

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS

Food and Drugs Analysis

Composition of Shell Eggs. L. C. Mitchell. (*J. Assoc. Off. Agric. Chem.*, 1932, **15**, 310–326.)—The methods used for determining total solids, chlorine, phosphorus, organic and ammoniacal nitrogen, and dextrose, are those described in *Methods of Analysis (A.O.A.C.)*, 1930, 244–249. Crude albumin nitrogen is taken to be the water-soluble nitrogen precipitable by 40 per cent. alcohol. The fat was determined by hydrolysis as follows:—Approximately 2 grms. of yolks, or 3 grms. of whole eggs, or 5 grms. of whites, are weighed accurately by difference from the well-mixed sample into a Mojonnier fat-extraction tube, 10 c.c. of

concentrated hydrochloric acid being then added slowly and with vigorous shaking. The tube is placed in a water-bath at 70° C., this being then brought to the boiling point and kept boiling for 30 minutes, during which time the tube is carefully shaken at five-minute intervals. The tube is afterwards removed from the bath, and, after addition of water (better than alcohol) to fill the lower bulb of the tube, cooled to room temperature. The contents of the tube are next mixed, first with 25 c.c. of ether and then with 25 c.c. of redistilled petroleum spirit (b.pt. below 60° C.), and allowed to stand until the ethereal layer becomes clear. This layer is decanted into a weighed 125-c.c. beaker-flask containing two or three porcelain chips, the solvent being evaporated slowly on a water-bath. The residual liquid is subjected to two further extractions, 15 c.c. of each of the two solvents being used each time and mixed in separately as before. The clear solvent layer is again decanted into the beaker-flask, and the residue left on evaporation of the solvent is dried at 100° C. to minimum weight. The vessel is then left in the air until it no longer changes in weight. The weight of the residue thus obtained is corrected by a blank determination on the reagents.

Numerous data, referring to two-day-old eggs, commercial fresh eggs, and storage eggs separately, are given. The outstanding feature of the results is the uniformity in composition shown by fresh eggs. Certain changes in composition with increasing age of the egg are indicated, and further work in this direction will be reported later. Both whites and yolks separated from fresh and storage eggs exhibit differences in composition which indicate osmotic action, and suggest a possible means of ascertaining the freshness of eggs. T. H. P.

Decomposition of Lecithin in Eggs. L. C. Mitchell. (*J. Assoc. Off. Agric. Chem.*, 1932, 15, 282-284.)—A sample of whole eggs, broken under presumably aseptic conditions just before being sent by post to various analysts, gave percentages of lipid phosphorus (as P_2O_5), varying from 0.37 to 0.08. The control sample was divided into a number of portions, which were analysed from time to time until they became putrid. With some of these portions, which exhibited normal putridity, the proportion of lipid and of phosphoric anhydride in the lipid suffered little change. With others, which developed a creamy colour and a pasty consistence, the percentage of lipid diminished slightly, and that of phosphoric anhydride in the lipid enormously; the nitrogen also decreased markedly, and the acidity showed a large increase. For the lipoids of two of the portions the following results were obtained:

	Lipoid present Per Cent.	Phosphoric anhydride Per Cent.	Nitrogen Per Cent.	Acidity in c.c. of C_2H_5ONa per gram.
Putridity normal	12.68	2.67	0.55	4.0
Putridity abnormal	11.54	0.17	0.16	60.0

The decomposition of lipid material occurring in the abnormal cases evidently involves a breakdown of the lecithin, with formation of fatty acid and disappearance of lipid phosphorus and choline nitrogen. That this change is due to bacterial growth is shown by the observation that inoculation of fresh eggs with the abnormal eggs induces similar changes. The bacteria apparently produce the enzyme lecithinase, which attacks lecithin. T. H. P.

Determination of Nitrogen in Yeast by the Hydrogenation Method. H. ter Meulen and K. Peeren. (*J. Inst. Brewing*, 1932, **38**, 330–331.)—As a result of the erratic results obtained by Thorne (*id.*, 1932, **38**, 28), further details of ter Meulen's method (*Rec. Trav. Chim. Pays-Bas*, 1924, **43**, 643; 1930, **49**, 396) are given. One c.c. of a 1 per cent. suspension of the sample in water is mixed with 0.5 grm. of nickel formate in a porcelain boat, and the mixture is then dried and heated in a current of hydrogen at 250° C., the gases being passed through 10 grms. of soda-lime (to trap any unchanged formic acid) and nickel-asbestos. The resulting ammonia is titrated with 0.01 *N* acid from a burette reading to 0.001 c.c. The determination takes 30 minutes, and the values for 10 samples of brewer's or baker's yeast, which were accurate to within about 0.002 mgrm., confirm the previous results, namely, that the method gives results from 0.8 to 19.1 per cent. higher than those obtained by the Kjeldahl–Gunning method, these differences being, on the whole, greater than those between the Gunning and Christensen–Fulmer modifications.

ABSTRACTOR'S NOTE.—It has been found that the most suitable apparatus for this method is a resistance-glass tube about 40 cm. long and 2 cm. in diameter, passed through holes in the ends of an asbestos or tin box 25 cm. long. The portion of the tube inside the box is packed with a catalyst consisting of an intimate mixture of asbestos and reduced nickel prepared by heating black nickel oxide in hydrogen at 320° C. Soda-lime is also required if halogens or sulphur are present. Nickel formate is prepared by the action of formic acid on precipitated nickel carbonate, and if 0.1 grm. is mixed with 0.05 grm. of sample and 0.5 grm. of reduced nickel, reduction is greatly facilitated. A blank determination should first be made to ensure that the apparatus is free from nitrogen, and the boat containing the sample is then inserted in the portion of the tube outside the box and heated in a current of hydrogen, gently at first, and, finally, to a red heat, the portion of the tube inside the box being maintained at 250 to 300° C. (inside temperature). J. G.

Volumetric Method for the Determination of Formic Acid in Fruit Juices and Fruit Syrups. G. v. Szelényi. (*Z. Unters. Lebensm.*, 1932, **63**, 534–541.)—In Fincke's method (*id.*, 1911, **21**, 1; **22**, 88; *ANALYST*, 1911, **36**, 103, 496) such quantity of reaction mixture should be chosen that at least 0.01 per cent. of mercuric chloride (added in the form of a solution containing 100 grms. of mercuric chloride and 30 grms. of sodium chloride per litre) is present. Preferable methods are as follows:—(1) The sample is subjected to preliminary treatment, as in Fincke's method, and a portion of the filtered distillate, corresponding with 10 to 30 c.c. of juice or 30 grms. of syrup, is mixed in a stoppered flask with 5 c.c. of a 10 per cent. solution of sodium acetate and 15 to 20 c.c. (x c.c.) of 0.1 *N* bromoacetic acid, the extent of the decolorisation being an indication of the amount of formic acid present ($\text{HCOOH} + \text{Br}_2 = \text{CO}_2 + 2\text{HBr}$). After 30 minutes 5 c.c. of a 10 per cent. solution of potassium iodide are added, and the mixture is titrated with 0.1 *N* sodium thiosulphate solution (y c.c.). Then $2.3(x - y)$ gives the formic acid in mgrms. (allowance being made for any blank), with an error of 0.05 to 0.5 mgrm., whilst Fincke's method gives lower results. Blank tests with

samples of raspberry juice and syrup free from formic acid yielded 0.5 per cent. (on 10 c.c.) and 1 to 1.5 per cent. (on 30 grms.), respectively, the corresponding figures obtained by Fincke's method being appreciably higher. (2) Calcium formate is produced by the addition of calcium carbonate (*cf.* Fincke, *loc. cit.*), and 200 to 300 c.c. of the mixture are heated with 10 c.c. of 10 per cent. sodium carbonate solution, 20 c.c. of 0.1 *N* potassium permanganate solution being then added rapidly, followed, after 1 minute, by 1 or 2 c.c. of a 10 per cent. solution of zinc sulphate. This removes any colloidal manganese compounds and sharpens the end-point of the final titration, which is carried out with 0.1 *N* arsenic acid. Then 1 c.c. of 0.1 *N* potassium permanganate solution \equiv 2.3 mgrms. of formic acid (*cf.* Hanak and Kürschner, *ANALYST*, 1931, **56**, 116) is added. If 45 minutes are allowed for the oxidation stage, the procedure may be carried out in the cold, and the results, though slightly higher, have an error of only 0.02 to 0.3 mgrm., and, again, are more accurate than those obtained by Fincke's method. Method (2) is better for fruit juices than for syrups, as the latter give higher blanks and develop a yellow colour due to decomposition products of the sugars. J. G.

Aluminium in the Ash of Plant Materials, Fruit Juices, and Similar Products. L. Hart. (*J. Assoc. Off. Agric. Chem.*, 1932, **15**, 285–289).—With the accepted methods based on the initial precipitation of iron and aluminium as phosphates, if the aluminium is calculated by difference after the iron and phosphoric acid have been determined, the result is subject to the errors of such determinations, whilst the colorimetric method with aluminon (ammonium aurin-tricarboxylate) requires very small amounts (less than 0.1 mgrm.) of aluminium in the comparison solutions. In the method now proposed, aluminium and iron are separated at pH 4 as phosphates, which are then dissolved in acid; the iron is removed by means of cupferron, and the aluminium is precipitated with 8-hydroxyquinoline. The method has been developed primarily to determine aluminium in apple vinegar, and gives good results with a synthetic mixture having the composition of apple ash.

The reagents used are: (a) 0.04 per cent. solution of bromocresol green in alcohol; (b) 5 per cent. ammonium nitrate solution adjusted to pH 4 with acetic acid [yellow-green with (a)]; (c) 6 per cent. aqueous cupferron solution; (d) 2.5 per cent. 8-hydroxyquinoline solution, prepared by triturating 2.5 grms. of the reagent and 5 c.c. of glacial acetic acid, pouring into water at 60° C., cooling, filtering, and diluting to 100 c.c.; (e) aluminium-free ammonia, prepared by distilling ammonia solution into water until the resulting solution is at least of 20 *N* concentration; to be kept in a bottle lined with paraffin wax or ceresin; (f) 5 per cent. ammonia solution. A quantity of the substance containing 2 to 10 mgrms. of aluminium is ashed at a red heat in an electric muffle until carbon-free, the ash being moistened, dried and again heated, if necessary. The ash is dissolved in dilute hydrochloric acid (1 + 4), and any iron is oxidised with a few drops of nitric acid. Any residue is filtered off, ignited, fused with sodium and potassium carbonates, and dissolved in a little hydrochloric acid (1 + 4), the solution being added to the filtrate. Unless a fivefold excess of P_2O_5 over the equivalent of iron and aluminium is assured, 0.1 gm. of monopotassium phosphate is added. After addition of 1 to 2 c.c. of (a) and 10 c.c. of ammonium acetate solution the liquid is partly

neutralised with redistilled ammonia, and treated gradually, while gently boiling, with ammonium acetate solution until it assumes a yellow-green colour. It is boiled gently for 1 to 2 minutes to coagulate the precipitate, which is allowed to settle, filtered off on a 7 cm. Whatman No. 41 filter paper, washed two or three times with the cold ammonium nitrate solution, and transferred to the original beaker. The filter is washed into the beaker with hydrochloric acid, and then with hot water, the ferric and aluminium phosphates being dissolved, by heating if necessary. A few crystals of potassium monophosphate are added and re-precipitation at pH 4, as above, is effected. The precipitate is filtered off, washed two or three times with cold ammonium nitrate solution and transferred to a 150-c.c. Pyrex beaker, the residue being dissolved in a known quantity (not over 50 c.c.) of sulphuric acid (1 + 4). The percentage of sulphuric acid in the whole liquid (not above 100 c.c.) is adjusted to 10 to 12, the solution being treated at $10^{\circ}C.$, and with gentle stirring, with a slight excess of fresh cupferron solution—formation of a white precipitate, which immediately redissolves, indicates excess. After settling for 2 or 3 minutes, the precipitate is collected, by means of gentle suction, on filter-paper supported by a platinum cone or in a Gooch crucible; the filtrate is caught in a 250-c.c. Pyrex beaker containing cupferron solution to indicate if precipitation is complete, and the precipitate is washed with cold 10 per cent. sulphuric acid containing 1.5 gm. of cupferron per litre. The filtrate is concentrated to about 50 c.c. on the steam-bath, and then evaporated with 10 c.c. of concentrated nitric acid until dense fumes appear. If the solution is not colourless it must be heated with a little more of the acid. The cold liquid is diluted to about 60 c.c. and filtered to remove silica, the filtrate being treated with a slight excess of 8-hydroxyquinoline (1 c.c. of the 2.5 per cent. solution \equiv 1.54 mgrm. of Al), and then with redistilled ammonia until precipitation occurs, a 5 c.c. excess of the ammonia being added. The liquid is digested at 60° to $70^{\circ}C.$, until the precipitate coagulates, cooled in ice-water, and filtered through a tared Gooch crucible. The aluminium quinolate precipitate is washed with 5 per cent. ammonia containing a few drops of 8-hydroxyquinoline solution, and dried, at $110^{\circ}C.$, to constant weight; it contains 5.87 per cent. of aluminium or 11.10 per cent. of aluminium oxide. A blank experiment on the reagents, together with about 0.01 gm. of iron as ferric salt, should not give more than 0.2 or 0.3 mgrm. of aluminium. If the iron is to be determined, the cupferron precipitate is washed with 10 per cent. ammonia solution, which converts it into ferric hydroxide. If a cloudiness is produced, a second filtration becomes necessary. T. H. P.

Determination of Starch in Feeding Stuffs. G. S. Fraps. (*J. Assoc. Off. Agric. Chem.*, 1932, 15, 304–307.)—The use of taka-diastrase in the determination of starch in feeding stuffs is preferable to that of malt, as the correction for the sugars dissolved is smaller. By the official method of acid hydrolysis large amounts of pentosans are included, but the use of weaker acid (0.02 *N*) reduces the quantity of pentosans dissolved, and thereby increases the accuracy of the method. Correction may be made for the pentosans either by determining these or by applying a factor, the value of which is dependent on the nature of the material analysed. Since 1 part of dextrose corresponds with 0.9 part of starch,

and 1 part of pentose with 0.8799 part of pentosan, sufficiently exact results are obtained by subtracting the percentage of pentosan from that found for starch. As the proportions of the various pentose sugars produced by the hydrolysis of the pentosans are not known, the exact figures to be used for the reducing factors for copper and for conversion to pentosans are also unknown, but errors thus introduced are small if the percentage of pentosans dissolved by the acid is small. When the results are corrected, hydrolysis with 0.02 *N* acid gives approximately the same results for starch as that with either malt-diatase or taka-diatase.

The procedure recommended is as follows: 2.25 grms. of the material are extracted on a hardened filter paper with five successive 10-c.c. portions of ether, and are then washed, first with 150 c.c. of 10 per cent. alcohol solution, and afterwards with a little strong alcohol, and allowed to become nearly dry. The residue is transferred, with exactly 200 c.c. of water, to a crude fibre beaker. The mixture is heated to boiling point, treated with 20 c.c. of 0.2 *N* hydrochloric acid, boiled for 30 minutes, and filtered through asbestos into a flask; the residue is washed two or three times with hot water into the same flask, the total volume of liquid obtained being 300 c.c. The solution, mixed with 30 c.c. of hydrochloric acid (sp.gr. 1.125), is heated for 2½ hours in a boiling water-bath, cooled, and made up to 500 c.c. On 20 or 40 c.c., neutralised with sodium carbonate, the sugars are determined by the Munson and Walker method. To determine the pentosans, 150 c.c. of the 500 c.c. of solution are distilled with 65 c.c. (measured with a pipette) of concentrated hydrochloric acid in the usual way.

T. H. P.

Fatty Acids Associated with Cassava Starch. L. Lehrman. (*J. Amer. Chem. Soc.*, 1932, 54, 2527–2530.)—Fat was removed from cassava starch by extraction with petroleum spirit, and the residue left on evaporation of the solvent was hydrolysed with hydrochloric acid. The resulting 0.1 per cent. of yellow semi-solid oil (iodine value 78.8) was removed by filtration, and the filtrate was tested for glycerol, with negative results. The unsaturated fatty acids were separated from the saturated fatty acids by the magnesium soap and alcohol method; the latter were identified by their neutralisation values, and the former by oxidation with an alkaline solution of potassium permanganate and extraction of the oxidation-products with chloroform, and then with hot water. Analysis of the resulting hydroxy-stearic acids showed that the original fatty acids contained palmitic, oleic and linolic acids. Bromination of a solution of the unsaturated fatty acids in cold anhydrous ether, and extraction of the resulting white precipitate (after being washed with cold ether and dried) with hot petroleum spirit, yielded hexabromostearic acid (m.pt. 80 to 81° C.), indicating the presence of linolenic acid; attempts to find hexahydroxystearic acid in the oxidation products (*vide supra*) were unsuccessful. The portion soluble in petroleum spirit was found to consist of tetrabromostearic acid, and this confirmed the presence of linolic acid, which was also indicated by the production of tetrahydroxystearic acid on oxidation. Tests for nitrogen, sulphur, phosphorus, halogens, and unsaponifiable matter in the fatty acids gave negative results.

J. G.

Determination of Reducing Sugars by Colorimetric Determination of Unreduced Copper. E. M. Emmert. (*J. Assoc. Off. Agric. Chem.*, 1932, **15**, 327-329.)—The method described results in considerable saving of time when compared with methods in which the cuprous oxide formed is separated quantitatively. The reagents required are as follows: (a) A solution containing 40 grms. of copper sulphate per litre; (b) a solution containing 200 grms. of Rochelle salt and 150 grms. of sodium hydroxide per litre; (c) a solution containing 0.25 gm. of pure dextrose per litre; (d) 20 per cent. (by vol.) ammonia solution.

To standardise the copper solution, 10 c.c. of (c), 5 c.c. of (a), and 5 c.c. of (b), in a 150 c.c.-Erlenmeyer flask, are heated to boiling on an asbestos gauze and boiled gently for exactly 3 minutes. The hot liquid is made up to 25 c.c. in a measuring flask, and then filtered as rapidly as possible through any ordinary filter paper able to retain the cuprous oxide, which is discarded. Exactly 5 c.c. of the filtrate are made up to 50 c.c. with reagent (d) in a measuring flask. The blue colour of this solution is compared with that produced by 5 c.c. of reagent (a) when subjected to the same procedure, except that the 10 c.c. of dextrose solution is replaced by 10 c.c. of water. The number of mgrms. (X) of dextrose equivalent to 5 c.c. of the copper sulphate solution is calculated from the formula: $X - XR/U = Y$, in which U is the colorimetric reading of reagent (a) without added dextrose, R the reading after reduction with dextrose, and Y the number of mgrms. of dextrose added. This standardisation should be made in duplicate or triplicate.

The approximate sugar content of the solution to be analysed is determined by boiling 10 c.c.-portions of it with different quantities of the mixture of copper sulphate and alkaline tartrate solutions. The sugar solution, if necessary, is diluted or evaporated until 10 c.c. contains from 5 to 20 mgrms. of reducing sugar, expressed as dextrose. Ten c.c. of the solution are then treated exactly as in the standardisation of the copper solution. If it is not possible to adjust the concentration of the sugar solution without destroying some of the reducing power, the amount of copper sulphate solution used may be lessened somewhat. When tested with 5 to 6 mgrms. of pure dextrose this method showed errors of +4 to +5 per cent.; with quantities ranging from 7 to 20 mgrms. of the sugar the errors varied from -3 to +3.3 per cent.

T. H. P.

Determination of Reducing Sugars in Raw Sugars, etc., by the Pot Method. H. Main. (*Int. Sugar J.*, 1932, **34**, 213-217.)—For routine determination of reducing sugars, incremental titration with Fehling's solution in presence of methylene blue as internal indicator, as described by Lane and Eynon (*ANALYST*, 1923, **48**, 220, 277), gives satisfactory results, but the personal element may introduce inaccuracies. Moreover, the standard method of titration given by these authors involves factors difficult to standardise, namely, the time of heating to boiling and the rate of ebullition, and, in addition, any local heating of the concentrated alkaline solution may cause destruction of both sucrose and invert sugar. The volumetric method now described overcomes these and other objections, and yields extremely accurate results in the hands of operators with a minimum of experience.

It consists essentially in heating, in boiling water, three or more large test-tubes, containing Fehling's solution, methylene blue, and such different amounts of the sugar that, at the end of a definite time, some tubes are still blue, whilst one, at least, shows complete reduction of the cupric salt. The tubes used should be of nearly the same size and weight, those employed by the author being of Monax glass and having the length 150 mm., the internal diameter 38 mm., and the weight 50 to 55 grms. Floats inserted to prevent access of air to the solutions during the determination consist of similar tubes, making a sliding fit in the others and being conveniently drawn out to a taper about 100 mm. from the closed end to make a total length of 170 mm. The water-bath may be a 3-gallon oval iron kitchen pot, tinned inside and fitted with an overflow and with a sight-feed through which hot water is added continuously to replace loss by evaporation. The water must be kept boiling. While in the water-bath, the tubes are held in clips fitted to a frame.

Use is made of Soxhlet's modification of Fehling's solution, which contains: (1) 34.639 grms. of pure crystallised copper sulphate dissolved to 500 c.c.; (2) 173 grms. of Rochelle salt and 50 grms. of sodium hydroxide dissolved to 500 c.c. Equal volumes of (1) and (2) are mixed as required. The methylene blue indicator is a 1 per cent. aqueous solution. The standard invert sugar solution is prepared as described by Lane and Eynon (*loc. cit.*), and tested as follows:—Into three of the test-tubes are placed in order: 10 ml. of Fehling's solution (into each); 24.5, 25.0 and 25.5 ml., respectively, of the diluted sugar solution, prepared by neutralising 50 ml. of the standard invert sugar solution with caustic soda solution and diluting to 250 ml.; 2 drops of methylene blue indicator. The contents of each tube are mixed by gentle rotation, the floats being then inserted so that they rest on the liquid and entrap no air-bubbles. The tubes in the frame are placed in the boiling water and removed and inspected after exactly five minutes. If the middle tube shows complete reduction, and that with 24.5 ml. of sugar solution is still blue, 10 ml. of Fehling's solution are taken as equivalent to 24.75×0.002 gm. = 0.0495 gm. of invert sugar. Closer approximation may be attained by lessening the differences between the volumes of sugar solution in the different tubes. The mean between the volumes in the last blue and the first red tube is always taken as correct, except that, when the blue colour in any tube is seen to fade on removal from the bath, the actual volume in that tube is regarded as the true volume.

When sucrose is present together with invert sugar, the oxidising power of the Fehling's solution is apparently decreased, probably owing to partial inversion of the sucrose. Tables are, therefore, given for the volumes of Fehling's solution reduced by solutions of invert sugar containing various proportions of sucrose. When very small amounts of reducing sugars are to be determined, the action of the Fehling's solution is accelerated by mixing it with 5 *N* sodium hydroxide solution (1 vol. to 1 vol.). In this way the small percentages of invert sugar (0.01 to 0.001) in commercial white sugars may be determined accurately. The difficulty of judging the end-point in such cases, owing to the dichroism caused by the very fine state of division of the cuprous oxide, may be overcome by adding to the mixture of sugar solution and Fehling's solution, before heating, potassium ferrocyanide in the proportion of 1 mol. per 4 mols. of copper sulphate present; the ferrocyanide

(14.647 grms. of $K_4FeCy_6 \cdot 3H_2O$) may be incorporated in the 500 c.c. of Fehling's solution (2) containing the Rochelle salt and sodium hydroxide. This solution keeps unchanged for months. The procedure followed is that described above, except that for sugars containing less than 0.01 per cent. of invert sugar the time of heating in the boiling water-bath is increased to 10 minutes. This modification is particularly suitable for use at night-time. A table is given also for this method of working.

T. H. P.

New Volumetric Method for the Determination of Reducing Sugars.

E. Haddon. (*La Revue Agricole*, 1931, No. 59, 131; *International Sugar Journal*, 1932, **34**, 43.)—The determination is made on the ordinary clarified solution without de-leading. To about 100 c.c. of the solution to be examined, four drops of a 1 per cent. methylene blue solution are added, and the coloured solution is introduced into the burette. Two c.c. or 4 c.c. of a 10 per cent. solution of potassium ferrocyanide are added to 5 c.c. or 10 c.c. of Fehling's solution, and the solution is titrated by adding the coloured solution gradually without letting the boiling slacken. The end of the reaction is a *sudden* disappearance of the blue colour; it is very sharp, and the results are concordant. This method, which is a simplification of published methods, will probably be useful to sugar-house chemists, especially when determinations are to be made at night.

Quince Seed Oil. **W. H. Dickhart.** (*Amer. J. Pharm.*, 1932, **104**, 335–336.)—*Cydonium* (quince) seeds contain about 22 per cent. of mucilage, and, when dried, about 15 per cent. of a fixed oil, amygdalin, tannin, colouring matter, and 13 per cent. of ash. As obtained by extraction with petroleum spirit a specimen of the oil had the following characteristics:—Specific gravity at 15° C., 0.9251; n_D^{40} , 1.4696; saponification value, 187.7; iodine value (Wijs), 112.4; free fatty acids (as oleic), 6.49 per cent.; unsaponifiable matter, 9.35 per cent.; n_D^{40} of free fatty acids, 1.4639; and iodine value, 100.5; total fatty acids, 90.4 per cent. The Halphen and Villavecchia tests were negative, but the Bellier test showed a trace of arachidic acid.

D. G. H.

Oil from the Nuts of *Calophyllum inophyllum* (Dilo Oil). **K. W. R. Glasgow.** (*J. Soc. Chem. Ind.*, 1932, **51**, 172–174r.)—*Calophyllum inophyllum*, a tree indigenous to Fiji, is closely related to the trees producing such oils as laurel nut, domba, and Alexandrian laurel oils; also to tacamahaca fat, and poon-seed oil, Calabar, Njamplung and pinnay oils. The dry nuts contain about 40 per cent. of kernels, yielding, on extraction, about 43.5 per cent. of an amber oil, and, on a large scale by discontinuous extraction with hot ether, about 58 per cent. of oil. The oil melts at 50° C., but, when once completely melted, takes a considerable time to set again; it is a non-drying oil. The sample examined had sp.gr. at 21° C., 0.929; n_D^{60} , 1.4680; saponification value, 200.9; iodine value, 81.7; free fatty acids (oleic), 33.9 per cent.; unsaponifiable matter, 0.25 per cent.; optical rotation, -9.8° . Six hours' treatment with pure oxygen decreased the iodine value to 64, the acid value remaining unaltered. The liquid and solid acids of the free acid portion were separated and examined, and these acids showed no radical difference from the acids separated from the neutral oil. The whole oil

was saponified, and the liberated acids, when examined by the Twitchell method, yielded 9·7 per cent. of resin acids, only abietic acid being identified. The remaining fatty acids yielded 31 per cent. of solid, and 69 per cent. of liquid acids, and, after separation and redistillation, the 90·30 per cent. present were found to consist of palmitic, 14·1; stearic, 11·0; erucic, 3·0; oleic, 48·0; and linolic acid, 14·3 per cent. The unsaponifiable matter contained sitosterol.

D. G. H.

Determination of Veronal and of Mercury Tannate. **A. Ionescu-Matiu and A. Popesco.** (*J. Pharm. Chim.*, 1932, **124**, 551–554.)—*Veronal*: Two to 4 c.c. of a saturated solution of veronal are treated with 5 c.c. of a mercuric sulphate solution made by dissolving 5 grms. of mercuric oxide in 20 c.c. of concentrated sulphuric acid and 93 c.c. of water. A white precipitate is formed on shaking, and, after centrifuging, the opalescent supernatant liquid is decanted, and the precipitate is washed with 3 portions of 3 to 4 c.c. of water, and, finally, dissolved in a hot mixture of sulphuric and nitric acids (strength not given), the solution on dilution to 100 c.c. remaining clear. After addition of a few drops of permanganate solution (to destroy the nitrous compounds), the mercury is precipitated with 12 drops of a 10 per cent. solution of sodium nitroprusside. By means of a micro-burette a 0·1 *N* solution of sodium chloride is added until the cloudiness has disappeared. One c.c. of 0·1 *N* sodium chloride solution is equivalent to 0·01393 of veronal. The amounts of veronal found by this method agreed very closely with the amounts taken.

Mercury Tannate.—The tannate (0·02 to 0·15 gm.) is treated with 10 c.c. of a hot mixture of sulphuric and nitric acids (strength not given), the clear solution is diluted to 100 c.c., a few drops of 2 per cent. potassium permanganate solution are added, followed by 20 drops of 10 per cent. sodium nitroprusside solution to precipitate the mercury. Titration with 0·1 *N* sodium chloride solution then follows, and the number of c.c., multiplied by the factor 0·01785, gives the amount of mercury tannate originally present.

D. G. H.