid has been rendered more probable by the isolation of a thick oil which appears to be a liquid form of this acid.

2. A rapid and accurate micro-method for determining the unsaturation value of non-volatile oils in general has been devised and tested in a large number of cases. The method involves the preparation of brominated oil films, and these are found to be largely specific in appearance. The values for most unoxidised oils obtained agree well with the iodine values as usually determined.

3. The behaviour of tung oil has been found to be unique, and a method of determining the active constituent of this oil has been devised.

The author wishes to acknowledge his indebtedness to the Director of the Imperial Institute for gifts of oil; also to Mr. C. Ainsworth Mitchell and to Mr. A. Chaston Chapman for gifts of oil and for checking some of the results here recorded. He would also like to thank Mr. T. J. Ward for making photomicrographs, slides and prints of some of the oil bromide films.

BIRKBECK COLLEGE,
UNIVERSITY OF LONDON.

DISCUSSION.

The PRESIDENT congratulated Mr. Toms on his further contribution to the chemistry of the bromine compounds of fatty oils. In the past he had often experienced difficulty in getting an iodine value for tung oil which agreed with that found by other chemists. The explanation of this difficulty was to be found in the work of Mr. Toms, which would thus have a direct practical value.

Mr. C. A. MITCHELL recalled the fact that Hehner, many years ago, had brought before the Society a gravimetric bromine method (ANALYST, 1895, **20**, 49). This had given fairly good results under strictly empirical conditions, but it had the drawback that the bromine and oil were mixed in a solution, and that the heat required for complete removal of the solvent caused the bromine to combine by substitution, as well as addition, and thus the weight of the oil bromide never became quite constant. In Mr. Toms's micro-method constant weight was obtained without difficulty. The principle of the method might be tried with other reagents in the form of a vapour, such as chlorine, osmium tetroxide, and especially sulphur trioxide. The method might also give useful results in the examination of heavy lubricating oils and vaseline.

Mr. M. S. SALAMON said that he, too, had had difficulties with regard to the iodine value of tung oil, and he welcomed the new method, since it promised to put an end to disputes. He suggested the application of the method to the examination of resins and especially of shellac.

Mr. E. HINKS asked for information as to the thickness of the films.

Mr. TOMS, replying, said that he would try the various reagents that had been suggested on fatty oils and eventually on resins. It was essential that the film should be as thin as possible; otherwise a skin formed on the surface and protected the oil beneath from the action of the bromine. His practice was to spread a single drop of the oil with the finger so as to cover about two-thirds of the area of the slide.

ADDENDUM.—Since this paper was in print my attention has been drawn to a paper by Sabalitschka and Dietrich (*Pharm. Ztg.*, 1924, **69**, 425), in which a method based on the same principle (absorption of bromine vapour) is described. As quantities of 0·1 to 0·4 grm. of the oil are used on plates, 14 cm. by 5 cm., and from 2 to 3 hours' heating are required to expel the excess of bromine, it is not surprising that these authors have found that some substitution occurs, and that their results are uniformly higher than those obtained by the Hübl method, even in the case of solid fats and non-drying oils. As is mentioned above, the oil film must be sufficiently thin to prevent the formation of a pellicle on the surface, which impedes the removal of the excess of bromine; this is not readily practicable with quantities of 0·1 grm. and upwards.—H. TOMS.

The Composition of Human Milk.

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

MANY observers have previously recorded the composition of human milk. Their results have been ably summarised by Richmond (*Dairy Chemistry*, 3rd Edition, 1920, pp. 316–322), and by J. A. Gardner and F. W. Fox (*The Practitioner*, 1925, **114**, 153–168), which publications may be consulted for further details than are given in this paper. A few recent analyses are given by Miss May Deacon (*National Health*, July, 1926, p. 11), but the number of samples involved is too small to afford much information, particularly as the conclusions which she draws differ somewhat widely from those usually accepted and substantiated by the present series of figures.

The present series commenced a few years ago, as the result of some milks submitted for analysis by Dr. Hilda K. Brade-Birks, the Senior Maternity and Child-Welfare Medical Officer for the City of Salford. Dr. Brade-Birks has certain facilities for obtaining samples of this nature, and she willingly acceded to the suggestion of the writer that a more extended series would be desirable. The indebtedness of the writer to Dr. Brade-Birks and her staff will be sufficiently obvious from the contents of this paper; the results cannot fail to have added interest and weight from the fact that they are based on samples taken under skilled supervision. Dr. Brade-Birks will deal elsewhere with the physiological and nutritional questions involved, the chemical results being given in this paper.

In all, 529 samples have been examined as completely as the quantity available would allow. The samples have been obtained from eighty-six different subjects, the number of samples from each subject varying from one to thirty-four.* Table I contains in tabular form the results obtained, each horizontal line dealing with a separate subject.

TABLE I. (ALL SAMPLES).

Number of Samples.	Total solids, per cent.			Solids-not-fat, per cent.			Fat, per cent.		
	Aver.	Max.	Min.	Aver.	Max.	Min.	Aver.	Max.	Min.
7	12·0	13·1	10·7	9·0	9·9	8·5	3·0	4·2	2·0
7	12·2	14·3	10·1	8·8	9·7	8·1	3·4	4·6	2·0
6	11·1	12·8	9·3	8·7	9·5	7·3	2·4	4·0	1·2
5	11·1	11·9	10·7	8·8	9·4	8·0	2·3	3·3	1·3
6	12·6	13·9	11·0	8·7	9·5	8·0	3·9	5·9	2·1
12	12·5	15·6	9·0	8·7	9·6	7·2	3·8	6·0	1·7
7	12·5	15·3	10·7	9·1	11·1	7·3	3·4	5·2	2·2
11	12·8	16·3	10·3	8·9	9·3	6·5	3·9	6·5	1·8
5	12·8	13·4	11·2	9·3	10·6	8·6	3·5	5·4	1·2
10	12·0	16·0	10·0	9·1	10·3	8·4	2·9	7·2	1·2
9	11·3	13·1	10·2	8·8	10·0	7·9	2·5	4·2	1·0
13	11·6	14·6	9·4	8·0	9·1	7·0	3·6	5·8	0·4

* The period of lactation varying from two days to nine months.

Number of	Total solids, per cent.			Solids-not-fat, per cent.			Fat, per cent.		
Samples	Aver.	Max.	Min.	Aver.	Max.	Min.	Aver.	Max.	Min.
4	11·7	12·4	10·9	9·1	9·6	8·7	2·6	2·8	2·0
5	13·4	16·6	9·7	10·5	14·4	7·9	2·9	4·8	1·8
6	10·7	12·0	10·0	8·5	9·2	7·6	2·2	3·7	1·3
2	11·4	12·2	10·7	8·5	8·8	8·2	2·9	3·4	2·5
4	12·6	13·6	11·4	8·8	9·5	8·2	3·8	5·0	3·0
6	12·6	13·2	11·8	9·6	9·8	9·4	3·0	3·7	2·3
4	12·5	13·7	11·4	8·7	9·7	8·3	3·8	4·4	2·8
2	11·7	12·4	11·1	9·1	9·5	8·8	2·6	2·9	2·3
1	13·3	13·3	13·3	9·1	9·1	9·1	4·2	4·2	4·2
3	11·0	11·8	9·6	8·6	8·8	8·3	2·4	3·1	1·3
1	10·1	10·1	10·1	8·7	8·7	8·7	1·4	1·4	1·4
6	11·1	11·9	10·5	8·7	9·3	7·5	2·4	3·3	1·6
5	10·9	13·1	9·1	8·2	9·4	5·2	2·7	3·9	1·5
12	12·0	13·8	10·3	8·5	10·9	6·8	3·5	5·6	1·8
6	11·7	13·7	10·9	9·0	9·4	8·6	2·7	4·3	1·7
4	11·5	11·9	11·0	8·6	9·4	7·0	2·9	4·4	1·6
9	12·5	13·9	10·7	9·1	9·9	8·0	3·4	5·1	2·1
4	11·3	12·5	10·4	9·1	9·5	8·8	2·2	3·2	1·2
2	13·5	14·2	12·8	8·3	9·0	7·6	5·2	5·2	5·2
1	10·2	10·2	10·2	9·0	9·0	9·0	1·2	1·2	1·2
1	10·2	10·2	10·2	9·2	9·2	9·2	1·0	1·0	1·0
4	12·1	13·0	11·3	9·2	9·4	8·9	2·9	3·6	2·4
4	12·5	13·8	10·6	8·9	9·4	8·7	3·6	5·0	1·2
1	11·0	11·0	11·0	9·9	9·9	9·9	1·1	1·1	1·1
5	11·6	12·3	11·2	9·6	10·3	9·1	2·0	2·4	1·2
1	12·5	12·5	12·5	9·3	9·3	9·3	3·2	3·2	3·2
9	12·5	16·1	5·7	7·9	10·0	3·8	4·6	7·6	1·9
6	12·6	15·5	11·1	8·9	9·1	8·8	3·7	6·7	2·3
10	10·7	13·0	8·7	7·8	8·8	6·6	2·9	3·9	1·6
4	13·7	16·1	11·7	8·9	9·4	8·5	4·8	7·6	3·2
4	12·2	12·9	11·4	9·1	9·8	8·5	3·1	4·4	1·6
5	11·3	13·1	10·3	8·7	9·2	7·2	2·6	4·0	1·2
6	11·0	12·0	10·1	8·9	9·8	8·3	2·1	2·8	1·2
8	11·8	13·9	10·4	8·7	9·4	7·9	3·1	5·6	1·5
6	12·2	13·9	10·6	8·9	9·7	8·1	3·3	4·8	2·0
7	11·2	12·3	10·1	9·0	9·9	7·9	2·2	3·6	0·8
4	11·1	14·2	9·2	8·0	9·2	7·0	3·1	5·0	2·2
3	12·9	13·2	12·6	9·2	11·0	7·9	3·7	5·2	1·6
5	12·8	14·8	11·3	8·9	9·6	8·2	3·9	6·2	2·0
11	11·1	14·6	10·0	8·6	9·1	8·1	2·5	5·9	1·1
3	10·9	12·6	9·6	7·9	8·6	7·2	3·0	4·0	2·8
10	11·7	13·5	9·0	8·7	11·4	6·6	3·0	4·1	0·9
3	10·5	10·8	10·4	9·1	10·0	8·3	1·4	2·2	0·4
11	12·0	12·8	10·7	9·1	9·5	8·4	2·9	4·0	2·3
3	11·5	12·1	10·6	9·0	9·7	8·2	2·5	3·0	2·0
1	13·3	13·3	13·3	8·9	8·9	8·9	4·4	4·4	4·4
6	12·8	15·2	11·9	9·1	9·8	8·7	3·7	6·4	3·0
1	12·5	12·5	12·5	8·3	8·3	8·3	4·2	4·2	4·2
12	12·5	14·5	9·9	8·9	9·6	8·4	3·6	6·1	1·3
3	11·4	12·6	9·5	8·6	9·8	7·1	2·8	3·5	2·4
4	14·6	15·7	14·0	9·5	10·2	9·1	5·1	6·4	4·4
6	11·3	12·5	8·8	8·1	9·0	7·4	3·2	4·8	1·4

Number of Samples.	Total solids, per cent.			Solids-not-fat, per cent.			Fat, per cent.		
	Aver.	Max.	Min.	Aver.	Max.	Min.	Aver.	Max.	Min.
3	11·9	14·0	10·2	8·8	9·2	8·2	3·1	4·8	2·0
8	13·0	15·7	11·1	8·7	9·4	7·3	4·3	7·0	2·0
6	13·1	13·8	11·9	9·4	11·7	8·8	3·7	4·7	1·9
4	12·1	17·0	9·8	8·6	9·6	7·4	3·5	8·0	1·6
6	12·8	14·5	11·3	8·6	9·1	7·9	4·2	6·5	2·0
7	12·6	14·4	11·4	9·4	11·0	8·2	3·2	4·9	1·6
4	10·1	11·0	8·6	8·0	8·6	7·0	2·1	2·4	1·6
6	11·3	11·9	10·5	8·9	10·3	8·4	2·4	3·5	1·6
5	10·9	11·5	9·7	9·2	10·1	8·5	1·7	2·4	1·0
9	12·4	15·9	10·1	8·6	9·2	6·7	3·8	7·0	1·2
5	11·6	12·6	10·3	9·1	9·9	8·6	2·5	4·0	1·2
34	13·1	21·6	10·7	9·0	17·3	6·5	4·1	10·2	1·1
15	11·7	12·9	7·2	8·2	9·2	7·2	3·5	4·7	2·5
10	13·1	14·8	11·2	9·3	9·8	8·8	3·8	5·9	2·2
8	11·0	12·4	9·8	8·4	9·2	6·3	2·6	3·5	2·1
7	12·0	13·1	10·9	8·6	9·5	7·4	3·4	4·8	2·5
6	11·8	12·5	10·3	8·9	9·6	8·2	2·9	3·4	1·6
8	11·5	12·7	10·6	8·7	9·3	7·4	2·8	3·8	2·2
3	10·5	11·0	9·9	8·6	9·5	7·6	1·9	3·4	1·1
11	13·5	15·3	11·4	8·2	9·2	5·4	5·3	7·0	2·5
8	12·4	14·3	10·4	9·1	11·0	7·9	3·3	6·4	1·4
4	10·9	12·4	9·4	8·2	10·8	6·6	2·7	3·6	1·2
				Average of all samples.					
529				8.80			3·27		

A number of the samples are known to have been obtained from the left breast and a number from the right. Table II contains the results obtained, which are, as might be expected, practically identical.

TABLE II.

No. of samples.		Solids-not-fat, per cent.			Fat, per cent.		
		Aver.	Max.	Min.	Aver.	Max.	Min.
Left breast	244	8·73	11·7	3·8	3·29	10·2	0·4
Right breast	217	8·78	11·4	5·2	3·27	8·0	0·8

The samples have been further divided (in those cases where the information was available) into those taken within one hour of nursing and those when the period exceeded one hour. Table III contains the results obtained:

TABLE III.

	No. of samples.	Solids-not-fat, per cent.			Fat, per cent.		
		Aver.	Max.	Min.	Aver.	Max.	Min.
Below 1 hour	113	8·76	11·7	5·2	3·27	7·2	1·0
Above 1 hour	320	8·49	14·4	5·4	3·22	8·0	0·4

A greater variation than that found in the results for fat might have been expected. It is possible, however, that there is not quite the same freedom of choice in Table III as in Table II.*

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* That is to say, the two series may have been taken, respectively, always from the same group of subjects.

The average composition, as deduced from the present series, is compared in Table IV with those from a previous series, and also with those given by Richmond and by Gardner and Fox.

TABLE IV.

	Solids-not-fat. Per Cent.	Proteins. Per Cent.	Fat. Per Cent.	Ash. Per Cent.
Elsdon, 1927	8·8	1·8	3·3	0·29
Elsdon, 1916	8·5	1·2	3·2	0·21
Richmond	8·3	1·3	3·3	0·2
Gardner and Fox	8·7	1·5	3·7	0·2

Taking into account the fact that the methods of sampling in the present series would tend to give low results for fat, there is a good agreement with Gardner and Fox. The higher ash and proteins figures than those given by Gardner and Fox are due to the somewhat large proportion of samples early in lactation in the present series.

In general, the quantities of milk were not sufficient for a more complete analysis to be carried out, but this was done in a few cases. The figures obtained are summarised in the following table.

TABLE V.

	Max.	Aver.	Min.
Protein ($N \times 6{\cdot}38$), per cent. ..	3·25	1·81	0·51
Ash, per cent.	0·52	0·29	0·08

The variation in the composition of human milk with the period of lactation has been considered by several workers. The consensus of opinion is that the percentage of fat increases as the lactation advances, whilst at the same time there is a diminution of the protein and ash. These conclusions have been confirmed by the present series in which the following average results have been obtained:

TABLE VI.

	Proteins. Per Cent.	Ash. Per Cent.
1 day	2·20	0·37
2 days	2·04	0·35
3 ,, ...	2·11	0·35
4 ,, ...	1·98	0·29
5 ,, ...	2·08	0·31
6 ,, ...	1·91	0·33
7 ,, ...	1·76	0·28
8 ,, ...	2·25	0·24
9–12 days	1·77	0·27
13–20 ,, ...	1·60	0·28
21–50 ,, ...	1·26	0·27
50 days to 9 months	1·23	0·20
Average	1·81 for 85 samples.	0·29 for 117 samples.

In the following table results for fat and solids-not-fat are given:

TABLE VII.

Period of lactation.	No. of samples.	Fat. Per Cent.	Solids-not-fat. Per Cent.
Up to 7 days	223	2·86	9·01
8 to 28 days	280	3·58	8·58
1 to 9 months	26	3·44	8·48

It will be obvious from the results obtained by various workers that the composition of human milk depends so much upon the stage of lactation that average figures can be of little practical value. From the results of various workers the following may be taken as average figures for the various stages.

TABLE VIII.

Period of lactation.	Fat. Per Cent.	Proteins. Per Cent.	Lactose. Per Cent.	Ash. Per Cent.
1 to 7 days	3·0	2·1	6·8	0·30
7 to 28 days	3·5	1·6	6·8	0·25
1 to 9 months	3·5	1·3	6·9	0·22

The mixed fat from a considerable number of samples taken very early in lactation has been examined. The iodine value was 35·9, the Reichert (Reichert-Meissl-Wollny) 3·4, Polenske value 1·9, and Kirschner value 2·0. The Reichert value is somewhat higher than those previously recorded; this might possibly be due to the fat having become rancid on keeping, but the Kirschner figure does not substantiate this.

It is with great pleasure that I acknowledge the assistance which I have received from my former assistants, Messrs. D. Foster, P. Smith and H. Holden, who have carried out the bulk of the analytical work on which this paper is based.

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LIVERPOOL.

Arsenic in Coated Papers and Boards.

By HAROLD J. STERN, B.Sc., Ph.D., A.I.C.

(*Read at the Meeting, December* 7, 1927.)

BARRY (ANALYST, 1927, **52**, 217) has considered the question of arsenic in printing ink. The presence of arsenic in coated papers is of more importance, for several reasons. With the case of a printed box or label both the area covered by the printing and the actual weight of material printed on are comparatively small, and, of this small weight, only a portion consists of actual pigment which may contain arsenic. In the case of a coated board or paper the whole surface is covered, and the quantity of pigment present is many times greater than in printing. Figures may be given to illustrate this point. Barry, in his paper, gives 0·5 grm. per square metre as an approximate maximum for the weight of printing ink employed, but there is no doubt that this is too low, and experiments with actual foodstuff packages show that the figure should be nearer 5 grms.; in one case the weight of ink used was 4·173 grms.*

On the other hand, the coating on an ordinary coated board or paper weighs, on the average, from 25 to 30 grms. per square m., and 50 grms. is not an uncommon figure. Whereas the process of printing is well known, it should be explained that in paper-coating a pigment, bound by glue or some other aqueous medium, is applied to the whole surface of the paper by special machinery. The arsenic here is important, because of the comparative ease with which such coatings may be removed, especially when wet, and therefore Barry's remark about moist confectionery wrappers coming into the hands of children has far greater force when the case of coated wrappers is to be considered. It is probable that earlier workers on this subject have not clearly distinguished between coated and printed papers, and that cases reported as due to arsenic in printing ink were, in fact, due to arsenical coatings.

In the production of coated papers with a safe arsenic content, the constituents involved must be considered separately. It has already been explained that the coating is carried out, generally, in an aqueous medium consisting of pigment and adhesive. We have, therefore, to consider: (1) the raw paper or board, (2) the adhesive, and (3) the pigment.

The raw paper or board is of interest, quite apart from the coating question, as it is often employed in the uncoated state. In the course of the examination of paper and boards suitable for wrapping foodstuffs, extending over several years, it is only on rare occasions that I have noted any appreciable quantities of arsenic. Sulphite materials have been found to contain up to 10 parts per million, but it may be said, in general, that there is little likelihood of a paper or board, in its uncoated state, containing any dangerous amount of arsenic.

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* These figures refer to cardboard containers. Barry (private communication) worked with light wrapping papers.

The same may be said of the adhesives employed in the coating process. These are mainly natural products, and the possibility of contamination with arsenic is remote. Glue or gelatin, and casein are the adhesives chiefly employed, but starch, dextrin and certain gums also find a place. The case of dextrin has received attention in the past, and does not call for further comment, beyond the fact that the commercial samples examined by me have always been satisfactory, so far as arsenic content was concerned. Gelatin has been reported to contain arsenic (O. Köpke, *Chem. Centrallblatt,* 1912, p. 281), and, according to Bogue, (*The Chemistry and Technology of Gelatin and Glue*) it may be found in glue, but here, again, the highest arsenic content that I found in any of a number of samples examined was about 5 parts per million.

PIGMENTS FOR COATING.—Turning to the pigment, there is a different set of conditions. It is outside the scope of this paper to review the pigments employed, but it may be said that any and every type of pigment may be found in the coating on a modern coated paper or board. Some of the classes may, however, be mentioned—mineral pigments, such as china clay and the ochres, synthetic inorganic pigments (for instance, barium sulphate), lead chromate, Prussian blue, the lakes of natural dyestuffs (such as logwood), and, finally, with the possible exception of one or two small groups, the lakes of every type of synthetic dyestuff, and all types of synthetic organic pigments. From the point of view of arsenic content these must be considered more closely.

Mineral pigments are generally so inert and insoluble as to cause little danger. The mineral azurite (basic copper carbonate) is the only poisonous mineral pigment which might be used, but the possibility is very remote, and there is no question of arsenic. There is, however, a class of pigment which may be conveniently mentioned here; it is sold under a variety of trade names, and consists of almost pure antimony oxide. This antimony oxide pigment may be used as part of the base for colour lakes, and may therefore be found in coloured, as well as in white coatings. Its presence in the coating on foodstuff packages is of course undesirable.

SYNTHETIC INORGANIC PIGMENTS.—In the class of synthetic inorganic pigments are included the so-called arsenical greens. The general method of preparation is to dissolve arsenious oxide in sodium carbonate solution and to precipitate the pigment by means of copper sulphate. The composition varies according to the details of the process employed by different manufacturers. These greens are sold under a great variety of names; Scheele's green is, perhaps, the best known description. A sample of a green of this type, sold under the name of emerald green, was found to contain over 30 per cent. of arsenious oxide. If acetic acid is added during manufacture, Schweinfurt's green is obtained. In view of the high arsenic content and the weight of coating involved, it will be seen that these pigments constitute a danger. As an example, consider a paper coated with the emerald green just mentioned. Taking a coating of 30 grms. per square m., there are approximately 20 grms. of pigment (the remainder consisting of the dried adhesive, etc.), so that the paper in this case would have over 6 grms. of arsenious oxide per square metre.

By way of contrast, take a paper printed with an ink, and suppose that the pigment consists of this same emerald green. As mentioned earlier, the heaviest printing likely to be found would weigh 5 grms. per square m. Of this weight, 3 grms. may be reckoned as actual pigment—the proportion of pigment varies with its density, but 60 per cent. is a fair average, based on actual works' figures. Thus the printed paper has only 0·9 grm. of arsenious oxide per square m., as compared with 6 grms. in the case of a coated paper.

The use of these pigments in paper coating was at one time considerable, and even now there is always the danger of their application for foodstuff wrappers by people ignorant of their nature. Fortunately the arsenical pigments are deficient in covering power, but they still find use for tinting, *i.e.* for mixing with a white pigment in the production of pale green and blue shades, especially as the fastness to light is good.

LAKES.—There appears to be no danger from the lakes of natural dyewoods. The lakes of synthetic dyestuffs are of greater interest, in view of the large number of types employed, and of considerations other than the presence of arsenic which are involved, such as brilliance and fastness. With the exceptions to be mentioned, synthetic dyestuffs and pigments are generally free from arsenic. There is, however, a process for making lakes from basic dyestuffs in which arsenious oxide (dissolved in alkali solution) is used as a precipitant. The dyestuffs usually precipitated in this way are Magenta and Methyl Violet, or mixtures of these, and a lake of great brilliance, but of no great permanence, is produced. Bright violet or magenta coloured papers may, therefore, be suspected; a case reported by Elsdon (ANALYST, **1924, 49,** 33) is, no doubt, due to the use of a pigment of this type. The pigments are sold for paper-coating purposes in the form of a paste; calculating the results on the solid matter present, a typical specimen of a magenta pigment was found to contain 40 per cent. of arsenious oxide, and a violet pigment 57 per cent. The resulting papers or boards may therefore have 10 or 12 grms. of arsenious oxide per square m., and, in fact, in these cases the arsenic content is measurable, not as parts per million, but as a percentage.

Another method of producing a pigment from these basic dyestuffs involves the use of tartar emetic and results in an undesirably large content of antimony in the finished lake. These lakes are a little faster to light than those produced with arsenic, but are not quite so brilliant. In both cases far more satisfactory results may be obtained by the use of other dyestuffs which do not require arsenic or antimony in their application, although the cost of the lake is a little higher.

It has been said that, in general, the dyestuffs themselves are free from arsenic, but there is always the possibility of contamination during manufacturing processes. Thus a lake may be made in a vessel which has previously been used for an arsenical colour, and the sudden appearance of arsenic at any stage in the making of coated papers may usually be traced to an analogous source.

Certain dyes and pigments seem particularly liable to contain arsenic, though not usually in dangerous amounts. Pigment Scarlet 3 B is one of the worst offenders, as is also Orange II, and it is quite common to find in these dyes 50 to 100 parts per million, or even more. In spite of statements to the contrary—which no doubt originate from interested manufacturers—no superior fastness to light has been observed in such dyes over samples of the same dyestuff which contained only one or two parts per million of arsenic. Pigment Fast Red PRL and Fast Orange RL are also liable to contain arsenic, as are also colours of the type of Patent Blue A (Disulphine Blue A).

LIMITS FOR ARSENIC.—With regard to the safe limits for arsenic in coated boards and papers, for some years a provisional arrangement has been in force with certain important users, by which the arsenic content is limited to 10 parts per million. This could, no doubt, be greatly exceeded without the slightest danger, but generally there is no difficulty in keeping below the limit, and the boards and papers now in use seldom contain more than 2 or 3 parts per million. This method of defining the limit allows more arsenic to be present in a given area of a light paper than in a heavy board, and in this respect a definition based on unit area would be more accurate. The limit set is, however, so low that the question is not of great importance.

Finally, a word may be said on the method of determining the arsenic. This is really a matter for a separate paper which I hope to publish, but it may be mentioned that in most cases digestion with hydrochloric acid, followed where necessary by distillation and determination of the arsenic in the distillate or pulp by the Gutzeit method, has been found to give results which are sufficiently accurate for the work in question, and which are quickly obtained—a matter of great importance in the average works' laboratory where the determinations are made.

I have to thank Mr. Leonard Wild, B.Sc.Techn., for information concerning printing processes, and Mr. A. L. Sharpe, Managing Director of Backhouse & Coppock, Ltd., for permission to publish this paper.

Adulteration Statistics.

BY J. F. LIVERSEEGE, F.I.C.

THE Annual Report of the Ministry of Health each year gives a table showing the percentage of adulteration in each district in England and Wales. If the uncorrected figures are used misleading comparisons may be made.

The table divides the samples into five groups. In 1925 the percentage of adulteration of each group was as follows:—Milk 8·3, butter 1·5, spirits 13·8, drugs 5·4, and other articles 4·7. It is obvious that the total percentage of adulteration will depend not only on the composition of each group, but also on the relative number of samples of each group taken.

With a constant amount of adulteration in a district, the apparent percentage of adulteration in it might be varied considerably by administration. For instance, if the number of samples of milk (8·3 per cent. of adulteration) were halved and a corresponding increase were made in the number of samples of butter (1·5 per cent. of adulteration), the apparent percentage of adulteration would fall considerably, though the real amount of adulteration might be unaltered.

The table of the Ministry of Health shows that the practice of different districts varies considerably. In Town A, 32·2 per cent. of the samples taken were milk, whilst in Town B, the proportion was 61·4, and in England and Wales, as a whole, 52·1 per cent. of the samples were milk. A correct comparison can only be made on a common basis, a convenient one being that of the proportion of England and Wales as a whole, which does not vary much from year to year.

The revised figure is obtained by calculating, for a particular district, how many samples of milk out of 52 are adulterated, and adding to it the number of samples of butter adulterated out of 9, the number of spirits adulterated out of 2, the number of drugs adulterated out of 5, and the number of other articles adulterated out of 32.

The following table will illustrate the method:

	Milk.	Butter.	Spirits.	Drugs.	Other Articles.	Total.
England and Wales, 1925.						
No. of samples	61,909	11,201	2,140	5,175	38,505	118,930
Percentage of total samples	52·1	9·4	1·8	4·4	32·3	100
No. adulterated	5,163	168	296	277	1,810	7,714
Percentage of adulteration	8·3	1·5	13·8	5·4	4·7	6·5
Standard sampling.						
No. of samples	52	9	2	5	32	100
No. of them adulterated	4·32	0·13	0·28	0·27	1·50	6·5
Town A.						
No. of samples	668	94	96	344	873	2,075
Percentage of total samples	32·2	4·5	4·6	16·6	42·1	100·0
No. adulterated	144	0	4	12	18	178
Percentage of adulteration	20·9	0	4·2	3·5	2·1	8·6
Standard sampling.						
No. of samples	52	9	2	5	32	100
No. of them adulterated	11·21	0	0·08	0·17	0·66	12·12
Town B.						
No. of samples	330	13	19	67	109	538
Percentage of total samples	61·4	2·4	3·5	12·4	20·3	100
No. adulterated	14	0	3	8	0	25
Percentage	4·2	0	15·8	11·9	0	4·6
Standard sampling.						
No. of samples	52	9	2	5	32	100
No. of them adulterated	2·21	0	0·32	0·60	0	3·13

As the percentages of each group of samples analysed in England and Wales is taken as "Standard Sampling," the same figure (6·5 per cent. of adulteration) is obtained by either way of calculation.

For correct comparison with England and Wales, however, the percentage of adulteration of Town A is not 8·6, as stated, but the figure on standard sampling is *increased* to 12·12. If the same method of calculation is applied to Town B, the percentage of adulteration is *decreased* from 4·6 to 3·13. According to the stated percentage of adulteration the relative figures for the two towns are 8·6 per cent. and 4·6 per cent., whilst the actual relations with standard sampling are 12·12 per cent. and 3·13 per cent. These are extreme cases, but the illustration shows that the uncorrected percentages of adulteration are unreliable for comparison.

Another illustration may be found in the Scottish figures for adulteration. If the stated 8·1 per cent. of adulteration of formal samples is calculated on the basis of sampling in England and Wales, as shown above, the figure is reduced to 7·0 per cent.

There is a fashion in adulteration, and sampling ought to follow it. The discovery of cotton seed oil in lard and of olive stones in pepper caused a large increase in the number of these samples taken, with a consequent alteration in the proportion of the number of different groups of samples. All alterations of this kind may make comparison of different *years* in the same district unreliable.

Some time ago Birmingham had 13·3 per cent. of adulteration, and for the next year the figure was 9·4 per cent., showing an apparent *decrease* of adulteration of 3·9 per cent. The two years, however, are not comparable, as there was a considerable difference in the group distribution of the samples taken. When the figures are corrected to standard sampling, as shown above, they are 10·5 per cent. and 10·9 per cent., reversing the relations of the years, and making an *increase* of adulteration of 0·4 per cent.

Series sampling of milk may introduce inequalities in the comparison of various towns. When all samples were taken formally only one or two adulterated samples could be taken from a vendor, but when a series of informal samples are taken at a railway station, as many as 23 adulterated samples have been taken from one farmer, with a corresponding apparent increase in milk adulteration, which is entirely due to administration.

In a recent quarter, 27 out of 604 Birmingham samples (4·5 per cent.) of milk from 12 different vendors were adulterated, and 12 of the adulterated ones were from one farmer. If, after obtaining the first two adulterated samples from him, the Inspector had stopped sampling his milk, for that quarter 580 samples would have been taken, with 17 adulterated, (2·9 per cent.). The Inspector, by series sampling, had increased the apparent percentage of adulteration by 1·6 per cent. Similarly, comparison of different groups of milk samples by the percentage of adulteration may be fallacious. It is rare to have more than one adulterated sample from a milk shop and quite usual to have two or more adulterated samples from a farmer at a railway station.

The most satisfactory comparison would be on the percentage of vendors selling one or more adulterated samples, but as this is impracticable, a good substitute is found in the number of vendors selling adulterated milk per 100 samples bought. This has been used in Birmingham for a number of years under the term, "comparative adulteration figure," which in the above example would be 2·0. At present such a comparison can only be used locally, but if the Ministry of Health obtained and published the number of vendors of adulterated samples, better comparisons of different localities could be made.

It might be worth while pointing out that milk stands on a different footing from other articles with regard to adulteration.

The adulterated samples of the tables are mostly those samples which are below the Board of Agriculture limit for fat or solids-not-fat. The test of the genuineness of a milk is now: "Is the milk as it came from the cow?"—a test which can only be applied in the comparatively few cases where the inspectors visited the farm.

The adulterated samples of milk, therefore, will *include* a few poor samples which were "as they came from the cow," though below the official limit, but will *exclude* some adulterated samples where the adulteration was insufficient to bring the composition of the milk below a limit.

Occasionally the classification of a sample as genuine or adulterated requires consideration. Suppose "cream" was asked for, and cream free from preservative was sold, and the carton was marked, "preserved cream." The proper course appears to be to classify it as genuine "cream" with a false label. By Section 8 of the 1875 Act a mixed article must be judged by its label, but it is not probable that the converse will hold. The sample in question is *not* condemned by Section 6 as "Perserved Cream" which is "not of the nature of the article demanded." In other words, if the article asked for is supplied, an incorrect description on the label does not make it adulterated.

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.

CHESHIRE BUTTERS AND CHEESES OF LOW REICHERT-MEISSL VALUE.

During the month of November a number of samples of butter and cheese which had been made in the County of Cheshire were found with Reichert-Meissl values below 24, whilst at the same time there was nothing to indicate adulteration with foreign fat. Similar results have been found in previous years during the months of November and December.

Some of the latest samples gave Reichert-Meissl and Polenske values as follows:—

	Reichert-Meissl value.	Polenske value.
A. Butter	23·5	1·7
B. Butter	23·9	1·6
C. Butter	22·6	1·7
A. Cheese	23·4	2·0
B. Cheese	22·9	1·9

To obtain confirmation of these results, a gallon sample of milk was taken from the mixed milk of the cows from which butter C was produced. The milk was churned in the laboratory, and the resulting butter fat was found to have a Reichert-Meissl value of 22·1 and Polenske value of 1·6.

It was not possible to obtain fuller information as to the cows giving the milk, beyond the fact that it was the mixed milk of forty cows, many of which were said to be "going dry." Although it is now very generally known that, as the period of lactation draws to a close, the Reichert-Meissl value of the fat falls, and that genuine butters are found with Reichert values below 24, yet I have known of several cases where such butters have been condemned as adulterated. Whenever there is any doubt as to the genuineness of a butter or cheese it is advisable, if possible, to obtain a sample of the milk for a comparison of the figures given by the fat.

HAROLD LOWE.

ASSAY OFFICE, CHESTER.

APPARATUS FOR THE GUTZEIT TEST.*

MR. JOHN WHITE (ANALYST, 1927, **52**, 699) is mistaken in attributing the spring clip for the Gutzeit apparatus to the late J. M. Wilkie; it was devised by Mr. J. C. Hibbert (see *J. Soc. Chem. Ind.*, 1916, **35**, 672).

The arrangement of two corks, described by Mr. Stubbs (ANALYST, *loc. cit.*), is a modification of Harvey's glass apparatus (*Chemist and Druggist*, 1905, 168), in which the mercuric chloride paper was put between two glass tubes with ground edges.

The device, used by Mr. Cribb, of gumming a disc of mercuric chloride paper to the upper ground ends of two thick-walled glass tubes is essentially a simplification of the all-glass apparatus described by Mr. F. C. J. Bird (ANALYST, 1901, **26**, 181).

Although the three papers to which I have referred were published many years ago, they are still worth reading at the present time.*

H. DROOP RICHMOND.

USE OF STRIPS OF MERCURIC CHLORIDE PAPER IN THE GUTZEIT TEST.

THREE notes from different sources, recently published in this journal (ANALYST, 1927, **52**, 699), indicate that the production of uniform stains in the Gutzeit test is frequently a matter of some difficulty and uncertainty.

Until a comparatively recent period the difficulty was met in this laboratory by fixing the mercuric chloride paper to the top of the tube by means of a minute amount of seccotine. This method ensures all the evolved arsenic passing through the paper, but the paper is sometimes difficult to remove without tearing, and the tube will require cleaning before being used again. The whole trouble may be avoided by using the *strips* of mercuric chloride paper suggested by Sanger and Black (*J. Soc. Chem. Ind.*, 1907, **26**, 1115; ANALYST, 1908, **33**, 28). In carrying

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* In justice to the authors of the notes quoted by Mr. Richmond, it should be mentioned that it was the wish of the Publication Committee to publish information on the various devices now used in the test, and that originality was not claimed for the apparatus described.—EDITOR.

out the test the Gutzeit tube is packed with plumbised cotton wool up to such a height that the paper strip (4 × 70 mm.), when placed in the tube and just projecting from it, is just supported.

The evolved gases, after passing through the cotton wool, are then free to pass up the tube to the outer air, and, at first sight, one would suppose that only a portion of the arsenic would come in contact with the mercuric chloride paper. Experiment shows, however, that this is not so, and that all the arsenic in normal quantities (*i.e.* up to 0·05 mgrm. As_2O_3) is retained at the lower end of the paper, forming a stain whose length and intensity are proportional to the amount present.

Some attention must be paid to the quantities of zinc, acid and water used, so as to regulate the rate of evolution of hydrogen, which, of course, will affect the nature of the stain.

This method has been in use in this laboratory for eighteen months and gives satisfactory results with a minimum amount of manipulation; the writer recommends its trial to all whose duties may include frequent Gutzeit tests.

The use of mercuric bromide paper instead of mercuric chloride, has been recommended (ANALYST, 1926, **51**, 478), but after it had been used here for an extended period its superiority was not very apparent.

A. F. LERRIGO.

CITY ANALYST'S LABORATORY,
BIRMINGHAM.

Notes from the Reports of Public Analysts.

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

CITY OF BIRMINGHAM.

REPORT OF THE CITY ANALYST FOR THE THIRD QUARTER, 1927.

OF the 1268 samples submitted during the quarter, 1128 were taken under the Food and Drugs Acts. Of these, 1072 were bought informally (24 adulterated), and 56 were formal samples (2 adulterated).

MILK. LEAKAGE OF COOLER DEFENCE.—Two samples of milk taken on successive days from a farmer, delivering Grade A (Tuberculin Tested) Milk to a children's hospital, contained 36 per cent. and 29 per cent. of added water. Other samples from the same farmer were genuine.

The farmer's explanation was that the water in the milk was due to a leak in the cooler, which he discovered on the morning of the third day. The adulterated sample of milk was said to have been the last cooled and to have been left under the cooler a little longer than the others. It was difficult to believe that if the adulteration was due to a leaky cooler, three churns of milk, containing 32 gallons of milk, could have passed through the cooler and showed no signs of adulteration, whilst 1¼ of the next 4 gallons passed through the cooler was water.

An experiment made in the laboratory showed that, with a constant head of about two feet of water, 17 minutes was required to pass $1\frac{1}{4}$ gallons of water through a hole of $\frac{1}{16}$ inch in diameter—a hole which gives quite an appreciable jet.

The Magistrates were not satisfied with the explanation and fined the defendant £10 and 11s. special costs.

Treacle, Table Syrup.—The single sample of treacle was a sugar product, but two samples of table syrup contained about 20 per cent. and 50 per cent. of glucose syrup, respectively. These figures illustrate the fact that "table syrup" is an indefinite mixture of golden syrup and glucose syrup.

Salol Tablets.—Four of the 5 informal samples of 5 grains tablets contained 4·8 to 5·1 grains of salol; one had only 4 grains. These tablets were also irregular in weight, 40 per cent. of them differing from 5 to 10 per cent. from the mean weight, and 38 per cent. differing more than 10 per cent. from the mean weight. None of the other four samples contained any tablets which were more than 5 per cent. from the mean weight. That sample, also, contained 12·2 per cent. of talc and showed no signs of disintegration after being kept in water a month. The vendor, who supplied the article to one of the Birmingham hospitals, was cautioned. Three of the other 4 samples contained no talc and the fourth 4·7 per cent. The latter did not disintegrate so well as the others.

Aspirin Tablets.—Five informal samples of 5 grain tablets contained from 4·9 to 5·2 grains of aspirin. None of them differed more than 10 per cent. from the mean weight. One sample contained 5·7 per cent. of talc and disintegrated poorly in water. Another sample contained 2·6 per cent. of talc, and the other three did not exceed 0·1 per cent.

Calcium Lactate Tablets.—The British Pharmacopoeia allows a range of 7 per cent. in the composition of calcium lactate, and the 10 grain tablets should contain at least 6·6 grains of anhydrous lactate. One sample was just on this limit. Five other informal samples of 5 grain tablets contained from 3·3 to 3·6 grains of anhydrous lactate, 3·3 being the lower limit. Only one sample contained tablets which varied more than 10 per cent. from the mean weight, and all of the tablets of 3 of the 5 samples were within 5 per cent. of the mean weight. One sample had 0·1 per cent. of talc and disintegrated poorly. Others had from 3·1 to 5·2 per cent. In these tablets the presence of a small amount of talc appears to aid disintegration.

Barium Sulphate for X-ray Work.—This article is not in the B.P., but the British Pharmaceutical Codex requires that 10 grms. shall not contain any appreciable amount of soluble barium salts. Three of the 5 samples complied with this requirement, but two samples had soluble barium equivalent to about 0·01 per cent. of barium chloride. This percentage of impurity is very small; but, as 16 ounces of the barium sulphate may be given in a test meal for X-ray diagnosis, a high degree of purity is necessary. In this quantity the soluble barium would be equivalent to about $\frac{3}{4}$ grain of barium chloride, an amount which might not have any injurious effects, but one feels that pure samples should be preferred for this work.

J. F. Liverseege.

METROPOLITAN BOROUGH OF STEPNEY.

Annual Report of the Borough Analyst for the Year 1926.

The number of samples examined during the year was 1382, of which 1341 were taken under the Food and Drugs Acts; of these, 844 were formal and 497 informal. The number of samples found to be adulterated was 37, or 2·8 per cent.

MINERAL OIL IN MARGARINE.—A sample of margarine contained 9·5 per cent. of mineral oil (=11·9 per cent. on the fat). The solicitor for the defence produced two invoices stamped with a warranty bearing June dates, but, as the sample had been taken on May 18th, the vendor was fined £1, with £2 2s. costs.

Two other samples, obtained from a wholesale dealer, contained 10·0 per cent. and 10·5 per cent. of mineral oil, respectively. The dealer produced a warranty from the agents supplying him, and proceedings were taken against this firm for issuing a false warranty. It was stated that the oil came from Holland to the factory, where it was converted into margarine, and then sold through agents and wholesalers to the retailers. The agents supplying the vendor paid £3 3s. costs.

Another sample (informal) contained 13 per cent. of mineral oil, but a further (formal) sample consisted of genuine margarine.

CREAM CHEESE.—The following varieties of cheese were supplied in reply to a demand for cream cheese: (1) "Creamy cheese" or Full Cream Dutch; it consisted of whole milk cheese containing 25·5 per cent. of fat. (2) "Cream Cheese," whole milk soft cheese containing 15·5 per cent. of fat. (3) "Cream Cheese," whole milk soft cheese containing 21·4 per cent. of fat. (4) "Cream Cheese" (same vendor as No. 3), soft cheese containing 8·5 per cent. of fat, and made from partly skimmed milk. The vendor was fined £3, with £1 1s. costs.

In a leaflet on Dairying and Cheesemaking, issued by the Ministry of Agriculture and Fisheries, it is stated that, in general, two varieties of cream cheese are manufactured, one from cream containing about 50 per cent. of fat, and the other from cream containing from 25 to 30 per cent. of fat. Cheeses made from cream containing such percentages of fat will naturally contain a still higher proportion, namely, from 60 per cent. and upwards in the first variety, and from 40 to 50 per cent. and upwards in the second variety. The average proportion of fat in the cream sold in the Borough was 50·6 per cent.

DOUGLAS HENVILLE.

Legal Notes.

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

RUM AND BUTTER TOFFEE.

ON December 15, 1927, an appeal was heard in the King's Bench Division Court, before Justices Avory, Branson and Talbot, against a conviction by the Bishop Auckland magistrates for breach of warranty by the manufacturers of "rum and butter" toffee, in selling it under that name to a firm of grocers.

Mr. Lowenthal, K.C., for the appellants, said that the toffee was sold at 1d. per ounce, and there was no question about its purity. But a sample, taken by an inspector under the Food and Drugs Acts, had been analysed and found to contain coconut oil, in addition to butter. This was added to give the necessary consistence, but it was said that, since the toffee was called "rum and butter" toffee, and some other fat had been used as well as butter, there had been misdescription and a breach of warranty, because the purchaser did not get a sweetmeat of the nature, substance, and quality demanded. In his (counsel's) view, the point was an artificial one, although it was of great importance, because, if the decision went against them, the appellants would be compelled to destroy a large quantity of the toffee.

Mr. Justice Avory observed that if an old-fashioned cook, not knowing anything about synthetic flavours, were asked what toffee was made of, she would say "sugar and butter or treacle."

Mr. Lowenthal replied that it was not a question of synthetic flavours. A nutritious fat had been used.

Mr. Justice Talbot said that he was of opinion that the conviction was wrong, because "toffee" was a name given to a sweet, and so long as "rum and butter" toffee contained a substantial amount of butter, no fault could be found with it.

Mr. Justice Avory and Mr. Justice Branson held a different view. In their opinion the use of the word "butter" in this connection implied that butter only, and no other fat, was an ingredient of the toffee. Hence, as some other fat had been used, there had been a sale to the prejudice of the purchaser, who did not receive an article of the nature, substance and quality demanded.

The appeal was accordingly dismissed with costs by a majority decision.

CHEMICAL EVIDENCE OF DRUNKENNESS.

On December 28, 1927, a motorist was charged at Marlborough Street Police Court with being drunk while in charge of a car in South Carriage Road, Hyde Park.

Dr. Gorsky, Police Surgeon, gave evidence that, in addition to the usual tests for drunkenness, he had asked the defendant to inflate a football bladder with his breath. Subsequent analysis of the contents of the bladder had shown that two litres of the breath contained 1·5 mgrm. of alcohol, and from this the witness concluded that the man was "50 per cent. drunk." The test had only been used to confirm the results of his other tests.

The Magistrate (Mr. Cancellor) imposed a fine of £30, with £5 costs, and ordered the defendant's licence to be endorsed.

Government of Palestine.

ANNUAL REPORT OF THE DEPARTMENT OF PUBLIC HEALTH FOR THE YEAR 1926.

The Government Analyst, Mr. G. W. Baker, reports that, in the Chemical Sub-section, 5525 samples were examined, as against 5512 in 1925 (Analyst, 1927, **52**, 230). Of the 3800 samples of milk examined, 113 (or 3 per cent.) were the subject of prosecutions, as compared with 5·3 per cent. in the preceding year.

Semneh.—A special request for samples of semneh (native butter) brought in from the various districts 213, of which 45 were adulterated. There is some evidence that intensive sampling for a short period has more moral effect than occasional sampling over a longer period.

Legal, Judicial and Police Departments.—Arsenic was found in the viscera in two cases of suspected human poisoning. Yellow sulphide of arsenic was found in one specimen of dried figs and in two specimens of bread.

Forged Documents.—Thirteen documents were submitted for expert examination. In one of these alteration in a date was detected by chemical and photographic means. In the case of three bills it was shown that parts of the signatures were forged, the inference being that stamps bearing parts of the genuine signatures had been transferred from other documents.

Thirty samples of beer concerned in a forged label case were examined and compared with genuine samples obtained from Germany.

DEPARTMENT OF CUSTOMS, EXCISE AND TRADE.—The samples submitted by this Department were chiefly alcoholic liquors, confectionery, drugs, dyes, perfumes and essences. Two samples of matches have been submitted under the new phosphorus ordinance. Yellow phosphorus was absent from both samples.

The Analyst was called upon to give evidence in a case of the smuggling of motor spirit as a "turpentine substitute," and of lubricating oil as "crude oil." The duty on motor spirit is 4 piastres per gallon, which is equivalent to about 25 per cent. *ad valorem*; that on turpentine substitute would be 12 or 15 per cent. *ad valorem*. Lubricating oil has a duty of 12 per cent. *ad valorem*, and "crude oil" is free of duty.

In all such cases one is handicapped by the absence of official definitions. In this case it was shown that the so-called "turpentine substitute" was a volatile petroleum distillate which was suitable for use in ordinary motor cars, and that the flash point and distillation test did not conform to the American specifications for "petroleum thinners." The so-called "crude oil" was shown to be a petroleum product lacking volatile fractions and asphaltic constituents and to be suitable for use as a lubricating oil. A conviction was obtained.

DEPARTMENT OF AGRICULTURE AND FORESTS.—Twenty-nine specimens of viscera in connection with cases of animal poisoning were submitted by veterinary officers. Arsenic was found in 21, mercury in 1, and strychnine in 1.

NOTES AND SPECIAL INVESTIGATIONS.—(*a*) LEGISLATION.—The Ottoman Penal Code (Art. 194, Sec. 1, 2 and 3) requires proof of danger to health, and for that reason is of little value in most cases of adulteration. Although Art. 194, Sec. 5, leaves much to be desired, successful prosecutions may be obtained, so long as the magistrate is ready to accept the analyst's opinion as to what constitutes "imitation and adulteration" in any particular article of food and drink.

Article 257 is of use chiefly to enforce municipal regulations, but presumably the expression "articles of food or drink on which the natural composition is changed by fermentation . . . or from any other cause" might be interpreted to cover adulteration.

The Trade Marks Ordinance has been found very useful in all cases of false labelling, so long as it can be shown that there is intention to deceive a purchaser as to the origin of the article or the identity of the manufacturer. The cases of the Jamaica rum and arak Zahlawi noted below are instances. If there is a choice between the Ottoman Penal Code and the Trade Marks Ordinance, it is invariably more satisfactory to base a prosecution on the latter, as it is not open to so many interpretations as the penal code.

Practical experience in working with the above articles of the penal code during the past seven years has demonstrated the need for a special law to deal with the manufacture and sale of food and drink. Regulations could then be made defining the nature and composition of the important articles of diet and imposing certain standards of purity and methods of labelling.

With this object in view, recent legislation in England, New Zealand, Cyprus and other countries is being studied, and analytical data on locally produced food stuffs are being accumulated.

(*b*) Edible Oils.—Three important edible oils are produced in Palestine, *viz.* olive oil, sesame oil, and sunflower seed oil.

An intensive sampling of edible oils, started in July, 1926, and still being continued, has made it very evident that imported cottonseed oil is finding an increasing use as an adulterant of, and as a substitute for, olive oil and sesame oil.

The production of sunflower seed oil is a recent enterprise by a company with a large, modern, well-equipped factory which, in the face of competition from cotton seed oil, found it impossible to continue the production of sesame oil. Even sunflower seed oil, however, cannot be sold to compete with the lower grades of cotton seed oil.

(*c*) Alcoholic Liquors.—As the result of a prosecution instituted by this Laboratory under the above-mentioned section of the Trade Marks Ordinance, it was established in Court that a liquor made in Palestine cannot be sold legally as "Jamaica Rum," and that the name "Arak Zahlawi" can only be applied legally to arak made in Zahle. The defence claimed that "Jamaica" and "Zahlawi" were generic terms, and were intended to describe a particular kind of flavour or quality. The Court, however, held that, to the ordinary purchaser, they represented territorial descriptions.

These and other prosecutions appear to have had a very marked effect upon the use of spurious or deceptive labels, as was shown by recent inspections of distilleries and retail establishments. The present situation regarding denatured alcohol differs little from that described in the 1925 report.

Molasses Alcohol.—There are several factories now making alcohol from molasses, and the sale of this for drinking purposes, and, presumably, for the manufacture of spurious liquors, adds another complication to an already complex situation. The proposed Excise Ordinance mentioned in last year's report is not yet in operation.

Excise duty under existing regulations is paid on the raw material on the basis of the amount of alcohol obtainable. As molasses alcohol is a new product here, it has become necessary to determine the yield of alcohol from that material. With this end in view, samples of molasses and of alcohol obtained therefrom in a local distillery were submitted for analysis. The distillation process and measurements of vessels and distillates were under the direct supervision of an Excise Officer. As the results may be of interest, they are given here:

Total reducing sugars in the molasses (average of 5 samples, representing 13,899 kilos) ..	52·5 per cent.
Total yield of alcohol calculated to absolute alcohol after one fractional redistillation ..	18·6 ,,

Approximately 95 per cent. of this yield of alcohol was collected at a strength of 82 to 94 per cent. by volume, the remaining 5 per cent. being at a strength of 40 per cent. by volume.

(*d*) Arsenical Poisoning.—Arsenic is undoubtedly the favourite poison in Palestine, as in Egypt. In the year under review the arsenical poisoning of cattle was almost epidemic during the latter part of the summer. This coincided with the cattle plague outbreak, and it is possible that the cattle poisoners considered it a favourable opportunity.

Two forms of arsenic are used, *viz.* (1) white arsenic (arsenious oxide), and (2) artificial yellow or orange sulphide of arsenic.

It is a favourite practice to conceal the arsenic in dried figs which (if for cattle poisoning) are then enclosed in a bunch of grass or straw. It appears that the artificial sulphide owes its poisonous properties to the white arsenic it contains as an impurity. One specimen of the sulphide examined contained 50 per cent. of arsenic soluble in 2 per cent. hydrochloric acid. If sulphide of arsenic is the poison used, one expects to find some of it remaining in the stomach and intestines in the solid state. Even after much vomiting, it may still be found adhering to the walls of these organs.

In a case of human poisoning in the Haifa District granules of the yellow sulphide were found adhering to the stomach, and arsenic in solution was found in the liver and kidneys. In this case the police, on searching the house of one of the accused persons, found a packet containing red sulphide of arsenic, but no yellow sulphide. To satisfy the Court that the red sulphide produced could not have been the poisoning agent, it was analysed, and found to contain less than 1 per cent. of arsenic soluble in dilute hydrochloric acid. One-tenth of a grm. was also given to a rat, with no ill effects, whilst the same quantity of artificial yellow sulphide caused death within 24 hours.

In this same case the *post-mortem* examination had revealed the presence of a strangulated hernia, and in the trial the question naturally arose as to whether the strangulation had been brought about by the violent symptoms produced by the poison, or whether it was an independent condition which could have itself accounted for the death of the individual. The Court found that death was due to arsenical poisoning. The finding of a few grains of rice and fragments of marrow skin in the stomach was an important point in connecting the accused with the crime, as there was evidence that the poison had been concealed in a marrow stuffed with rice.

The sale of arsenic is subject to control under Public Health Ordinance No. 4, but there is evidence that some of the sulphide is smuggled into the country. It is used locally as an ingredient in depilatory pastes. The purchase of white arsenic, sodium arsenite, lead arsenate and Paris Green for use as insecticides is possible under permit. Recently a sample of four tons of white powder stored in sacks in the old Acre arsenal since Turkish times was submitted for analysis and proved to be white arsenic. It is remembered that some years ago a man was arrested in the same district for being in unlawful possession of a sack of what he appeared to think was flour. As this also proved to be white arsenic, it is probable that it came from the same source.

(*e*) LEAD IN SODA WATER.—Although, as mentioned in the 1925 report (*loc. cit.*), the presence of lead has been almost eliminated from bottled soda water produced in local factories, the same precautions cannot be applied to the small "soda fountains" existing in considerable numbers in all the larger towns. Samples taken from these fountains have contained as much as ten parts per million of lead in solution.

The fountains consist of a tin-plated copper carbonating chamber connected with a water supply and a cylinder of carbon dioxide. From this chamber the carbonated water passes through a lead cooling coil (kept in ice water) to the delivery tap on the fountain. The lead contamination is derived from the cooling pipe, and the amount of lead passing into solution depends upon the length of time the carbonated water is allowed to stand in the pipe.

Orders have been given to reject any carbonated water that has been standing overnight, but this does not eliminate the possibility of contamination, and, if frequent rejections were enforced, the function of the cooling pipe would, of course, be destroyed.

Pipes of pure tin are expensive and are not at present readily obtainable. Experiments are now being carried out with tinned copper cooling devices of local manufacture submitted by the S.M.O. Haifa. (*Cf.* ANALYST, 1928, 39.)

Ministry of Health.

STATUTORY RULES AND ORDERS, 1927, No. 1092 and No. 1093.*

PUBLIC HEALTH, ENGLAND.

(No. 1092) THE PUBLIC HEALTH (CONDENSED MILK) AMENDMENT REGULATIONS, 1927, DATED NOVEMBER 14, 1927, MADE BY THE MINISTER OF HEALTH.

(No. 1093) THE PUBLIC HEALTH (DRIED MILK) AMENDMENT REGULATIONS, 1927, DATED NOVEMBER 14, 1927, MADE BY THE MINISTER OF HEALTH.

The Minister of Health in the exercise of the powers conferred upon him by the Public Health Act, 1875, the Public Health (London) Act, 1891, the Public Health Act, 1896, the Public Health (Regulations as to Food) Act, 1907, and by Section 8 of the Milk and Dairies (Amendment) Act, 1922, and of every other power enabling him in that behalf, hereby makes the following Regulations, with the consent of the Commissioners of Customs and Excise, so far as they apply to the Officers of Customs and excise, that is to say:—

1. These Regulations may be cited as the Public Health (Condensed Milk) [Dried Milk] Amendment Regulations, 1927, and these Regulations and the Public Health (Condensed Milk) [Dried Milk] Regulations, 1923 (hereinafter called "the principal Regulations") shall be construed together and may be cited together as the Public Health (Condensed Milk) [Dried [Milk] Regulations, 1923 and 1927.

2. These Regulations shall come into operation on the 1st day of May, 1928, so far as they amend Part III of the principal Regulations (relating to importation of Condensed Milk), and in other respects on the 1st day of September, 1928:

Provided that the provisions of paragraphs 1 and 3 of the First Schedule to the principal Regulations shall not at any time after the date of these Regulations apply to a tin or other receptacle of condensed milk which is labelled in the manner prescribed in that Schedule as amended by these Regulations.

3. The following provision shall be added to and form part of the principal Regulations immediately after Article 4 thereof, that is to say,—

> "*4a.*—Where a tin or other receptacle containing condensed [dried] skimmed milk is required by Article 4 of these Regulations to be labelled, no person shall expose or offer for sale such a tin or receptacle in a paper or other wrapper unless such wrapper has printed on the outside thereof the words 'unfit for babies,' such words being contained within a surrounding line. The type used for the words shall be not less than a quarter of an inch in height and the printing shall otherwise conform with the rules prescribed for the printing of the same matter on the label affixed to the tin or other receptacle."

4. The following paragraphs shall be substituted for paragraphs 1 and 3 of the First Schedule to the principal Regulations:—

> "1. Every tin or other receptacle containing condensed milk shall bear a label upon which is printed such one of the following declarations as may be applicable or such other declaration substantially to the like effect as may be allowed by the Minister:—

(i) In the case of full cream milk (unsweetened):—

CONDENSED FULL CREAM MILK, UNSWEETENED. THIS TIN CONTAINS THE EQUIVALENT OF (*a*) PINTS OF MILK.

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* H.M. Stationery Office, each price 1d. net.

(ii) In the case of full cream milk (sweetened):—

> CONDENSED FULL CREAM MILK, SWEETENED.
>
> THIS TIN CONTAINS THE EQUIVALENT OF
> (*a*) PINTS OF MILK, WITH SUGAR ADDED.

(iii) In the case of skimmed milk (unsweetened):—

> CONDENSED MACHINE-SKIMMED MILK [or CONDENSED
> SKIMMED MILK], UNSWEETENED.
>
> UNFIT FOR BABIES.
>
> THIS TIN CONTAINS THE EQUIVALENT OF
> (*a*) PINTS OF SKIMMED MILK.

(iv) In the case of skimmed milk (sweetened):—

> CONDENSED MACHINE-SKIMMED MILK [or CONDENSED
> SKIMMED MILK], SWEETENED.
>
> UNFIT FOR BABIES.
>
> THIS TIN CONTAINS THE EQUIVALENT OF
> (*a*) PINTS OF SKIMMED MILK, WITH SUGAR ADDED.

"3.—(*a*) The prescribed declaration shall be printed in dark block type upon a light coloured ground.

(*b*) There shall be a surrounding line enclosing the declaration and in the cases in which the words 'unfit for babies' are required to be used there shall be another such line enclosing those words.

(*c*) The distance between any part of the words 'unfit for babies' and the surrounding line enclosing those words shall be not less than one-sixteenth of an inch.

(*d*) No matter other than that hereinbefore prescribed shall be printed within either surrounding line.

(*e*) The type used for the declaration shall not in any part be less than one-eighth of an inch in height (or if the gross weight of the tin or other receptacle does not exceed twelve ounces, one-sixteenth of an inch in height) and the type used for the words 'unfit for babies' shall not be less than twice the height of any other part of the declaration."

5. Copies of the Public Health (Condensed Milk) [Dried Milk] Regulations, 1923, printed under the authority of His Majesty's Stationery Office may be printed with any additions, omissions or substitutions directed to be made by these or any other amending Regulations but with a footnote in each instance referring to such amending Regulations; and the principal Regulations so printed may be cited as the Public Health (Condensed Milk) [Dried Milk] Regulations.

Given under the Official Seal of the Minister of Health this Fourteenth day of November, in the year One thousand nine hundred and twenty-seven.

R. B. CROSS (Assistant Secretary, Ministry of Health).

W. YOUNG, C. B. GRYLLS.

The Commissioners of Customs and Excise hereby consent to the foregoing Regulations so far as they apply to the Officers of Customs and Excise.

SALE OF FOOD AND DRUGS ACT, 1927.

GEO. V. CHAPTER 5.

AN ACT TO AMEND THE SALE OF FOOD AND DRUGS ACTS, 1875 TO 1907.

[12TH APRIL, 1927.]

Whereas it is expedient to give effect to the recommendations of the Departmental Committee on the use of preservatives and colouring matters in food:

Be it therefore enacted by the King's most Excellent Majesty, by and with the advice and consent of the Lords Spiritual and Temporal, and Commons, in this present Parliament assembled, and by the authority of the same, as follows:—

1.—(1) Where any regulations made under the Public Health (Regulations as to Food) Act, 1907, as amended by any subsequent enactment and the enactments mentioned in that Act, prescribe the composition of any article of food or drink intended for sale or prohibit or restrict the addition of any preservative or other ingredient or material to any such article, a purchaser of such article shall, unless the contrary is proved, be deemed for the purposes of section six of the Sale of Food and Drugs Act, 1875, to have demanded an article complying with the provisions of the regulations, as regards the presence or amount of any constituent, ingredient or material specified in the regulations, and the addition of any such ingredient or material, in contravention of the regulations, shall, for the purposes of the said Act of 1875, be deemed to render the article injurious to health.

(2) Where any such regulations restrict the addition of any preservative, or other ingredient or material, to an article of food or drink, the addition of any such ingredient or material to an amount not exceeding the limit specified by the regulations shall not, for the purposes of the Sale of Food and Drugs Act, 1875, be deemed to render the article injurious to health.

2.—This Act may be cited as the Sale of Food and Drugs Act, 1927, and shall be construed as one with the Sale of Food and Drugs Acts, 1875 to 1907, and those Acts and this Act may be cited together as the Sale of Food and Drugs Acts, 1875 to 1927.

3.—This Act shall come into operation at the date of the passing of this Act.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Composition of the Fat of Mare's Milk. O. Laxa. (*Ann. Falsif.*, 1927, **20**, 462–464.)—The fat from a sample obtained by mixing the milk of two mares gave the following figures:—M.pt., 8–9° C.; solidification pt., 5–6° C.; refractometer reading at 40° C., 50°; saponification value, 227·8; iodine value, 61; Reichert-Meissl value, 7·0; Wauters-Polenske value, 6·1; mol. wt. of soluble volatile acids, 163·8; mol. wt. of insoluble volatile acids, 194; of the non-volatile acids, 283; of the saturated insoluble acids, 249; m.pt. of non-volatile acids, 34–35° C.; solidification pt., 20° C. The composition of the fat was taken as caprylic acid, 2·29; capric, 2·19; oleic, 67·80; lauric, myristic, palmitic acids and glycerol, 27·72 per cent. D. G. H.

Detection and Determination of Rice Flour in other Flours and in Spices. M. Wagenaar. (*Z. Unters. Lebensm.*, 1927, **54**, 357–369.)—The structural and microscopical differences between rice and buckwheat flours are discussed from the point of view of the detection of adulteration by the former. Rice flour is more easily attacked by acid, alkali and fermentation reactions, but produces less decomposition of a 1 per cent. solution of hydrogen peroxide in the presence of acetic acid. The intensity of the blue colour produced by the addition of a solution of potassium iodide after 10 minutes is a measure of the undecomposed hydrogen peroxide. The physical effects of alkali on rice and buckwheat flours are best demonstrated by the use of Bellier's reagent (5 grms. of potassium hydroxide and 15 grms. of glycerin in 85 c.c. of water). The author has detected 0·5 per cent., and determined 5 per cent. of rice flour in buckwheat flour by counting and measuring under the microscope the cells in 50 mgrms. of the ground mixture. The cells were stained with 0·5 c.c. of a filtered solution of 0·5 grm. of magenta in 25 c.c. each of acetic acid and methyl alcohol, when a section through a rice grain showed a colour gradually decreasing in intensity towards the centre, whilst in the case of buckwheat only the outer layer and the embryo were coloured, and the starch remained unstained. In doubtful cases the presence of starch or protein matter was tested by the addition of a drop of a dilute solution of iodine in potassium iodide. For the quantitative determination, the flour (100 mgrms.) was washed and centrifuged, and suspended in 15 grms. of glycerin. The rice flour cells were counted and measured, and from the previously determined relations between their sizes, weights and volumes, the percentage present was calculated. There is risk of personal error, especially for large quantities of rice flour, but determinations by two observers gave fairly reliable results. The method was also applied to oatmeal, wheat flour, pepper, mustard, mace, nutmeg, cloves, and cinnamon. J. G.

Sugar Content of Water-Melons and other Types of Melon. S. N. Lutochin. (*Z. Unters. Lebensm.*, 1927, **54**, 281–289.)—Determinations of the sugar contents of the juices of a large number of samples of different types of water and other melons by the methods of Fresenius (ANALYST, 1921, **46**, 136) and of Willstätter and Schudel (*id.*, 1918, **43**, 416) have shown that the total sugars (monosaccharides after inversion) vary from 5·5 to 9·5 per cent. and from 4·25 to 13·25 per cent., respectively. The variation for the juices of water-melons from different plants of the same type is 0·25 to 2·5 per cent., and for a single fruit from the same stock, from 0 to 1 per cent. of total sugars. For the majority of the samples of water-melons the mean values of 52·1, 33·4 and 14·5 per cent. were obtained for the fructose, glucose and sucrose, respectively, whilst for melons the corresponding values varied from 0·09 to 3·70, 1·85 to 3·92, and 0·24 to 7·60 per cent., respectively. The absolute yield of sugar from the fruit varied from 35·19 to 442·75 grms. and from 20·9 to 178·83 grms. for the water-melons and other melons, respectively, and was not constant for fruits of the same type or from the same plant. The formation of sugar in the fruit is dependent on the soil, climate and nature of the plant. Statistics of melon production are also discussed. J. G.

Detection of Apple Juice in Pure Fruit Preserves (Cherries and Strawberries). C. F. Muttelet. (*Ann. Falsificat.*, 1927, **20**, 535–537.)—The presence of filtered apple juice in a pure fruit preserve or jelly may be detected by determining the sugars, the apple juice introducing an excess of laevulose. Moreover, in the case of citric acid fruits, such as gooseberries, black currants, raspberries, strawberries, or oranges, the results may be confirmed by applying tests for malic acid. The numbers of grms. of various sugars contained in 100 c.c. of the juice of cherries and strawberries were as follows:

	Sucrose.	Laevulose.	Dextrose.
Bigarreau cherries	0·10	3·60	4·30
Montmorency cherries	0·20	4·50	5·30
Cherries	0·30	5·00	6·50
Strawberries	0·35	1·90	2·00
Héricart strawberries	0·10	3·10	3·30
Tomato strawberries	2·50	2·10	2·10

The numbers of grms. of the sugars found in 100 grms. of various cherry and strawberry preserves were as follows:

		Sucrose.	Laevulose.	Dextrose.
Strawberry:	Household preserve ..	26·25	20·00	20·25
	Pure fruit jam	0·55	29·50	30·00
	Pure fruit preserve ..	9·75	26·05	25·90
	,, ,, ,, ..	12·50	25·10	24·90
	,, ,, ,, ..	27·65	19·80	19·10
Cherry:	Household preserve	39·25	14·00	14·15
	Pure fruit preserve	26·50	20·40	19·75
	Cherry and gooseberry preserve	6·75	28·00	26·40

The last of the strawberry preserves, which contained malic acid, and also the last two of the cherry preserves, undoubtedly contained added apple juice. (See ANALYST, 1922, **47**, 398; 1923, **48**, 181; 1927, **52**, 160.) T. H. P.

Almond, Apricot-kernel and Peach-kernel Oils. J. Pritzker and R. Jungkunz. (*Z. Unters. Lebensm.*, 1927, **54**, 233–242.)—The analytical values and reactions to qualitative tests of a number of samples of bitter and sweet almond oils, and apricot-kernel and peach-kernel oils, and of their fatty acids, have been determined and tabulated for oils extracted with ether from the dried and ground nuts, and for a number of trade products. It is concluded that pure almond oil does not give Bellier's reaction, and that, though the Kreis reaction (ANALYST, 1902, **27**, 330) gives a strong magenta-red colour with apricot-kernel oil, and will detect 5 per cent. of it with certainty in almond oil, it is not a reliable test for peach-kernel oil. (This reaction, in which a 0·1 per cent. ethereal solution of phloroglucinol is added to a mixture of the oil with an equal volume of nitric acid of sp. gr. 1·4, differs from the Kreis rancidity reaction, which gave negative results throughout.) Bieber's reaction is not recommended, and the Maben, Baudouin and Halphen reactions also gave negative results. The trade names of these oils are often misleading. Thus, so-called peach-kernel oil is usually adulterated with apricot-kernel oil, whilst the French custom of referring to the latter as "huile de marmotte" leads to its confusion with marmot fat. J. G.

Accidental Green Coloration of Olive Oil. (Test for Copper in Water.) R. Marcille. (*Ann. Falsificat.*, 1927, **20**, 538–539.)—The green colour sometimes exhibited by olive oil is due to the well-known compound of chlorophyll with copper. Its development in the oil stored in tinned iron vessels is due to the presence, in the tin, of traces of copper, which dissolves the more readily in the more acid oils. These green oils are obtained only during the early months of the extraction, since, when the olives are stored for some time, the skins undergo an oxidation which prevents the formation of the green compound. The presence of traces of copper in ordinary or distilled water may be readily detected by the appearance of the green colour when about 10 c.c. of olive oil are boiled for 10 minutes with 200–250 c.c. of the water in presence of a little pumice or a small Guernez tube to regulate the boiling. T. H. P.

Determination of Extractable Materials in Coffee. A. Schugowitsch. (*Z. Unters. Lebensm.*, 1927, **54**, 330–336.)—Comparative determinations of the materials soluble in alcohol, in ether, and in water (extracted from whole coffee beans) have been carried out by the Swiss and Austrian official methods. The amounts of extract obtained increased with the degree of roasting of the coffee in all cases, and for highly roasted beans were in excess of that allowed. For normally roasted beans (*i.e.* brown coloured with a matte surface) extracts of less than 1, 0·5 and 1 per cent. were always obtained for the alcoholic, ethereal and aqueous extracts, respectively. Only the alcoholic and ethereal extracts varied with the brand of coffee. Re-treatment of the extracted beans with fresh solvent, and the number and surface area of the beans taken, also influenced the results. The use of resin (colophony) as a fining material is detectable to within 0·5 per cent. by extraction of the abietic acid. The beans (10 grms.) are shaken with 30 c.c. of 0·5 *N* sodium hydroxide solution and kieselguhr for 3 minutes, and the filtered liquid acidified with sulphuric acid and extracted with ether. The residue left after evaporation of the ethereal extract is dissolved in 1 c.c. of acetic anhydride and tested with a drop of sulphuric acid (62·5 per cent.). A blue-violet colour, which changes through brown to yellow when the solutions mix, denotes the presence of abietic acid. J. G.

Rapid Test for an Excessive Amount of Lime in Ground Pepper. W. Plahl. (*Z. Unters. Lebensm.*, 1927, **54**, 369–371.)—The evolution of carbon dioxide from the calcium in pepper powders is visible under the microscope immediately after the addition of 2 drops of a solution of chloral hydrate containing 1 per cent. of dilute hydrochloric acid. Black and white peppers with ash-contents less than 7 and 4 per cent., respectively, gave no visible reaction, whilst for those with 13 per cent. or more of ash, it was visible to the naked eye. J. G.

Preparation of Saponin Products from Horse Chestnuts. R. Vadas. (*Chem. Ztg.*, 1927, **51**, 895.)—A sample of horse chestnuts contained starch and sugar, 63 per cent.; saponin, 11·90 per cent.; and fat, 6·12 per cent. (calculated on the dry material), with ash, fibre, and colouring matter. The nuts are used in the starch and fermentation industries, but the saponin must first be removed on account of its toxicity to yeast. According to the author's method of separation (D.R.P., 396,029) the dried material is de-fatted with petroleum spirit and extracted with 70 per cent. alcohol at 55° C. The alcohol is recovered by evaporation *in vacuo*, and the brown residue re-dissolved in alcohol, and precipitated by the addition of ether. The crude saponin is then purified by acetylation with acetic anhydride in the presence of zinc chloride, mixed with freshly precipitated lead hydroxide, and finally filtered and dried in a vacuum. The properties and uses of saponin and its derivatives are briefly mentioned. J. G.

Modification of the Gravimetric Method for the Evaluation of Hops. J. J. H. Hastings and T. K. Walker. (*J. Inst. Brew.*, 1928, 34, 9–13.)—The gravimetric method for the determination of resins, now used by the authors, is as follows :—The given sample of hops (25–30 grms.) is minced, the first portion (about five grms.), which passes through the machine being discarded. Ten grms. of the remainder are transferred to a Soxhlet extractor, the capacity of which is 100 c.c. to the top of the siphon tube, a small pad of cotton wool, previously extracted with ether, being placed in the bottom of the Soxhlet, so that it covers completely the outlet to the siphon tube. The extraction is made with about 200 c.c. of ether (methylated, sp. gr. 0·730). The receiver is a 250 c.c. bolt-head flask, placed on a water bath heated by an electric heater to a temperature not exceeding 55° C., which distils the ether at a rate sufficient to cause the siphon to function at intervals of about twelve minutes. A four-bulb water-jacketed reflux condenser is adapted to the top of the Soxhlet, and in warm weather it may be necessary to use, in addition, a ball condenser. Extraction of the hops is allowed to proceed for three hours, after which the Soxhlet is removed and the ether distilled off from the flask through a condenser, the last portion of the solvent (about 30 c.c.) being removed under diminished pressure in order to avoid overheating the resin. The resin is then extracted with successive portions (20 c.c. each) of warm (50°–60° C.) methyl alcohol (b.pt., 65° C.), and the extracts mixed together, their total volume amounting to 100 c.c. at 15·5° C. The combined extracts are filtered through paper, and 40 c.c. of the filtrate are removed, warmed to 60° C., and the α-resin content precipitated with a one per cent. solution of lead acetate in methyl alcohol. In conducting this precipitation the end-point is found by "spotting" on sodium sulphide paper, an excess of 1–2 c.c. of lead acetate solution being added to ensure that the end-point is indicated quite definitely. The α-resin lead salt is filtered off in a tared Gooch crucible, washed carefully with methyl alcohol, and dried at 100° C. for 1 hour. The precipitate should be allowed to stand for half-an-hour before filtration, as this allows it to settle and makes filtration much quicker. For the determination of the total soft resin 20 c.c. of the remainder of the methyl alcohol solution are diluted with 40 c.c. of water containing a little common salt (not more than one per cent.), and the resin is subsequently extracted by shaking with four successive volumes, each of 50 c.c., of petroleum spirit (b.pt., below 40° C.). The combined petroleum extracts are filtered into a tared flask, the filter paper being carefully washed with more petroleum spirit. The petroleum is then distilled off through a condenser, and the flask dried at 100° C. for 1½ hours.

Weight of total soft resin × 50 = per cent. of total soft resin.
Weight of α-resin lead salt × 15·75 = per cent. of α-resin.
Per cent. T.S.R.—per cent. α-resin = per cent. β-resin.

Assay of Sulphonal Tablets. L. E. Warren. (*J. Assoc. Off. Agr. Chem.*, 1927, **10**, 523–526.)—The following method, which assumes the absence of extraneous substances soluble in chloroform, yields results of moderate accuracy. Ten tablets are weighed separately to ascertain the average weight and the individual variations, and are then powdered to pass a No. 60 sieve. Sufficient of the powder to represent at least 5 grms. of sulphonal is exhausted with chloroform either in a Bailey or Soxhlet extractor or by maceration with successive quantities of 10 c.c. of the solvent, followed by filtration and washing of the filter. The extract is evaporated in a tared dish at the ordinary temperature in a gentle current of air, and the residue dried over sulphuric acid to constant weight.

T. H. P.

An Unrecorded Constituent of Commercial Ethyl Ether. H. King. (*Nature*, 1927, **120**, 843.)—During the course of some experiments on the bromine absorption of commercial anaesthetic ethers, for which a standard solution of bromine in glacial acetic acid was added with exclusion of light to avoid direct substitution of the ether by bromine, the different ethers from six manufacturers absorbed as follows :—A, 0·6 ; B, 2·5 ; C, 7·5 ; D, 11·6 ; E, 13·6 ; and F, 15·9 c.c. of 0·1 N bromine in 100 c.c. of the ether in the dark in one hour, and these numbers, in the absence of aldehydes, give a measure of the degree of unsaturation of the ethers. One grm. of αβ-dibromovinylethyl ether was identified as a major product from the bromination products of 2250 c.c. of ether C. It must have arisen from vinylethyl ether, $CH_2:CH.O.CH_2.CH_3$, b.pt. 35·5°, present in the original ether. Vinylethyl ether was present to a similar extent in freshly prepared manufacturer's ether which had never been exposed to light. It doubtless owes its origin to vinyl alcohol behaving in the same way as ethyl alcohol in the Williamson process. The instability of vinylethyl ether to acids, with production of acetaldehyde, is on record, but it also develops aldehyde on storage without acid and reacts towards Schiff's and Tollens' reagents as an aldehyde. The question whether vinylethyl ether is the first product of the action of light on diethyl ether, as postulated by Wieland, is being investigated, but an old sample of solvent ether, rich in peroxides, was found to contain only a small amount of vinylethyl ether. P. H. P.

Biochemical.

Preservation of Blood for Chemical Analysis by the Use of Sodium Fluoride. J. H. Roe, O. J. Irish and J. I. Boyd. (*J. Biol. Chem.*, 1927, **75**, 685–695.)—It has been concluded by previous workers, whose results are discussed, that sodium fluoride is an efficient preservative of blood for chemical analysis.

Further experiments have now been performed to ascertain (1) the concentration of sodium fluoride necessary for satisfactory preservation; (2) the advisability of sterilisation when fluorides are used as preservatives; and (3) possible interference with standard methods of analysis. All the experiments were carried out upon human blood. Sodium fluoride in concentrations of 10 mgrms. per c.c. of blood will prevent changes in the values for non-protein nitrogen, uric acid, creatinine, sugar and cholesterol, in sterile blood for at least 10 days; preservation will occur for 30 days under these conditions for all except non-protein nitrogen values. A concentration of at least 20 mgrms. of sodium fluoride per c.c. of blood has been found necessary to prevent marked changes in non-sterile blood (blood inoculated with *Bacillus coli communis* and *Bacillus subtilis*), but even increased amounts of fluoride do not give as satisfactory preservation as can be obtained with blood submitted to sterile conditions. Sodium fluoride interferes with the determination of urea by methods which involve urease hydrolysis. The interference is proportional to the concentration of fluoride in the hydrolysate. This difficulty can be overcome in bloods which contain not over 30 mgrms. of sodium fluoride per c.c. by dilution with 7 to 10 volumes of water before treatment with urease. Precipitation of the fluoride ions with magnesium is a beneficial procedure for concentrations of more than 30 mgrms. of sodium fluoride per c.c. P. H. P.

The Contents of Vitamins *A* and *C* in Watercress. K. H. Coward and P. Eggleton. (*Lancet*, 1928, **214**, 97–98.)—Watercress is a remarkably rich source of vitamin A, 0·1 grm. only of the green leaf being necessary to promote normal growth in a rat which has become steady in weight on a diet lacking this factor only. It contains also small amounts of vitamin D. Its growth-promoting properties are even stronger in the spring and summer than in the winter. It is a very rich source of vitamin *C*, 1 grm. daily being enough to protect guinea-pigs completely for a period of 70 days.

Bacteriological.

Substitution of Janus Green for Methylene Blue in the Reductase Test for Milk. L. Soep. (*Le Lait*, No. 70, Dec., 1927.)—Christiansen (*Milchwirtschaft. Ztg. Lubeck*, No. 42, 1926; *Molkerei Ztg.*, No. 102, 1926) suggested the use of Janus Green (safrano-azo-dimethylaniline) in place of methylene blue in the reductase test, and his modification has been adopted by many of the large dairy establishments in Germany. Janus green is bluish green in acid solution (bright green in alkaline solution) and, where reduced, forms a bright red compound, changing to violet, and finally becoming colourless. The red compound first formed is stable towards atmospheric oxygen. The time of reduction is taken as the period between the beginning of the test and that at which the formation of the red compound is complete. One c.c. of a solution of 1 part of the dye in 10,000 parts of water is added to 10 c.c. of the milk, and the test then carried out in the same way as the methylene blue test. The classification of milks in Scandinavian countries, based on that of Barthel and Orla Jensen, is as follows :

Class I.—Good milk: Reduction in not less than 5½ hours; number of bacteria less than 0·5 million.

Class II.—Milk of average quality: Reduction in 2 to 5½ hours; 0·5 to 4 million bacteria.

Class III.—Bad milk: Reduction in ⅓ to 2 hours; 4 to 20 million bacteria.

Class IV.—Very bad milk: Reduction in less than 20 minutes; more than 20 million bacteria.

Christiansen's classification is somewhat different, the times of decolorisation (change to red) being : Class I, 0 to 1 hour ; Class II, 1 to 3 hours ; Class III, 3 to 6 hours ; and, Class IV, more than 6 hours.

The author has made a series of parallel tests by means of Bathel's original method and Christiansen's modification on 1036 samples of milk, as normally received by the Dutch Food Control during the months of August and September, 1927. The results obtained were as follows :

Change of Colour after.	Methylene blue.		Janus green.	
Hours.	Total.	Per Cent.	Total.	Per Cent.
½	275	26·5	105	10·1
1	454	43·8	228	22·0
2	644	62·2	439	42·5
3	773	74·6	619	59·8
4	864	85·3	793	76·5
5	924	89·2	884	85·3
6	945	91·2	945	91·2

The conclusion drawn from the experiments made is that Barthel's reductase test gives too high results, owing to diffusion of atmospheric oxygen into the liquid and resulting re-oxidation of the leuco compound formed by the reduction of the methylene blue. For this reason a dye such as Janus green, the red reduction compound of which is not re-oxidised in this way, is preferable for the reductase test, for which, however, a different classification scale is then required.

Toxicological and Forensic.

Preparation, Purification and some Properties of Gossypol, the Toxic Principle of Cottonseed. E. P. Clark. (*J. Biol. Chem.*, 1927, **75**, 725–739.)—A brief summary is given of all that has previously been recorded concerning the chemistry of the peculiar phenolic body gossypol, the toxic principle of cottonseed. It has now been prepared in a state of analytical purity, and its molecular formula has been found to be $C_{30}H_{30}O_8$. The gossypol obtained was a bright canary-yellow crystalline substance with a melting point of 214° C. (corr.). In solution no optical activity was exhibited. It was soluble in ether and acetone, sparingly soluble in other common organic solvents, and insoluble in water. Various other properties are also recorded. Analytical data, including optical properties in certain cases, have been obtained with several derivatives of gossypol, *viz.*: the so-called " acetate," *i.e.* gossypol+1 molecule of acetic acid, anhydrogossypol (B gossypol of Carruth (*J. Amer. Chem. Soc.*, 1918, **40**, 647)), the dianilide, the dioxime and the hexacetyl derivative. The dianilide, which is used for the quantitative determination of gossypol, has been found not to be a salt of gossypol (1 mol. of gossypol+2 mols. of aniline), as Carruth has suggested, but a condensation product formed by 1 mol. of gossypol condensing with 2 mols. of aniline, with the elimination of 2 mols. of water. Gossypol has 2 oxygen atoms present as carbonyl groups and 6 as hydroxyl groups. Two of these hydroxyl groups behave differently from the remaining 4, as they are much more acidic and require drastic treatment for the hydrolysis of their acetyl derivative. A short report concerning some interesting physiological effects of gossypol upon rats is made.

P. H. P.

Organic Analysis.

Determination of Water in Dynamite Glycerin. T. Berth. (*Chem. Ztg.*, 1927, **100**, 975.)—The sample of dynamite glycerin is distilled with tetrachlorethane, which has the advantage over xylene of being denser than water and non-inflammable. The aqueous layer of the distillate is measured in a capillary graduated in 0·01 c.c. In the case of colourless or very pale samples the results agree closely with those obtained by sp. gr. and acetin determinations ; but with darker samples the results only agree if a little finely powdered silver nitrate is present in the distilling flask. This effect is not yet explained. In making a determination 2·5 grms. of finely powdered silver nitrate are placed in a boiling flask with 150 c.c. of tetrachlorethane and well shaken. One hundred grms. of the sample are now weighed into the flask, which is connected with a condenser. The distillation must be commenced with a small flame and carried out slowly, with an asbestos guard to prevent condensation in the neck of the flask. The volume of the water distilled gives the result expressed as a percentage. The time of distillation is less than 1 hour, and the results are far more accurate than those obtained by the International Standard Method, according to which the sample is exposed in an evacuated desiccator over sulphuric acid for 48 hours.

R. F. I.

Fatty Acids of Seed Oils of *Brassica* Species. Composition of Rape, Ravison, and Mustard Seed Oils. T. P. Hilditch, T. Riley, and N. L. Vidyarthi. (*J. Soc. Chem. Ind.*, 1927, **46**, 457–462T.)—The fatty acids combined with glycerol in the fats of these representative oils of the genus *Brassica* have been determined by preliminary separation of the mixed fatty acids into two or three groups by means of the varying solubility of the lead salts in alcohol and ether, followed by quantitative fractional distillation of the methyl esters at a pressure of about 1 mm. The percentages of the different acids in the total fatty acids were as follows :

Oil.	English rape seed.	Danubian ravison.	English black mustard (charlock).	English white mustard.
Source.	*Brassica campestris.*	*B. campestris* (var.).	*B.* (*Sinapis*) *nigra.*	*B.* (*Sinapis*) *alba.*
Palmitic	1	2	2	2
Stearic	—	—	Trace	Trace
Arachidic	—	—	Trace	1
Behenic	—	Trace	—	—
Lignoceric	1	2	2	1
Oleic	32	20·5	24·5	28
Linolic	15	25·5	19·5	14·5
Linolenic	1	2	2	1
Erucic	50	47	50	52·5

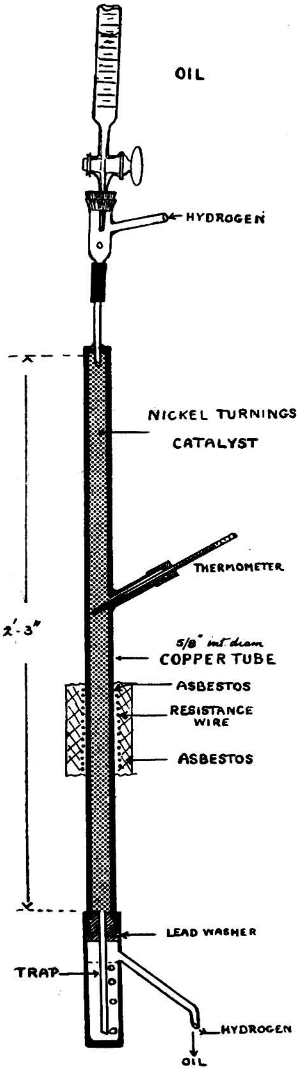
The chief chemical difference between these oils, connoted by variation in the iodine absorption, consists in the extent of the unsaturation in the oleic acid and linolic acid group, in which a small amount of linolenic acid is also usually present. The total proportion of the unsaturated acids containing 18 carbon atoms ranges from 43·5 to 48 per cent. T. H. P.

Ill-defined Acids of the Oleic Series. Part III. "Rapic Acid" and other Acids of Rape and Mustard-Seed Oils. T. P. Hilditch, T. Riley, and N. L. Vidyarthi. (*J. Soc. Chem. Ind.*, 1927, **46**, 462–467T.)—In rape, ravison, and mustard-seed oils, the unsaturated acids containing 18 carbon atoms consist mainly of ordinary oleic and linolic acids, with a small proportion, probably not more than 1 per cent., of a liquid acid (or acids), which is isomeric with oleic acid, forms a lead salt (or salts) sparingly soluble in cold alcohol or ether, and yields a dihydroxystearic acid (or a mixture of such acids) melting at 117–118° C. When subjected to controlled oxidation, this acid or its most prominent component yields a dicarboxylic acid, $C_{10}H_{18}O_4$, m.pt. 137–138° C., and probably an octoic acid. The lower saturated fatty acid present is almost entirely palmitic acid (2 per cent.); and myristic acid, which Sudborough, Watson and Ayyar (*J. Indian Inst. Sci.*, 1926, **9A**, 25) assert to be present in place of palmitic acid in Indian rape and mustard oils, cannot form more than 0·2 per cent. of the acids of the oils now considered. The erucic acid found is $\Delta^{13:14}$-dodecenoic acid, and no indication is obtained of any isomeric dodecenoic acids. T. H. P.

Ill-defined Acids of the Oleic Series. Part IV. "Cheiranthic Acid" of Wallflower Seed Oil. T. P. Hilditch and E. E. Jones. (*J. Soc. Chem. Ind.*, 1927, **46**, 467–469T.)—The so-called cheiranthic acid of wallflower seed oil, described by Matthes and Boltze (*Arch. Pharm.*, 1912, **250**, 211), does not exist, the oil containing a mixture of oleic, linolic, linolenic, and erucic acids, together with smaller proportions of palmitic and lignoceric acids. T. H. P.

Purification of Palmitic and Stearic Acids. A. L. Wilkie. (*J. Soc. Chem. Ind.*, 1927, **46**, 471–472T.)—Samples of palmitic and stearic acids, purchased as reasonably pure, were found to contain, in each case, a proportion of the other acid. The usual methods of purification, involving either the fractional crystallisation of the acids from suitable solvents or the fractional precipitation of their salts, are laborious and wasteful, but it is found that moderately pure acids may be obtained by fractional distillation of the ethyl esters (see Armstrong, Allan, and Moore, ANALYST, 1925, **50**, **191**) in a special apparatus embodying the Dufton column (ANALYST, 1919, **44**, 148). Acids of a higher degree of purity may then be prepared by subsequent recrystallisations from petroleum spirit, as described by Stratton and Partington (*Phil. Mag.*, 1924, (VI), **48**, 1085).

T. H. P.



Hydrogenation of Fatty Acids and of Mixtures of Fatty Acids with Neutral Oils. R. G. Pelly. (*J. Soc. Chem. Ind.*, 1927, **46**, 449T–454T.)—The rate of hydrogenation of mixtures of neutral oil and fatty acids was investigated for a large number of different mixtures, both activated nickel turnings and powder catalyst being used. The apparatus shown in the diagram was used for the nickel turnings catalyst. The trap at the exit end is to prevent access of air. The copper catalyst tube is electrically-heated, and the oil flows in from a burette, fitted, in the case of day and night experiments, with a capillary nozzle. Where powder catalyst was used, it was first reduced for one hour at 300–320° C. and the oil run into the flask and hydrogenated with vigorous stirring with a large excess of hydrogen passing through at 180° C. Two flasks were run in the same oil bath, and samples were drawn at stated times. With both types of catalyst it was found that fatty acids alone were less rapidly hydrogenated than neutral oil alone, and that in mixtures of fatty acids and neutral oil the degree of hydrogenation of the fatty acids is very considerably increased beyond that for fatty acids alone, or even beyond that for neutral oil alone, while the degree of hydrogenation of the neutral oil is greatly decreased. Further, the presence even of saturated fatty acids greatly retards the rate of hydrogenation of unsaturated glycerides. So far, a satisfactory theory for the above facts is not available. D. G. H.

Melting Point of Hydrogenated Cottonseed Oil. K. A. Williams. (*J. Soc Chem. Ind.*, 1927, **46**, 448T–449T.)—By plotting the m.pts. and iodine values of samples of cottonseed oil, hydrogenated at different temperatures, a series of curves is obtained with the same initial and final points, *i.e* for the original and fully hydrogenated oil. The m.pt. of the oil of iodine value 67 was obtained for each curve, and showed that over the range of temperature from 120 to 200° C. the figure is inversely proportional to the temperature. Since variation from complete selection was also found inversely proportional to temperature of hydrogenation over the same range, the m.pt. of hardened cottonseed oil of a given iodine value is directly proportional to the divergence from complete selection when the oil is hardened between 120 and 200° C. D. G. H.

Salts of Alpha-Linolic Tetrabromide from Candlenut (Lumbang) Oil. C. M. Jovellanos and A. P. West. (*Philippine J. Sci.*, 1927, **33**, 349–356.)—Bromination of the mixed fatty acids of lumbang oil yielded linolenic hexabromide, and a mixture of crystalline (alpha) and oily (gamma) linolic tetrabromides. The purified alpha-linolic tetrabromide melted at 112·3 to 114·3° C. Its salts were prepared by first converting it into the potassium salt, an alcoholic solution of which was then treated with a solution of the inorganic salt such as cobalt chloride. Chloroform was found to be the best solvent for the copper and cadmium salts; ethyl benzoate for the manganese salt, and, to a less extent, for the cadmium salt; whilst the cobalt and magnesium salts were only slightly soluble in a few solvents (hot benzyl alcohol, ethyl bromide, and (magnesium salt) hot toluene or xylene.

Iodimetric Evaluation of Methylene Blue. W. C. Holmes. (*J. Assoc. Off. Agr. Chem.*, 1927, **10**, 505–507.)—According to Moraw (*ibid.*, 1923, **7**, **20**), addition of acetic acid helps to complete the reaction between iodine and methylene blue, but the author finds that such addition restricts the absorption of iodine by the dye. The proportion of iodine taken up depends primarily on the concentration of residual iodine in the solution. If accurate results are to be obtained by the "official" iodimetric method for the evaluation of methylene blue, allowance must be made in this method for variation in the concentration of the dye.

T. H. P.

Inorganic Analysis.

Electrometric Titrations. Chloramine-T as Titrating Reagent. A. McMillan and W. Easton. (*J. Soc. Chem. Ind.*, 1927, **46**, 472T.)—The end-point in titrations with chloramine-T (see Noll, ANALYST, 1925, **50**, **34**) may be determined very accurately electrometrically by means of an apparatus similar to that used by Kelley (*J. Ind. Eng. Chem.*, 1917, **9**, 780). The method gave accurate results when applied to the determination of mixed arsenious and arsenic oxides, tin in tin-foil or phosphor-tin, antimony chloride, and iron. T. H. P.

Test for Small Quantities of Free Ammonia or Alkali (Hydroxl-ions). E. Schmiz. (*Arch. Pharm.*, **265**, 2, 15; *Pharm. J.*, 1927, **119**, 645.)—One c.c. of 1 per cent. copper sulphate solution is mixed with 1 c.c. of 3 per cent. hydrogen peroxide, and 0·5 c.c. of the diluted ammonia or alkali solution is carefully superimposed. A brownish-black or light brown flaky precipitate of oxides of copper is immediately formed, and, on greatly diluting, a dark or light yellow colour appears. Reagent plus ammonia:—Dilution, 1:100, brownish-black zone; 1:1000, dark brown; 1:10,000, light brown; 1:100,000, yellow; 1:1,000,000, yellow tint just noticeable. Reagent plus potassium hydroxide or sodium hydroxide: 1:100, dark brown zone ; 1:1000, light brown ; 1:10,000, yellow tint just noticeable; 1:100,000, no colouration. Reagent plus calcium hydroxide, lime water freshly prepared from *Calc. oxid. e marmore* ; 1·7:1000, dark brown zone ; 1·7:10,000, dark yellow ; 1·7:100,000, no coloration. One c.c. of freshly prepared Nessler's reagent overlaid with 1 c.c. of 1:100,000 ammonia gives a light reddish-yellow zone and with 1:1,000,000 ammonia a light yellow tint with a white ring. D. G. H.

Determination of the Constituents of Gaseous Mixtures containing Hydrogen Sulphide, Carbon Dioxide, Hydrogen Arsenide or Phosphide, and Acetylene. M. Wilmet. (*Compt. Rend.*, 1927, **185**, 1136–1138.)—The constituents of the mixture are absorbed in turn by suitable reagents: hydrogen sulphide by zinc acetate in the form of a crystalline mass; carbon dioxide by potassium hydroxide; arsine by an 80 per cent. solution of cadmium acetate; phosphine by a 30 per cent. solution of selenous anhydride, and acetylene by Lebeau and Damien's alkaline potassium iodomercuric reagent (*Ann. Chim.*, 1917, **8**, 340). Absorption takes place rapidly in all cases except arsine and the last traces of acetylene. D. G. H.

Volumetric Determination of Antimony and Arsenic. P. E. Winkler. (*Helv. Chim. Acta*, 1927, **10**, 837–842.)—After the reduction of quinquivalent antimony and arsenic to the trivalent state by means of iodine, the liberated iodine may be titrated with thiosulphate ; this is unsatisfactory. Volatilisation with steam while accelerating the liberation by bubbling carbon dioxide through the liquid is satisfactory, however, the iodine being titrated in a bicarbonate medium. For antimony, 10 c.c. of concentrated hydrochloric acid are added to 100 c.c. of neutral antimony solution containing up to 0·2 grm. of antimony, together with 10 c.c. of 10 per cent. potassium iodide solution and a few c.c. of starch solution. For arsenic, the same conditions hold except that 10 c.c. of 50 per cent. sulphuric acid are added before the hydrochloric acid and 20 c.c. of potassium iodide are taken. For larger quantities of metal the volumes of the solutions are increased. Excellent results were obtained, and it is suggested that the method may advantageously replace gravimetric methods. D. G. H.

Differentiation of Aluminium Alloys by the use of Indicators of the P_H Value. Cu. Quillard. (*Comptes rend.*, 1927, **185**, 1281–1283.)—If samples of industrial aluminium or aluminium alloys are submerged in doubly distilled water or in a strong electolyte the P_H value is characteristically altered. Strips of metal, 7·3 sq. cm., left for two hours in a solution of pure sodium chloride of P_H value 5·4, with bromcresol purple as indicator, altered the P_H value from 5·6 to 7·0. A series of samples of aluminium silicon alloys with magnesium and manganese was found to be of the following ascending order of liability to attack : Manganese 0·5 per cent. ; silicon 1 ; silicon 2 ; silicon 4 ; silicon 5 ; manganese 1 ; manganese 2 ; manganese 1·5 ; magnesium 0·5 ; magnesium 1 ; magnesium 1·5 ; magnesium 2 per cent. Points of attack of different samples, and even certain differences of methods of attack, may be demonstrated by this method. On prolonging the immersion in water characteristic coloured marks are produced on the metals. American metal was thus found more liable to corrosion than

French industrial metal. Mechanical cleaning by sand increased the action with metals, but not with certain industrial alloys. D. G. H.

Volumetric Determination of Cerium. G. Autié. (*Bull. Soc. Chim.*, 1927, **41**, 1535–1546.)—As the result of an examination of the published volumetric methods, the author recommends the three following :—(1) Von Knorre's method as modified by Bielz and Pieper (*Z. anorg. Chem.*, 1924, **134**, 13), in which the oxidation by persulphate is effected in alkaline solution ; (2) Meyer and Schweitzer's method (*ibid.*, 1907, **54**, 104) ; oxidation by permanganate in hot alkaline solution, with addition of zinc oxide or magnesia ; and (3) Tomiček's potentiometric method (ANALYST, 1925, **50**, 360). (*Cf.* also ANALYST, 1927, **52**, 365.) W. R. S.

Physical Methods, Apparatus, etc.

Arc-Spectrographic Detection and Determination of Germanium. J. Papish, F. M. Brewer and D. A. Holt. (*J. Amer. Chem. Soc.*, 1927, **49**, 3028.) —A table is given showing the position and visibility of the spectral lines of solutions of potassium germanate at varying concentrations. A stock solution of pure germanium dioxide in the smallest possible amount of potassium hydroxide is prepared so that it contains 1 grm. of germanium per 100 c.c., and dilute solutions of this are made up from 0·1 to 0·00001 grm. of germanium per 100 c.c. The solution to be tested (0·1 c.c.) is deposited on the graphite electrode of an arc lamp (already heated by a short discharge), the electrode being renewed for each solution tested. By comparing the position of the spectral lines of an unknown solution with those in the table, and also the dilution at which they are visible, an idea may be obtained of the amount of germanium present. The results are not affected by the presence of tin. Germanium has been detected by this method in several varieties of cassiterite and other tin minerals. Germanium has been found in enargite to the extent of 0·01 to 0·03 per cent., and a method of extracting it from this mineral is described. R. F. I.

Estimation of the Age of Eggs by means of Ultra-Violet Fluorescence. J. E. H. van Waegeningh and J. E. Heesterman. (*Chem. Weekblad*, 1927, **24**, 622–623.)—The ultra-violet fluorescence of egg-white, diluted with water to a convenient extent (up to 10 per cent.), increases with the age of the egg, and may be measured by comparison with that of a series of sterile, standard solutions of gelatin (0·5 to 10 per cent.). If yellow and blue colour filters are used, the slight variations in fluorescence between the sample and comparison solutions may be neutralised. At high temperatures the differences in fluorescence due to ageing are less marked. J. G.

"Greasy" Burettes. W. Lowson. (*Nature*, 1928, **121**, 14).—Burettes used with standard acid become "greasy" after being in use for a short time, with the result that small drops of the standard solution adhere to the surface of the glass above the level of the liquid. The addition of a minute trace of saponin to the standard acid will prevent this, and, if the solution is not unduly shaken, will not cause any inconvenience through frothing.

Nitrocellulose Membranes of Graded Permeability. H. F. Pierce. (*J. Biol. Chem.*, 1927, **75**, 795–815.)—The lack of precision and the number of uncontrolled conditions in the usual methods of membrane preparation led the author to endeavour to standardise the factors which enter into the process, and to simplify it as much as was consistent with uniformity of results. A method for the preparation of nitrocellulose filtering membranes is given in which, if the constitution of the collodion, the temperature of the drying film, and the condition of the atmosphere in which the film is dried, are kept constant, reproducible membranes of different degrees of permeability may be prepared if the drying time is varied. A second, simpler method is described in which the permeability of the membranes is varied by the incorporation of different percentages of a water-soluble, non-volatile swelling agent in anhydrous ether-alcohol collodion, complete evaporation of the volatile solvents in a water-free atmosphere and immersion of the resulting gels in water. For example, 4 c.c. of a 1 per cent. ether and alcohol collodion solution, containing ethylene glycol (1 to 15 per cent.), as volatile swelling agent, are evaporated on a 7·5 cm. plate in a moisture-free atmosphere. This gives a membrane, weighing about 0·039 grm., the filtration rate of which (in c.c. per sq. cm. per minute) increases with the proportion of ethylene glycol in the solvent up to a certain limit. The prime determinant of permeability of a membrane is the ratio between the swelling agent or solvent and the nitrocellulose in the film at the time of its immersion in water. The structure, strength, uniformity and permeability of a membrane depend upon the structure of the gel before immersion in water. A theory of membrane formation is proposed and used to explain the characteristics of membranes prepared in this and other researches. Diagrams show the drying apparatus for alcohol membranes and ethylene glycol membranes. P. H. P.

References to Scientific Articles not Abstracted.

RECENT ADVANCES IN TOXICOLOGY. W. WILLCOX. (Presidential Address to the Medico-Legal Society.) *Brit. Med. J.*, 1927, No. 3487, 828.

Advances in the methods of determining arsenic—Illustrative cases—Arseno-benzene compounds—Extraction of alkaloids—New drugs—Barbituric acid—Thallium acetate—Advances in forensic medicine.

UNRECOGNISED TOXIC SUBSTANCES IN HUMAN FAECES. D. C. WATSON. *Lancet*, 1928, **214**, 174 (Jan. 28).

Erroneous teaching on normal faeces—Toxins in human faeces—Experiments with the faeces of animals—Proof of presence of toxic substances in certain diseases—Diagrams of effects on blood pressure.

SOME NEW PRINCIPLES IN CHEMOTHERAPY. M. COPLANS and A. G. GREEN. *J. Soc. Dyers & Col.*, 1928, **44**, 5.

Chemotherapy of dyestuffs—Experiments with leuco dyes—Action of leuco dyes upon the animal organism and bacteria—Experiments with dyes containing labile acidic groups—Internal antisepsis by means of sulphato compounds—Summary.

Reviews.

THE HIGHER COAL-TAR HYDROCARBONS. By ARTHUR ERNEST EVEREST, D.Sc., PhD., F.I.C. Pp. xiv+334. With Diagrams. London: Longmans, Green & Co., Ltd. 1927. Price 18s. net.

This book is a critical summary of the present knowledge of the higher coal-tar hydrocarbons. It is divided into four chapters dealing, respectively, with (1) the acenaphthene group (acenaphthene and aceanthrene), (2) the fluorene group (fluorene, fluoranthrene, and truxene), (3) the phenanthrene group (phenanthrene, retene, chrysene, pyrene, picene, perylene, and phenanthroanthracene), and (4) a miscellaneous group (benzerythrene, crackene, chrysogene, succisterene, and synanthrene). Some of the hydrocarbons discussed have not been found in coal-tar, but have been included owing to their relationship to those which are present.

The section on each hydrocarbon opens with an account of its discovery, and of the analytical and synthetic methods which have been employed to fix its structure, the reasons for the adoption of the graphical formula finally assigned being clearly set out. Derivatives of the hydrocarbon are then discussed, very thoroughly, as the subject index shows. The author has paid particular attention to the industrial applications of the compounds described, and to stressing points where further research is necessary.

The lack of a standard method of numbering condensed ring systems is a real difficulty in the study of the higher hydrocarbons. The author has met this difficulty by using the numberings tentatively proposed by the "Committee on Organic Nomenclature of the International Union of Pure and Applied Chemistry," except for those hydrocarbons for which a different system is generally accepted.

Complete references are given to the academic, technical, and patent literature, but it is to be regretted that for all journals, except the *Journal of the Chemical Society*, the volume number has usually been employed in the citations without the year of issue. It is difficult to obtain historical perspective from a series of references based on volume numbers only. It might also be of advantage to include, with the numbers of the patents cited, references to the corresponding abstracts in, for example, the "B" issue of the Bureau of Chemical Abstracts, since patent specifications are in no way as available as journals.

The author is to be congratulated on this monograph, which fills a real gap in chemical literature. A few printer's errors have been noticed, particularly in the graphical formulae, but, in view of the complexity of these, this is only to be expected.

T. S. WHEELER.

DIE IODZAHLSCHNELL METHODE UND DIE UEBERIODZAHL DER FETTE. By B. M. MARGOSCHES. Pp. viii+227. Stuttgart: F. Enke. Price 16.20 *m.* paper covers, 17.10 *m.* bound.

This is Volume XXV in a series of monographs covering the whole range of chemical and physico-chemical analysis. It is based, in the main, on experimental work carried out in the laboratory of the Technical High School in Brünn, and is essentially a detailed study of the effects of the combined action of alcohol and water on the absorption of iodine by fats and fatty acids. Hübl's method of iodine absorption was for many years taken as the standard of comparison for any new method of determining the iodine value of oils and fats. Then Wijs showed that the action of iodine monochloride was analogous to that of the Hübl reagent, but was much more rapid, and the Wijs method in turn became the standard. Later work has shown that the unsaturated double bonds of fats may behave differently in their absorptive capacity, and it has been proved that, even with the Wijs reagent, bonds in certain positions in the molecule react much more slowly than those in other positions; this different absorptive capacity accounts for the fact, recently demonstrated by Toms, that one of the double bonds of tung oil fatty acid absorbs bromine vapour very rapidly, whereas days are required to saturate it with Wijs's iodine monochloride solution. It is this difference in the behaviour of the double bonds of fatty acids which justifies the study of further methods of halogen absorption; if results were always identical with those given by the Wijs reagent, there would be little object in adding to the number of reagents already described; as it is, preferential absorption is a hopeful avenue to explore.

In the author's method, which, as mentioned above, consists in the direct absorption of iodine in presence of alcohol and water, the conditions for rapidly obtaining accurate results have been exactly determined. Advantages of the method are that only one halogen is used, and that under specified conditions, which have been worked out in detail, it is possible to distinguish between certain oils and fatty acids, such as olive oil and castor oil, which have similar iodine values, as determined by the Wijs method. For this purpose the ordinary iodine value, as given by the author's reagent after five minutes' absorption, is compared with the "super-iodine value," which is defined as twice the amount of hypo-iodous acid (expressed as percentage of iodine) which is consumed in 24 hours by the action of alcoholic iodine solution acting, in the presence of water, on fats or fatty acids. It is proved, by a long series of curves, that a definite ratio exists between the rate of absorption of the iodine and the constitution of the different series of fatty acids and their glycerides. It is not possible to go into the details of these ratios here, but enough has been said to indicate the nature of this work, which is a very valuable contribution to the analytical chemistry of oils and fats.

EDITOR.

COLLOIDS. By H. R. KRUYT. Translated by H. S. VAN KLOOSTER. xi+262. London: Chapman & Hall, Ltd. 1927. Price 17s. 6d. net.

The author states that it is his purpose "to offer a main line of orientation to students who wish to become acquainted with the general trend of Colloid Chemistry or who desire to undertake research in this particular branch of Chemistry." The methods of physical chemistry have been chosen to outline the path of the necessary studies, and personal opinion is frequently given. This is an advantage, as the author is deservedly eminent in this field.

The book is divided into four parts: General Introduction; Suspensoids; Emulsoids; Special Cases. The text is very clear, the arguments logical, and the subjects treated are of fundamental importance. Particularly good are the pages devoted to capillary electric phenomena and charge and stability. The Helmholtz double layer is clearly dealt with.

The reviewer is of the opinion that the book is well suited as an introduction to the standard work by Freundlich. The treatment is not too elementary, emphasis is placed on the really important points of theory, and modern work and conclusions are kept constantly to the fore.

The indexes are good, and the publishers have also done their part well.

WILLIAM CLAYTON.