NOVEMBER, 1923. Vol. XLVIII., No. 572.

THE ANALYST

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

An Ordinary Meeting of the Society was held on Wednesday, October 3rd, at the Chemical Society's Rooms, Burlington House. The President, Mr. P. A. Ellis Richards, F.I.C., was in the chair.

Certificates were read for the first time in favour of:—Messrs. Laurence Eversley Campbell, M.Sc. (Lond.), F.I.C.; John Troubridge Hannen, B.A.(Cantab.), A.R.C.Sc., A.I.C.; Cyril Langley Hinton, F.I.C.; Douglas William Kent-Jones, B.Sc. (Lond.), F.I.C.; Thomas William Alan Shaw, M.Sc. (Liv.); William Hall Simmons, A.I.C.; Kenneth Edward Nethercoate Williams; Percy Noel Williams, M.Sc. (Liv.), A.I.C.

Mr. Robert C. Grimwood, A.C.G.F.C., A.I.C., was elected a member of the Society.

The following papers were read:—"The Sampling of Coal; the General Problem and some Experiments," by J. H. Coste, F.I.C., E. R. Andrews, F.I.C., and W. E. F. Powney, F.I.C.; "A New Test for Distinguishing Castor Oil," by H. G. Stocks, F.I.C.; "The Volumetric Estimation of Vanadium in Steel," by A. T. Etheridge, B.Sc., F.I.C., M.B.E.; and "The Iodimetric Determination of Sugars," by C. L. Hinton, F.I.C., and T. Macara, F.I.C.

Obituary.

WILLIAM THOMSON.

By the death of William Thomson, on October 4th, an outstanding figure in local scientific circles, and probably the doyen of the analytical profession in the North of England, has been removed with tragic suddenness. Ever a man of activity, concentrating on the problem of the moment, he suffered a sudden attack of cerebral hæmorrhage when examining some photographic plates in his laboratory, and, to those of us who knew him well, it appears strangely fitting that he should have passed away so painlessly amidst familiar sounds and surrounded by his valued "instruments of office."

Born at Glasgow in 1851, and educated at the Andersonian College, Thomson, when in his nineteenth year of age, came to Manchester to act as assistant to the late Dr. Crace-Calvert, whose laboratory, occupying the basement of what was formerly the Old Royal Institution building, and is now the City Art Gallery, dates back to, at least, the fifties of last century. Some four years later, and within a few months of Dr. Crace-Calvert's death, he was taken into partnership, and for half a century continued the analytical and consulting practice which, since the passing of the Sale of Food and Drugs Act, included the appointment of Public Analyst for Stockport. In the centre of Britain's busiest textile area, and surrounded by a multiplicity of chemical and other manufacturing interests, our deceased colleague steadily increased a reputation for accuracy and thoroughness, and his professional services in such directions as the technology of bleaching, dyeing, calico printing, etc., chemico-legal inquiries, arbitration proceedings, and so forth, were eagerly sought and highly appreciated. Frequently appearing in the Law Courts, his evidence, particularly in cases of water pollution and riparian rights, was essentially practical and unambiguous, and often won the encomiums of the Bench. In his official capacity of Public Analyst, his services gained the complete confidence of the Stockport Authority for whom, also, he acted as Water Examiner.

With an alert and inquiring mind, Thomson took an exceptionally active part in the work of the various scientific and technical societies of Manchester; indeed, the writer can think of no more familiar figure at the accustomed meetings throughout the session, and both the diverse character of original papers presented, and his many-sided contributions to the ensuing discussions, were sufficiently indicative of his ripe experience and broad mental outlook. With a seat on the Council for many years, and President from 1917–19, this year marked his jubilee as a member of the Manchester Literary and Philosophical Society, to which he freely contributed in service and material. About a year ago, he presented to the Society the original specimens of the sulphides of calcium, barium, etc., to the phosphorescent properties of which he drew the attention of Crookes in 1877. In a personal letter, dated April 28, 1897, Crookes wrote:

> "I was much interested in reading a copy of our correspondence in 1877 and 1879. It takes one back to the very beginnings of my radiant matter experiments and phosphorescence in high vacua which have been so splendidly carried on by Lenard and Röntgen. When I have occasion to write anything about the early history of the X rays it will be very useful to have these letters to refer to, and I will not fail to mention the part you played in the progress of the research when you drew my attention to the phosphorescent properties of the calcium sulphide."

Other papers of special interest to be found in the Proceedings of the Society, include the results of a painstaking investigation of the degree of atmospheric pollution in the centre of the city, and a record of his work on the electrolytic determination of arsenic in beer and foodstuffs generally.

An original member of the Society of Chemical Industry, he took a very live interest in the work of the local Section, occupying the chair from 1917–1919,

and freely did he give of his fund of knowledge and experience in the papers he presented. Other Societies, etc., whose welfare he had at heart were The Dyers and Colourists, the Manchester Geographical, the Textile Institute, and the Manchester and Salford Sanitary Association, whilst, for relaxation, the social gatherings of the local Caledonian Association and membership of the Manchester Playgoers' Club afforded him keen pleasure.

An original Fellow of the Institute of Chemistry (Member of Council, 1887–90, 93–96), and a Fellow of the Chemical Society (elected 1872), Thomson was awarded the Fellowship of the Royal Society of Edinburgh at the early age of 23.

His association with the Society of Public Analysts goes back to the dim and distant pioneer days, and just about 46 years ago we find him contributing his first paper to THE ANALYST "On the Incongruity of the Mode generally adopted in stating the Results of Milk Analyses." His other communications to our journal included "The Detection and Estimation of Mineral Oil," "Estimation of Fat in Milk," "Ferment produced by the morbid Growth of the Bioplasm of the Yolk of Egg," "Presence of Arsenic in the Body and its Secretion by the Kidney," "Estimation of Minute quantities of Arsenic," and "Estimation of Indigotin in the Presence of Starch."

The recital of a man's success in life, his business or professional attainments, however admirable, is incomplete without a brief reference to his personal qualities. There was a richness and sympathy in Thomson's nature, despite his quiet reticence, which endeared him to many. Given the environment, he was a most companionable soul, possessed of a shy geniality which was tinctured with the pawky humour typical of so many of his compatriots. Just and generous in his dealings, and of a charitable disposition, the record of a life such as his cannot fail to leave a hopeful impulse behind it in the world and better the tradition of mankind.

S. ERNEST MELLING.

The Estimation of Lactose by the Polarimetric and the Gravimetric Methods.

BY A. L. BACHARACH, B.A., A.I.C.

(*Read at the Meeting, May* 2, 1923.)

IT became necessary in this laboratory to make use of the polarimetric constants for lactose when the strong green band of the mercury vapour arc is the source of illumination.

Records of only two previous recorded determinations of $[\alpha]^{20}_{\lambda=546}$ were found. An examination of the value of $[\alpha]^{20}$ for different values of λ was made in 1912 (Grossman and Bloch, *Zeitsch. Ver. Deutsch. Zuck. Ind.* [*Tech. Teil*], **1912**, **62**, 61); the figures, however, do not include a direct determination for $\lambda=546$, the corresponding value for $[\alpha]^{20}$ being obtained by interpolation.

Moreover, no determination of the change with temperature of $[\alpha]$ for a given value of λ was made. This last consideration also rendered unacceptable to us the figure given by Quisumbing and Thomas (*J. Amer. Chem. Soc.*, 1921, **43**, 1503). Further, their value for $[\alpha]^{25}_{\lambda=546}$, 61·36, is open to other objections which are discussed later.

It is to be regretted that a certain amount of confusion has been introduced into the nomenclature of lactose. Four modifications of lactose may be recognised. First, there is the crystalline hydrate, which shows the characteristic phenomenon of bi-rotation. This is universally referred to as α-lactose (*cf.* Maquenne, *Les Sucres et leurs Derives Principaux*, 1900; Tollens, *Kurzes Handbuch der Kohlenhydrate*, 1898; Richmond, *Dairy Chemistry*, etc.). Secondly, there is the anhydride of α-lactose, a hygroscopic substance prepared by heating α-lactose at about 130° C. This has been designated β-lactose by Tollens, though "β-lactose" is used for at least two other modifications by other writers (Maquenne, Richmond, Hudson). The use of the term α-lactose-anhydride for this substance avoids confusion.

Thirdly, there is the non-hygroscopic anhydrous modification of lactose, prepared by concentrating aqueous solutions of α-lactose at or above 100° C. This substance exhibits the phenomenon of "hemirotation," and is designated γ-lactose by both Tollens and Maquenne, whereas both Richmond and Hudson (*J. Amer. Chem. Soc.*, 1908, **30**, 1767) call it β-lactose. We shall throughout use the term β-lactose for this modification, since it has been shown to have the constitution β-glucose-β-galactoside.

Finally, there is the modification which exhibits on solution in water neither bi-rotation nor hemi-rotation, and is prepared by evaporation or alcoholic precipitation of an aqueous solution of α-lactose under carefully controlled conditions. This is presumably not a fresh modification at all, but simply a mixture of α-lactose and β-lactose in the proportions in which they are present when in equilibrium in aqueous solution. This is called β-lactose by Maquenne; the other authors mentioned above either make no reference to it, or give it no special name. This substance has been described in various degrees of hydration; obviously the composition of the true equilibrium mixture would correspond with the formula $C_{12}H_{22}O_{11}$, $\frac{1}{2}H_2O$. Any departure from this composition must be due to excess of one or the other of the α- or β- modifications.

It was decided to make, on a sample of purified lactose, the following determinations:

(1) Value of $[\alpha]^{20}_{D}$; (2) value of $[\alpha]^{20}_{\lambda=546}$; (3) Reducing action on Fehling solution.

Attempts at establishing the purity of this lactose by other quantitative methods failed, never even approaching an accuracy of 1 in 1000. It will be seen from the method of preparation that the aim was to obtain a specimen of crystalline lactose monohydrate, $C_{12}H_{22}O_{11}$, H_2O, with a maximum impurity of 0·1 per cent.

PREPARATION OF PURE LACTOSE.—A sample of lactose of B.P. standard was re-crystallised twice by dissolving it in boiling distilled water, and then cooling the solution to room temperature. The product was washed with cold water, air-dried at atmospheric temperature, and finally dried for one hour at 102° to 103° C. After it had been ground and passed through a copper-gauze sieve of about 30-mesh, the product was bottled and kept in a vacuum desiccator. On being examined for impurities, it showed no ash, negative test for the presence of sulphate, and the merest traces of protein, chloride, and phosphate.

The sample showed no loss of weight at 98° to 99° C., either under atmospheric pressure or under diminished pressure. When, however, it was heated under atmospheric pressure at 102° to 103° C. it showed a small progressive loss in weight, which was roughly proportional to the time of heating; this amounted to less than 2 per cent. in 20 hours, and can only have been due to slow removal of part of the water of crystallisation.

An attempt to dehydrate this specimen of α-lactose quantitatively failed, as the temperature necessary to remove the whole of the water of crystallisation was also sufficient to bring about partial decomposition of the carbohydrate molecules. It was also found impossible to convert a sample quantitatively into the β-modification; only 4·8 per cent. of water was lost, instead of the theoretical quantity of 5·0 per cent.

It was decided, therefore, to rely on the qualitative examination as proof of purity, and to check this by a polarimetric determination with sodium light, since the value of $[\alpha]_D^{20}$ has been accurately determined. The value given by Grossman and Bloch is 52·42°, and this is accepted by, amongst others, Pfansteil and Black (*J. Ind. Eng. Chem.*, 1921, **13**, 685).

Accordingly, an accurately prepared five per cent. solution was made up at 15·5° C. in a previously checked standard (D.R.) flask, and the rotation of this solution was determined in a water-jacketed 400 mm. tube, certified by the N.P.L. as accurate at 16·5° C. to $\pm$0·1 mm. Three readings were taken at three different temperatures, which were measured by one of a set of Anschutz thermometers; this had been checked against a N.P.L. standard thermometer, and showed a maximum divergence of $\pm$0·1 degree.

The values found were:

Temperature.	Angular Rotation.
15·3° C.	10·55°
19·2° C.	10·46°
28·1° C.	10·37°

These values were plotted as shown in the lower part of Fig. 1; after making a correction for the change in concentration between 15·5° C. and 20° C., the following expression was obtained:

$$[\alpha]_D^t(c=5)=52{\cdot}42+(20-t)\times 0{\cdot}072.$$

This, it will be seen, agrees with Grossman and Bloch's figure. It is known that concentration has, within wide limits, no effect on the specific rotation; it was not,

therefore, considered necessary in this case, or with the mercury-lamp, to take readings with solutions of different concentrations.

DETERMINATION OF $[\alpha]^t_{\lambda=546}$.—The same solution used above was examined by means of the green light of the mercury-vapour arc (Fig. 1, upper part). The readings obtained were:

Temperature.	Angular Rotation.
15·05° C.	12·48°
19·50° C.	12·35°
26·50° C.	12·28°

From this we obtain:

$$[\alpha]^t_{\lambda=546}(c=5) = 61{\cdot}94 + (20-t) \times 0{\cdot}085.$$

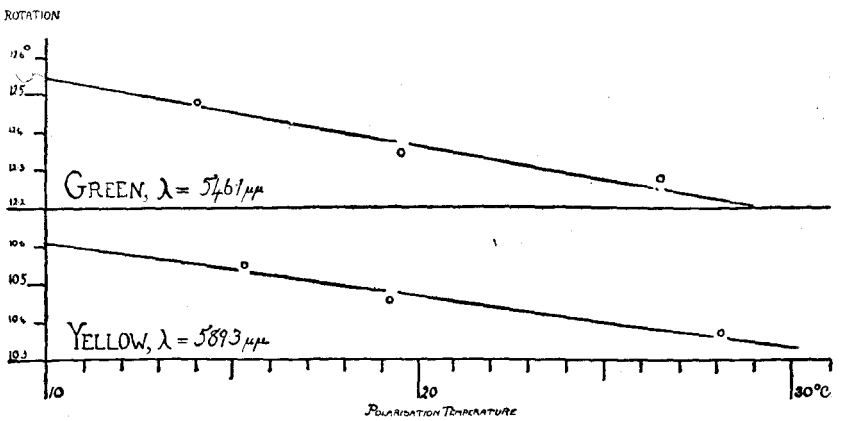

FIG. 1.

The figure obtained by interpolation, or by graphical methods, from Grossman and Bloch's determination is between 60·0° and 60·5°; the figure given by Quisumbing and Thomas for $[\alpha]^{25}_{\lambda=546}$ is 61·36°, whereas from the above formula we get: $[\alpha]^{25}_{\lambda=546} = 61{\cdot}51°$.

By calculation from the simple expression

$$\frac{[\alpha]_{\lambda_2}}{[\alpha]_{\lambda_1}} = \left[\frac{\lambda_1}{\lambda_2}\right]^2$$ and by taking $[\alpha]^{20}_{D} = 52{\cdot}42$ we get $[\alpha]^{20}_{\lambda=546} = 61{\cdot}03$.

Tabulating these results, there are obtained the following values for $[\alpha]^{20}_{\lambda=546}$:

Value found	61·94°
Value calculated	61·03°
Quisumbing and Thomas	61·79°
Grossman and Bloch	60·0–60·5°

Neither the calculated value, which is at best only a first approximation, nor Grossman and Bloch's figure, which is not derived from a direct determination,

need be considered; the question arises whether this new value is to be preferred to that given by Quisumbing and Thomas.

Putting $$K=\frac{[\alpha]^t_{\lambda=546}}{[\alpha]^t_D},$$

then from the determinations described above $K=1{\cdot}180$;

and from Quisumbing and Thomas's determinations $K=1{\cdot}160$.

The discrepancy here is so great as to demand further examination of Quisumbing and Thomas's figure. They give $[\alpha]^{25}_D=52{\cdot}90°$, a figure far higher than any previously recorded. If this be merely an overlooked printer's error for 51·90°, then their value for K is 1·181, that is, identical with that calculated from the determinations here described.

In order to examine this point further, careful determinations were made of $[\alpha]_D$ and $[\alpha]_{\lambda=546}$ for a pure specimen of sucrose, supplied by the courtesy of the Chief Chemist of Messrs. S. Tate and Sons, Ltd., and dried for about 2 hours at 102°–103° C. The values found were:

$$[\alpha]_D=66{\cdot}47. \qquad [\alpha]_{\lambda=546}=78{\cdot}63.$$

Whence $K=1{\cdot}183$, a very good agreement with the figure calculated for lactose. If now the value of K be calculated for the various sugars used by Quisumbing and Thomas, the following results are obtained:

Lactose (corrected for printer's error)	1·180
Sucrose	1·177
Dextrose	1·182
Lævulose	1·174
Invert sugar	1·169
Maltose	1·171

The agreement here does not appear to be satisfactory. Since their "invert sugar" was made by taking equimolar solutions of dextrose and lævulose, it is particularly surprising not to find K for "invert sugar" intermediate in value between K for dextrose and K for lævulose.

In view, therefore, of the doubts regarding the polarimetric determinations of Quisumbing and Thomas, which were not integral parts of their work, but merely incidental thereto, the expression for lactose given above is suggested:

$$[\alpha]^t_{\lambda=546}(c=5)=61{\cdot}94+(t-20)\times 0{\cdot}085.$$

THE REDUCING POWER OF LACTOSE ON FEHLING SOLUTION.—Experience in this laboratory of the Government Laboratory method for estimating lactose in dried milk showed that it was by no means trustworthy, at any rate for the degree of accuracy required. Not only did the results fail to agree with polarimetric determinations (sodium light), but they were not consistent among themselves.

Several series of determinations were made on solutions of pure lactose, with the same unsatisfactory results. Thus, in one case a series of eleven determinations gave amounts of cupric oxide varying from 0·4135 to 0·4210 grm., of which only three were within ±0·0020 grm. of the mean. Fourteen different determinations of the "blank" were made alongside of these and other reductions, and the weight of cupric oxide formed was found to vary from 0·0010 grm. to 0·0105 grm., the variation being unco-ordinated with the shape, size, or material of the crucible used.

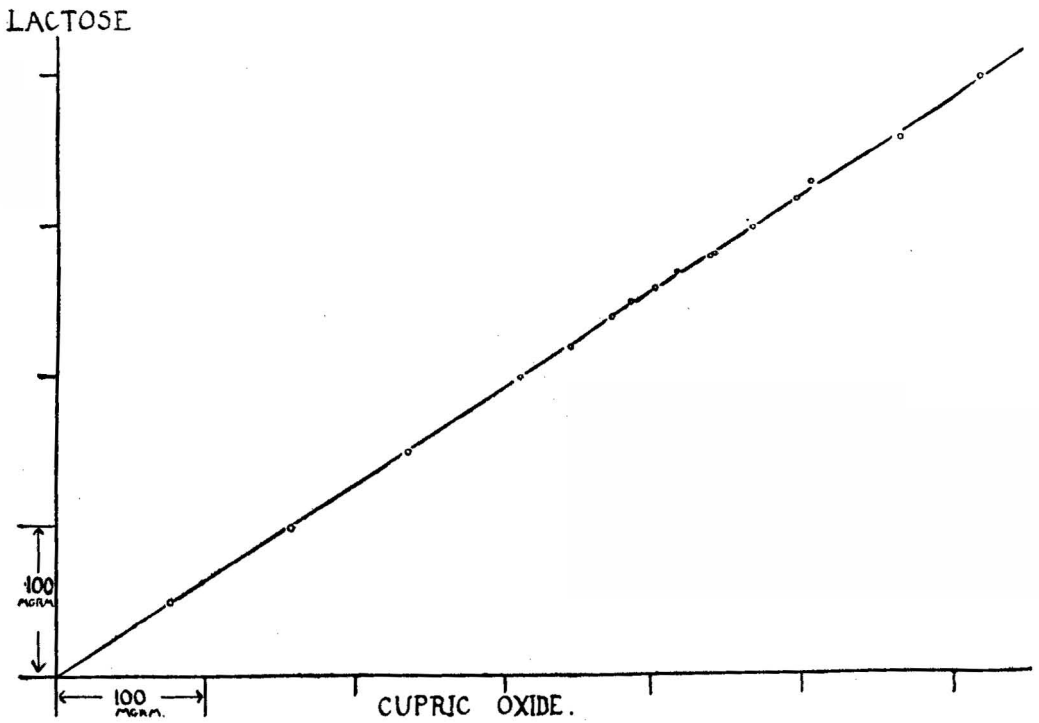

FIG. 2.

The criticism levelled by Quisumbing and Thomas at various gravimetric methods of estimating sugars, as well as the modifications in the Fehling's method proposed by them, seemed so reasonable that it was decided to make a series of determinations, following their technique as closely as was practicable. The chief features of their method are described in the following abstract (ANALYST, 1922, 47, 27):

> Twenty-five c.c. each of the copper sulphate solution and the alkaline tartrate solution are placed in a beaker 9 cm. in diameter, 50 c.c. of the sugar solution are added, and the beaker is covered and placed in a water-bath at 80° C. After exactly thirty minutes the cuprous oxide is collected on an asbestos filter, washed in the usual way, and the copper then determined, preferably by the electrolytic method. The copper sulphate solution should contain 82·4 grms. of crystallised copper sulphate per litre; the alkaline tartrate solution is prepared by dissolving 346 grms. of crystallised Rochelle salt in water, adding 130 grms. of sodium hydroxide (in the form of a concentrated solution made by dissolving purified sodium hydroxide in a small quantity of water and separating the clear solution from settled carbonates, etc.), and diluting the mixture to 1 litre.

The method used by us differed from theirs in one important respect, and in a number of details. The copper was throughout estimated by igniting the cuprous oxide formed. The oxidation to cupric oxide was carried out in a porcelain Gooch crucible heated to redness inside an opaque silica crucible. It has also been found that oxidation takes place quite easily in an electric muffle furnace. Temperature control during reduction was maintained by means of an ordinary gas-mercury thermo-regulator, whereby the temperature could be kept within 0·5° of 80° C. when the regulator was working well. On other occasions a somewhat larger divergence from the mean was observed, but it was always possible, when greater accuracy was required, to keep the temperature within the limits mentioned.

The claims made by Quisumbing and Thomas have, in general, been completely substantiated; indeed, their method has been introduced for routine sugar determinations, when these have to be done gravimetrically, in this laboratory.

On no occasion has a blank of more than 0·0005 grm. been found; generally no change in weight at all could be observed in the crucible after passing the solution through it, and igniting.

A series of preliminary determinations was made, and the results are plotted in the graph shown as Fig. 2. This entirely confirms Quisumbing and Thomas's claim that the amount of copper reduced is directly proportional to the amount of sugar present. The departures from the straight-line relationship are, it will be noticed, quite haphazard, and can be accounted for by slight variations in the quantity of solution delivered by the checked standard (D.R.) pipettes used and by inadequate temperature control.

From the values so obtained:

$$R = \frac{100 \times \text{weight of copper oxide formed}}{\text{weight of sugar reacting}} = 156{\cdot}6.$$

From Quisumbing and Thomas's factor—R = 154·0.

There is obviously a considerable discrepancy here, and a further series of reductions was accordingly made. In this series the temperature of the bath was very carefully watched, and was kept between 79·5° C. and 80·5° C. during the whole of the reductions. Further, the solution was delivered from a standard (D.R.) burette, carefully cleaned and dried; the reading of the burette was only taken when, after three or four minutes, there was no rise in the level of the meniscus. It will be noted that a remarkably consistent set of results was thus obtained; these are shown in Fig. 3.

The value for R given by these determinations was 156·5, still further removed from Quisumbing and Thomas's value. As these workers had invariably weighed, and not measured, their sugar solutions, the precaution was taken of carrying out two determinations with weighed quantities of lactose.

These determinations established clearly that no error commensurate with the difference between the figures given above and those of Quisumbing and Thomas was introduced by measuring the lactose solution for reduction.

The sample of lactose which gave R=156·5 was identical with that used for the polarimetric determinations.

If the difference between the values given above and those found by Quisumbing and Thomas is solely due to differing degrees of purity in the samples of lactose, then, provided the impurities are optically inactive and do not affect the

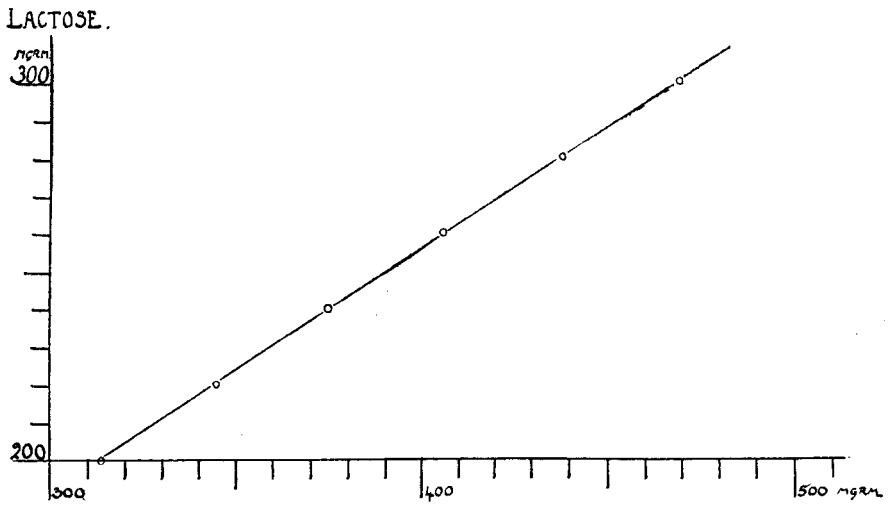

FIG. 3.

reduction of Fehling solution (*e.g.* moisture, mineral salts, etc.), the ratio $\frac{10\times[\alpha]^{20}_{\lambda=546}}{R}$ should be the same in both cases.

	$10\times[\alpha]^{20}_{\lambda=546}$	R	Ratio
Quisumbing and Thomas	618·1	154·0	4·01
Bacharach	619·4	156·5	3·96

There is thus a considerable difference between the set of results given here and those obtained by Quisumbing and Thomas; about 1½ per cent., of which only ½ per cent. can be explained by the presence of moisture, mineral matter, etc., in the lactose used by them.

SUMMARY.—(1) Careful redetermination of the polarimetric constants of lactose gave the following results:

$$[\alpha]^{t}_{D} = 52\cdot40+(t-20)\times0\cdot072,$$
$$[\alpha]^{t}_{\lambda=546}=61\cdot94+(t-20)\times0\cdot085.$$

(2) The modified Fehling method described by Quisumbing and Thomas was found satisfactory in the respects claimed by them.

(3) The ratio $\frac{100\times\text{cupric oxide}}{\text{Lactose}}$ found was 156·5.

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Estimation of Fat, Lactose, and Moisture in Dried Milks.

By H. JEPHCOTT, M.Sc., F.I.C.

(*Read at the Meeting, May* 2, 1923.)

Experience has shown that the examination of milk powders presents, particularly with respect to the estimation of fat and sugars, certain difficulties which are not apparent at first sight.

These difficulties are largely due to two factors. The first, and probably the most important when the methods commonly used for liquid milks are employed, is the eightfold multiplication of any error; a difference which would be passed as experimental error on a liquid milk becomes, upon a dried milk, one of considerable consequence. The second is the peculiar nature of milk powder.

The control of the manufacture of a standardised dried milk has involved the examination of a very considerable number of dried milks. The opinions here expressed are based upon the examination of more than 25,000 samples.

Moisture.—Owing to the hygroscopic nature of milk powder, it is essential to use weighing-bottles with well-fitting stoppers for moisture estimations.

By using a water-oven working at 99 to 100° C., and taking about 1 grm. of powder in a weighing-bottle 1″ × 1″, constant weight is reached in 2 hours, even with samples containing 6-8 per cent. of moisture. In most cases all moisture is lost in 1½ hours.

No considerable darkening occurs either in a water-oven or in an electric-oven working at 102 to 103° C. in less than 4 hours. After that time, appreciable decomposition may result at 103° C.

Milk powders may contain from 1–8 per cent. of moisture. On exposure, powders of low moisture-content rapidly absorb moisture until saturation is reached, usually at 6 to 8 per cent. of moisture; the total amount of moisture absorbed depends upon the powder and the humidity of the atmosphere.

It is of great importance that samples under examination should be kept in well-stoppered bottles and, if samples were originally from packages not themselves airtight, it needs to be remembered that the moisture-content and, consequently, the analysis generally, may differ considerably from that when the powder was manufactured or packed.

Estimation of Fat: *By Direct Ether Extraction.*—All attempts to estimate the fat-content of milk powder by extraction in a Soxhlet apparatus have given results so far below those obtained by other methods that direct extraction may

be eliminated as inapplicable to milk powders. The figures obtained in one experiment, typical of several, were:

Time (hrs.)	Fat Extract. Per Cent.
1	21·95
3	22·35
7	22·70
9	23·70
21	23·70
Fat by Werner-Schmidt	= 25·0
,, ,, Röse-Gottlieb	= 24·8

Werner-Schmidt Method.—This method was employed at the Government Laboratory in the examination of the dried milks for the Local Government Board Report, and the details are described in Appendix I. of that report. For ordinary laboratory practice the boiling with acid in a beaker and subsequent transference to a tube is cumbersome, and it has been our custom to modify the process slightly as follows:

One grm. of milk powder is weighed into a hard glass boiling tube, 8″ × 1″, 8 c.c. of water are added, and 2 drops conc. ammonia solution (0·880). The mixture is gently boiled until all lumps are dissolved. Ten c.c. conc. hydrochloric acid (1·16) are then added, and the tube heated in a bunsen flame with constant gentle agitation. The time of first boiling is noted, and gentle boiling continued for 3 minutes. The tube is then allowed to cool somewhat, about 50 to 70 c.c. of ether added, and the ethereal layer blown off, after standing for a short time, by means of the usual wash-bottle fitting. On this first extraction the ether is not shaken with the acid mixture. The major portion of the fat is on the surface of the acid liquor, and readily dissolves in the ether. The omission of shaking reduces any possibility of loss with the first ethereal solution, which is by far the most concentrated in fat.

A second portion of 50 to 70 c.c. of ether is run on, and the tube well shaken, allowed to stand for at least 30 minutes, and then blown off into the same flask. A further third extraction may then be made if necessary. This will depend upon the fat-content of the sample under examination, and how closely to the acid layer the ether has been blown off.

The ether is recovered by distillation, the flask dried at 102° C. for 1 hour, any ether remaining being removed by blowing with a small bellows, and the flask cooled and weighed. The fat is extracted from the flask with petroleum spirit, three rinsings of about 10 c.c. each being used, and the flask again dried as before, cooled, and weighed. The difference in the weight is taken as fat.

The precise time of boiling does not appear to be material, provided that it is sufficient and not excessive. Experiments carried out on the same sample with varying periods of boiling showed but little variation.

Time of Boiling.	Extract Per Cent.
2 minutes	25·05
2½ ,,	25·02
3 ,,	25·08
4 ,,	25·16
4½ ,,	25·37

It is noticeable, however, that with increasing time of boiling, an increasing quantity of substance soluble in ether but insoluble in petroleum spirit is produced, and in the course of the petroleum spirit extraction some of this may be mechanically lost, and consequently reckoned as fat. We find that with 3 minutes' boiling,

such insoluble matter is not produced, and error from this source unlikely, whilst the emulsion is entirely destroyed, and there is little or no risk of incomplete solution, even with a stale milk powder, the proteins of which are practically insoluble in water.

With a view to determining the ability of different workers to obtain concordant results, several estimations upon the same sample of milk powder were made by five workers, the method employed being as described above, except that the flasks were dried to constant weight both before and after dissolving out the fat with petroleum spirit. All weighings were made upon the same balance by one person.

Boiled by.	Extracted by.	Condition of Fat.	Fat Found Per Cent.
B	B	free from pet. spirit insoluble	25·15
B	B	,, ,, ,,	25·15
B	F	,, ,, ,,	25·15
F	B	,, ,, ,,	25·25
F	F	slight pet. spirit insoluble	25·05
F	F	,, ,, ,,	25·25
F	J	,, ,, ,,	25·15
J	J	,, ,, ,,	25·25
J	F	free from pet. spirit insoluble	25·15
D	D	slight pet. spirit insoluble	25·20
D	A	appreciable pet. spirit insoluble	25·25
A	A	slight pet. spirit insoluble	25·0
A	A	,, ,, ,,	25·1
A	D	free from pet. spirit insoluble	25·15
		Average ..	25·16

These results would appear to prove that the method will give concordant results.

There are one or two precautions which it is desirable should be taken, since it should be remembered that an error of 1 mgrm. is equivalent to 0·1 per cent. of fat.

Unless the indiarubber stoppers used in the extraction are well-fitting and rinsed into the tube, slight losses may occur. If tubes are placed in cold or running water after agitation until quite cold, there will be no liability to spitting on removing the stopper.

Ether, water, and petroleum spirit vapour must be completely removed from the flask; lack of care, especially with respect to the first weighing, may result in an error of as much as 6 mgrms., and will often amount to 2 to 3 mgrms., *i.e.* may make the result 0·5 per cent. in error. Flasks should be blown out three or four times during drying.

Care should be taken to avoid blowing over any of the acid liquor. If this is done, it is usually better to decant into a clean flask, and rinse the dirty flask with a little ether. The presence of any considerable amount of "tish" insoluble in petroleum spirit involves distinct danger of loss during petroleum spirit extraction, and consequently an apparently high figure for the fat.

Röse-Gottlieb Method.—When carried out with care, we find this method to give satisfactory results, but there is considerable liability to obtain incomplete solution of the milk powder and, consequently, to fail to extract all the fat. This is particularly the case when dealing with stale and insoluble powders. Estimations by this method showing low results have frequently come under our notice.

Moreover, where many determinations have to be carried out the accumulation of mixed ether and petroleum spirit residues is a nuisance, and the method proves expensive without any added advantage.

A modified Röse-Gottlieb method is used in the United States Department of Agriculture, as follows:

> Weigh 1 grm. of the powder into a 30 c.c. lipped beaker, rub up with 9 c.c. of water and 2 c.c. of conc. ammonia, digest on steam-bath until casein is well softened, and the whole resembles milk. Cool, transfer to Rohrig tube or similar apparatus, using 10 c.c. of 95 per cent. alcohol for rinsing, followed after shaking contents of tube, by 25 c.c. washed ethyl ether. Shake vigorously for one-half minute, and proceed as in the Röse-Gottlieb method.

This, we have shown, causes considerable and variable saponification of the fat, giving results as much as 2 per cent. too low. On acidifying the residual liquor a portion of, but, in our experience, not all, the saponified fat can be extracted as fatty acids.

The Association of Official and Agricultural Chemists have had three methods tested by their members:

(*a*) Practically the Werner-Schmidt, but without actual boiling. (*b*) The Röse-Gottlieb, with the use of 1 c.c. of ammonia solution (conc.). (*c*) The Röse-Gottlieb as in (*b*), but followed by boiling with 10 c.c. of hydrochloric acid for 5 minutes.

They state that too few dependable results were obtained for a definite opinion to be formed. The results certainly appear to include first efforts by workers unfamiliar with the methods.

Gerber Method.—For rough routine work where great accuracy is not required, we have found the Gerber method, modified as follows, to give good results:

> Ten c.c. of sulphuric acid (1·825) are delivered into the tube, a little cold water is added (to form a "cushion" and prevent charring of the powder), then 1 c.c. of amyl alcohol. 1·69 grms. of powder are next brushed into the tube through a glass funnel, and the tube filled to about $\frac{1}{8}''$ from the neck with hot water. The tubes are shaken until all lumps are broken down, whirled for 8 minutes, immersed in water at 70° C. for 3 minutes, read off, re-whirled, and again read. Taking reading to bottom of meniscus, $\frac{2}{3}$ reading = percentage of fat in powder.

It is very essential, however, that Gerber tubes of great accuracy (maximum error 0·25 of a small division) be used. All our tubes are standardised in our own laboratory. We find the results to agree, in general, with those obtained by the Werner-Schmidt with a variation of 0·3 per cent. In the case of a hundred samples recently examined, in which the fat was estimated in New Zealand by the Gerber method as described above, and in London by the Werner-Schmidt method, the results were as follows:

	Werner-Schmidt.					Gerber. (modif.)		
Average fat-content	25·57 per cent.					25·63 per cent.		
Difference from Werner-Schmidt, per cent.	0·7	0·6	0·5	0·4	0·3	0·2	0·1	0·0
No. of samples	1	1	9	7	22	21	24	15

These figures have all been taken from the laboratory records, and no special care had been taken in the estimations. They have an added usefulness in that they confirm the results obtained by the Werner-Schmidt method, while being entirely independent of that method.

LACTOSE.—For the routine estimation of lactose in milk powders polarimetric methods are rapid and convenient, but, as will be shown below, for other than routine estimations we have found certain difficulties which cast doubt upon their absolute accuracy.

Re-determination of the specific rotation of pure lactose has been made with very great care, and we find lactose ($C_{12}H_{22}O_{11}$, H_2O) to have specific rotations for sodium light, and for the green band of the mercury vapour arc as follows:

$$[\alpha]^t_D = 53{\cdot}86 - 0{\cdot}072t,$$
$$[\alpha]^t_{\lambda=546} = 63{\cdot}74 - 0{\cdot}09t.$$

We now use the green band of the mercury vapour lamp exclusively, since by this means one can obtain a light which is both steadier and very much more intense than any sodium flame with which we are familiar. By this means it is possible to use a 400 mm. tube, and to take readings to 0·02 of a degree.

Ten grms. of milk powder are thoroughly mixed with water in a mortar and washed into a 100 c.c. flask, boiling water being added until the total volume is about 75 c.c. This solution is kept in a boiling water-bath for 35 minutes, and then cooled, 10 c.c. of acid mercuric nitrate solution added, and the whole made up to the mark, with thorough shaking.

The mixture is filtered, and the rotation of the filtrate determined in a 400 mm. tube. The percentage of lactose in the powder is calculated in the usual manner, allowance being made for the volume of precipitated protein and fat on the basis of 0·8 for specific volume of protein, and 1·08 for specific volume of fat.

In order to ensure complete solubility and to destroy "bi-rotation," it is essential that the solution should be heated to boiling, and kept at 100° C. for a short time. During this heating, other changes besides bi-rotation appear to take place, and the angle steadily decreases with the length of time for which the solution is kept at 100° C., as the following instance will show:

A 10 per cent. solution of milk powder, prepared as described above, gave the following readings:

	Angle.
Raised to boiling point only	11·68°
1 hour on waterbath	11·29°
2 hours ,, ,, ...	10·90°
3 hours ,, ,, ...	10·64°
4 hours ,, ,, ...	10·26°
Raised to boiling point and left for 12 hours	11·49°

It is clear from these readings that some other change besides bi-rotation occurs on prolonged heating at 100° C. This change is clearly *not* the inversion of lactose, the rotation of which increases on inversion.

GRAVIMETRIC METHOD.—Our experiences with the method described in the Local Government Board Report upon Dried Milk have not been entirely satisfactory. High and variable blanks were frequently obtained, and there was often difficulty in obtaining concordant results. Whether this was due to faulty solutions or to other causes, we are not in a position to say.

A modified gravimetric method was described by Quisumbing and Thomas in the *Journal of the American Chemical Society*, **1921, 43**, 1503–1526; ANALYST, **1922, 42**, 37. These workers, after a fairly exhaustive investigation of the Fehling reduction, recommended solutions of a particular concentration and reductions at 80° C. Their cuprous oxide was ultimately estimated as copper by electrolysis.

We have investigated this method with some care, and have substituted estimation of the copper as cupric oxide as in the Brown, Morris and Millar method. On no occasion have we found a blank exceeding 0·5 mgrm., and, generally, no change in weight at all could be observed. Estimations with varying quantities of pure lactose up to the limit for 50 c.c. of the Fehling solution showed the ratio $\frac{\text{lactose}}{\text{CuO}}$ to be constant, and equal to 0·639, as the following table shows:

Lactose ($C_{12}H_{22}O_{11}$, H_2O) Mgrms.	CuO Mgrms.	Lactose / CuO Mgrms.
100	156·5	0·639
200	313·5	0·638
220	345·0	0·638
240	375·0	0·640
260	406·0	0·640
280	438·0	0·640
300	469·5	0·640
	Average	0·639

Adapting this method to dried milk we find the following procedure convenient:

A solution of the powder, 3 grms. in 250 c.c., is made up in the ordinary way with hot water, heated to boiling, and cooled to atmospheric temperature; the powder should be weighed to 5 mgrms. Fehling's copper solution is used as precipitant, the quantity taken being just slightly in excess of that needed to produce complete precipitation. The solution is then made up to the required volume, and shaken very vigorously for about half a minute. After filtration through a pleated filter-paper, 50 c.c. of the solution are reduced with 50 c.c. of Fehling solution for half-an-hour at 80° C. in a 240 c.c. "Kavalierglass" beaker, the beaker being immersed in the water-bath so that the level of the water outside is the same as that of the solution inside; the beaker is covered with a clock-glass of appropriate size. After the half-hour, the beaker is removed from the bath, the clock-glass rinsed with hot water, and the whole solution filtered through a porcelain Gooch crucible. The precipitate is collected in the usual manner and finally washed with a little 95 per cent. alcohol. After 10 minutes' drying in the oven at 102° C. the crucible is ignited inside a larger silica crucible or in a muffle, and the weight of cupric oxide determined after the crucible has been cooled in a desiccator.

Per cent. of lactose = CuO (in grams) × 106·5.

This result needs to be corrected for the volume of precipitated fat and protein.

In conclusion, I desire to acknowledge valuable suggestions and assistance from Mr. Norman Ratcliffe, F.I.C., of the Glaxo Laboratory, Hamilton, New Zealand.

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DISCUSSION.

Mr. H. DROOP RICHMOND said that it was well-known that if you heated milk sugar in a solution which contained ionisable salts the rotation decreased greatly. He had been in the habit of estimating milk sugar by polarisation, and he thought that, so long as you ensured that your milk proteins were removed, results of sufficient accuracy for ordinary purposes were obtained. He would like to draw attention to the fact that one could make a rapid and accurate estimation of milk sugar, after removal of fat and of proteins, by the iodine absorption method. He had made some careful comparisons of this method with other methods, which had proved quite satisfactory. He would like to suggest to the author that he should investigate the iodine method of estimating the milk sugar.

Mr. A. CHASTON CHAPMAN said that he felt that a careful revision of the properties of important compounds such as had been undertaken by the authors of these papers was of the highest importance. With regard to the copper reduction of lactose he had himself a good many years ago prepared some very pure lactose and had found that the amount of copper reduced was substantially proportional to the concentration of the solution,—or in other words, that a factor could be used for the calculation of percentages of lactose without any appreciable error. As regards the relation between the rotations for sodium light and ordinary white light he (Mr. Chapman) was under the impression that for the ordinary carbohydrates the relationship was an approximately constant one. In some of their classical work Brown and Morris had adopted the method of drying sugars in an evacuated vessel over phosphorus pentoxide, and he (Mr. Chapman) had found this method exceedingly satisfactory from the point of view of the avoidance of any decomposition. The Soxhlet method for the estimation of fat in materials such as infants' foods was liable to lead to very incorrect results, and was quite useless in the case of materials like casein. In such cases the Werner-Schmidt or some similar method should be adopted.

Mr. C. C. ROBERTS enquired if the method referred to in a previous paper in THE ANALYST, of drying oxalic acid in a current of air of definite humidity, would be applicable in the case of sugars such as lactose.

Mr. JEPHCOTT, referring to Mr. H. Droop Richmond's method, said that he had gone on blissfully with the polarimeter until he had found a half to two per cent. difference in his results; after that he had had no further confidence in it. He would try the iodine method put forward, and report his results to the Society. With regard to Mr. Chaston Chapman's contention that the Soxhlet method was obsolete, he agreed that the method was useless in the case of dried milk and similar material, and regretted that the fact was, nevertheless, not recognised by all chemists.

The Melting Point and Iodine Value of Refined Natural *D*-Camphor.

By MAURICE S. SALAMON, B.Sc.

(*Read at the Meeting, May* 2, 1923.)

While there are several methods available for estimating the percentage of camphor in preparations, such as spirits of camphor or camphorated oil, in which the camphor forms only a small proportion of the whole, there is no reliable method by which one can even approximately determine the camphor content of refined *d*-camphor, that is, camphor containing 99 per cent. and upwards of actual camphor.

The usual impurities present in refined camphor are traces of dirt and oil, and it is customary to judge the quality of refined camphor from the percentage of non-volatile residue and the melting point, the latter, of course, indicating whether or not there is much or little oil present.

Lane (*J. Soc. Chem. Ind.*, **1922, 41,** 32T.) has suggested the determination of the iodine value as a means of approximately estimating the oil percentage in crude camphor, and concludes from his figures that the iodine value of natural camphor is 1·4, whilst that of the oil is 130. Hence, from a determination of the iodine value, he deduces the approximate percentage of oil present in the crude camphor. Richardson and Walton (Analyst, **1908, 33, 466**) assume that the iodine value of camphor is 5 and that of the oil 180.

If the iodine value and melting point are to be reliable guides as to the quality of a sample of refined camphor, it is necessary to assure oneself that the published figures are approximately correct, but the examination of a considerable number of samples of refined camphor made over the last few years, leads me to believe that the melting point generally ascribed to pure *d*-camphor is too low, whilst the published iodine values both for camphor and camphor oil are too high.

Although all the authorities are agreed as to what is the melting point of inactive camphor, there appears to be considerable diversity of opinion as to what is the melting point of *d*-camphor.

The standard works give the following figures:

	d-Camphor.	*l*-Camphor.	Inactive Camphor.
Allen (latest edition)	175° C.		179° C.
Richter	175·0° C.		178·6° C.
Beilstein	176·4° C.	178·6° C.	178·6° C.
Victor Mayer	178·7° C.		
Thorpe's Dictionary (latest edition)	175–178·75° C.	175–178·75° C.	
B.P. 1914	About 175° C.		
U.S.P.IX.	174–177° C.		

If the melting point given for inactive camphor is correct, it is remarkable that the melting point of *d*-camphor should be 2 or 3 degrees lower, unless *l*-camphor

has a very high melting point; but all the authorities agree that this is not so, and state that the properties of *l*-camphor, apart from the rotation, are identical with *d*-camphor.

If, on the other hand, the figure given by Victor Mayer is correct, the melting point of inactive camphor should be identical with the melting point of *d*- and *l*-camphor, and this, in fact, is the case.

The figure of 175° C., given in the new edition of Allen, is a melting point obtained by Sadtler, who, in order, to obtain pure camphor, re-sublimed ordinary refined camphor with the aid of paraffin-wax; his theory being that the paraffin-wax would hold back any traces of oil that might be present in the camphor.

He apparently overlooked the fact that paraffin-wax and other waxes usually contain a trace of matter that is volatile at the high temperature necessary to sublime the camphor; and in all the experiments that I have made, using either paraffin-wax, carnauba-wax or beeswax, the melting point of the sublimed camphor has actually been lower than the melting point of the sample that I started with.

In order to prepare pure camphor, it is best to sublime it without the aid of any admixture, using about 5 grms. of camphor, subliming this in a fairly tall 100 c.c. beaker, and rejecting the first and last portions sublimed.

By this method there is no difficulty in obtaining samples of *d*-camphor having a melting point of practically 179° C., which melting point remained constant after repeated sublimation.

As regards the iodine value of pure *d*-camphor, which, according to Lane, is 1·4, determinations made on resublimed camphor of high melting point show that the iodine value does not exceed 0·1, and Lane's high value is probably due to the fact that the camphor he used for his experiments still contained a trace of oil.

It is extremely difficult to remove the last traces of oil, and it is probable that even the iodine value of 0·1 that I have obtained is due to a minute trace of oil still present in the camphor.

The iodine value of oil of camphor must vary according to the type of oil, but if the iodine value of the oil is to be used in order to determine how much oil is present in the camphor, it is necessary that the samples of oil taken for the iodine value determinations should be oil actually expressed from crude camphor.

It is almost useless to determine the iodine value of samples of camphor oil purchased in the ordinary way, as such oils are practically never identical with the oil present in crude camphor, but are oils obtained by fractionating the crude camphor oil, and they will therefore vary in iodine value according to whether they are what is known as light camphor oil or heavy camphor oil.

The oil that is expressed from ordinary crude camphor still contains, of course, a considerable amount of camphor in solution, and this must be removed if one wishes to ascertain what is the iodine value of the oil itself.

For my experiments I used samples of oil that I expressed from both Chinese and Japanese crude camphor, and having obtained the oil, I removed the dissolved camphor as completely as possible by repeated freezing and fractionation.

Mr. Lane has been good enough to communicate to me the iodine value of various samples of oil which he has examined; these are as follows:

		Specific gravity.	Iodine value.
A.	Oil purchased from local druggist (Hong Kong)	0·896	128
B.	Oil purchased from England	0·976	73
C.	Oil purchased locally	0·920	130
D.	Fraction boiling 164–167	0·900	129
	,, ,, 180–190	—	89
	,, ,, 197–199	0·889	213

The iodine values that I have obtained with the samples of oil special prepared are:

	Oil.	Specific gravity at 15·5° C.	Iodine value.
No. 1.	White in colour, obtained from Chinese camphor	0·937	91·7
No. 2.	Yellow in colour, obtained from Chinese camphor	0·975	86·8
No. 3.	Brown in colour, obtained from Japanese camphor	0·951	86·0

It will be seen that the values given by Mr. Lane are, as a whole, generally higher than the values that I have obtained. I ascribe this to the fact that the oils used by Mr. Lane are probably fractionated oils, and it will be seen from his experiments how much the iodine value varies in different fractions.

On the other hand, my values are probably a little on the low side, as it is practically impossible to free the oil completely from the dissolved camphor, but the amount of camphor still left in my samples of oil could only have been small.

If one assumes that the camphor present is about 10 per cent., and takes 100 for the iodine value of the oil, one is, I am certain, erring on the generous side, and by using this value one may slightly underestimate, rather than overestimate, the oil present in a sample of refined camphor.

The following are some of the figures obtained in the course of my experiments:

Sample.	Melting point. °C.	Iodine value.
English refined flowers	178·6–179·2	0·1
English refined flowers	178 –179	—
English refined slab	177·6–178·8	0·4
English refined tablet	177·4–178·8	0·5
Japanese refined flowers	176·6–177·4	0·4
Japanese refined flowers	178 –179	—
Japanese refined flowers	178 –179	—
Japanese refined slab	176 –177	0·5
Japanese refined slab	173 –175	—
Chinese refined slab	176 –177	0·6
Chinese refined slab	176·4–177·4	0·7
Chinese refined slab	171 –174	—
The same slab once sublimed	175 –177	—
Chinese refined tablets	176 –178	—
Camphor obtained by pressing Chinese crude camphor	175·8–176·6	1·3
The same after subliming 3 times	177·4–177·8	-
The same after subliming 4 times	177·8–178·6	—
The same after subliming 6 times	178 –178·8	0·1
*Chinese crude camphor	172 –175	3·3
The same after pressing	175·8–176·8	0·9

The melting points were determined in a capillary tube sealed at one end, the first temperature given being when the first distinct visible change was observed, and the final temperature being the temperature at which the camphor was completely melted. An Anschutz thermometer was used, thus avoiding the necessity for any stem correction.

The iodine value of the camphor was determined on about 1½ grms. of camphor, 10 c.c. of Wijs' solution and 10 c.c. of carbon tetrachloride being used; whilst the iodine value of the oil was determined on about 0·2 grms. of oil, one hour's standing being allowed in each case. I have varied both the amount of Wijs' solution and also the proportion of camphor without obtaining any different results.

The sample marked with an asterisk (*) contained approximately 2 per cent. of water, and it is therefore probable that the iodine value is a little low, but, taking it at the figure obtained and allowing for the 2 per cent. of water, the actual camphor content indicated would be approximately 94·5 per cent.

On pressing, the camphor lost 4·8 per cent., but gave a pressed cake that still had an iodine value of 0·9; and therefore contained a little under 1·6 per cent. of oil; hence from this experiment one would deduce that the original sample contained slightly over 94 per cent. of camphor, which really agrees well with the percentage deduced from the original iodine value and moisture content of the sample.

As regards refined camphor, there appears to be no difficulty in producing commercially a camphor having an iodine value of 0·7 or less and a melting point of at least 176° C., and I suggest that these might be the minimum requirements with which refined camphor that is to be used for pharmaceutical purposes should comply.

Discussion.

Mr. H. Droop Richmond said that he would like to suggest that the author had not removed the whole of the camphor by freezing out. In his experience with commercial camphor oil, after freezing out, the oil still contained about 15 per cent. of camphor. He would like to draw attention to the fact that the whole of the commercial camphor on the market had a melting point which agreed closely with the British Pharmacopœia, *i.e.* 176° C. He had found, in the course of the examination of hundreds of samples, that the melting point was remarkably constant about that point, which was a practical working limit.

Mr. M. S. Salamon, in replying, said he did not think that the amount of camphor left in the samples of camphor oil referred to in the paper was anything like 15 per cent. The method he used was first to freeze out, then distil and fractionate, then take the camphor fraction, freeze it again, remove the camphor, and add the portion not camphor to the remainder of the oil. He thought there might be 5 per cent. of comphor left in, certainly no more. He considered you could very well take, as a round working figure, an iodine value of 100. Much of the camphor on the market, known as "Japanese and Chinese refined slab," had a lower melting point than English flowers; English flowers usually had a melting point of 178° to 179° C.

Preservatives in Food.

HULL STATISTICS FOR THE TEN YEARS 1913 to 1922.

By ARNOLD R. TANKARD, F.I.C.

Over 7000 foods were examined, of which 9·0 per cent. contained preservatives. Excluding milks, 19·3 per cent. of the remaining foods were chemically preserved. Of all foods which habitually contain preservatives (excluding milks), 55·1 per cent. were chemically preserved by one or more of the preservatives referred to in the following tables:

(1.) FOODS CONTAINING BORON COMPOUNDS.

Food.	Total number of samples.	Number of samples preserved.	Percentage containing boron preservative.	Amounts of preservative expressed as boric acid, H_3BO_3. Maximum.	Minimum.	Ratio of maximum to minimum amounts of preservative.
				Grains per pint.		
Milk ..	3991	41	1·0	11·0	1·9	5¾:1
				Grains per pound.		
Dried milk ..	38	6	15·8	7·7	2·1	3½:1
				Per cent.		
Cream ..	255	225	88·2	0·67	0·05	13:1
Butter ..	275	46	16·7	0·43	0·03	14:1
Cheese ..	59	2	3·4	0·11	0·10	1:1
Margarine ..	164	149	90·9	0·36	0·02	18:1
Lard substitutes ..	33	4	12·1	Boric acid present in small quantity.		
Liquid eggs ..	6	6	100·0	1·70	0·80	2:1
Cake ..	9	8	88·9	0·48	0·03	16:1
Potted and other meats	69	50	72·5	1·27	0·02	63:1
Potted fish ..	20	12	60·0	2·52	0·04	63:1
Totals (including milks)	4919	549	11·0(average)	—	—	—
Totals (excluding milks)	890	502	56·4(average)	—	—	—

(2.) FOODS CONTAINING FORMIC ALDEHYDE.

Food.	Total number of samples.	Number of samples preserved.	Percentage containing formic aldehyde.	Amounts of preservative expressed as formic aldehyde, HCHO. Maximum.	Minimum.	Ratio of maximum to minimum amounts of preservative.
				Per Cent.		
Milk ..	3991	6	0·15	0·0025 (1 in 40,000)	0·00015 (1 in 670,000)	17:1
Ham	1	1	100·0	(present)	—	—

(3.) FOODS CONTAINING SALICYLIC ACID.

Food.	Total number of samples.	Number of samples preserved.	Percentage containing salicylic acid.	Amounts of preservative expressed as salicylic acid. Maximum.	Minimum.	Ratio of maximum to minimum amounts of Preservative.
				Grains per pint.		
Fruit juices, &c.* ..	108	43	39·8	8·4	0·1	84:1
				Grains per pound.		
Table jellies ..	9	4	44·4	7·0	2·8	2½:1
Jams ..	30	5	16·7	0·84	0·46	2:1
Bottled fruits ..	2	1	50·0	1·75		—
	149	53	35·6(average)	—		—

* Under this heading are included lime and lemon juices and cordials, medicated wines, non-alcoholic "wines," aerated waters and fruit syrups. One sample of "Sherry Cordial" contained 1·6 grain of benzoic acid per pint.

(4.) FOODS CONTAINING SULPHUROUS ACID.

Food.	Total number of samples.	Number of samples preserved.	Percentage containing sulphurous acid.	Amounts of preservative expressed as SO_2. Maximum.	Minimum.	Ratio of maximum to minimum amounts of Preservative.
				Grains per pint.		
Fruit juices, &c. ..	108	10	9·3	3·80	0·34	11:1

A sample of lime juice cordial, examined in 1920, was found to contain 1·2 grain of sulphurous acid (SO_2) per pint, and, in addition, 39·4 grains of free sulphuric acid (H_2SO_4) per pint.

(5.) FOODS CONTAINING BOTH SALICYLIC AND SULPHUROUS ACIDS.

Food.	Total number of samples.	Number of samples preserved.	Percentage containing both salicylic and sulphurous acids.		Amounts of Preservative. Maximum.	Minimum.	Ratio of maximum to minimum amounts of Preservative.
					Grains per pint.		
Fruit juices, &c. ..	108	8	7·4				
Showing maxima and minima of salicylic acid.				Salicylic acid	2·8 together with	0·28 together with	10:1
				SO_2	1·1	0·59	2:1
Showing maxima and minima of sulphurous acid.				SO_2	2·1 together with	0·59 together with	3½:1
				Salicylic acid	2·5	0·28	9:1

The total percentage of fruit-juices, etc., preserved in one way or another was therefore 56·5.

CITY ANALYST'S LABORATORIES,
HULL.

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.

EGG CUSTARD POWDER.

RECENT notices in THE ANALYST (1922, **47**, 512; 1921, **46**, 271) have called attention to the composition of "egg powders," and have shewn, in the instances given, that these articles were devoid of egg. Two specimens of "Real Egg Custard Powders" which I have recently examined gave the following analytical results:

	A. Per Cent.	B. Per Cent.
Moisture (loss at 100° C.)	10·9	12·75
Ash	0·97	1·60
Oil or fat (chloroform extraction) ..	6·2	7·1
Nitrogen	0·98	1·39
Organic phosphorus (as P_2O_5)	0·20	0·24
Copper reducing power (as anhydrous lactose)	0·23	3·71

From these figures it is to be concluded that A contained dried egg and that B contained both dried egg and dried milk. In B, lactose was positively identified. In each case the powder had a starch basis.

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16, SOUTHWARK STREET, S.E.1.

THE GELATIN TEST AND CATECHIN.

FREUDENBERG asserts that catechin is precipitated by gelatin [*Ber.*, 1920, **53**, 236; 1922, **55**, 1734, 1940; *Chemie d. naturlichen Gerbstoffe*, p. 120 (1920); *Handb. d. biologischen Arbeitsmethoden*, Sec. I., Vol. X., p. 524 (1920)]. This is contrary to all previous data [compare for example, Procter, *Leather Industries Laboratory Book*, p. 139 (1908), who states that: "Unlike tannins, it does not precipitate gelatin"]. It is also contrary to my own observations (*J. Chem. Soc.*, 1922, **121**, 26; *Berichte*, 1922, **55**, 3832). In this connection reference must also be made to Freudenberg's statement that Zwenger (Freudenberg gives *Annalen*, 1920, **37**, 324, the correct reference is, however, *Annalen*, 1841, **37**, 324) has also found that gelatin is precipitated by catechin. This is not correct, since Zwenger definitely states that: "Leimsolution wird nicht gefällt."

A recent statement by R. J. Browne (*J. Soc. Leather Trades' Chemists*, 1923, **7**, 375) deserves comment, as it apparently confirms Freudenberg's observations. Browne states that: ". . . the original catechin solution, on evaporating to dryness and dissolving in a minimum amount of water, also gave the gelatin-salt test." This is quite in agreement with my results, as prolonged boiling with water converts catechin into a tannin-like substance which precipitates gelatin. I therefore wish to emphasise the fact that a *freshly-prepared* solution of *pure* catechin does not precipitate gelatin, and that prolonged standing or boiling of the solution leads to the formation of tannin-like substances which are precipitated by gelatin. This obviously accounts for Browne's results, who *evaporates his catechin solution to dryness.*

M. NIERENSTEIN.

THE UNIVERSITY, BRISTOL.

A CASE IN WHICH WINCKLER'S MANGANOUS PROCESS FOR DISSOLVED OXYGEN IS UNTRUSTWORTHY.

WHEN it is sought to estimate the dissolved oxygen in solutions of ammonium chloride of concentration M/2 and upwards by Winckler's manganous process the precipitation of manganous hydroxide and its subsequent oxidation is very greatly retarded for the reasons which enable $Fe^{\cdots}$, $Al^{\cdots}$ and $Cr^{\cdots}$ to be separated from $Mn^{\cdot\cdot}$ in qualitative analyses. Gasometric estimations of the dissolved gases by Winckler's calcite and hydrochloric acid process show that ammonium chloride in solution has an effect of the same order as that of sodium chloride in reducing the solubility in water of the gases of the air.

The solubilities of oxygen in ammonium chloride given by MacArthur (*J. Phys. Chem.*, 1916, **20**, 495), are therefore erroneous.

J. H. COSTE and E. R. ANDREWS.

TEDDINGTON.

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 interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

CITY OF BIRMINGHAM.

REPORT OF THE PUBLIC ANALYST FOR THE SECOND QUARTER, 1923.

DURING the second quarter of this year 1311 samples were submitted for analysis, of which 1161 were analysed under the Sale of Food and Drugs Acts. Of these 997 were bought informally, of which 56 were adulterated, and 164 were bought under the provisions of the Act, of which 19 were adulterated.

MILK.—Of the 649 samples analysed, 61 were adulterated. The average composition of all the samples was: Fat, 3·43; and solids-not-fat, 8·8 per cent. Of 398 samples taken from farmers, chiefly at the railway stations, 19 were deficient in solids-not-fat, and 36 in fat only. One farmer sent milk containing from 2·2 to 4·6 per cent. of fat. On inspection of the farm, it was found that the milk was from six cows which were only partly milked, the later and richer part being left for the calves to suck. In the appeal case of *Grigg* v. *Smith* this proceeding was considered legal, and it is to be hoped that in any new legislation this defect in the law will be remedied.

ACTION OF POTASSIUM CARBONATE ON GLASS.—The samples of potassium carbonate used contained 17·1, 17·6 and 18·2 per cent. of water respectively, and complied with the British Pharmacopœia requirements. They were mixed with about equal quantities of ground glass containing lead and arsenic, and put in well-corked white glass bottles for three months. At the end of that time the soluble lead had increased, in each case, to about 1400 parts of lead per million, and the samples contained from 70 to 100 parts of arsenic per million. The test was a very severe one, and it shows that in these conditions the solvent action of B.P. potassium carbonate on glass containing lead and arsenic was considerable.

In another test 5 grms. of potassium carbonate containing 18·2 per cent. of moisture were put in a corked 4 oz. glass bottle. At the end of three months the soluble lead had increased from 2 to 20 parts per million. In a similar experiment

when the sample contained 17·1 per cent. of moisture the soluble lead increased from 1 to 8 parts per million. In these two cases it was observed that the varying temperature of the room caused droplets of moisture to appear on the sides of the bottle, and that when the bottle was shaken, some of the potassium carbonate stuck to the sides of the bottle at these wet places. In a pharmacy it is probable that the variations in temperature would be greater than in my laboratory, and the condensation of moisture on the upper part of the bottle might be sufficient, when part of the contents of the bottle were shaken out for sale, to make a strongly alkaline solution, which would have local solvent action on the glass.

These experiments show that if potassium carbonate be kept in bottles which contain arsenic and lead, there is a risk of the drug being seriously contaminated, and that it is desirable this drug should be kept from contact with such glass. (*Cf.* ANALYST, **1923, 215, 260.**)

J. F. LIVERSEEGE.

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.

"GOLDEN SYRUP" WARRANTY.

ON September 3, I. and J. Sanders were summoned at Croydon for selling as golden syrup a substance not of the nature and quality demanded.

The defendants pleaded a warranty from the wholesale firm, who had written: "We have sold you 100 cases each of 36 tins, labelled 'Amolco Golden Syrup'." The Town Clerk, however, contended that this did not protect the defendants, since it merely stated that the tins were labelled "golden syrup," not that the article sold was golden syrup.

Mr. H. S. Baron, for the defence, said that Sanders Bros. bought the goods as golden syrup, and that in the contract note they were described as "pure cane sugar." It had been bought from Messrs. Field and Co. at 4½d. per lb., and had been bought with a warranty which had been regarded as sufficient to cover the buyers.

The Bench held that the article sold was not golden syrup within the meaning of the Act, and imposed a fine of 40s. and costs. They were asked to state a case for a higher Court on the question of the warranty.

VINEGAR.

ON September 7th, Wrenson's Ltd., were summoned at Birmingham for selling vinegar containing less than 4 per cent. of acetic acid.

The inspector said that the youth who served the vinegar fetched it from a cellar, and said that he had taken it from a barrel marked "Wrensons." Thereupon the manager remarked that he was wondering whether the vinegar was cloudy, as it was getting to the bottom of the cask. He then shook one of the sample bottles and observed, "It looks all right."

Mr. J. F. Liverseege, the Public Analyst, stated that the sample analysed by him on August 2 contained only 2·9 per cent. of acetic acid, and by September 5

the acidity had fallen to 2·7 per cent., and the vinegar showed signs of decomposition. There was no evidence of adulteration with water, and it was probable that the deficiency was due to decomposition. He also pointed out that the appeal case of *Robinson* v. *Newman*, in 1917, was applicable to this case. In his opinion, evaporation by itself would not cause the deficiency, which, however, might possibly be due to chemical change.

Mr. Oxley, for the defence, said that the business, which had been started in 1865, comprised 56 retail shops, which together sold over 20,000 gallons of vinegar a year, and that there had never been a previous prosecution against the company. The vinegar in question was from a 12½ gallon cask bought in October, 1922, and there was only half a gallon in it when the purchase was made. Instructions had now been given that only six gallon casks should be ordered in future.

The Bench decided that a technical offence had been committed, and dismissed the case on payment of costs.

BARLEY IN GROUND ALMONDS.

ON September 8, P. Davies was summoned at Ashton-under-Lyne, for the sale of ground almonds containing 5 per cent. of foreign cereal agreeing in characteristics with barley.

Mr. B. F. Robinson, for the prosecution, said that, although it might be urged that almonds were difficult to grind, and that the addition of a cereal such as barley was required to enable them to be ground, yet it was the opinion of grocers that almonds could be ground without any such addition. Therefore, if a purchaser asked for ground almonds, he should be supplied with ground almonds. He did not suggest that this was a serious case, but submitted that there had been a technical offence.

Mr. Fleming, for the defence, said that the original order form guaranteed the product to be pure ground almonds, but, as this warranty had not been put in in time, he must plead guilty to a technical offence. There was no intention on the part of the defendant to defraud the public. He understood that if some substance such as barley or rice were not used in grinding almonds, the product would be a pulpy mass, and therefore it was frequently used, especially, as in this case, with foreign ground almonds.

The magistrates agreed that the defendant had not acted wilfully, and said that they would make the fine as light as possible. A fine of 10s. with £3 3s. costs was imposed.

Report of the Government Chemist upon the Work of the Government Laboratory.

FOR THE YEAR ENDING MARCH 31ST, 1923.*

THE chemical work of the following departments is performed wholly or in part in the Government Laboratories—Admiralty, Ministry of Agriculture and Fisheries, Air Ministry, Colonial Office and Crown Agents for Colonies, Board of Customs and Excise, Geological Survey, Ministry of Health, Board of Inland Revenue, Ministry of Pensions, Post Office, Department of Scientific and Industrial Research, Home Office, Board of Trade, War Office, Office of Woods and Forests, and Office of Works.

* H.M. Stationery Office, Kingsway, W.C. Price 1s. 6d. net.

The total number of samples examined in the course of the year is 343,453, as compared with 302,562 in the preceding year, an increase of 40,891. The samples of wine have increased from 61,683 to 85,776, of sugar from 42,715 to 49,172, and of tobacco offal and waste from 31,851 to 35,545, while there are notable increases in the numbers of samples of wort and exported and imported spirituous preparations. The number of samples of beer for the detection of dilution is again high, the number being 5,079, as compared with 4,650 in the previous year and 854 two years ago. There has been a decrease of 5,500 in the number of samples of tea. In connection with the Safeguarding of Industries Act the number of samples in the present year is nearly 8,000. The Dangerous Drugs Act, and the Dyestuffs (Import Regulation) Act, continue to impose considerable work on the Department.

IMPORTED DAIRY PRODUCE AND MARGARINE.—During the year 793 samples of butter were examined, of which 14 contained more than 16 per cent. of water. Seven of 531 samples of margarine also contained more than the maximum limit of water.

Cheese.—The proportion of fat in the 143 samples examined varied from 9·6 to 36·6 per cent. (20·2 to 54·4 per cent. on the dry matter). As there are no regulations as to the marking of skimmed or partly skimmed milk cheese, no exception could be taken to the importations.

Cream.—Fourteen samples of preserved cream and three samples of fresh cream were examined. Twenty samples of tinned sterilised cream were examined. This was of two kinds, one containing 7 to 10 per cent. of fat, and the other from 22 to 28 per cent.

Condensed Milk.—The 244 samples included milk powders. In nine cases the condensed milk had been made from skimmed milk, and the outer packages were properly marked, but there were no labels on the tins.

The composition of the "whole" milk samples varied considerably, the fat ranging from 6·88 to 10·9 per cent., with a corresponding range in the amounts of non-fatty solids.

SHEEP DIPS.—The regulations of the Ministry of Agriculture require the periodical dipping of sheep in a bath prepared from a dip which has received the approval of the Ministry. Where the formula submitted by the manufacturer is satisfactory samples of the dip are analysed. Samples of the bath in use are also occasionally examined. Of 180 samples submitted during the year 27 were defective.

POLLUTION OF RIVERS BY TAR DRAINAGE.—Experiments made in the Government Laboratory showed that pure bitumens were free from tar acids and bases, and did not contain toxic ingredients likely to be dissolved in road drainage. From this point of view bituminous road dressings would appear to be reasonably free from risk to fishing streams. The question was investigated at a site at Alresford on the road previously used for testing tarred surfaces. A report (No. 40 of Standing Committee on Rivers Pollution, Ministry of Agriculture and Fisheries), dealing with these experiments has now been issued, and it is shown that under the conditions stated no toxic effects resulted from the bituminous road dressing.

FERTILISERS AND FEEDING STUFFS ACT.—During the year 22 fertilisers and 22 feeding stuffs were examined. In most cases the amounts of nitrogen, phosphates or potash were less than the guarantee stated. Two samples of basic slag were found to contain mineral phosphate, and a sample of sulphate of ammonia was found to be deficient in nitrogen, and to contain 0·33 per cent. of free sulphuric acid and 5 per cent. of water, the deficiency in nitrogen probably being due to the absorption of water by the free acid in the sulphate.

The feeding stuffs included ground oats, clover meals, fish meals, coconut cake and pea meal, alleged to contain foreign materials, and various meals and cakes deficient in oil and albuminoids when compared with the guarantee on the invoice. The ground oats, in some cases described as "Sussex Ground Oats," were adulterated to an extent varying from 20 per cent. of barley meal to 90 per cent. of barley, maize, weed seeds and oat-shudes. The clover meals contained 50 per cent. of material not derived from clover. A sample of coconut cake contained 17 per cent. of castor oil cake; fish meal 30 per cent. of vegetable matter and sand and a sample of pea meal, 45 per cent. of wheat and maize. Among the samples deficient in oil and albuminoids were a meat-and-bone meal containing only two-thirds, and a meat meal with less than one-half of the guaranteed quantities of albuminoids.

It is interesting to record that in all the samples there was substantial agreement with the results of the agricultural analysts who had examined the samples in the first instance.

MERCHANDISE MARK ACT: *Blue Vitriol.*—One inquiry, in the course of which 71 samples were received, related to the quality of the blue vitriol supplied for horticultural or wheat-dressing purposes throughout the country. Sixty-five of the samples were sold under the descriptions "Blue vitriol," "Blue stone," or "Copper sulphate," and of these 53 contained more than 98 per cent. of crystallised copper sulphate, while 12 were almost up to this standard. Two other samples, consisting of mixtures of copper sulphate and ferrous sulphate in approximately equal proportions, and sold under the description "Commercial blue vitriol," were the subject of legal proceedings, at which it was contended that it was a trade practice to apply the term to copper sulphate containing large quantities of ferrous sulphate. This contention was negatived by the result, the seller being fined.

Ground Oats.—Another enquiry into the composition of ground oats involved the analysis of 50 samples taken in different parts of the country. Nineteen of the samples were described as "ground oats," and 31 as "Sussex Ground Oats." Six of the former description and 11 of the latter were found to be substantially pure oats, but the majority of the samples were mixed with other materials. Some of the samples, including all those taken at the outset of the investigation, were mixed with barley only, and contained quantities of barley ranging from traces to 95 per cent. The samples taken at a later period, in addition to barley, contained tapioca, maize flour, dari, and oat husk, the most highly adulterated sample containing 50 per cent. of tapioca and 40 per cent. of oat husk, with only 10 per cent. of ground oats.

CUSTOMS AND EXCISE.—The number of samples tested for arsenic, including beer, wort, malt, sugar, and other materials used in brewing, was 462. Of these 66 were found to contain arsenic in excess of the limit laid down by the Royal Commission on Arsenical Poisoning—namely, the equivalent of one-hundredth of a grain of arsenious oxide per pound in the case of solids, or per gallon in the case of liquids. Of the 208 samples of malt and sugar tested, only three contained arsenic in excess of the limit, but the limit was exceeded in three instances out of 61 samples of beer and wort.

Cocoa and Chocolate.—During the year the question of the presence in chocolate of fat other than cocoa butter has continued to receive attention. Fat expressed from the cocoa nib during the manufacture of cocoa powder was formerly available in large quantities for incorporation in chocolate in which a varying proportion of extra fat is desirable. For various reasons manufacturers have been constrained to use a substitute which, whilst not derived from the cocoa bean, closely resembles cocoa butter in its physical and chemical properties. The estimation, and even

the detection of this fat when incorporated in chocolate, presents great difficulties, but the method devised in this Laboratory has been applied with satisfactory results (*cf.* ANALYST, 1921, **46,** 229).

Dangerous Drugs Act.—Three of 105 samples contravened the provisions of the Act. One sample bearing the seal of a foreign company and marked "Cocainum hydrochloricum," was seized by officers, but it consisted of quinine hydrochloride, and two samples with indistinct markings, but bearing labels of a foreign cocaine company were found to consist of benzoic acid.

Dyestuffs (Import Regulation) Act, 1920.—Six hundred and thirty-three samples of imported colours, lakes, etc., were examined.

Matches.—None of the 107 samples contained white phosphorus.

Safeguarding of Industries Act.—Nearly 8000 articles were examined, including many samples of medicines, perfumes and proprietary articles.

Spirits.—Of 822 samples of wood naphtha and mineral naphtha, 877 were approved as suitable for methylating purposes. Further work has been done in connection with the enquiry into the characteristics of denaturants and colouring substances for industrial alcohol. Fifteen of 108 samples of fusel oil contained over 15 per cent. of proof spirit and, as an alternative to payment of duty, the importers were allowed to reduce the proportion of proof spirit below 15 per cent.

Commercial amyl acetate is allowed to contain a proportion of ethyl aetate equivalent to the alcohol allowed free in fusel oil.

Of 82 samples of wood spirit (methyl alcohol) 46 were found to be liable to duty on the ground that they had been sufficiently purified to be used as a substitute or ordinary spirit.

Sugar, Glucose and Saccharin.—The number of samples of sugar and articles containing sugar or other sweetening matter examined was 49,172, as compared with 42,715 the previous year.

Examinations of glucose and glucose syrup were made in 1131 cases, and 179 samples of imported goods were tested for saccharin. Forty-nine samples of saccharin and materials used in its production were examined in connection with the assessment of duty on saccharin made in this country.

Tea.—The total number of samples examined was 36,518, of which 1266 were reported against. The number of samples of denatured tea examined was 645.

Tobacco.—The percentage of moisture was estimated in 10,570 and the percentage of oil in 1960 samples taken from the stocks of manufacturers and retailers. The samples taken from tobacco exported on drawback numbered 8262, as compared with 8210 in the previous year. During the year 22,067 samples of tobacco stalks for drawback purposes, and 7287 samples for tests as to uniformity of condition, were received, together with 6,191 samples of offal snuff, shorts and smalls.

DEPARTMENT OF SCIENTIFIC AND INDUSTRIAL RESEARCH.—Samples of natural gas from bore-holes in this country were found to contain from 0·005 to about 0·2 per cent. of helium by volume.

BOARD OF TRADE: *Lime and Lemon Juice.*—The number of samples of raw juice examined was 219, and that of fortified juice 109. The quantity of juice approved was 26,179 gallons.

OFFICE OF WORKS, LONDON.—The samples examined consisted largely of materials purchased for the public service. Some "preservatives" for stone and wood were examined, but did not show any advance on older preparations.

Excellent results have been obtained by the treatment of the old tiles in the Chapter House of Westminster with aqueous 3 per cent. casein, containing a little formaldehyde, followed by a thin coat of matt varnish.

SALE OF FOOD AND DRUGS ACTS.—Thirty-one samples of food and four drugs were sent to the Laboratory for analysis. The foods consisted of 19 milks, four samples of tinned peas and one of tinned asparagus, two jams, and one sample each of rum, whisky, salmon and shrimp paste, sausage meat, and malt vinegar. The drugs were a lime water, a spirit of nitrous ether, and two compounded drugs.

In 29 cases the results were in agreement, and in six cases in disagreement with those put forward by the prosecution.

The following were the cases in which there was disagreement:—A milk, alleged to be adulterated with added water, contained 9·79 per cent. of non-fatty solids (not taking into account the loss during fermentation), 4·29 per cent. of fat, and 0·81 per cent. of ash.

In a jam alleged to contain 7½ per cent. of apple pulp there was only a trace of pulp.

A sample of rum, alleged to be more than 35 degrees under proof, indicated an alcoholic strength of 37·5 degrees under proof if the strength was calculated from the specific gravity of the rum without distillation, but when the obscuration was removed by distillation the strength was 34·7 degrees under proof.

A sample of salmon and shrimp paste was found to be free from boric acid, alleged to be present; a sample of spirit of nitrous ether, alleged to be deficient in ethyl nitrites, satisfied the requirements of the British Pharmacopœia; and a sample of malt vinegar, alleged to contain vinegar made from materials other than malt, gave analytical results in conformity with its having been derived from malt or from a mixture of malt and unmalted barley.

Department of Scientific and Industrial Research.

FOOD INVESTIGATION BOARD.

MOULD GROWTHS UPON COLD STORE MEAT.*

THE moulds found on cold store meat during this investigation are limited in kind but, with one exception, they have been frequently encountered on meat from all the exporting countries of the southern hemisphere.

The chief classes include "black spot," *Cladosporium herbarum* (see ANALYST, **1921, 46,** 292), moulds which produce white spots (*Sporotrichium carnis*), moulds producing "whiskers" (*Thamnidium* and *Mucor*), bluish-green moulds due to certain species of *Penicillium*, and white or pink *Saccharomyces*.

A fungus typical of a new genus was isolated from a consignment of skinned Australian rabbits, and was named *Wardomyces anomola* (after Marshall Ward). It produced circular white fuscous colonies, adpressed to the substratum, had creeping, branched heptate hyphæ, 2 to 4 μ wide, and brown to black, smooth sub-spherical to oval conidia usually with slightly pointed ends, 5 to 8μ by 4 to 6μ.

It was found that many forms of *Cladosporium* were strains of *C. herbarum*, and not distinct species. Some strains will develop from spores at −6° C., and will give rise to considerable growths under prolonged cold-storage conditions.

Torula botryoides, Sporotrichium carnis, Penicillium expansum and *Thamnidium* species will sometimes develop at −6° C., but develop readily at 0° C. *Mucor* species, *Saccharomyces* species and *Wardomyces anomala* do not develop at −6° C., but grow readily at 0° C. or just above that temperature.

* Special Report, No. 17. By F. T. Brooks and C. G. Hansford. Pp. 142. H.M. Stationery Office, Kingsway, W.C. Price 1s. 6d. net.

Spores, and young mycelia of certain of these moulds, notably *Thamnidium* spp. and *Penicillium expansum*, retain their vitality for more than 2 years at −6° C., and germinate or continue to develop on removal to ordinary temperatures.

The growth of these moulds on meat is superficial, and even in the case of "black spot," the mycelium penetrates only to a maximum depth of 4 mm. These fungi do not confer poisonous properties on the meat, and, unless associated with putrefactive bacteria, do not render the meat unfit for food. Their growth may be prevented by controlling the temperature and humidity conditions in the cold stores.

The report concludes with a bibliography of 18 references from 1886 to 1921.

RESEARCH ASSOCIATION OF BRITISH FLOUR MILLERS.

THE Secretary of the Department of Scientific and Industrial Research begs to announce that a license, under Section 20 of the Companies' (Consolidation) Act, 1908, has been issued by the Board of Trade to the Research Association of British Flour Millers which has been approved by the Department as complying with the conditions laid down in the Government Scheme for the encouragement of industrial research.

The Secretary of this Association is Mr. G. H. Ball, LL.B., 40, Trinity Square, E.C.

Ministry of Health.

THE PUBLIC HEALTH (CONDENSED MILK) REGULATIONS (No. 2), 1923.

PROVISIONAL REGULATIONS, DATED SEPTEMBER 24, 1923, MADE BY THE MINISTER OF HEALTH.

68,765.

The Minister of Health certifies under Section 2 of the Rules Publication Act, 1893, that on account of urgency the following Regulations should come into immediate operation, and hereby makes the following Regulations to come into operation forthwith as Provisional Regulations:—

1. Notwithstanding anything contained in Article 1 of the Public Health (Condensed Milk) Regulations, 1923, Part II of those Regulations, so far as it relates to the sale, or the exposure for sale, by retail, or the deposit in any place for the purposes of such sale, of any condensed milk intended for human consumption, shall not come into operation until the 1st day of November, 1923.

2. These Regulations may be cited as "The Public Health (Condensed Milk) Regulations (No. 2) 1923."

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Detection of Abnormal Milk by the Alcohol Test. A. C. Weimar. (*J. Dairy Sci.*, 1923, **6**, 95–101; *Chem. Abstr.*, 1923, **17**, 2331.)—In the case of milk of different cows from 3 herds whose milk had given positive results with the alcohol test (*cf.* ANALYST, 1922, **47**, 25), the fermentation test, acid test, bacterial count, flavour and odour gave no clue as to the cause of the alcohol reaction. Visits to the farms, however, showed that the cows whose milk had given positive reactions had been fed on mouldy silage, whereas the milk of cows which had refused the silage gave negative results. A relationship was established between the alcohol test and the coagulating point of evaporated milk, those milks giving a positive reaction in the test coagulating at a lower temperature than those giving a negative result. Hence the conclusion is drawn that the alcohol test is of value in detecting some cases of abnormal milk which could not be detected by chemical analysis.

Meat Extracts and their Substitutes. K. Berk and W. Schneider. (*Zeitsch. Unters. Nahr. Genussm.*, 1923, **45**, 307–336.)—Detailed analyses are given of several meat, crab and yeast extracts, and similar products, showing the distribution of the nitrogenous bases therein. The method adopted for the separation of the different nitrogenous bodies is as follows:—Fifty c.c. of aqueous solution, containing about 10 per cent. of dry extract, are diluted to 250 c.c. and heated on the steam bath for four or five hours, with the addition of a little water to maintain the volume constant, then for a further half-hour with the addition of 2·5 grms. of tartaric acid. This treatment hydrolyses the glutin to glutose. Any coagulated albumin is filtered off (precipitate 1). The solution is now neutralised, 25 c.c. of saturated solution of zinc sulphate are added, the volume made up to 300 c.c., and, after settling, the precipitate (No. 2) of albumoses and proteoses is filtered off. To the filtrate are added 25 c.c. of a solution containing 7 grms. of nitrogen-free tannin and 3 c.c. of acetic acid per 100 c.c., and the precipitate (No. 3) containing the glutin nitrogen is filtered off. The filtrate is now diluted to 500 c.c. The nitrogen in each precipitate and in an aliquot portion of the filtrate is then estimated by Kjeldahl's method. Genuine meat extracts contain 4 to 7 per cent. of the total nitrogen as albumoses. The high results recorded for the albumose in meat extracts is probably due to incomplete separation of glutin nitrogen. Meat extracts contain about 20 per cent. of glutin, whereas crab extracts contain little albumose and glutin, the two together being only

10 per cent. of the total nitrogen. The decomposition products of these extracts are also discussed in detail; estimation of the ammonia and amino-nitrogen affords the best indication of the state of freshness of meat extract. In a normal sample there is about 4 per cent. of ammonia and 16 per cent. of amino-nitrogen. Yeast extracts show much higher total ammoniacal and amino-nitrogen, usually about 30 per cent., and in crab extract this total reaches 52 per cent. The following table shows the distribution of the nitrogen in some of the fresh materials:

NITROGEN DISTRIBUTION IN PER CENT. OF THE TOTAL NITROGEN.

	N. in aqueous solution.	N. pptd. by heating	N. in $ZnSO_4$ ppt.	N. in the tannin ppt.	N. in filtrate from the tannin ppt.	Total creatinine N.	Ammoniacal N.	Amino-N.
Meat extract 1	1·8	3·7	7·2	22·9	64·4	11·0	2·6	10·6
Meat extract 2	7·0	2·1	4·4	17·0	70·5	12·2	5·0	19·5
Beef tea extract	3·3	0	0	39·7	55·9	2·4	6·0	29·0
Meat extract substitute	0·8	0·9	0	2·7	93·9	0	10·3	57·8
Yeast extract	—	4·2	10·5	17·9	68·8	0	3·4	27·7
Crab extract 1	12·6	trace	0	9·5	77·5	0	4·3	34·3
Crab extract 2	17·3	0·7	0	11·4	70·5	0	2·5	32·0
Gelatin	0	1·9	0	87·8	10·3	0	1·0	7·4

H. E. C.

Caviare. G. Hinard. (*Ann. Falsificat.*, **1923**, **16**, 524–332.)—Genuine fresh caviare contains much more fat and protein than caviare substitutes, and has the following percentage composition: Water, 45 to 55; total proteins (N × 6·25), 23 to 28; fat, 12 to 18; ash, 3·8 to 6·8; sodium chloride, 2·8 to 5·3; and hydrocarbons up to 1·0. A sample of German *caviare de brochet* (which is **not** true caviare) contained: Water, 78·31; total proteins, 11·5; fat, 3·47; and sodium chloride, 4·57 per cent. The palatability of caviare depends upon its freshness, and this can be interpreted chemically in terms of its acidity and the formaldehyde titration of the nitrogen (Sörensen); the following figures show how closely these two factors are related:

Acidity in c.c. *N* per 100 grms.	Nitrogen titratable with formaldehyde. Per Cent.	Acidity c.c. *N*. Per 100 grms.	Nitrogen titratable with formaldehyde. Per Cent
1·7	0·046	1·4	0·024
1·6	0·034	1·3	0·029
1·6	0·027	1·0	0·018
1·5	0·030	—	—

It is not possible to titrate the acidity directly in alcoholic or ether solution, as the addition of alkali at once forms a coherent jelly which prevents an accurate result; and if the protein is first dried or coagulated by heat there is loss of volatile

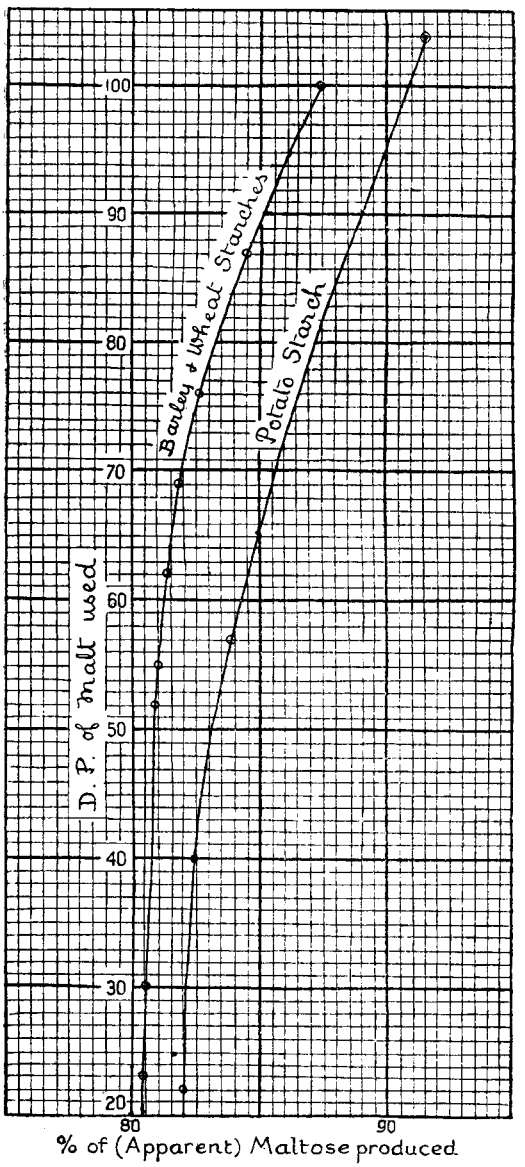
acids. The procedure recommended is to grind 20 grms. of the sample in a mortar with 50 c.c. of water, adding 50 c.c. of alchohol (96 per cent.), with constant trituration. Then the mixture is transferred to a flask, well shaken, allowed to settle, and decanted. In this way a clear liquid is obtained, 25 c.c. of which are diluted with 75 c.c. of water and titrated with the use of phenolphthalein as indicator. The results are conveniently expressed in c.c. of N alkali per 100 grms. For the formaldehyde titration 10 c.c. of neutralised formaldehyde solution are added to the neutralised solution and followed by a second titration. Reasonable limits for fresh caviare are not more than 2 c.c. per cent. for acidity and not less than 0·05 per cent. of nitrogen titratable with formalin. When caviare begins to putrify, the principal products of the fermentation are aldehydes and formic acid, together with traces of butyric and valeric acids. The figures below show the effect of the degradation of proteins and amino-acids:

Time.	Acidity c.c.	Ammoniacal N. Per Cent.	Amino-N. Per Cent.	Formaldehyde titration. Per Cent.
—	2·0	0·011	0·025	0·036
2 weeks	2·2	0·028	0·038	0·066
4 ,,	2·9	0·031	0·061	0·092
10 ,,	3·5	0·041	0·085	0·126
12 ,,	3·8	0·046	0·112	0·158

H. E. C.

Histology and Chemistry of the Avocado. W. J. Stoneback and R. Calvert. (*Amer. J. Pharm.*, 1923, **95**, 598–612.)—The paper gives a general history of the avocado (the fruit of *Persea gratissima* and *P. drymifolia*), the habit of the tree, a description of the fruit and a detailed account of its histology. The Guatemala variety, known as the "Collins," is dealt with more particularly; the chemical analysis of a sample was as follows: Moisture, 66·0; protein, 1·21; fat, 25·26; carbohydrates, 6·44; and ash, 1·09 per cent. The butyro-refractometer reading of the oil at 25° C. was 62·8. In the United States the avocado is commonly used in the form of a salad, but in Central America it is used as a substitute for meat. A lamp oil is expressed from the pulp, and the seeds, which contain tannin, are used for the production of an indelible marking ink. D. G. H.

Formation of Maltose in Sweet Potatoes during Cooking. H. C. Gore. (*J. Ind. Eng. Chem.*, 1923, **15**, 938–940.)—When sweet potatoes are boiled or baked a considerable proportion of the starch is converted into maltose. From 13 to 19 per cent. of maltose was found to be present in baked sweet potatoes, whilst only 0·4 per cent. was found in the raw potatoes. The formation of the maltose takes place during the initial stages of the cooking; it is not formed at the boiling point. W. P. S.



Estimation of Starch in Potatoes. A. R. Ling and W. J. Price. (*J. Inst. Brew.*, 1923, **29**, 732–734.)—A method previously described (ANALYST, 1923, 29) has been applied to the estimation of starch in potatoes. The accompanying curve was constructed by the use of prime potato starch treated with malts of various diastatic powers, and shows that this starch gives a higher yield of apparent maltose than those derived from barley and wheat. Approximately 8 grms. of potato in thin slices are ground to pulp in a mortar and washed into a beaker with 100 c.c. of water, in which the mixture is allowed to stand, with occasional stirring, for 30 minutes. The supernatant liquid is poured off through a folded filter, and the residue is again treated in a similar manner with a second 100 c.c. of water, after which the pulp and filter are well washed with water to remove reducing sugars. The filter and its contents are transferred to the beaker containing the pulp, and are boiled for 10 minutes with 100 c.c. of water, after which the liquid is cooled, treated with malt extract, and titrated with Fehling solution as previously described. Three duplicate estimations with extracts prepared from malts ranging in diastatic power from 28° to 88° Lintner gave results varying from 17·3 to 17·8 per cent. of starch.

T. J. W.

Alcohol Content of Bread. T. Sundberg. (*Svensk. Kem. Tids.*, 1923, **35**, 109–115; *Chem. Abstr.*, 1923, **17**, 2155.)—Nineteen samples of bread, as sold, were cut into small pieces and placed in a 2-litre flask, which was heated in a calcium chloride bath maintained at 105° C. The alcohol was distilled with

steam, and when 100 c.c. of distillate had been obtained, the receiver was changed, the temperature raised to 110°–115° C., and a second 100 c.c. obtained. These distillates were titrated, neutralised and re-distilled. The acidity expressed in terms of c.c. of 0·1 *N* alkali ranged from 1·8 to 21·6, and the alcohol content from nil to 0·84 per cent. The lowest figures for alcohol were from sliced bread which had been exposed for 6 or more days, and the highest were from fresh bread in which syrup had been mixed with the dough. In no case did the first 100 c.c. contain all the alcohol (when more than 0·02 per cent. was present).

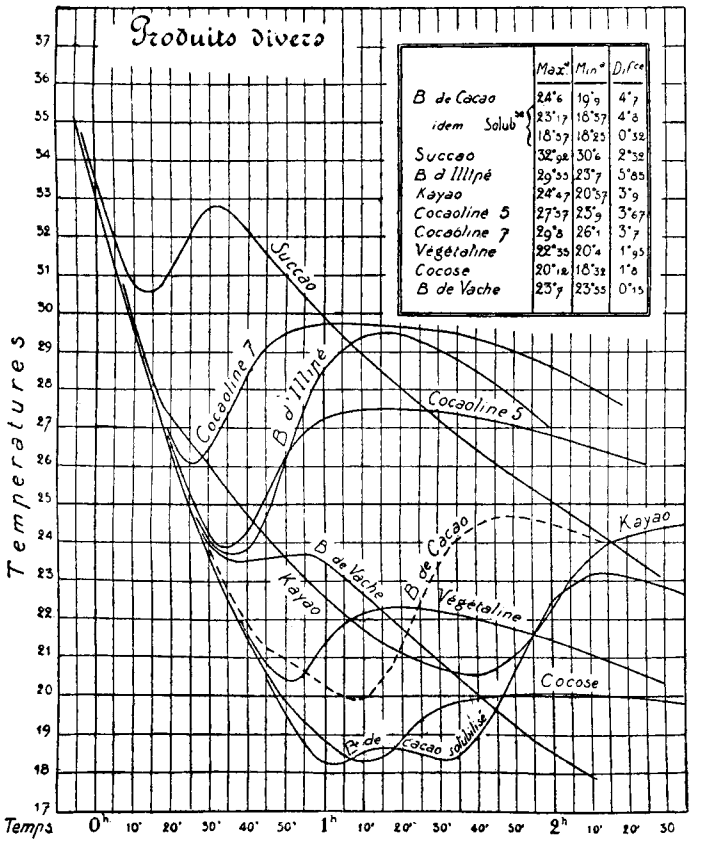
Estimation of Fatty Acids in Butter Fat. II. E. B. Holland and others. (*J. Agric. Res. Washington*, 1923, **24**, 365–397.)—Tables are given showing the general analyses of a large number of samples of butter fat from the milk of a mixed herd of Holstein and Jersey cows, and from individual animals. The original paper should be consulted for the detailed results, but it is interesting to note that for both grades the effect of lactation was to increase the percentage of total fatty acids, the soluble fatty acids decreasing and the insoluble increasing, and the change was more gradual in the case of the Jerseys. The addition of coconut fat to the ration caused a decrease in all the soluble acids in increasing amounts from butyric to capric acid; an increase in lauric and myristic acids; and a decrease in oleic acid. Less saturated oils, such as arachis, maize and soya oils, increased the butyric acid, decreased all the other acids from caproic to palmitic (except caprylic in the case of maize and soya oils), and increased the stearic and oleic acids. The neutralisation values of the soluble, insoluble and total fatty acids were decreased by the liquid oils, and that of the insoluble acids was increased by coconut oil.

Taking into consideration data obtained from all experiments at the Massachusetts station, extending over many years, it appears evident that proteins and carbohydrates have but little influence on the chemical composition of the butter fat, whilst different fats and oils appear to exert a definite effect. Analyses of 29 samples of butter fat from cows fed on normal rations gave the following average figures:—Saponification value, 231·4; acid value, 2·04; iodine value, 28·46; total fatty acids, 94·82 per cent.; with neutralisation value, 244·05; free fatty acids, 0·84 per cent.; soluble fatty acids, 7·29, with neutralisation value, 504·79; insoluble fatty acids (by alcoholic potassium hydroxide), 87·52; with neutralisation value, 222·36; stearic acid (by crystallisation), 15·01 per cent.; glycerol, 12·54 per cent.; refractive index n_D^{40}, 1·4536; colorimeter (Lovibond), $\frac{1}{2}$ inch cell, 3·2 yellow, 1·4 orange.

The average composition of 21 samples of butter fat from cows fed on normal rations was: *Soluble acids*: butyric, 2·93; caproic, 1·89; caprylic, 0·78; capric, 1·57. *Insoluble acids*: lauric, 5·85; myristic, 19·78; palmitic, 15·17; stearic, 14·9; and oleic acid, 31·89 per cent. The amounts of stearic acid ranged from 7·08 to 15 per cent., of palmitic acid from 5·78 to 22·86 per cent., of myristic acid from 15·5 to 22·6 per cent., of lauric acid from 4·53 to 7·68 per cent., and of oleic acid from 25·2 to 40·3 per cent. No appreciable quantities of unsaturated acids higher than oleic acid were detected, even in the case of butters from cows receiving maize and soya oils. D. G. H.

The Phytosterols of Maize, Cottonseed and Linseed Oils. R. J. Anderson and M. G. Moore. (*J. Amer. Chem. Soc.*, 1923, **45**, 1944–1953.)—Examination of the phytosterol separated after the saponification of maize oil shows it to be a homogenous substance identical with sitosterol and free from stigmasterol; it has m.pt. 137·5° C., and $[\alpha]_{20}^{D} = -34{\cdot}38$, and forms an acetate of m.pt. 127° C. The unsaponifiable matter of cottonseed oil contains at least two phytosterols, which may be separated by fractional crystallisation; one of these has m.pt. 138 to 139° C., $[\alpha]_{20}^{D} = -34{\cdot}19$, and acetate of m.pt. 124° C., and the other m.pt. 134 to 135° C., $[\alpha]_{20}^{D} = -33{\cdot}61$, and acetate of m.pt. 119° C. Linseed oil also contains at least two phytosterols, neither of which, however, can be completely separated by fractional crystallisation, as the solubilities in all the usual solvents are so nearly alike. The phytosterols of this oil have the following properties respectively: (*a*) m.pt. 138° C., $[\alpha]_{20}^{D} = -34{\cdot}22$, and acetate m.pt. 129 to 130° C., and (*b*) m.pt. 134° C. (not sharp), $[\alpha]_{20}^{D} = -31{\cdot}16$, and acetate m.pt. 124° C. In the case of each of the phytosterols examined the loss in weight on drying was approximately equivalent to one-half molecule of water of crystallisation. H. E. C.

Adulterations of Cacao Butter. M. Pichard. (*Ann. Falsificat.*, 1923, **16**, 197–215.)—The total fatty matter extracted from the product to be analysed is subjected, after melting, to slow cooling in a suitable vessel. In the case of pure cacao butters, the curves representing the variation of the temperature with the time are similar and almost superposable, no matter what the origin of the cacao beans, their degree of maturity, or the extent of the torrefaction or pressure applied in extracting the butter. For other fatty materials capable of fraudulent employment in the manufacture of chocolates, or coatings, or of direct admixture with cacao butter without sensibly modifying the appearance or the chemical and physical constants of the butter, the curves obtained differ widely, not only from that of cacao butter, but also among themselves. For mixtures of cacao butter with foreign fats, the curves are not exactly intermediate to those given by the separate constituents, but either lie outside of these curves or follow quite different courses, as is seen in the accompanying diagram, which represents the form of the curves of "succao" (a commercial substitute for cacao butter), cocaoline (a substitute), illipé butter, "çocaoline," cows' butter, "kayao" (a substitute with the same chemical and physical constants as cacao butter), cacao butter, "vegetaline," "cocose," and "solubilised" cacao butter. The method used is to introduce 90 grms. of the melted fat at a temperature not exceeding 50° C. into a corked tubular vessel surrounded by an outer tube, with side tubulures at the top and the bottom, to act as an air condenser. A thermometer is passed through the cork at the top, and the whole apparatus is then kept at 9° to 11° C. The thermometer readings are taken at intervals of 5 minutes, and the times are plotted as abscissæ on the scale of 2 mm. to the minute, whilst the temperatures are plotted as ordinates on the scale of 20 mm. per degree. The following table shows the results obtained with cacao butters of different origin and modes of preparation. In each case the



maximum represents the rise of temperature following the incipient solidification at the minimum:—

	Maximum ° C.	Minimum ° C.	Difference ° C.
Sanchez	23·75	19·4	4·35
Arriba	24·55	19·925	4·625
Puerto Cabello	24·475	19·775	4·7
Martinique	23·62	19·325	4·3
Lome	24·475	19·9	4·575
Gaudeloupe	24·5	19·525	4·975
Bahia	23·4	18·5	4·9
Congo	24·3	20·3	4·5
Cameroons	24·8	20·15	4·65
Caraque	25·0	20·00	5·00
Haiti	24·25	19·85	4·4

The curves of the fat from "solubilised" cocoa differ materially in form from those of the fats of the same cocoas which have not been thus treated, as is shown in the diagram. After reaching the first minimum they rise slightly, then fall, and then

rise to a final maximum. The difference between the second maximum and minimum, however, agrees with that of a normal cacao butter. For example, the following results were obtained with the fats from three samples of Dutch cocoa:—

	Maximum ° C.	Minimum ° C.	Difference ° C.
Franken ...	19·35	19·2	0·15
	23·1	18·8	4·3
Korf	19·3	18·96	0·35
	23·4	18·9	4·5
Boon	18·575	18·25	0·32
	23·175	18·325	4·85

In the case of mixtures of illipé butter with cacao butter the minimum point is always higher than that of pure cacao butter, and the curves are above those of pure products. For example, the following results were obtained with mixtures:—

	Maximum ° C.	Minimum ° C.	Difference ° C.
Cacao butter	24·675	19·95	4·725
Illipé butter	29·5	23·7	5·8
Cacao butter 20 per cent., illipé butter 80 per cent. ...	25·878	20·6	5·275
Cacao butter 66·6 per cent., illipé butter 33·3 per cent.	26·5	20·9	5·6
Cacao butter 50 per cent., illipé butter 50 per cent. ...	27·45	21·2	6·25
Cacao butter 33·3 per cent., illipé butter 66·6 per cent.	28·55	21·8	6·75
Cacao butter 25 per cent., illipé butter 75 per cent. ...	28·925	21·05	7·875

The figures given by karité butter and the form of its curve are quite different from those of cacao butter, *e.g.* maximum, 25·00° C.; minimum, 19·85° C.; and difference, 5·15° C. The solidification begins at 25·6° C., and the rise from the minimum to the maximum is so abrupt that the curve forms an acute angle.

T. H. P.

Detection of Phthalyldiethyl-ester in Spirits. S. Eilles. (*Zeitsch. Unters. Nahr. Genussm.*, 1923, **45**, 379–380.)—The resorcinol and sulphuric acid or phenol and sodium hydroxide tests are not satisfactory for the detection of this ester, which is now used for denaturing alcohol. The best reagent for its identification is pyrogallol, which gives with it a distinctive violet colour which is not given by any other substance; the brick-red colour given with formic acid is easily distinguishable. In the case of alcohol or spirits free from formic acid or from extractive matters which would give a dark colour with sulphuric acid, 5 drops of 15 per cent. sodium hydroxide solution are added to about 30 c.c., and the mixture evaporated on the water bath. When formic acid or extractive matters are present, the sample is diluted with an equal volume of water and, if necessary, protein matter is precipitated with lead acetate, and the mixture shaken out with 20 c.c. of petroleum spirit (b.pt., 30° to 50° C.). The petroleum spirit is evaporated in a porcelain basin without addition of alkali. To the dried residue are added 5 or 10 drops of sulphuric acid (sp. gr. 1·84), and the basin is heated over

a small flame until the acid begins to fume. A small quantity of pyrogallol is melted on another part of the basin, and the two substances allowed to run together. In the presence of the ester there is at once a violet coloration. The test as described will detect about 0·01 per cent. of the ester in a strong spirit, or about 0·1 per cent. in a tincture. H. E. C.

Tannin of Wild Cherry Bark. J. C. and B. L. De G. Peacock. (*Amer. J. Pharm.*, 1923, **95**, 613–623.)—The authors have isolated the tannin of wild cherry bark (3·4 per cent.) and have found it to be responsible for both the bitterness and astringency of the drug. The presence of gallic acid in the extract was not indicated, and it was found that the fluorescent principle is not the tannin. Cold water extracted only part of the tannin from the bark, leaving the insoluble phlobaphene. An aqueous solution of the tannin gave a white precipitate with gelatin, dark green colorations and precipitates with ferric chloride and ferric acetate, and a canary-yellow precipitate with lead acetate. Considerable quantities of benzoic acid were found in the bark during the course of preparation and purification of the tannin, and a study of conditions suggests that whenever benzaldehyde is present in living plant material, it may be the source of benzoic acid found in the dried tissue. It should be noted that the free benzoic acid contained in preparations of wild cherry bark causes decomposition of the cold-water-soluble tannin, producing cloudiness or sediment, and the practice of the addition of glycerin to the water used for extracting the bark is sound. D. G. H.

New Sources of Santonin. A. Viehoever and R. G. Capen. (*J. Amer. Chem. Soc.*, 1923, **45**, 1941–1944.)—On account of the scarcity of santonin, most of which is obtained from *Artemisia cina* and comes from Russia, some 56 species of *Artemisia* have been examined for santonin. This glucoside has been found in *A. Mexicana*, *A. Neo-Mexicana* and *A. Wrightii*, all of which are indigenous to Mexico or New Mexico. The method of examination consisted in simple sublimation; the flower heads were placed in cups heated to 150–170° C., and the sublimate collected on cool glass plates. The santonin was then identified by its crystalline form, m.pt. (170° C.), the furfural reaction and the periodide test. H. E. C.

Estimation of Santonin in Wormseed. L. M. van den Berg. (*Pharm. Weekblad*, 1923, **60**, 858–870.)—Five grms. of the wormseed (unopened flower buds of *Artemisia maritima*) are heated for 15 minutes on a steam bath with 30 c.c. of water, after which 10 c.c. of dilute hydrochloric acid are added, and the heating continued for another 15 minutes. The mixture is then cooled and vigorously shaken for 5 minutes with 60 c.c. of chloroform, 5 grms. of tragacanth added, and the shaking repeated until the chloroform separates. The chloroform extract is filtered through cotton wool, 40 c.c. of the filtrate are distilled, and the residue is boiled for 10 minutes with 50 c.c. of 15 per cent. alcohol beneath a reflux condenser. The solution is filtered, the residue washed twice with 10 c.c. of the boiling dilute alcohol, and the filtrate and washings left for 24 hours in a weighed flask in a cool place and then weighed. The liquid is next filtered through a weighed

filter, the crystalline deposit transferred to the filter by means of a jet of 3·5 c.c. of the alcohol, and washed twice with 10 c.c. of the same solvent, and the flask and filter dried at 100° to 105° C. An addition of 6 mgrms. is made to the weight of santonin for each 10 grms. of the liquid in the flask prior to the filtration. Ten samples of wormseed thus examined yielded from 1·55 to 3·36 per cent. of santonin.

Colour Reactions of Iron Salts with Tinctures of various *Boleti*. R. Gruyot. (*Bull. Soc. Pharm. Bordeaux*, 1922, **61**, 31; *J. Pharm. Chim.*, 1923, **28**, 159.)—The tincture is prepared from different species of *Boletus* (*luridus, satanas, scaber, bovidus*) by slicing and treating the fungus with boiling water or alcohol to destroy oxidases, and then macerating it with alcohol at 30° to 60° C. If, after filtration, a trace of an iron salt is added to the tincture diluted 50 per cent., a green coloration appears which is very stable and lasts for 2 months, provided the fungi were treated when fresh. Acids turn the colour yellow, but the green colour is restored by adding a sodium salt, and the tincture may be used as an indicator in acidimetry, as well as to denote the presence of ferrous or ferric iron. The reaction appears to be specific for iron, and neither copper nor manganese interfere with it. Iron present in complex acid salts, such as ferrocyanide and ferricyanide, or in hæmoglobin, does not give the green colour. The reaction appears to be analogous to that obtained with salts of iron and a fresh solution of hydroquinone.

D. G. H.

Estimation of Sparteine in Tablets. P. W. Jewel. (*J. Amer. Pharm. Assoc.*, 1923, **12**, 107–112; *Chem. Abstr.*, 1923, **17**, 1691.)—The tablets, which usually contain the sparteine in the form of a salt, are dissolved in slightly acidified water, and the alkaloid is separated by shaking the solution with two successive portions of chloroform. The solvent is evaporated on the steam bath, the final traces being removed by means of a current of air and the addition of a little ether, and the residue is cooled and weighed. The alkaloid is slightly volatile at 100° C., the loss being 3·19 per cent. after 1 hour's and 6·6 per cent. after 7 hours' heating. Sparteine hydrosulphate may be titrated with 0·02 *N* sodium hydroxide solution, with methyl red or phenolphthalein as indicator. It is not possible to titrate the separated alkaloid after extraction with chloroform, owing to the uncertain end-point. The picric acid method is also unsatisfactory, because of the solubility of the picrate in water. Separation of the alkaloid by shaking its ethereal solution with acid and titration of the excess of acid is not practicable.

Biochemical, Bacteriological, etc.

Age and Chemical Development in Mammals. C. R. Moulton. (*J. Biol. Chem.*, 1923, **57**, 79–97.)—The author presents in graphical form extensive data collected by himself and puts forward what appear to be general laws of chemical development in mammals. To make apparent the effects of age or abnormal development the composition of animals should be compared on the fat-free basis, on which the fatness of the individual has no effect on the composition.

It is shown that mammals show a rapid decrease in relative water content and increase in protein (nitrogen) and ash content from earliest life until the time of chemical maturity is reached, the point at which the composition (concentration of water, proteins and salts) in the fat-free cell becomes comparatively constant. They vary at birth, those relatively mature having a low water content and those less mature a high water content. Œdema, under-development and atrophy, give abnormal water percentages. The change in chemical development at birth follows the degree of physical development. Mammals reach chemical maturity at different ages, but these ages are a fairly constant relative part of the total life cycle. P. H. P.

Observations in Regard to Growth-Promoting Substances of Bacterial Origin. S. R. Damon. (*J. Biol. Chem.*, 1923, **56**, 895–902.)—The author has carried out a series of experiments to extend the investigation of these substances and to confirm the findings of Bottomley (*Proc. Roy. Soc., London, B*, 1914–15, 88, 237) and Thjötta (*J. Exper. Med.*, 1921, **33**, 763), who suggest that substances found in bacterial cultures, which apparently stimulate the growth of other micro-organisms, are vitamins, or possibly related to vitamin *B*. To demonstrate the presence or absence of vitamin the substances must be subjected to the biological test on young rats. Again, the organic constituents of the culture medium, *i.e.* commercial peptone and beef extract, were found to be devoid of vitamin *B*. Organisms were chosen for testing for their content of vitamin *B* representing (1) the group of aerobic spore formers, (2) the mucoid organisms of the *Bacillus mucosus capsulatus* group, and (3) the acid-fast bacteria, because they may be grown and desiccated in adequate quantities for feeding experiments fairly easily. Five per cent. of a spore-forming aerobic organism, *Bacillus adhærens*, did not supply the deficiency of vitamin *B* in an otherwise adequate diet. Of two mucoid organisms used, 5 per cent. of Friedländer's bacillus failed to induce growth in young rats, whilst 5 per cent. of Pfeiffer's bacillus added to a diet deficient in vitamin *B* maintained the animals at a constant weight or induced rapid growth. Five per cent. of *Bacillus timothy*, an acid-fast organism identical with *Bacterium phlei*, acted similarly to Pfeiffer's bacillus, whilst 10 per cent. induced more rapid and continuous growth in the rats. Tests are being carried out to find an explanation of the discordant results of the mucoid organisms, Friedländer's bacillus and Pfeiffer's bacillus (see ANALYST, 1923, 393). P. H. P.

Vitamins in Molluscs. Presence of the Antiscorbutic Factor in the Oyster. (Mme.) Randoin. (*Comptes rend.*, 1923, **177**, 498–501.)—The results of experiments made in the summer, during the emission of the fry, show that the oyster contains vitamin *C* in marked proportion; in the winter large amounts are probably present. This vitamin is evidently derived from the diatoms serving as food for the oyster. T. H. P.

Vitamins from Yeast and Rice Polishings. C. Funk, B. Harrow and J. B. Paton. (*J. Biol. Chem.*, 1923, **57**, 153–162.)—An investigation was carried out to find the best solvent for a vitamin, *viz.* one which extracts it quantitatively

while extracting a minimum amount of other materials. Extracts were made from yeast and rice polishings, and these and the residues were tested on pigeons and rats. Tables of results are given. The extracts were also tested with regard to their yeast growth-promoting power (due to vitamin *D*) and their content of co-ferment, total nitrogen and total solids. The following solvents were used:—Ethyl alcohol (50, 60, 70, and 80 per cent.), methyl alcohol (60 and 70 per cent.), and 70 per cent. solutions of propyl alcohol, butyl alcohol, isobutyl alcohol, acetone, methylethyl ketone and acetic acid. Taking inactivity of residue as a criterion, 70 per cent. alcohol was found to be the best among the solvents used for vitamin from yeast, and 60 per cent. alcohol when rice polishings were used in place of yeast; but, considering also the low percentage of nitrogenous and other impurities accompanying the vitamin in the extract from yeast, acetone was the best solvent. On the whole, for the yeast extractions, the higher the nitrogen content the greater the percentage of total solids and the greater the activity, *i.e.* the nitrogen content is a fairly good criterion of the vitamin activity of an extract; also vitamin *B* (as measured by means of pigeons) and vitamin *D* (as measured by yeast growth) tend to run parallel with one another, so that generally the higher the content of vitamin *B*, the higher is that of vitamin *D*. The extracts from rice polishings, although particularly active when tested on rats, were far less so by comparison when tested on pigeons. The co-ferment shows no definite relation to either vitamin *B* or *D*. However, two suggestive facts are pointed out: (1) that the butyl and isobutyl extracts of yeast were rich in co-ferment, though they quite lacked vitamins *B* and *D*; and (2) that there was complete absence of co-ferment in the methylethyl-ketone extract of yeast and in the 50 and 70 (hot) per cent. alcoholic extracts of rice polishings. The hope was not realised that a single solvent would have a selective dissolving power for a particular vitamin, or that a particular solvent would yield an extract that would induce growth in pigeons and not in rats (or *vice-versa*). The results suggest that the vitamin for rats may be less soluble in the solvents used than the vitamin for pigeons. P. H. P.

Colour Test for Vitamin *B*. **A. Jendrassik.** (*J. Biol. Chem.*, 1923, **57**, [illegible]om the results of Abderhalden and Schmidt (*Arch. ges. Physiol.*, **1920, 185,** 141), Abderhalden (*Arch. ges. Physiol.*, **1922, 193,** 329), and Hess (*Z. physiol. Chem.*, 1921, **117,** 284), it was thought desirable to apply a test which would involve reduction. In this test the reagent is a solution of ferric chloride and potassium ferricyanide in grm. molecular ratios; *i.e.* ferric ferricyanide used in acid media, which goes blue on reduction. The method is as follows:—Acetic acid is added to the concentrated aqueous solution of the preparation in question to make the concentration about 2 per cent. The reagent, freshly prepared by mixing equal volumes of tenth molar ferric chloride and potassium ferricyanide solutions, is added as long as the depth of the blue colour increases. The colour is observed after the mixture has been left standing for 10 minutes in a stoppered test-tube, and again after a convenient dilution has been reached by adding 1 to 5 volumes of distilled water. The test is negative if there is not a distinct

blue colour or, after standing for some time, a bright blue precipitate. Extracts were made from foods, botanically unrelated, which contain vitamin B (*e.g.* carrots, a leaf and a fleshy root) and foods which do not (*e.g.* polished rice and beef steak), with the use, in each case, of solvents which dissolve it out and others which do not. Positive results were obtained with all extracts from foods containing vitamin *B*, when they were made with solvents which dissolve out the latter, and negative results were obtained when the solvents used did not dissolve out vitamin *B*, and also when the extracts were from foods which do not contain vitamin *B*, even if the solvents used extract that principle. Water, glacial acetic acid and dilute alcohol extract it—certain organic solvents do not. Certain chemical reactions common to all preparations containing vitamin *B* are described. Chemical investigation of the extracts apparently excluded phenols, amino-acids and most alkaloids as substances giving the reaction for vitamin *B*, and it would seem that this vitamin is the substance which reduces the ferric-ferricyanide under the conditions described. P. H. P.

Experiments with *B. botulinus* under Household Conditions. R. B. Edmondson, C. Thom and L. T. Giltner. (*Amer. Food J.*, 1923, **18,** 143–146; *Chem. Abstr.*, 1923, **17,** 2230–2231.)—Experiments were made to determine the effect of household storage conditions on the development of botulinus toxin in various foods, including milk, meat and bread. The results showed that, even when *B. botulinus* was present, it did not develop to any appreciable extent when the food was kept for 1 or 2 days in an efficient ice box, and was quite safe for cooking. Even after 3 or 4 days there is little risk, provided there are no indications of spoilage (mould, souring, odour), but in every case thorough cooking is essential.

Estimation of the Titratable Alkali of the Blood with Dinitrosalicylic Acid. J. B. Sumner, R. S. Hubbard and L. L. Finner. (*J. Biol. Chem.*, 1923, **56,** 701–709.)—The following rapid method is a modification of one described by Greenwald and Lewman (*J. Biol. Chem.*, 1922, **54,** 263). Dinitrosalicylic acid is used instead of picric acid, for, unlike the latter, it can be estimated colorimetrically, giving with ferric chloride an intense red colour which is directly proportional to the amount present if too great an excess of the iron salt is not used. The following solutions for standard end-points must be prepared: *Methyl Red Standard End-point*: Five grms. of citric acid dissolved in about 600 c.c. of water, 90 c.c. of 1·32 per cent. dinitrosalicylic acid solution, and 80 drops of methyl red solution are treated with sodium hydroxide until a satisfactory end-point is obtained, and the solution is diluted to a litre and mixed. When 5 c.c. of the blood filtrate are titrated, with methyl red as indicator, 6 c.c. of the above solution with one more drop of methyl red solution in a test-tube controls the end-point; but when using 5 c.c. of blood and 20 c.c. of the blood filtrate and thereby decreasing the percentage error, 25 c.c. of the above solution are required. *Ferric Chloride Dinitrosalicylic Standard*:—Five c.c. of 1·32 per cent. dinitrosalicylic acid solution are mixed with 50 c.c. of 10 per cent. ferric chloride solution, and diluted to a litre. This standard keeps for one week. *Methyl Red Solution*: One hundred mgrms. of

methyl red are ground in an agate mortar with 7·4 c.c. of 0·05 *N* sodium hydroxide until dissolved, and the solution diluted to 250 c.c., and filtered. *Thymolphthalein Solution*: One grm. of thymolphthalein is dissolved in 100 c.c. of alcohol and 0·1 *N* sodium hydroxide added until the colour becomes slightly blue.

The acid is titrated with 0·01 *N* sodium hydroxide until neutralised, methyl red being used. Two c.c. of oxalated blood are diluted with 8 c.c. of water and mixed with 10 c.c. of 1·32 per cent. dinitrosalicylic acid solution, with constant rotation, which is continued for one minute before filtering the mixture into a test-tube. Five c.c. of the filtrate are pipetted into a large hard glass test-tube, heated to boiling, with shaking, boiled for 10 to 15 seconds to expel carbon dioxide, cooled in running water, and titrated with 0·01 *N* alkali after the addition of one drop of methyl red solution, and the coloration compared with the standard end-point. It is titrated again, after addition of 1 drop of thymolphthalein solution, to the first appearance of a green colour. This gives the free dinitrosalicylic acid. To 5 c.c. of the filtrate in a 100 c.c. volumetric flask, 5 c.c. of 10 per cent. ferric chloride are added, diluted to volume, mixed, and compared with the standard. This estimates the free and combined dinitrosalicylic acid. By subtracting the free acid from the total of free and combined acid the titratable alkali of the blood as c.c. of 0·1 *N* alkali per 100 c.c. of blood is obtained. Estimations of the titratable alkali content of the blood in comparison with the carbon dioxide-combining capacity of the plasma gave roughly parallel results in typical cases, but the increase in the former was out of proportion to that of the latter in a case which was receiving alkali therapy. P. H. P.

Micro-Colorimetric Estimation of Hydrogen Ion Concentration of the Blood. V. C. Myers, H. W. Schmitz and L. E. Booker. (*J. Biol. Chem.*, 1923, **57**, 209–216.)—The colorimetric method of Cullen (*J. Biol. Chem.*, 1922, **52**, 501) is adapted to the bicolorimeter of Myers (*J. Biol. Chem.*, **1922, 54,** 675) for the estimation of the hydrogen ion concentration of blood plasma. With this method the error in colour comparison falls within $\pm P_H$, 0·02. The method of taking the blood and separating the plasma is described, and the preparation and preservation of the saline solution. One-tenth of a c.c. of the plasma is discharged from a tuberculin syringe graduated in 0·01 c.c. into 2 c.c. of the saline solution in the cup of a bicolorimeter under oil. This solution is stirred with a rod and its colour is compared with Sörensen's buffer phosphate solutions which have different P_H values (8·0 and 6·8) in wedges of the bicolorimeter. By a simple technique, with the use of oil, contact of the blood with air is entirely excluded. The final estimation requires less than 10 minutes after the blood has been obtained. An adaptation of special tubes for estimating the carbon dioxide content or carbon-dioxide combining power is described. P_H estimations have been made on the blood plasma of more than 100 human cases with this method. In about 25 miscellaneous hospital cases, when abnormal values were not expected, the figures varied between P_H 7·35 and 7·43, with an average of about 7·39. The highest P_H value so far obtained was 7·52 with a carbon dioxide combining power of 87, and the lowest, a P_H of 6·98 with a carbon dioxide combining power of 12. P. H. P.

"Permutit" as a Reagent for Amines. J. C. Whitehorn. (*J. Biol. Chem.*, 1923, **56**, 751–764.)—"Permutit," a synthetic zeolite of the approximate composition $2SiO_2$, Al_2O_3, $Na_2O.6H_2O$, removes relatively strong bases from solution. It separates relatively strong nitrogen bases (those having a basic dissociation constant of about 5×10^{-9} or greater) from weaker nitrogen bases and non-basic substances. In general, alkylamines are removed by permutit, whilst arylamines are not. The interaction of bases and permutit is reversible, and is subject to the Mass Law. The system ammonium chloride and sodium permutit in water shews this: $NH_4Cl + Na\ permutit \rightleftarrows NH_4\ permutit + NaCl$. In this system the equilibrium constant K has a value 0·75 in the expression:— $(NH_4Cl)(Na\ permutit) = K(NH_4\ permutit)(NaCl)$. The temperature perceptibly influences the rate but not the equilibrium. In the expression $k_{t_2} = k_{t_1} \varphi_{10}^{\frac{(t_2-t_1)}{10}}$ in the range between room temperature and 0° C., the temperature coefficient φ_{10} has a value 1·6. The hydrogen ion concentration considerably affects these reactions, for in half-hour tests more of a given base is removed from a neutral solution than from an acid or alkaline solution. Permutit removes bases from solution in certain organic solvents, as well as in aqueous solutions. These are varying concentrations of ethyl alcohol, amyl alcohol and ether. For quantitatively removing bases from solution, filtration through permutit has several advantages over agitation with permutit. More base is removed by a given quantity of permutit, since the sodium salt formed by the reaction which would tend to reverse it does not accumulate; also, successive portions of a relatively large volume of fluid can be rapidly brought into intimate relation with the permutit; and, easily oxidisable substances are saved from destruction. A saturated aqueous solution of potassium chloride is a good general reagent for the recovery of bases from permutit. Special procedures can be used in special cases. *Estimation of Adrenalin*:—The author has developed a procedure for the colorimetric estimation of adrenalin based upon its separation by permutit from uric acid and non-basic polyphenols, the reduction of alkaline phosphotungstate solution and the use of a polarimeter tube in colorimetric comparison as used by Folin and Denis for blood ammonia estimations. (*J. Biol. Chem.*, 1912, **11**, 527.) Known quantities as small as 0·006 mgrm. of adrenalin in 50 c.c. can be estimated with a 90 per cent. accuracy. It is not yet a wholly satisfactory method of adrenalin estimation for biological fluids in general. P. H. P.

Toxicological and Forensic.

Contribution to the Study of the Toxicity of Mercuric Cyanide. R. Fabre and J. Josset. (*J. Pharm. Chim.*, 1923, **28**, 81–89.)—Acids such as sulphuric, phosphoric, lactic, tartaric, oxalic and glycollic in concentrations of 1 per cent. only decompose mercuric cyanide to a small extent, and 1·25 to 3·7 per cent. of hydrocyanic acid is liberated. In concentrations of 10 per cent. the proportion of hydrocyanic acid liberated varies for the different acids from 5 to 17·6 per cent. Proteins, however, exert a much more powerful influence, and it

was found that a 1 per cent. solution of mercuric cyanide in contact with blood, serum, milk, gastric juice, urine, broth, and meat caused a liberation of hydrocyanic acid amounting, in the case of gastric juice, to as much as 88 per cent. It therefore follows that in toxicological investigations for volatile poisons hydrocyanic acid found in the distillate may have been derived from mercuric cyanide, and not necessarily from an alkali cyanide. Mercury would have to be looked for in the residue. In cases of suspected poisoning by mercuric cyanide or alkali cyanides, it is important to carry out the investigation at the earliest possible moment, as the hydrocyanic acid is rapidly transformed into ammonium formate and thiocyanic acid. The fact that mercuric cyanide so soon undergoes decomposition in the body accounts for the fluidity of the blood in such cases of poisoning.

D. G. H.

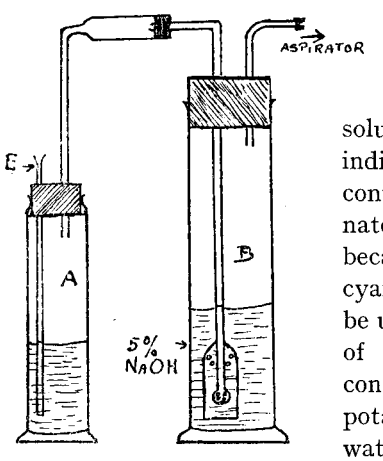
Agricultural Analysis.

Comparative Study of Certain Methods of Estimating Humus in Soils. V. Agafonoff. (*Comptes rend.*, 1923, **177**, 404–406.)—Tests made with the black soils of Russia, Poland and Rumania, show that the percentages of carbon found by calcining the soil and weighing the absorbed carbon dioxide, agree closely with those obtained by Simon's method, in which the humus is oxidised by means of silver bichromate and sulphuric acid (*Comptes rend.*, **1923, 176,** 1409). The chlorine index appears to indicate approximately the proportion of humus in a soil.

T. H. P.

Organic Analysis.

Estimation of Cyanide by Aeration. J. H. Roe. (*J. Amer. Chem. Soc.*, 1923, **45,** 1878–1883.)—The method consists in aspirating air through an acidified solution of the cyanide and washing the cyanide-laden air by passing it through dilute sodium hydroxide solution. The resulting sodium cyanide is titrated with silver nitrate solution, with the use of potassium iodide as indicator. The process is applicable to all mixtures containing soluble cyanogen compounds, and eliminates the errors and disadvantages of distillation, because there is no loss due to hydrolysis of hydrogen cyanide and no danger to the operator; also it may be used for the estimation of cyanide in the presence of ferrocyanides. A quantity of the substance containing the equivalent of about 0·05 grm. of potassium cyanide is dissolved in about 100 c.c. of water in the vessel A, 25 c.c. of saturated solution of tartaric acid are added through E, and air is drawn through the apparatus at the rate of about 3 litres per minute for 2 hours; the absorbing liquid in B is



5 per cent. sodium hydroxide solution. A few drops of amyl alcohol may be added to prevent frothing, if necessary. In the case of a non-dissociating cyanide, such as mercuric cyanide, the compound is reduced to the mercurous state by adding to the solution in A a few crystals of sodium chloride and 10 c.c. of 5 per cent. solution of stannous chloride. For the titration, 1 drop of 10 per cent. potassium iodide for each 15 c.c. of sodium hydroxide solution is added, and standard silver nitrate solution run in until a permanent precipitate of silver iodide appears. The aeration method is accurate to 0·00005 grm. per c.c. of solution; the air aspirated must be free from hydrogen sulphide, but other common impurities do not interfere. H. E. C.

Estimation of Eugenol in Essential Oils. P. N. van Eck. (*Pharm. Weekblad*, 1923, **60**, 937–940.)—A weighed quantity (2 to 3 drops) of clove oil, or other essential oil containing eugenol, is treated with 25 c.c. of 0·1 *N* silver nitrate solution, to which sufficient ammonia has been added just to dissolve the precipitate first formed. The flask is then heated beneath a reflux condenser for a short time until a silver mirror is formed on the sides, after which the liquid is filtered, the filter and flask washed, the filtrate slightly acidified, with nitric acid, and the unreduced silver titrated with 0·1 *N* thiocyanate solution. Experiments with pure eugenol showed that the mgrms. of reduced silver divided by the mean factor 1·75 give the corresponding weight of eugenol.

Occurrence of *l*-Menthone in Pine Oil. A. H. Gill. (*J. Ind. Eng. Chem.*, 1923, **15**, 887.)—A fourteen-year-old specimen of pine oil, prepared originally by the dry distillation of wood, was distilled under 4 mm. pressure. About 60 per cent. of the oil distilled between 74° and 79° C. By repeated fractional distillation this portion was separated into two fractions; the first fraction, amounting to 8 per cent. of the original oil, boiled at 202° to 203° C., and consisted of inactive fenchyl alcohol. The second fraction, constituting 20 per cent. of the original oil, boiled at 208·5° to 209·5° C., had an optical rotation of −20°28′, and was identified as *l*-menthone by converting it into its semi-carbazone, m.pt. 184° C. W. P. S.

Separation of Asphaltenes from Mineral Oils by means of Cyclohexane. M. Jakeš. (*Chem. Zeit.*, 1923, **113/114**, 757.)—The author recommends the use of purified cyclohexane instead of petroleum spirit (benzine) for the separation and estimation of asphaltenes in mineral oils. The purification is carried out as follows: One kilo of commercial cyclohexane (b.pt. 81° C.; sp. gr. 0·783 at 15° C.) is treated with concentrated sulphuric acid, shaken 3 times for half-an-hour with a solution of 30 grms. of trioxymethylene in 100 grms. of sulphuric acid, and then again with sulphuric acid alone. The product is washed with dilute sodium hydroxide solution and with water, dried with calcium chloride and distilled. Comparative analyses with benzine and cyclohexane of a cylinder oil rich in asphaltenes showed that the former solvent gives a higher result (1·38 per cent.) than the latter (0·36 per cent.). Moreover, the separated asphaltenes were viscous and brown-black in colour if benzine was used, whereas with cyclohexane they were more granular and black. R. F. I.

Method for Determining the Detergent Action of Soaps. J. W. McBain, R. S. Harborne and A. M. King. (*J. Soc. Chem. Ind.*, 1923, **42**, 373–378T.)—A method is described for the rapid direct determination of the amount of finely-divided carbon which various soap solutions carry through filter papers. The "carbon number" thus obtained is characteristic of any soap solution, and may be taken as a measure of the detergent action of the material. A gravimetric method of estimating the carbon is given, and this is essential as an absolute standard, but satisfactory results are obtainable more rapidly by colorimetric comparison with a solution containing Nigrosine and Bismarck Brown. The results obtained show that by a slight alteration of conditions the detergent action of a soap may be enormously increased. An optimum concentration of the soap exists for which the effect is a maximum, but very slight addition of either acid, or, preferably, alkali, greatly enhances the detergent power. This is diminished, at first slowly and afterwards more rapidly, by rise of temperature, but this effect may be completely overshadowed by the vital necessity of using a sufficiently high temperature to dissolve the soap. When dissolved, soaps as different as potassium myristate and potassium oleate exhibit remarkably little difference in detergent power. The possibility that the action of soap on carbon black is to a certain extent specific, and hence not quite parallel to the effect on oily matter, or on emulsification, or on frothing, may restrict the value of the above results.

T. H. P.

Estimation of β-Naphthol in α-Naphthol and of α-Naphthol in β-Naphthol. J. Prochazka. (*J. Ind. Eng. Chem.*, **1923**, **15**, 944–945.)—To estimate β-naphthol in α-naphthol 0·36 grm. of the sample is dissolved in 40 c.c. of alcohol, and the solution is cooled below 5° C., and treated with *p*-nitro-diazobenzene solution, of which 100 c.c. are equivalent to 1 grm. of nitrite; this solution should contain only a very slight excess of nitrous acid, and not too large an excess of hydrochloric acid. If the α-naphthol is pure (100 per cent.), 17·25 c.c. of the diazo solution would be required to convert it into the azo compound. If the sample is supposed to contain about 10 per cent. of β-naphthol, 14·6 c.c. of the diazo solution are added; the coloured compound begins to separate at once, and, after thirty minutes, a drop of the solution is treated on a piece of filter paper with a drop of dilute sodium hydroxide solution. A blue line appears at the junction of the two spots if unchanged α-naphthol is present. A further quantity of the diazo solution is then added, and the mixture again tested, and so on, until a red line (para-red) due to the β-naphthol compound appears on the spot tested. The quantity of diazo solution used is a measure of the amount of α-naphthol present. For the estimation of α-naphthol in β-naphthol, 1·44 grm. of the sample is dissolved in 50 c.c. of alcohol, and the solution is treated with 3 c.c. of *p*-nitrodiazobenzene solution and 0·03 grm. of nitrite. If the percentage of α-naphthol is less than 0·5, all of it, together with some of the β-naphthol, will be precipitated as the *p*-nitroazobenzene compound. After thirty minutes the mixture is diluted with 60 c.c. of boiling water, and the precipitate is collected on

a filter and washed with hot water. All the uncombined β-naphthol passes into the filtrate, together with some impure azo compound of the α-naphthol. The precipitate is washed off the filter, boiled with 50 c.c. of hot water containing 1 c.c. of 25 per cent. sodium hydroxide solution, the mixture is filtered, the filtrate diluted to 100 c.c., and the coloration compared with that of a standard *p*-nitroazobenzene α-naphthol solution. This is prepared by dissolving 0·1 grm. of the azo compound in 25 c.c. of warm alcohol containing 1 c.c. of 25 per cent. sodium hydroxide solution, and diluting the mixture to 50 c.c. with hot water. The alcoholic filtrate is also rendered alkaline and its colour compared with that of the standard. W. P. S.

Ultra-filtration of Tannin and other Solutions. R. J. Browne. (*J. Soc. Leather Trades Chem.*, 1923, **9**, 365.)—The paper suggests an alternative method of analysis of tannin solutions which rests on quite a different principle to the usual hide-powder method, and appears to overcome one of the recognised weaknesses of the older method, *viz.* that part of the gallic acid in a tan liquor is returned as tannin. The method depends on the fact that tannins are colloids, and by filtration through a suitable membrane can be separated from non-tans, which are crystalloids. The membrane described is prepared by soaking filter-paper in a 6 per cent. solution of collodion in alcohol-ether, allowing it to dry till quite opaque (not longer), and then keeping it under water for an hour or until required. Thus prepared, the membrane is of standard permeability. For use it is placed in a modified form of Bechhold's pressure-filtration apparatus, and the tannin solution containing 4 grms. of tan per litre is filtered through it. The method gives a figure for tannins and insolubles, and no attempt has yet been made to separate these. Comparative results obtained by this method and the hide-powder method are given, and these show agreement in the case of various fresh tanning materials. In the case of solutions containing gallic acid (*e.g.* spent tan-liquors) the results do not agree, since gallic acid passes through the membrane, but is largely retained by hide-powder. R. F. I.

Estimation of Total Sulphur in Rubber Goods. S. Collier, M. Levin and R. T. Mease. (*J. Ind. Eng. Chem.*, 1923, **15**, 953–955.)—The following procedure is recommended by the Bureau of Standards. The rubber (0·5 grm.) is treated with 15 c.c. of concentrated nitric acid saturated with bromine and the mixture is evaporated to dryness. The residue is mixed with 3 c.c. of nitric acid and 5 grms. of sodium carbonate, dried, and fused. After cooling, the fused mass is dissolved in water, the solution filtered, the filtrate acidified with hydrochloric acid, diluted to 300 c.c., and the sulphuric acid precipitated as barium sulphate. The weight of barium sulphate obtained must be corrected for the presence of occluded salts; this correction is found by precipitating known weights of sodium sulphate with barium chloride in the presence of the quantities of nitric acid, sodium carbonate, etc., mentioned. W. P. S.

Detection of Chondrin in Gelatin. M. A. Rakusin. (*Chem. Zeit.*, 1923, **47**, 602.)—All gelatin, after manufacture, especially if it is for photographic

purposes, must be tested for chondrin, which injures its properties. Until quite recently this was done by adding a saturated solution of chrome alum, with constant stirring, to a hot 10 per cent. solution of the gelatin, which was immediately gelatinised if any chondrin were present. Now the new analytical-chemical data serve for a qualitative and quantitative comparative analysis of gelatin and chondrin. Probably why so little is known of the physico-chemical nature of chondrin is owing to the powerful opalescence of all its solutions, one of 0·2 per cent. concentration appearing milky and opaque in an ordinary test-tube. In contrast to this, Rakusin and Brands (*J. russ. phys.-chem. Ges.*, 1917, 200) showed that cold gelatin solutions, quite clear up to 0·75 per cent., may be titrated with alkali, giving the expected glutinate. Pure gelatin gives only three of the eight colour reactions of the proteins, *viz.* the biuret, the Molisch and the Ostromyslenski reactions, whilst chondrin gives the xanthoprotein reaction as well. The author proposes a test for detecting chondrin and quantitatively estimating chondroitin-sulphonic acid in gelatin. This acid differs from sulphuric acid in exhibiting optical activity ($[\alpha]_D = +46·5$). By acting directly on a 0·2 per cent. chondrin solution with a dilute barium chloride solution no reaction is noticed owing to the pronounced opalescence, but by first treating the chondrin solution with 10 per cent. aluminium hydroxide, which combines with, or, as was formerly thought, adsorbs the protein residue, a perfectly clear filtrate is obtained, which only reacts with barium chloride, does not shew the albumin reaction and quantitatively contains all the chondroitin-sulphonic acid. Unlike chondrin, gelatin in aqueous solution reacts directly with barium chloride, and this reaction can also be used for the quantitative estimation of chondroitin-sulphonic acid in gelatin.

P. H. P.

Valuation of Dyestuffs by Titration Methods. R. B. Brown and H. Jordan. (*J. Soc. Dyers and Col.*, 1923, **39**, 203–208.)—Dyestuffs may be estimated with satisfactory accuracy by a volumetric method based on the mutual precipitation of acid and basic colours. The dyestuff to be estimated and its precipitant must be of totally different colours in solution, and the acid colour should be run into the solution of the basic dyestuff, the reverse procedure seldom giving a useful result. Use is made of solutions containing 1 grm. of dyestuff in 500 c.c., the end-point of the titration being recognised by the appearance in a spot made on filter paper of a ring of the colour of the precipitating solution; the tinctorial value is estimable in this way to within 2·5 per cent., which is much nearer than a practised eye can judge in the case of comparative dyeings. For auramine, indigo carmine is used as precipitant; for brilliant green or malachite green, orange II., and for magenta, brilliant green, both these precipitants being used in conjunction with tannin and sodium acetate; for methyl violet, naphthol yellow S; for methylene blue, tartrazine or crystal scarlet, together with tannin and sodium acetate; for rhodamine (B and 6G), orange MNO (concentrated metanil yellow) and tannin; for safranine, indigo carmine with tannin and sodium acetate; and for Victoria blue B, tartrazine alone. The method has been applied to only four acid dyestuffs,

namely, orange II., orange I., tartrazine and naphthol yellow S, the basic colour employed in all cases being Victoria blue B or night blue. With some dyestuffs, accurate estimation is possible only by adding excess of the precipitant and determining the excess by titration. T. H. P.

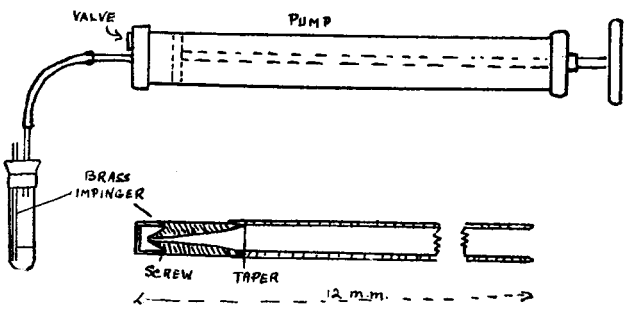
Inorganic Analysis.

Detection and Estimation of Indium. I. Wada and S. Ato. (*Rap. Inst. Phys. Chem. Research* (Japan), 1922, **1**, 57–77; *Chem. Abstr.*, 1923, **17**, 2250.)—Unlike the members of the tungsten and columbium group, indium nitrate solutions are not hydrolysed to form a salt insoluble in nitric acid when they are evaporated to dryness and the residue heated at 120° C. Indium cannot be separated from other elements by distillation of the bromide or precipitation of the chloride. The sulphide, In_2S_3, is not precipitated to any material extent by hydrogen sulphide in solutions of an acidity of 0·3 *N*. The sulphide is liable to be carried down with other sulphides, however, and it is therefore best, when precipitating the copper group, to pass hydrogen sulphide through the solution at an acidity of 0·6 *N*, to filter off the precipitate, dilute the filtrate with an equal volume of water, and to precipitate again with hydrogen sulphide. The second precipitate will contain some platinum, indium, rhodium, and lutecium sulphides, but all of these elements will be present in the filtrate. For the separation of indium from bismuth, the greater part of the bismuth should be removed by hydrolysis, and the remainder by precipitation with hydrogen sulphide in 0·6 *N* acid. A gelatinous precipitate of indium hydroxide, $In(OH)_3$, or of a sulphide or a carbonate is formed by ammonium hydroxide, ammonium sulphide, sodium hydroxide, sodium peroxide and sodium carbonate, so that indium behaves like titanium or zirconium with these reagents. In the presence of tartrate ions indium can be precipitated as sulphide, like iron, and thus separated from titanium and zirconium.

Physical Methods, Apparatus, etc.

Calculation of Flash Points of Pure Organic Substances. E. Mack, C. E. Board and H. N. Barham. (*J. Ind. Eng. Chem.*, 1923, **15**, 963–965.)—Thornton (*Phil. Mag.*, 1917, **33**, 190) has shown that in a mixture of air and a combustible gas or vapour, the minimum explosive mixture contains about twice as many oxygen atoms and the maximum mixture about one-half as many oxygen atoms as are required for complete combustion. Adopting this rule, the authors have calculated the flash points of a number of pure substances (hydrocarbons, esters, alcohols, phenols, etc.) by deducing from the vapour pressure the temperature to which the substance must be brought in order that its saturated vapour will furnish a mixture with air which is just explosive. The calculated flash points agreed very well with the values obtained by actual experiment. W. P. S.

New Method of Dust Estimation. R. N. Kotzé. (*J. Chem. Met. and Min. Soc., S. Africa*, **1923, 24,** 1–6.)—A simple form of apparatus, named the hydro-konimeter, is described which is suitable for counting the dust particles in mine air. It consists of a hand exhaust pump connected with a stout glass tube containing 2 c.c. of water and a brass impinger which dips just below the surface of the water, and has a nozzle of diameter 0·6 to 0·8 mm. at distance 1 mm. from the baffle plate (see fig.). The necessary high velocity of air through the impinger (200 to 300 m. per second) is maintained by a specially designed valve



on the pump, which prevents the partial vacuum in the pump from falling below about 1/3 atmosphere. This device maintains a constant velocity at the nozzle, which is essential. About 8 strokes of the pump are made for each test, which occupies about one minute; the volume delivered by each stroke is about 520 c.c. Calibration is effected by counting the number of strokes required to remove the air from a wide-mouthed bottle of known capacity inverted in a deep vessel of water. The limit of accuracy is within about 10 per cent. For the examination and enumeration of the dust particles which have been collected, 0·5 c.c. of hydrochloric acid is added to dissolve calcium sulphate and ferric hydroxide; then a few drops of the mixed turbid liquid are transferred to a cell made by drilling a hole of 1 mm. diameter in a flat square of brass 1 mm. thick placed on a microscope slide, and covering the cell with a greased cover slip. The number of particles which may be counted on the bottom of the cell by the aid of an eye-piece micrometer depends on the time allowed for the particles to settle. Particles of diameter 0·1μ require 8 hours to fall through 1 mm., and those of 5μ diameter fall in 0·2 minute in accordance with the approximate formula—

$$\text{Velocity (in mm. per sec.)} = K(d-1)D^2.$$

K for quartz in water is 0·0021, "d" is the density (2·65) and D the diameter in μ. A settling time for 20 minutes includes all particles over 0·5μ diameter and gives results comparable with those of the konimeter described by Greenburg and Smith (*Amer. Bureau of Mines*). H. E. C.

Spectrophotometric Identification of Dyes. W. C. Holmes. (*J. Ind. Eng. Chem.*, **1923, 15,** 833–836.)—The degree in which the intensity of the absorption of solutions of dyes is modified by variations in concentration, solvent,

alkalinity, acidity, and other conditions, depends on the degree of alteration of the dye in molecular form or aggregation, and this, in turn, depends on constitutional differences. The quantitative measurement of the intensity of absorption under such variation of conditions affords, therefore, spectrophotometric ratios or constants which are of value for purposes of identification. A scheme is outlined for the identification of acid blues of the Patent Blue type, and a comprehensive table of constants for these dyes is given. W. P. S.

Use of Dyes as Temperature Indicators. P. A. Kober. (*J. Ind. Eng. Chem.*, 1923, **15**, 837–838.)—There are numerous dyes which, when subjected to heat, decompose with complete disappearance of their colour; this change, however, is a function of the time of heating as well as of the temperature. For instance, the colour of erythrosin disappears after sixty minutes' heating at 350° C., and after six minutes' heating at 375° C. This method of measuring heat is useful in determining the heat distribution in any material, in the annealing of glass, in baking cements, for indicating overheating in electrical instruments, etc. W. P. S.

Radiator for Platinum Crucibles. M. M. Green. (*J. Ind. Eng. Chem.*, 1923, **15**, 890.)—A piece of wire gauze, 12·5 cm. square, is formed into a deep capsule by simply hammering in the centre as far as possible; the edges are bent over to provide a means of suspending the apparatus on a ring stand. A disc of gauze is fitted in the gauze capsule about 2 cm. from the bottom, and a triangle is fixed 1·5 cm. above the disc. The crucible is placed on the triangle and, when the apparatus is arranged over a burner, any liquid in the crucible may be evaporated smoothly to dryness. W. P. S.

Reviews.

PRACTICAL PHYSICAL CHEMISTRY. By A. FINDLAY, M.A., D.Sc., F.I.C. 4th Edition. Pp. xvi.+298. London: Longmans, Green & Co. 1923. Price 7s. 6d. net.

That a book of this kind, offered as a students' guide, should have reached a fourth edition, is evidence, if any is needed, of the advances made by physical chemistry, not only in correlating the various physical and chemical properties of bodies, but as an interesting and acceptable branch of experimental science. To the student of chemistry towards the later end of last century "physical chemistry" was merely a phrase disguising a nauseous variety of mathematics, unreal rather than merely abstract, and taking up time which was needed for the study of "real chemistry."

This book, like most others of its kind, inspired by Ostwald's *Physical Chemical Measurements*, leads the student *via* a preliminary consideration of limits of accuracy, methods of calculation and errors, which is necessary, although it might well receive proper treatment at school, through a representative course of physical measurements. When at an institution of university rank it is seriously considered whether students should be given a course of English to enable them to write

connected notes, it is perhaps not unreasonable for a book of this kind to start with a simple dissertation on accuracy and errors.

In dealing with the calibration of glass apparatus the author mentions the blowing-out method of using pipettes. Although Ostwald advises this, and it undoubtedly yields concordant results, it seems inadvisable to suggest its use, as pipettes verified by institutions like the National Physical Laboratory or the Reichsanstalt are all tested by touching off. The author does not mention the modern type of graduated apparatus which, by means of long graduations extending completely or nearly all round the tube, enable parallax errors to be avoided. He also, like other authors of books on physical chemistry, recommends the Schellbach burette, which, for exact work, is banned by the National Physical Laboratory.

Surface tension is usually treated in an unsatisfactory manner in books on physical chemistry. The methods given—the capillary rise and the drop methods—are undoubtedly convenient for chemists by reason of their relative simplicity, but neither is theoretically irreproachable unless the angle of contact is well known, which is seldom the case.

There seems to be no justification for attributing the equation $2\pi r.\gamma = W$ to Tate, who merely called attention to the approximate linear relationship between the radius (or diameter) of the tube from which a drop falls and the weight of the drop. Further, there is no justification, theoretically or in practice, for the equation at all. As long ago as 1899 the late Lord Rayleigh wrote: "Even if the tension at the circumference of the tube acted vertically and the whole of the liquid below this level passed into the drop, the calculation would still be vitiated by the assumption that the internal pressure at the level in question is atmospheric. It would be necessary to consider the curvatures of the fluid surface at the edge of attachment. If the surface could be treated as a cylindrical prolongation of the tube (radius a), the pressure would be T/a, and the resulting force acting downwards upon the drop would amount to one-half ($\pi a T$) of the direct upward pull of the surface tension along the circumference. At this rate the drop would be but one-half of that reckoned above." Actually he found that the substitution of $3{\cdot}8\ (=1{\cdot}2\pi)$ for 2π gave values similar to those found experimentally. Worthington, in 1881, had called attention to the inaccuracy both on theoretical grounds and in actual practice.

The author has given more attention than usual in such books to surface tension, including references to several recent papers, but a larger outer vessel for the capillary rise methods (at least 5 cm. diameter), and a much slower rate of dropping from the stalagmometer than 20 drops a minute is desirable.

The chapter on colloids is new, and includes useful matter which should give an insight into the methods used in the study of this "world of neglected dimensions."

A little more information on the determination of hydrion concentrations would have been useful, especially some reference to the use of indicators and buffer solutions.

The book is written in a clear and interesting style. It is not the author's fault that an overweighted curriculum may tend to make the student feel that science is a task rather than an adventure. Those who are really interested are given abundant references to original papers, the reading of which is a most fruitful way of grasping reality. It is to be hoped that many who are not, in the formal sense, students, will place this book on their shelves, or better, on their tables. The enterprising analyst will find in it many means of attack which are not to be found in the ordinary books on analytical chemistry.

The author uses the symbols recommended by the International Commission on Physico-chemical constants, of which he was secretary, a course much to be commended.

J. H. COSTE.

THE PHASE-RULE AND ITS APPLICATIONS. By A. FINDLAY, M.A., D.Sc., F.I.C. Fifth Edition. Pp. xvi.+298. London: Longmans, Green & Co. Price 10s. 6d. net.

This well-known work now appears in a revised, enlarged and entirely new form. During the last few years the Phase Rule has played a considerable part in the systematic development of chemistry, and the author therefore has re-written certain sections and included in the new edition such discussions as are representative of recent researches. Thus, in his treatment of phosphorus as an example of a one-component system advantage has been taken of the work of A. Smits, E. Cohen and their respective pupils. Then again, due emphasis has been placed on the recent practical applications of the Phase Rule. In the treatment of binary systems, iron-carbon alloys have received increased attention, and in the section on ternary systems a description of some thermal studies of minerals, illustrated by means of the elaborate and extremely important system, $CaO-Al_2O_3-SiO_2$, has been introduced. The bearing of this system on the formation of igneous rocks and of Portland cement clinker has been indicated. A study of the deposition of oceanic salts, such as has taken place at Stassfurt, now finds a place in the chapter on four component systems.

The developments in Phase Rule work have necessitated extra methods of graphical representation, for example, the methods devised by Jänecke to illustrate his studies on ocean salt solutions. For certain systems the Jänecke diagrams afford much more satisfactory representations. It is gratifying, therefore, to see that Prof. Findlay has described these methods in the present edition.

It can be safely said that we have in this text-book an admirable and concise exposition of the Phase Rule and its applications. The volume furnishes a remarkably complete outline of the subject, due, to a great extent, to the careful selection of the many systems considered.

Finally, mention must be made of the excellent manner in which the volume has been produced; it is now uniform in appearance with that of the later volumes of the Ramsay-Donnan Series of Text-books of Physical Chemistry.

HUBERT T. S. BRITTON.

THE BRITISH PHARMACEUTICAL CODEX. 1923. London: The Pharmaceutical Press. Pp. xxi.+1669. Price 30s. net.

In 1886 the British Pharmaceutical Conference passed the following resolution: "That in order to secure greater uniformity in composition and strength in non-official remedies, and also to enable the medical profession to prescribe them with definite knowledge of those qualities and without indicating any particular maker, the Conference undertakes the preparation of a formulary of non-official remedies." In the next year the first "Unofficial Formulary" of twelve pages was published as an addendum to the Year Book of Pharmacy. The letters "B.P.C." were to be used to distinguish such preparations. In subsequent years the formulary was enlarged, and had a considerable circulation, until, in 1904, the formulæ were acquired by the Pharmaceutical Society, and used in the first edition of the British Pharmaceutical Codex, published in 1907.

The "B.P.C." part of the third edition of the Codex consists of 341 pages of formulæ. It has been prepared by a committee of pharmacists, and supplements the Pharmacopœia by providing methods for making unofficial preparations, intended to be therapeutically effective as well as palatable and permanent, and is the result of experiment and experience. This part also contains general remarks on the preparation and uses of each class of medicaments, such as capsules, emulsions, pills, liniments, etc. Under "Mixtures," the following remarks are of interest: "Mixtures prescribed in concentrated form, or containing potent doses of medicament, and mixtures intended for administration to children or infants, should be made up to the exact volume prescribed, and dispensed in a bottle, bearing, in addition to the prescribed directions, instructions that each dose be measured in a properly graduated glass. The doses indicated by graduated bottles (especially short bottles bearing several graduations) are rarely sufficiently uniform for the administration of potent mixtures. By means of the larger sizes of graduated bottles more uniform doses can usually be measured, and these form a sufficiently accurate approximation to the intention of prescribers as regards the administration of ordinary forms of mixtures." As a rule, there are no tests, but a full account is given of surgical dressings, with standards and methods of analysis; also particulars of "witness tubes" for use as a test of efficient sterilisation. This part is authoritative for any substance mentioned in a prescription with the letters "B.P.C." after it. For instance, "Emulsio Olei Morrhuæ, B.P.C." should contain 50 per cent. of cod liver oil.

The preface claims that the book contains "information respecting all drugs and medicines in common use throughout the British Empire." The chief aim of the work is stated to be "The provision of accurate information for prescribers and dispensers." The first part of the work consists of 1174 pages, in which medicaments appear in alphabetical order, besides chemicals, both inorganic and organic, and crude drugs. There are monographs on sera, radium and its standardisation units, colloidal solutions and such animal substances as thyroid and insulin. Brandy and whisky are also given, and it is stated that whisky is "frequently preferred to brandy because it is more readily obtained unadulterated."

Articles which to the analyst are of more interest as reagents than as drugs are included, as, for instance, wood spirit, peptone, sodium taurocholate, pure dextrose, agar-agar, and methylene blue.

Vegetable drugs are described in full, including the characteristics of commercial varieties and adulterants, with microscopical details when required. In addition to the official oils, coconut, cotton seed, lard, palm, peach kernel and soya oils are included.

Under "preparations" there are directions for making tinctures, pills, etc., in which the drug is used. No less than 32 preparations are shown under "rhubarb." The preparations may be divided into past, present and future. The *past* is represented by articles still in use which were in former British, London or Edinburgh Pharmacopœias; the *present* by all the preparations in the last editions of the British and United States Pharmacopœias, and also by some contained in foreign pharmacopœias. The "B.P.C." formulary preparations offer a selection of tried remedies for *future* pharmacopœias. Chemicals of a definite composition have the formulæ indicated with molecular weights, based on the 1921 international atomic weights. Descriptions and methods of preparation are included.

Under "Action and Uses" appear details of the effects of drugs, their doses and methods of administration, as well as notes on incompatibility and antidotes for poisons. There is also an extensive pharmacological and therapeutic index. Under "Boric acid" is the following note: "The excretion of boric acid is very slow, so that it is cumulative. This, and the fact that it is without taste or odour, renders it more dangerous. Its use as a food preservative is highly undesirable." Some remarks are made on the use of preparations. The composition of "Brompton Cough Lozenges" is indicated, and then: "These lozenges are given to allay cough"; therefore there is no excuse for thinking they are to be used for another complaint, say, housemaids' knee. In drug prosecutions, questions are sometimes asked as to the common uses of the drug in question, but to the analyst who has consulted the Codex such questions will cause no difficulty.

The work contains much analytical information. The constituents of the drugs are stated, often quantitatively, and solubilities in water, cold and boiling, alcohol, ether, glycerin, oils, etc., appear as the case may require. The B.P. statement that strychnine hydrochloride is soluble 1 in 60 is corrected to 1 in 35·5. In ash and other characteristics maxima and minima values are often both given. The B.P. requires liquorice root to yield not less than 20 per cent. of aqueous extract, and not more than 6 per cent. of ash. The Codex limits are 20 to 27 per cent. and 3 to 6 per cent. respectively. When oils occur in the B.P. and the U.S.P., their official specific gravities as given by each authority are quoted. The B.P. tests are corrected where necessary; for instance, the need for addition of acid in the titration of sodium hypophosphite and reduced iron is pointed out. The official melting point of acetylsalicylic acid is 133 to 135° C., but the Codex states that pure dry samples melt at 137 to 139° C. As a rule, the B.P. methods of alkaloidal assay are not quoted, but under nux vomica assay it is pointed out

that the heating with nitric acid should be at 15 to 20° C., and not at the official 50° C.

Under "Olive oil" the percentage of unsaponifiable matter is given, in addition to the B.P. constants, and also the effect of adulteration on the constants. Three c.c. of water are added to the test for sesame oil to avoid a false colour, and there is a test for tea seed oil. There is no suggestion that the official limits for arsenic and lead are unduly stringent, except in the case of acid sodium phosphate where the official limit is two parts of arsenic, although its ingredients are allowed five parts per million. Occasional analytical facts are stated which are sometimes not easy to find, such as flash-points of ether and benzene, tests for rhapontic rhubarb in rhubarb, and for capsicum in essence of ginger, and the action of different degrees of heat on boric acid and olive oil. Some coefficients of expansion are given.

The B.P. is a "presumptive standard" for all articles contained in it, and the Codex published by the direction of the Council of the Pharmaceutical Society of Great Britain may be considered a similar standard for articles outside the official volume. Saffron, turned out of the B.P., has found a resting place here. "Extra Strong Seidlitz Powders" are not mentioned in the B.P., but the Codex requires them to have one and a half times the B.P. proportion of Rochelle salt. Recently the writer received samples of colourless tincture of iodine for analysis, and each of ten wholesale drug houses written to, replied that the firm accepted the formula of the Codex for the preparation.

The Codex is also useful in giving indications of what is called the "custom of the trade"; for instance, what should be given when "magnesia" is asked for? The answer of the Codex is "The practice is not uniform. In the majority of cases what is intended and supplied is the light carbonate." "Tincture of Rhubarb" may be considered to be the article given in the 1885 B.P. The Codex observes that the 1914 "Compound Tincture of Rhubarb" is also known as "Tincture of Rhubarb."

At the end of the book there are formulæ for test solutions and microscopical stains, and eleven pages of a seven columns table for converting percentages into grains or minims, per pint, pound, or ounce. The index is very full, occupying over a hundred pages.

The book is well printed, only one slight typographical error being noticed, and is substantially bound. J. F. LIVERSEEGE.

JOURNAL OF SCIENTIFIC INSTRUMENTS.

THE first number of this journal was published on October 15th.

CONTENTS.—"The Measurement of True Height by Aneroid," by L. N. G. Filon; "Two New Methods of Measuring the Internal Diameters of Transparent Tubes," by J. S. Anderson and G. Barr; A New Relay and its Application to Sustaining Pendulum Vibrations," by H. A. Thomas; "A General-Purpose Recording Drum," by C. V. Boys; "The Chain Balance," by Sir Flinders Petrie; "A New Recording Kata-Thermometer," by E. H. J. Schuster. Miscellanea. Reviews.

Published by the Institute of Physics. Price 2s. 6d. Annual Subscription 30s., post free.