

THE ANALYST

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

An Ordinary Meeting of the Society was held at the Chemical Society's Rooms, Burlington House, on Wednesday, March 3rd, 1926, Mr. E. Richards Bolton, F.I.C., President, being in the chair.

Certificates were read for the first time in favour of Messrs. John Allan, Michael Thomas Casey, B.Sc., M.Sc., George Henry Davis, Julius Grant, M.Sc., A.I.C., and Miss Monica Mary Ruston, B.Sc., F.I.C.

Certificates were read for the second time in favour of Messrs. Sydney Back, B.Sc., A.I.C., Howard Henry Bagnall, B.Sc., F.I.C., William Percival Crocker, Bernard William Alfred Crutchlow, B.Sc., A.I.C., Alec Munro Ferguson, M.A., B.Sc., A.I.C., Ralph Henry Klein, A.I.C., Oswald James Napier, M.A., A.I.C., George Stubbs, C.B.E., F.I.C., Joseph Hughes Williams, B.Sc., F.I.C., Kenneth Alan Williams, B.Sc., A.I.C.

The following were elected Members of the Society:—Messrs. Guy Chignell, B.Sc., A.I.C., Hugh Gower Watts, B.Sc., A.R.C.S., A.I.C., and Dr. K. Saito.

The following papers were read and discussed:—"An Accurate Method for the Determination of Mercury in Solution," by B. S. Evans, Ph.D., F.I.C., and S. G. Clarke, B.Sc., A.I.C.; "An Apparatus for Continuous Percolation and for Filtration in Neutral Atmospheres," by B. S. Evans, Ph.D., F.I.C.; and "Notes on the Determination of Moisture, Calcium and Phosphorus in the Bones of Rats," by A. L. Bacharach, B.A., A.I.C.

NORTH OF ENGLAND SECTION.

The first Annual General Meeting of the North of England Section of the Society of Public Analysts was held in Manchester on Saturday, February 27th, 1926.

Professor Roberts, was in the chair, and thirteen members were present.

The accounts for the year ending December 31st, 1925, were passed.

The following were elected members of the Committee for the present year:—
Messrs. J. Evans, H. Hurst, H. Lowe, S. E. Melling, W. H. Roberts, C. J. H. Stock,
J. R. Walmsley, and J. Wood.

Messrs. U. A. Coates and W. Marshall were re-elected Hon. Auditors.

The Determination of Ascaridole in Chenopodium Oil.

BY HUMPHREY PAGET, B.A. (Oxon.).

(Read at the Meeting, February 6, 1926.)

CHENOPODIUM or American wormseed oil was at one time extensively used as a general anthelmintic, but fell into disuse largely because of a number of cases of poisoning, due in most instances to over-dosage. In the last ten years it has acquired a special importance from its use on a large scale in campaigns against the hookworm, conducted, under the auspices of the International Health Board, in various tropical countries.

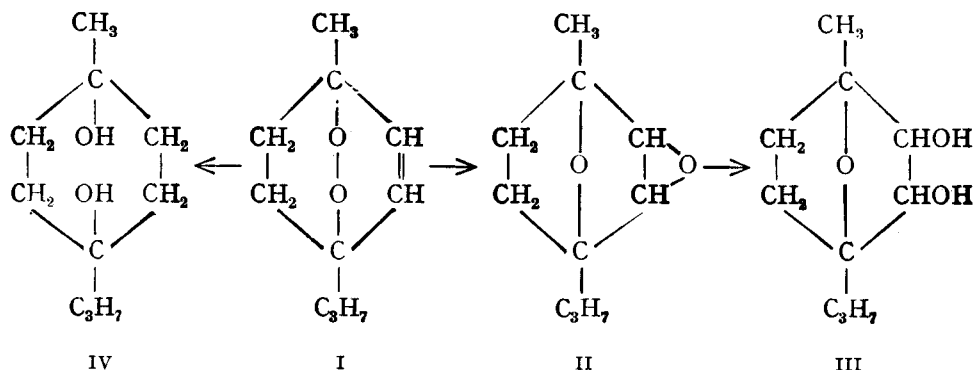
COMPOSITION AND CONSTANTS OF CHENOPODIUM OIL.—It is now known that the chief components of the oil are (a) ascaridole (Formula I), which is present to the extent of 60 to 75 per cent., and (b) a mixture of terpenes with *p*-cymene, which constitutes the residue, and which is here referred to as the hydrocarbon fraction. (Nelson, *J. Amer. Chem. Soc.*, 1911, 33, 1404; 1913, 35, 34; 1920, 42, 1204; Henry and Paget, *J. Chem. Soc.*, 1921, 119, 1715; 1925, 127, 1649.)

The oil is recognised in the United States Pharmacopoeia (10th revision), which specifies the following constants for it:—Sp. gr., 0.955 to 0.980 at 25° C.; optical rotation, -4° to -10° in a 100 mm. tube at 25° C.; refractive index, 1.4723 to 1.4770 at 20° C.; and solubility, 1 volume in not less than 8 volumes of 70 per cent. alcohol.

Apart from the statement of these data, little has been done to develop a means of ascertaining the quantity of ascaridole present, although the problem has now become of importance, since Smillie and Pessoa's results (*J. Pharm. Exp. Ther.*, 1924, 24, 359) no longer leave any doubt that ascaridole is the sole component of the oil which exhibits anthelmintic action against hookworm and roundworm, the parasites for which the oil is generally used.

THE U.S.P. METHOD OF DETERMINING ASCARIDOLE.—The hydrocarbon fraction is insoluble in 60 per cent. acetic acid, whilst ascaridole is miscible with this solvent, and it is on this fact that the method suggested by Nelson (*J. Amer. Pharm. Assoc.*, 1921, 10, 836), and adopted by the United States Pharmacopoeia, for the determination of ascaridole is based. This author, however, has overlooked

the fact that ascaridole readily undergoes intramolecular change to ascaridole glycol anhydride (Formula II), and that this is easily hydrated, forming ascaridole glycol (Formula III).



These two changes occur in succession by the mere application to ascaridole of (a) dry heat, (b) steam, and it is clear that in the distillation with steam involved in the manufacture of the oil from the seed such changes must occur. In the course of work on the components of the oil the author has fractionally distilled many litres of it, and has invariably found evidence of such decomposition. The anhydride and the glycol are, like ascaridole, miscible with 60 per cent. acetic acid, and therefore rank as ascaridole when general tests of this kind are applied. Further, chenopodium oil commands a fairly high price, and is in consequence liable to adulteration, as in the case of the "synthetic" chenopodium oils to which Messrs. Schimmel and Co. have called attention in their Reports for 1919 and 1921, and one of the substances found in such oils—cineole—is also miscible with 60 per cent. acetic acid (see p. 176).

QUANTITATIVE REDUCTION OF ASCARIDOLE.—It is therefore desirable that a method should be found by which ascaridole itself can be determined. In the work already referred to, the only method which has been found satisfactory is the isolation of ascaridole by distillation under reduced pressure, but, as ascaridole is liable to explode on the application of heat, this does not constitute a desirable process for general use. Formula I shows that ascaridole is a peroxide, and, like these substances in general, it forms no solid derivatives by which it can be isolated. Attention was therefore directed to the possibility of its quantitative reduction. It has long been known that in presence of ferrous sulphate ascaridole is converted into the corresponding glycol (Formula III), but, as will be seen from Formulae I, II and III, this is not a true reduction, but merely involves intramolecular change and hydration. Wallach (*Annalen*, 1912, 392, 60), however, has shown that hydrogen in presence of palladium reduces ascaridole to 1,4-terpin (Formula IV) by the addition of four atoms of hydrogen, and there therefore seemed a possibility of finding an agent which would effect such a change in a manner both quantitative

and measureable. Of the reducing agents tried, titanous chloride (or sulphate), which was employed as described by Knecht and Hibbert (*New Reduction Methods in Volumetric Analysis*, 1925), was found to be the most convenient for this purpose.

The products of this reduction are being examined, but the only information yet available is that they include neither ascaridole glycol nor its anhydride. If the reaction proceeds as in the catalytic hydrogenation described by Wallach, the amount of titanous chloride used should correspond to the addition of four atoms of hydrogen, but though the end of the titration is quite definite, the amount actually used is only one-third of this; even if only two atoms of hydrogen are added, the ethylene linkage not being reduced by titanous salts, the amount used is still only about three-quarters of the theoretical quantity. This point has been investigated by the use of carefully purified ascaridole in preliminary titration experiments. It appeared at first as if the reaction reached a definite equilibrium, but controlled variation of (1) the quantities of material, solvent and reagent used, (2) time of reaction, (3) nature of indicator, (4) the substitution of methylene blue for iron alum in determining the residual excess of reducing agent, and (5) the use of sodium hyposulphite in place of titanous salts, all failed to effect any material modification of the apparent end of the reaction first found (Table I). Until the products have been examined the process must rest on the empirical basis that one grm. of ascaridole is reduced by 1.2770 grms. of titanous chloride. This figure is taken, being the mean of results from several experiments varying from 1.2420 to 1.3040 grms.

While the author does not claim a high degree of accuracy for the process, it has the advantage over the existing general method of determination that, with the expenditure of a very small amount of the oil, it does give evidence of the amount of ascaridole really present.

The opportunity has been taken to determine to what extent the general methods already available are of value, and it is shown that with genuine samples of oil, they give quite useful results, but in case of adulterated oils, such as sample E, which was purchased in the open market as *Oleum chenopodii* U.S.P., they are quite useless.

EXPERIMENTAL.

PREPARATION AND CONSTANTS OF ASCARIDOLE.—Ascaridole was obtained from chenopodium oil by repeated fractional distillation at 15 mm. pressure (*J. Chem. Soc.*, 1921, 119, 1715), and isolation of the middle portion of a fraction boiling at 115° C. at 15 mm. It had the following constants:— D_{15}^{15} , 1.0111; D_{25}^{25} , 1.0050; α , -2.15° in a 1 dcm. tube; $[\alpha]_D^{25}$, -2.14° ; n_D^{20} , 1.4736; and on analysis gave: C = 71.18, H = 9.65 per cent. (ascaridole, $C_{10}H_{16}O_2$, requires: C = 71.43, H = 9.52 per cent.). This pure ascaridole is referred to later on as oil A.

The hydrocarbon fraction was obtained from the portion of the oil boiling below 115° C. at 15 mm. by repeated distillation, first at low pressure and finally over sodium at 760 mm. It then boiled between 170° and 185° C.; and had D_{15}^{15} , 0.8585; D_{25}^{25} , 0.8506; α , -18.21° in a 1 dcm. tube; $[\alpha]_D^{25}$, -21.41° ; n_D^{20} , 1.4862.

Of the five samples of chenopodium oil which were examined, oils B and C represented a commercial brand of ascaridole, the remaining three (D, E and F) being ordinary chenopodium oils of commerce, all of which fulfilled the requirements of the United States Pharmacopoeia (Rev. 10) as to specific gravity, optical rotation, and solubility in 70 per cent. alcohol and 60 per cent. acetic acid.

DETERMINATION BY MEANS OF SPECIFIC GRAVITY AND OPTICAL ROTATION.—The specific gravity and specific rotation at 25° C. of ascaridole and the hydrocarbon fraction, were determined, and from these data the percentages of ascaridole have been calculated (from the specific gravities and specific rotations) in the oils B to F (*cf.* Parry, *Chemistry of the Essential Oils*, 1921, Vol. I, p. 535), on the assumption that no change occurs on admixture of ascaridole with the hydrocarbon fraction (Table II).

DETERMINATION BY SOLUBILITY.—Nelson's method of determination was carried out by shaking 10 c.c. of each oil for 5 minutes in a cassia flask* with 60 per cent. acetic acid, the flask being then set aside at about 15° C. till separation was complete. All bubbles of oil were collected into the graduated neck, and from the volume of the insoluble hydrocarbons so obtained the apparent percentage of ascaridole was calculated. Oils B and C were completely soluble, except for a faint cloudiness in the solution; and though in every case the amount of ascaridole found was higher than that indicated by the specific gravity, reference to Table II, columns 4 and 6, will show that the results obtained by these two methods agreed well.

DETERMINATION BY DISTILLATION.—In order to check the results obtained above, each sample was separated by repeated distillation into four fractions:—(i) b.p. below 100° C. at 15 mm.; (ii) b.p. 100° to 110° C. at 15 mm.; (iii) b.p. 110° to 120° C. at 15 mm.; and (iv) the residue undistilled at 120° C. at 15 mm.

Experience gained during fractionation of considerable quantities of chenopodium oil has shown that of the fractions obtained by distilling about 250 c.c. at a time, (i) consists almost entirely of the hydrocarbons present in the oil, and (iii) of ascaridole, whilst (ii) is a mixture containing about 75 per cent. of ascaridole. The percentages shown in Table II, column 8, are arrived at on the basis of this assumption. In general, the previous results were confirmed (Table II), except in the case of Oil E, which was not easily separated into its constituents; on redistillation an ascaridole fraction was obtained from it representing, however, only about 38 per cent. of the oil. The quantity of Oil F available was insufficient for fractional distillation.

DETERMINATION BY MEANS OF REDUCING AGENTS.—Experiments with sodium metabisulphite and with sulphurous acid proved these reagents to be inactive. Sodium hyposulphite was also found to be unsuitable, since, in addition to effecting the reduction of ascaridole, it also reacted with the mixture of hydrocarbons. Titanous chloride was found to be the most convenient reducing agent.

* These are special flasks having the neck graduated in tenths of a c.c., which are used in the determination of cinnamic aldehyde in cassia oil; they are obtainable from the usual dealers in chemical apparatus.

Titanous chloride solution was prepared and standardised as described by Knecht and Hibbert, 66 c.c. of the commercial 15 per cent. solution being made up to 2250 c.c. One gram. of chenopodium oil was diluted with 96 per cent. alcohol to 100 c.c., and to 10 c.c. of this in a flask, through which a current of carbon dioxide was passing, an excess of titanous chloride, about 50 c.c., was added; the flask was then closed with a Bunsen valve, and its contents heated almost to boiling for one or two minutes. If the pale violet colour of the titanous chloride disappeared, more was added to ensure the presence of an excess. The formation of a precipitate of titanous oxide during heating did not interfere with the determination. About 1 c.c. of 5 per cent. potassium thiocyanate was then added, and the solution titrated back with a standard solution of iron alum until a permanent faint red colour was obtained. The amount of iron used, calculated in terms of titanous chloride, gave by difference the quantity of titanous chloride oxidised.

For example, (i), 10 c.c. of a 1 per cent. solution of pure ascaridole were heated, under the conditions described, with 50 c.c. of TiCl_3 (= 0.003856 gm. per c.c.); it then required 15.9 c.c. of iron alum (= 0.001485 gm. per c.c.), equivalent to 0.0651 gm. of TiCl_3 ; therefore 0.1 gm. of ascaridole oxidised 0.1277 gm. of TiCl_3 . (ii) 10 c.c. of a mixture, containing cymene, 18.3 per cent.; cineole, 27.4 per cent.; and ascaridole, 54.3 per cent., after being heated with 40 c.c. of TiCl_3 , oxidised 18.1 c.c., or 0.0698 gm. TiCl_3 , which is equivalent to 54.7 per cent. of ascaridole. (iii) 10 c.c. of a 1 per cent. solution of Oil F, after heating with 40 c.c. of TiCl_3 , required 12.85 c.c. of iron alum, equivalent to 0.0527 gm. of TiCl_3 ; therefore 0.1015 gm. of TiCl_3 was oxidised, equivalent to 79.5 per cent. of ascaridole.

Solutions in alcohol of the hydrocarbon fraction, of ascaridole glycol or of its anhydride were found to have no oxidising action on titanous chloride under these conditions.

MODIFICATIONS OF THE REDUCTION METHOD OF DETERMINATION.—As has been stated above, attempts were made to modify this method of determining ascaridole, so as to obtain results in agreement with those to be expected if the reduction followed a simple course. When the solution was left in the cold in a current of carbon dioxide, the reaction was complete at the end of one hour, and the same point was reached when the solution was heated or boiled for one or fifteen minutes. The presence or absence of a solvent was without influence on the reaction, except that when no solvent was used the rate was slower and the end-point less definite. By increasing the concentration of the titanous chloride solution from $N/50$ to about $N/10$, a little more titanous chloride was oxidised, corresponding to about 10 per cent. of ascaridole, but, if the concentration was raised much beyond this, the reduction proceeded, too vigorously. The addition of 10 c.c. of a 20 per cent. solution of Rochelle salt, did not further affect the result.

Variations in the conditions having failed to effect the object in view, the possibility of a re-oxidation of the primary reduction product by the ferric alum was considered. Methylene blue was tried, first as an indicator only, and then as an oxidising agent for the excess of titanous chloride; the end-point of the

titration was practically the same as before, but was less sharp, even when carried out at the boiling point. Sodium tungstate solution, when added to a solution of a titanous salt, forms a bright blue coloration which is destroyed on the addition of oxidising agents; the disappearance of the colour, however, was not sufficiently marked to allow of the use of this salt as an indicator. The determination of peroxides, such as hydrogen peroxide, by titanous chloride, is based on the production of an orange colour due to the formation of titanium trioxide, the disappearance of which coincides with the complete reduction of the peroxide. Under certain conditions this colour was produced with ascaridole, but it could not be made of use in a process of determination.

The results obtained by these modifications, which are given in Table I, below, display no advantage over the first reduction method described above.

TABLE I.

(Except where otherwise stated, 10 c.c. of a 1 per cent. solution of oil A was used.)

Oil used.	Reducing agent and conditions.	Ascaridole found. Per Cent.
(1) A	Left 65 min. cold with 60 c.c. TiCl_3 about N/50	97
(2) A	Heated 1 min. with 60 c.c. TiCl_3	101
(3) A	Boiled 10 min. with 50 c.c. TiCl_3	100
(4) 0.1675 grm. A	No solvent; heated 5 min. with 100 c.c. TiCl_3	108
(5) A	Heated 1 min. with 10 c.c. TiCl_3 about N/10	110
(6) A	As (2) using Rochelle salt	104
(7) A	As (2) titrating back with methylene blue	104
(8) 0.0960 grm. A	No solvent; titrating back with methylene blue	109
(9) 0.1158 grm. A	Reduced by sodium hyposulphite	101

APPLICABILITY OF THE METHODS.—A comparison of the results obtained by the application of the methods of determination described to the oils examined is made in Table II. From these it will be seen that, omitting Oil E, (i), the specific gravity is a useful guide to the ascaridole content; (ii) solubility in 60 per cent. acetic acid, which is the most rapid, and the most convenient process for field work, is also of value, although for the reasons given above, the results obtained with it are always high. But for the detection of adulterated or seriously deteriorated oils, such as Oil E, it is necessary to have recourse to the reduction or distillation methods

TABLE II.

PERCENTAGE OF ASCARIDOLE FOUND BY

Oil used.	D_{25}^{25}	$[\alpha]_D^{25}$	D_{25}^{25}	$[\alpha]_D^{25}$	Solubility in		
					60 per cent. acetic acid.	Reduction by TiCl_3 .	Distillation.
A	1.0050	-2.14°	100	100	100	100	100
B	1.0002	-2.21°	97	99	100	97	92
C	0.9995	-2.29°	96	98	100	96	90
D	0.9611	-5.83°	72	81	74	72	70
E	0.9586	-5.48°	70	83	73	48	45
F	0.9746	-5.57°	80	82	82	80	—

FURTHER EXAMINATION OF OIL E.—A comparison of the fractions obtained from Oil E with those obtained from a normal oil, such as Oil D, is given below.

TABLE III.

Oil used.	B.pt. below 100° C./15 mm. Per Cent.	B.pt. 100°– 110° C./15 mm. Per Cent.	B.pt. 110°– 120° C./15 mm. Per Cent.	B.pt. above 120° C./15 mm. Per Cent.
D	21	7	66	6
E	28	9	38	25

On shaking the fraction boiling below 100° C. at 15 mm. with a 50 per cent. solution of resorcinol in water, 39 per cent. was dissolved; and on distilling the resorcinol solution with steam an oil was recovered smelling strongly of cineole and boiling at 175° to 180° C., in which cineole was definitely identified by its crystalline compound with resorcinol, melting at 82° C. By repeated distillation of that part of the oil which boiled above 110° C. at 15 mm., fraction 3 (b.pt. 110° to 120° C. at 15 mm.), was raised to 43 per cent.; this was almost completely volatile in steam, but the oil so recovered contained only 73 per cent. of ascaridole (reduction method).

From the oil undistilled at 120° C. at 15 mm. no fraction of constant boiling point could be isolated. Twenty-seven per cent. of this oil was not volatile in steam, and it yielded ascaridole α - and β -glycols, probably representing ascaridole glycol anhydride in the crude oil. The portion volatile in steam contained about 20 per cent. of ascaridole. It was gently heated at atmospheric pressure, until conversion of ascaridole into the non-volatile glycol anhydride took place, and again distilled with steam; the resulting volatile oil distilled mainly at 220° to 245° C. at 760 mm. without decomposition, and was not ascaridole or any of its derivatives or a high-boiling ester.

This sample was therefore not a genuine chenopodium oil. It was probably an inferior sample brought to apparent conformity with the U.S.P. standards by the addition of cineole and a high-boiling constituent.

The author desires sincerely to thank Dr. T. A. Henry for much valuable criticism and advice.

A general glance at the figures shows that only two of the constants and variables fall during the oxidation, these being the iodine value and unsaponifiable matter, but that the other seven rise to a greater or less extent. Although the constants and variables rise or fall simultaneously at each stage, this does not take place at the same rate, so that there is no exact relationship between the rise in specific gravity, for example, and the rise in viscosity, or fall in iodine value. Speaking generally, such conclusions must be accepted, but there are several points of interest worth mentioning. As a rule, oils having a high iodine value have also a high refractive index, but with blown oils the refractive index rises as the iodine value falls. The rise in saponification and acid values is, as would be expected, concurrent with the rise in soluble volatile acids, as is shown by the Reichert-Wollny value. The rise in acid value takes place at the same time as the fall in unsaponifiable matter, so that the increase in acid value is apparently partly due to the oxidation of the unsaponifiable alcohols into fatty acids by the oxygen of the air used in blowing.

It may be noted that there is one exception to the irregularity referred to, as there is a practically regular rise in the viscosity and refractive index after the oil reaches a viscosity of 700 seconds and up to 1800 seconds, but below this the relationship is irregular.

It is also noteworthy that ether-insoluble bromides are not produced from blown shark, whale, or sperm oil. It is not necessary to go into detail as regards the various changes caused by blowing, as a study of the table will bring these out clearly.

ANALYSIS OF THE SAPONIFIED OILS.—A further analysis of the untreated shark oil and of the blown oil of sp. gr. 0.992 was made, after saponification of the oils with alcoholic caustic potash. The results were as follows:—

Untreated Oil.	Per Cent.
Fatty acids insoluble in hot water	83.56
Soluble volatile acids	0.07
Insoluble volatile acids	0.15
Glycerin	6.02
Unsaponifiable matter	15.64
Unknown organic matter	0.50

The molecular equivalent of the fatty acids insoluble in hot water was found to be approximately 286 (sodium hydroxide as 40).

Blown Oil.	Per Cent.	Molecular equivalent.
Fatty acids insoluble in hot water	73.20	284
Fatty acids soluble in hot, insoluble in cold water	1.96	212
Non-volatile fatty acids soluble in cold water	3.12	160
Volatile acids soluble in cold water	1.47	67
Acids insoluble in ether, soluble in alcohol	1.56	270
Glycerin	6.25	—
Unsaponifiable matter	10.02	—
Unknown organic matter	6.30	—

The acids referred to in the above analyses are, no doubt, mixtures of acids having different equivalents, and the equivalent for each is simply the average of these, but the figures can only be regarded as an approximation to the real value. The figures given explain themselves, and it is only necessary to note that the amount of glycerin produced has not been affected, but that a considerable quantity of an apparently neutral unknown organic matter has been formed.

VARIATIONS OF THE CONDITIONS OF BLOWING.—That the same conditions of blowing must be adhered to in order to obtain similar results, was clearly shown by another trial on the large scale, which was carried out on a somewhat different system. A portion of the same shark oil was used in this trial, and the following are the results of analysis of the oil after the blowing was considered complete. (Table II):—

TABLE II.

	Shark Oil. (Untreated.)	Shark Oil. (Blown.)
Specific gravity at 60° F.	0.914	0.993
Viscosity, in secs., at 200° F. (Redwood) ..	50	3000
Iodine value	130.32	62.02
Saponification value	163.62	214.26
Acid value	2.39	16.31
Reichert-Wollny value	0.30	5.30
Polenske value	0.40	0.50
Unsaponifiable matter, per cent.	15.64	12.24
Refractive index, n_D^{40}	1.4680	1.4766
Free fatty acids (as oleic acid), per cent. ..	1.20	8.20
Ether-insoluble bromides	29.35	None

Although the specific gravity of this blown oil is about the same as that of the oil last mentioned in Table I, the viscosity has increased very greatly, but, contrary to what might be expected, the acid value is lower and the iodine value higher. These anomalies are also noticeable in the saponification value, the Reichert-Wollny value, and the amount of saponifiable matter. The only value that conforms to the changes observed in other constants and variables, shown in Table I, is the re-fractive index, which has risen with the increase in viscosity.

SPERM, WHALE AND COTTONSEED OILS.—The results of the analyses of sperm, whale, and cottonseed oils, and of the corresponding blown oils from these, are given below (Table III).

With sperm oil the rise and fall of the constants and variables are similar in every case to those of shark oil. As will be observed, one blowing was done with the addition of oxide of cobalt (0.1 per cent.), which has a rather peculiar effect. Although the specific gravity is lower than by the ordinary method of blowing, without addition of any chemical, the viscosity and refractive index are higher, and other anomalies are noticeable in all the other constants and variables except the saponification value.

TABLE III.

	Sperm Oil.			Whale Oil.		Cottonseed Oil.		
	Un- treated.	Usual blowing.	Blown with cobalt oxide.	Un- treated.	Blown.	Un- treated.	Blown.	Blown.
Sp. gr. at 60° F.	0.880	0.968	0.964	0.920	0.984	0.922	0.967	0.981
Viscosity, in secs., at 200° F. (Redwood)	45	285	380	49	255	50	196	432
Iodine value	82.40	47.01	47.96	116.26	67.97	108.12	74.36	58.35
Saponification value	124.70	200.90	204.40	196.00	239.40	192.10	209.07	220.61
Acid value	2.98	24.68	20.41	24.86	32.84	0.81	8.41	11.40
Reichert-Wollny value	0.60	4.50	2.80	0.50	5.20	0.20	4.40	6.40
Polenske value	0.70	0.80	0.70	0.70	0.90	0.40	0.50	0.50
Unsaponifiable matter per cent.	39.20	24.43	26.70	2.41	2.04	—	—	—
Refractive index, n_D^{40}	1.4568	1.4702	1.4704	1.4640	1.4713	1.4648	1.4711	1.4727
Free fatty acids (as oleic acid), per cent.	1.50	12.41	10.26	12.50	16.51	0.41	4.23	5.73
Ether-insoluble bromides, per cent.	3.54	None	None	22.60	None	—	—	—

In every case the rise and fall of the constants and variables of whale and cottonseed oil are similar to those of shark oil.

It will be evident, from what has been stated, that the general conclusions which can be drawn from the results are that changes in composition are in the same direction as the blowing is continued, so long as the same conditions are observed. Under different conditions these changes are not comparable, except that the viscosity and refractive index rise simultaneously.

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.

RAPID SORTING TEST FOR SMALL QUANTITIES OF TARTARIC ACID IN SELF-RAISING FLOUR.

THE acidic constituent of self-raising flour may be either an acid phosphate or tartaric acid, or it may be a mixture of the two. If in a mixture, the tartaric acid (possibly present as potassium acid tartrate) may be present in such relatively small amounts, that its presence could easily be overlooked, unless it were the object of particular search.

The following method affords a means of rapidly detecting such small quantities, and readily indicates the presence of less than 0.1 per cent. of tartaric acid in a self-raising flour:—

About 4 grms. of flour are well shaken with 20 c.c. of water, allowed to stand two minutes, and then filtered into a test tube. When about 10 c.c. of filtrate have been collected, a few drops of dilute ammonia are added, with shaking. A little powdered silver nitrate (about 0.05 gm.) is then dropped into the liquid, and the test-tube is placed, without shaking, in water at about 70° C. After two minutes the tube is shaken.

A self-raising flour, free from tartrate, gives a pale yellow, turbid liquid. If tartrate is present, silver is reduced, and a turbid liquid, varying in colour from light grey to almost black, according to the amount of tartrate in the flour, is obtained.

The presence of relatively large quantities of calcium and sodium phosphates and of soluble constituents of the wheat flour appears to interfere with the formation of a mirror by the small amounts of tartrate which may be present. No mirror could be obtained with flours containing 0.1 to 0.5 per cent. of tartrate. Of course, if the tartaric acid is extracted with an organic solvent, and thus obtained in a much purer condition, a mirror is readily produced by the extract, but the object of the method described is to get the result more rapidly than by other methods. It will be found particularly useful when several samples have to be examined at the same time.

A. F. LERRIGO,

CITY ANALYST'S LABORATORY, BIRMINGHAM.

A COLOUR REACTION OF SAPONIN WITH NITRATES.

PURE commercial saponin (B.D.H.) can replace brucine or strychnine in the test for nitrates. On adding a drop of concentrated sulphuric acid to a mixture of minute quantities of the saponin and a nitrate, the well-known blood-red coloration is produced. The intensity of the coloration is proportional to the amount of nitrate, and apparently to that of the saponin. It has yet to be discovered whether the reaction is characteristic of all saponins or only of those from a particular source, and also whether a quantitative method of determining saponin can be based upon it. These questions are being studied, *inter alia*, in an investigation under the Analytical Research Scheme of the Society.

C. AINSWORTH MITCHELL.

DATA USED IN GRAPH FOR BEESWAX.

IN reply to an enquiry respecting my graph for the analysis of beeswax (*cf.* ANALYST, 1925, 50, 445), I may say that the exact numerical values taken as limits for pure beeswax were as follows:—

Specific gravity, 0.976 to 0.941; m.pt., 60.5° to 66.5° C.; butyrorefractometer reading, 43.2 to 45.7; iodine value, 5.42 to 17.1; saponification value, 87 to 106; acid value, 16.7 to 23.6; ester value, 65.9 to 85; ratio value, 2.8 to 4.5; and un-saponifiable matter, 48 to 53 per cent.

ARTHUR A. WEIR.

38, DELBRIDGE ST., NORTH FITZROY,
MELBOURNE.

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 CITY ANALYST FOR THE FOURTH QUARTER, 1925.

OF the 1298 samples submitted for analysis during the quarter, 1121 were analysed under the Sale of Food and Drugs Acts, and 177 were examined for various Corporation Departments. Among the 55 samples submitted by the Health Department were 4 samples of apples. One contained 1/100 grain of arsenic per lb. (present on the skin); the other three samples were free from arsenic.

Of the 1121 food and drug samples, 1084 were bought informally (21 adulterated), and 37 were bought under the provisions of the Acts (3 adulterated). The total percentage of adulteration was 2.1.

MILK.—Twenty-one of the 578 samples contained less than 11.5 per cent. of total solids. One of 153 samples of bottled milk contained only 8.1 per cent. of solids-not-fat, but a subsequent sample was genuine.

MARGARINE.—Thirteen of the 166 samples from 9 vendors were in wrappers which did not bear the word "Margarine."

LARD.—Two samples sold as "pastry lard" were found to consist of compound lard and were reported as adulterated.

CHICORY.—Five samples were genuine, but one sample was adulterated, the ash being 8.3 per cent., which included 3.7 per cent. of sandy matter.

DRUG TABLETS.—Forty-two samples were examined, and 2 of them had false labels (*vide infra*).

FRENCH CHALK.—The British Pharmaceutical Codex defines two varieties of talc, one of which is called "French Chalk," and the other, which has been treated with acid, is termed "Purified Talc." The "Powdered Talc" of the B.P. has been purified with acid.

Eight samples bought as *French chalk* yielded 92.0 to 98.3 per cent. of ash; the amount soluble in dilute acid varied from 1.9 to 13.2 per cent.; and the calcium in the acid soluble portion was equivalent to 1.9 to 10.6 per cent. of ordinary chalk.

Three samples were bought as *purified talc*. They yielded 95.7, 98.4 and 99.2 per cent. of ash; the amounts soluble in acid were 2.5, 2.0 and 0.4 per cent.; and the soluble calcium expressed as chalk, 1.0, 2.1 and 0.9 per cent. It is doubtful whether two or these samples had been treated with acid, as they resembled some of the samples of French chalk sold at a much lower price.

The amount of arsenic was trivial, being from 0 to 4 parts per million. The samples yielded almost a constant amount to cold water, the ash of the soluble matter being from 0.3 to 0.4 per cent. of the talc taken.

J. F. LIVERSEEGE.

EXAMINATION OF DRUG TABLETS.

MACHINES used for making tablets have a cavity of adjustable size which is automatically filled with the material; the die then descends and forms the tablets by compression. If the mixture is not in a uniform state of division, equal volumes will not correspond with equal weights, and some tablets will weigh more than others.

By weighing over 1700 individual tablets a good indication was obtained as to the uniformity of weight of the tablets in each bottle. It was found that the weight of 91.8 per cent. of the tablets did not vary more than 5 per cent. from the mean weight of the tablets of the sample; 7.3 per cent. showed a variation from the mean of 5 to 10 per cent.; and 0.9 per cent. an error exceeding 10 per cent. from the mean.

Some samples were remarkably uniform in weight. In one bottle of 100 sodium citrate tablets the lightest weighed 2.42 grains and the heaviest 2.57 grains. In another bottle of 50 the corresponding weights were 1.88 and 2.10 grains—figures which show how uniform tablets can be made.

The total weight, *i.e.* the weight of the drug with starch, talc, etc., showed a good deal of variation in different samples. Average weights of 5 grain tablets in a bottle varied from 6.7 to 4.5 grains, the excess being due to the talc, starch, etc., and the use of partly dried drug accounting for the lighter weights.

For the production of satisfactory tablets not only must the powder be uniform, but the cavity of the machine must be adjusted to contain a volume which represents the correct weight of the drug, otherwise the tablets may be uniform, but contain an excess or deficiency of the drug. Each tablet of one sample of aspirin tablets, for example, was within 5 per cent. of the mean weight, but 9 tablets showed an excess of 5 to 10 per cent. above the stated 5 grains of aspirin, and the other 16 tablets showed an excess of more than 10 per cent. Experiments showed that the composition of the heavier tablets was the same as that of the lighter ones, so that a heavy and a light tablet did not each contain the correct amount of drug with more or less of the binding material.

There was considerable difference in the rate of disintegration of the tablets. The appearance was not satisfactory evidence of the solubility of a tablet in cold water, as sometimes a skeleton of talc or starch retained the original appearance, although the soluble matter had been removed. Speaking generally, the tablets containing the largest proportion of French chalk or talc disintegrated the most slowly.

Sodium Salicylate Tablets (5 grains).—The average amount of sodium salicylate per tablet in 6 samples was 4.9 to 5.0 grains, but individual tablets varied from 4.4 to 5.6 grains. Four samples contained no talc, the other two, 5.2 and 6.9 per cent. The last also contained starch, whilst the other five contained glucose, but not starch.

Aspirin Tablets (5 grains).—The following table shows the composition of 12 samples:—

	Grains of aspirin per tablet.			No. of tablets incorrect.			Talc. Per Cent.
	Average.	Max.	Min.	Under 5 per cent.	5 to 10 per cent.	Over 10 per cent.	
1	4.8	5.1	4.3	19	4	2	2.1
2	5.1	5.3	4.9	20	5	0	2.3
3	4.7	4.9	4.6	12	13	0	1.9
4	5.6	5.8	5.3	0	9	16	0
5	4.8	5.5	4.3	12	9	4	2.7
6	4.9	5.1	4.7	22	3	0	4.5
7	4.9	5.3	4.6	17	8	0	0.9
8	5.0	5.2	4.8	25	0	0	0
9	5.0	5.1	4.8	25	0	0	0
10	4.6	4.9	4.5	5	20	0	2.1
11	4.8	4.9	4.7	10	6	0	0
12	4.9	5.2	4.7	25	0	0	0

The average weight of aspirin in No. 4 and No. 10 was 5.6 and 4.6 grains, respectively. The labels stating that they were 5 grain tablets were therefore false. None of the tablets of No. 4 was within 5 per cent. of the proper amount, and only 5 of No. 10. Five of the samples were free from talc, and the other 7 contained from 0.9 to 4.5 per cent. The British Pharmaceutical Codex orders 2 per cent. of purified talc to be used in the preparation of these tablets. The tablets containing 4.5 per cent. and 2.7 per cent. of talc were very slow in breaking down in cold water. In the author's opinion aspirin tablets which dissolve more rapidly are preferable.

Calcium Lactate Tablets.—Calcium lactate, according to the British Pharmacopoeia, should contain not less than 93 per cent. of pure calcium lactate. On drying, pure calcium lactate loses 29.2 per cent. of water. The five grain tablets may therefore contain from 3.29 to 3.54 grains of dried calcium lactate.

Eleven samples were marked "Calcium Lactate, 5 grains," and the average amount of the dried salt varied from 3.4 to 3.7 grains,—figures which suggest that the B.P. limit of 93 per cent. is unnecessarily low. In the remaining sample the average was only 3.1 grains. In 4 samples the average weight of the tablets varied from 4.5 to 4.8 grains, a result which at first suggested an insufficient amount of calcium lactate, but the proper amount of dried lactate was found to be present—showing that the drug had been partly dried, and a proportionately smaller quantity taken.

The amount of talc varied from a trace to 7.0 per cent. Starch was present in 5 samples, and in most cases the tablets disintegrated rapidly in water.

Sodium Citrate Tablets.—Sodium citrate is not included in the British Pharmacopoeia. The B.P. Codex of 1907 stated that the drug contained $5\frac{1}{2}$ molecules of water of crystallisation (= 27.7 per cent. of water). The 1923 edition of that work prefers 2 molecules of water (12.3 per cent.), but states, "Some of the sodium citrate in commerce contains $5\frac{1}{2}$ molecules of water." This position is very unsatisfactory, as five grains of the one are equivalent to 6 grains of the other. Undoubtedly the article should be added to the Pharmacopoeia.

The following table gives the results of the examination of 12 samples of tablets:—

No.	Grains stated on label.	Water-free equivalent. Grains.	Water-free average. Grains.	Talc. Per Cent.
1	5	4.4 or 3.6	4.3	0.5
2	2	1.8 or 1.4	1.7	0
3	2	"	1.4	0
4	2	"	1.6	3.1
5	2	"	1.7	2.2
6	2	"	1.6	2.5
7	2	"	1.6	3.6
8	2	"	1.7	1.0
9	2	"	1.7	0.1
10	2	"	1.4	3.8
11	2	"	1.8	2.9
12	1	0.9 or 0.7	0.7	0

The first three samples were made by one firm, the following four by another, and the next two by a third firm. Ten of the 12 samples were prepared with the drug containing 12.3 per cent. of water, and two (Nos. 10 and 12) with the drug containing 27.7 per cent. Sample No. 3, which was some years old, contained an

amount of the former equivalent to 2 grains of the latter. The quantity of either one salt or the other was near the stated amount in all the samples.

Seven of the samples contained talc (1.0 to 3.8 per cent.). As sodium citrate is largely used for adding to babies' milk it would seem better that an insoluble substance like talc should be absent, although the author has no information that its presence in milk is harmful. None of the samples contained starch. Although they differed in solubility, no objection could be taken to any of them for insolubility.

ANALYTICAL DEPARTMENT,
CITY OF BIRMINGHAM.

J. F. LIVERSEGE.

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.

LIABILITY FOR DAMAGE BY A SECRET CLEANING FLUID.

ANGLO-CELTIC SHIPPING CO. v. ELLIOTT AND JEFFERY AND OTHERS.

In this action, tried by Mr. Justice Roche, on February 11, 1926, the plaintiffs, owners of a steamship, claimed damages from two defendants alternatively for negligence and breach of duty.

The plaintiffs had sent a ship to the first defendants, a firm of ship repairers at Cardiff. Among other work to be done was the cleaning and repair of the condenser, and the plaintiffs told the defendants to use a cleaning fluid, known as "the Plu-perfect fluid," which was manufactured by the second defendants. This fluid had the property, unknown to the plaintiffs or the first defendants, of giving off hydrogen on contact with cast iron. The fluid was used for cleaning the brass tubes on the water side of the condenser; some of it leaked through to the steam side, and so came into contact with cast iron, and the hydrogen produced formed an explosive mixture with the air. A workman went to the spot carrying a lighted candle, and an explosion occurred, damage being done to the extent of £1200. The plaintiffs claimed that they were not liable to pay for this damage, and they also claimed £841 for loss of the use of the vessel during the period this damage was being repaired. Alternatively, they claimed these sums against the second defendants.

In their defence the defendants pleaded that they knew nothing about the Plu-perfect liquid themselves, that they used it in accordance with the plaintiff's instructions, and that plaintiffs assured them that it was harmless. The second defendants pleaded that the explosion was due to the negligence of the plaintiffs in not giving the defendants notice that the condenser tubes were leaking; and they said that if there was any risk in using the preparation, which they denied, the plaintiffs knew of the danger and elected to take the risk.

Evidence was given in support of the plaintiff's case, and it was stated that the manufacturers sent out a circular with the fluid, in which they pointed out that the liquid caused the formation of carbon dioxide which must be allowed to escape, but they said nothing of any danger of the formation of an explosive mixture.

Expert chemical evidence was given to the effect that analysis showed the Plu-perfect liquid to consist of a 10 per cent. solution of hydrochloric acid, with traces of other substances and a small amount of citronella oil to give a distinctive odour.

Counsel for the second defendants said that it was necessary for the plaintiffs to prove that the article was dangerous *per se*. The second defendants' business was carried on by the widow of the inventor of the fluid, and she and her son prepared it personally. It contained, besides the hydrochloric acid, about 1 per cent. of copper sulphate, the object of this being to form a film on iron which would prevent the liberation of hydrogen. The liquid was not dangerous *per se*, and the instructions issued with it provided for its safe use. The accident was due to an unfortunate combination of abnormal circumstances.

The Judge held that there must be judgment for the first defendants, with costs. As to the second defendants, it was sufficient for him to assume the truth of the proposition that if a dangerous thing was sold, a warning must be given. He was satisfied that the Plu-perfect liquid was dangerous of itself; it was sold under a fancy name, its composition was not disclosed, and it was disguised by a particular smell which was purposely imparted to it. The different analyses which had been given in evidence suggested that there were irregularities in manufacture. And not only was the article dangerous in itself, but the instructions failed to give any adequate warning. So there must be judgment for the plaintiffs, with costs.

SALE OF MEDICATED WINE WITHOUT A LICENSE.*

ON February 23rd a druggist was charged at the Brighton Police Court with having sold alcoholic liquor without a justices' licence.

A police constable gave evidence that he had asked the defendant for Wincarnis, and had been told to go to a wine store for it. He then bought a bottle of Wincarnis and quinine, on which a sixpenny stamp duty had been paid.

The Public Analyst (Dr. S. Woodhead) stated that the wine contained 13.99 per cent. of alcohol and 1.2 grains of quinine per fluid oz.

The defendant said that he had sold this preparation under a patent medicine licence for many years, and his solicitor contended that a medicine on which patent medicine duty had been paid was exempt from the provisions of the Licensing Act.

The Magistrates imposed a fine of 20s., with £1 11s. 6d. costs. Notice of appeal was given.

ARTIFICIALLY COLOURED GRAPE FRUIT.†

ON September 16, 1925, the U.S. Attorney for Porto Rico filed in the U.S.A. District Court for that district a libel praying for the seizure and condemnation of 361 cases of grape fruit shipped from the territory of Porto Rico into the State of New York, and charging adulteration in violation of the Food and Drugs Act.

Adulteration of the article was alleged for the reason that a product, *viz.* immature grape fruit artificially coloured, had been mixed and packed with, and substituted wholly, or in part, for the said article, and for the further reason that it had been coloured in a manner whereby inferiority was concealed.

On October 3, 1925, the manager of the fruit company admitted the allegations of the libel, and it was ordered by the Court that the product should be destroyed by the United States Marshal.

* It has long been accepted that a registered pharmacist may sell a medicated wine containing the above-mentioned amount of quinine without a licence.—EDITOR.

† U.S.A. Dept. of Agriculture. Service and Regulatory Announcements. Bureau of Chemistry. Suppl. 207. No. 13,848, Jan. 1926.

GROUND ALMONDS AND THE MERCHANDISE MARKS ACT.

ON February 16th a firm manufacturing marzipan and almond products was charged under the Merchandise Marks Act, 1887, before the Liverpool Stipendiary Magistrate, with applying a false trade description to one of their products. The defendant company had, it was stated by the prosecution, supplied to a firm in Liverpool certain cases which were found, on analysis, to contain ground apricot kernels, under the terms of a contract for pure sweet English ground almonds.

The goods were left at the station, and on October 16th, 1925, samples were taken by an inspector of the Food and Drugs Department from each of the boxes, and some were sent to the City Analyst (Mr. Roberts) and others to the defendants.

The analysis of the City Analyst indicated that the samples in question consisted of ground apricot kernel, and, although it was not possible to say that there might not be a small percentage of ground almonds present, it could not amount to more than 20 per cent.

Mr. Alfred Smetham stated that, in his opinion, the possible amount of ground almonds did not exceed 10 per cent., whilst Mr. S. E. Melling agreed that there might possibly be as much as 20 per cent. present.

Evidence was called to show that in the trade the term "ground almonds" meant, and always had meant, the kernel of the almond nut ground, and nothing else, and it was not a generic term applied to ground kernels of any description that could be used for the same purposes as ground almonds. Further evidence was given to show that in the trade the term "ground almonds" was an absolute guarantee that the goods to which it applied consisted of the almond nut itself, ground.

For the defence it was contended that an analysis made on their behalf had yielded results at variance with those for the prosecution. The analyst, in reply to the Magistrate, said that he could not explain why his result was different.

Witnesses engaged in the trade produced preparations made with apricot kernels, ground almonds, and some of the material made by the defendants, and invited the Magistrate to taste them. This Mr. Stuart Deacon declined to do, observing that he was there to judge by evidence and not by his own taste.

On February 26th the Magistrate gave a written judgment:—It appeared from the evidence, he observed, that at the time the contract was made the price of almonds ranged from 180s. to 220s. per cwt., that of bitter apricot kernels about 75s., and that of sweet apricot kernels about 100s., so that there would be considerable profit accruing to a person who could sell ground apricot kernels at the price of ground almond kernels.

No evidence as to the origin of the goods in question had been given by the defendant firm, but, an analyst for the defence had stated that, in his opinion, it was not possible to detect the difference between ground almonds and ground apricot kernels. In cross-examination this witness had said that he could not account for his results being so different from those of other analysts, unless there was some slight difference in the sample tested. Another witness for the defence had expressed the view that the only reliable test was to do what was done in the case of flours, and fall back upon the baking test. He pointed out that, if one baked apricot kernels, a bitter product instead of a sweet one was produced.

In his (the Stipendiary's) opinion, even if the various chemical and analytical tests applied by the other witnesses were to be regarded in one respect or another as unreliable, surely the suggested baking test was even more unreliable by reason of its being so much less scientific. He really had no option but to accept as more reliable the many tests which were applied to the many samples by the three

analysts for the prosecution. With deliberation, he expressed the view that it seemed an almost ludicrous suggestion to the Court that the baking test should be relied upon as an effective test in contrast to the scientific and analytical tests applied by scientific witnesses.

In imposing a fine of £20 with £90 6s. costs, the Stipendiary added that the case was an illuminating one, as being illustrative of the attempts which were made from time to time to supply a long-suffering and unsuspecting public with ingredients in their food which were of a nature totally different from those with which they believed they were being supplied, and that, of course, always at a price which put an extra illicit and unsuspected profit into the pockets of the vendors. The Health Committee of Liverpool Corporation were to be congratulated on having had the public spirit to institute these proceedings at the risk, had the decision been adverse to the prosecution, of having to incur and pay considerable costs to the defendants.

EGG CUSTARD.

ON March 2, a grocer was summoned at Marylebone Police Court by the Hampstead Borough Council for having sold to the prejudice of the purchaser, egg custard not of the nature, substance and quality demanded.

The certificate of the Public Analyst (Dr. H. E. Cox) stated that the substance contained, by weight, 12.28 per cent. of moisture, 83.7 per cent. of dry starchy matter, not more than 4 per cent. of dry egg, and a trace of artificial colour and flavour. This analysis, the certificate stated, showed that the sample was not real egg custard, but a mixture of coloured and flavoured maize starch, with a small proportion (not exceeding 4 per cent.) of dried egg; whereas real egg custard was a product of eggs and milk, and should not consist mainly of starch.

Mr. S. G. Turner, for the prosecution, said that there was a distinction to be drawn between articles sold as custard powder or egg substitute, as many were sold, and an article like this which was sold as real egg custard. The label of the packet bore the words: "Eggs is eggs, and so is Peterkin real egg custard." According to the instructions on the packet, the amount of the contents to be mixed with a pint of milk ought to correspond (to make a custard of the same quality as that commonly known as custard) to two whole eggs. Instead of that, the amount of egg present was not more than one-thirtieth of the quantity that would be present in a custard made in the usual way.

Dr. H. E. Cox said that two whole eggs, weighing about 4 ozs., would be equal to about $1\frac{1}{2}$ ozs. of dry egg, and that that would be enough to make 1 pint of custard. If sufficient of the sample were taken to make a pint of custard, there would be only about one-twenty-fifth of an ounce of dried egg in it.

In cross-examination he said that the amount of water in an egg was about 67 per cent., and he would disagree with Mr. E. Parry (the analyst for the defence) if he said 75 per cent. He agreed that, if Mr. Parry's figure was right, the sample would contain the equivalent of 16 per cent. of egg, and that if 4.5 per cent. of dried egg was present (as found by Mr. Parry) it would correspond to nearly 18 per cent. of egg.

Dr. Skrase, Medical Officer of Health for Hampstead, said that if a purchaser accepted this article, as real egg custard, he would be prejudiced as regards health; and if he were an invalid, it might be deleterious.

Mr. W. Frampton, for the defence, submitted that he had no case to answer. The directions disclosed that the article was egg custard powder, and no person in purchasing a pint packet for 1½d. could possibly be prejudiced because there was not in the packet 50 per cent. of egg and 50 per cent. of milk. When it was admitted that eggs cost 2½d. each it was absurd to expect two eggs in a 1½d. packet. On Dr. Cox's evidence there was 13 per cent. of egg in the sample, and this would be raised to 18 per cent. if the figures of the analyst for the defence were accepted. He submitted that the quantity of egg in the article was appreciable, having regard to the price charged and the way custards were made. Custard powders generally contained no eggs at all.

The Magistrate (Mr. Hay Halkett), having been informed that there was no standard for custard powder, and that he must make his own standard, then gave his decision. He did not think the sale was to the prejudice of the purchaser, since at the price charged he could not expect to get anything better. The amount of egg was very small, but he could not say that it was so small as to be to the prejudice of the purchaser. He therefore dismissed the summons.

Parliamentary Notes.

CERTIFIED MILK AND PASTEURISED MILK.—On February 25, the Minister of Health, replying to questions by Col. Fremantle, said that there were 96 producers' licences in operation in England and Wales for Certified milk, and 97 for Grade A (tuberculin tested) milk. On March 31, 1925 (the latest date for which figures were obtainable) there were 110 licences in operation for Grade A milk. The numbers of cows in the herds producing the first two grades of milk were 3500 and 3800 respectively. No exact information as to the number of cows in Grade A herds was available, but it was estimated at approximately 2000 to 2500.

Licences for the sale of pasteurised milk, issued by local authorities, showed that there were 62 pasteurising establishments working on March 31, 1925, and that there were 370 premises licensed to distribute pasteurised milk. As a rough estimate, the amount of milk annually pasteurised in England and Wales might be taken as between 15 and 20 million gallons.

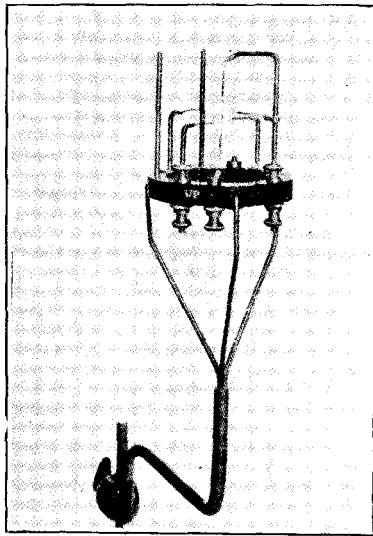
COLD STORAGE AND FOOD PRESERVATIVES.—Mr. Spoor asked whether, in view of the position about to be created by the prohibition of food preservatives, he would call for returns from Medical Officers of Health indicating the extent and adequacy of facilities for cold storage in their respective areas.

Mr. Neville Chamberlain replied that such returns would be unnecessary, since the Government was already in possession of information as to the cold storage accommodation in the country. As regards the other part of the question, he thought that the Hon. Member probably over-estimated the extent to which the Preservative Regulations would necessitate any increase in the facilities for refrigeration in the home, and he did not propose to take any steps in the matter.

Medical Research Council.

METHODS OF INVESTIGATING VENTILATION AND ITS EFFECTS.*

PART I. THE MEASUREMENT OF VARIATIONS IN THE VELOCITY AND TEMPERATURE OF AIR CURRENTS.—A portable “hot wire” anemometer has been designed to measure rapid oscillations of air movement vertically as well as horizontally. It consists of a modified Wheatstone bridge with the hot wire or “detector” portion constructed of three platinum wires, each 17 mm. long and 0.08 mm. diameter, soldered to thick brass supports in three planes at right angles, mounted on an ebonite ring, so that whatever the angle at which the air stream



strikes, the maximum value is obtained (see Figure). A 0.5 amp. current heats the wires to above 130° C., and the changes of resistance are read at intervals of $2\frac{1}{2}$ seconds on a galvanometer. The apparatus was calibrated against a kataba thermometer in a small wind tunnel.

When the apparatus was used the detector was first covered with an inverted cylindrical tin ($5 \times 3\frac{1}{4}$ inches), the mouth of which was packed with cottonwool to prevent access of air currents. When the circuit was completed the needle of the galvanometer attained a constant position in about 15 seconds. It was found that the change on galvanometer produced by a rise or fall of temperature was 4.5 scale divisions for each °C. The scale reading (with the detector covered up) was also noted at the finish, and the mean taken, but the readings seldom differed by more than two divisions. In order to measure variations in the temperature of air currents a delicate thermopile was constructed. The wires of nickel and iron were soldered together, the junctions hammered thin, and 20 junctions fixed in a boxwood ring, 5.3 cm. internal diameter, the lower ends being soldered together connected with three binding screws, and protected by an annular trough of brass.

* By Dr. H. M. Vernon and others. Special Report Series, No. 100. Pp. 71 Obtainable at Adastral House, Kingsway, W.C.2. Price 2s. net.

and the whole connected with a dead-beat galvanometer. The apparatus was calibrated by dipping the junctions (coated with lamp-black) alternately into water at different temperatures, and by taking from one room to another of a different temperature. The thermopile was not affected by changes in air velocity, nor the anemometer by temperature changes.

PART II. CALIBRATION OF THE KATA THERMOMETER.—The “factor” for the kata thermometer is not actually a constant, but there is an approximately linear relationship between it and the temperature of the chamber, and it is proposed that the temperature at which the factor is determined should be corrected to some standard temperature, *e.g.* 60° F. Partly owing to this and, to a less extent, to other factors, the relationship between H/θ values and air velocity is considerably affected by temperature, the correction necessary decreasing as the magnitude of H/θ increases, and ranges from 0.44 per cent. per °F., with an H/θ of 0.27, to one of 0.1 per cent., with an H/θ of 0.6 and upwards.

The response of the kata thermometer and hot wire anemometer to vertical air currents showed that the former is quite unreliable for measuring descending currents, but moderately accurate for ascending currents, and that the anemometer indicates about two-thirds the theoretical velocity (as measured by vane anemometer) of descending currents.

PART III. INFLUENCE OF COOLING POWER AND OF VARIABILITY OF AIR CURRENTS ON SENSATIONS OF AIR MOVEMENTS.—Nine classes were agreed upon for recording the sensation of the air, ranging from “very stagnant” to “very fresh,” and the cooling power of the air was determined after its cataloguing by “sensation,” also its oscillations of velocity and temperature.

Experiments were made in a large number of factories, 229 observations in summer and 203 in winter. Various degrees of “freshness” were found to be mainly due to differences in air velocity, whilst in the case of “stagnancy” they were entirely due to temperature differences, although allowances had to be made for “acclimatizing.” The measure of these two variables is best combined by the kata thermometer, and the correlation ratio of cooling power on air sensation was found to be 0.703 in summer and 0.780 in winter.

It could not be proved that oscillations of temperature increase the sensation of air movement, but oscillations in velocity do so moderately. Air velocity is not much affected by turning on the plenum ventilation in factories, but intermittent ventilation does increase the velocity of the oscillations.

PART IV. INFLUENCE OF TEMPERATURE, AIR VELOCITY AND CLOTHING ON THE RATE OF COOLING OF THE HUMAN BODY.—This section represents an attempt to ascertain the relative degrees of importance of air temperature and velocity and clothing on the human body, particularly with a view to establishing the best conditions for heavy workers in hot atmospheres, and the method of experiment adopted was to warm the body by step climbing, and subsequently to determine the rate of cooling. In the unclothed or very lightly clothed body an air current of 100 ft. per minute, as contrasted with still air, caused an extra cooling corresponding to a lowering of the air temperature by 1 to 4° F., and in a warmly clothed body a lowering of only 1° F. It was found that the unclothed body of the experimenter would remain in equilibrium at an air temperature of 81–83° F., and the warmly-clad body at 59–60° F., so that for cooling an overheated body it is most important first to reduce the temperature of the air, and then to reduce the amount of clothing, but an increase of air velocity has little effect.*

D. G. H.

* In connection with this Report reference may be made to Mr. R. Frederick's investigation of ventilation conditions and his observations on cooling power and the use of the wet and dry kata thermometer (*ANALYST*, 1925, 50, 213–224; 399).—EDITOR.

Revision of Inorganic Chemical Nomenclature.

I. REPORT BY PROF. M. DELÉPINE.*

CLASSIFICATION FOR TABLES AND INDEXES.—This should be based on the alphabetical order of the symbols and then on the increasing number of each of the elements, and this method is already used in the Formula Index of *Chemical Abstracts*. It presumes the unification of the symbols, started by the 1900 Congress, but which is not yet complete (*e.g.* niobium (Cb or Nb), and beryllium, Gl or Be). The small letters of symbols of two letters serve as a secondary classification, so that $\text{Ag}_3\text{S}_4\text{Sb}$ precedes Ag_3Sb , since S comes before Sb; and AlCl_3 precedes AuBr_3 , since Al comes before Au. Alphabetical order, however, will put substances in different positions according to language. It is generally agreed that the positive group in salts of metals shall precede the negative.

INDICATION OF VALENCY.—The Czech language allows of all degrees of combination being expressed by the appropriate designations with reference to oxygen. In Germany and Switzerland a system (that of Werner) is available, whereby vowels and diphthongs are inserted between the component parts of the name, *e.g.* nitro-pentammin-kobaltichlorid $[\text{Co}(\text{NO}_2)(\text{NH}_3)_5]\text{Cl}_2$, but the method offers difficulties in many languages. The simplest method would be the indication of degree of valency by amount, with a figure or the word valent preceded by a numeral. In teaching, salts of "iron II" or "iron III" would be referred to, so that analogies would show themselves normally and words like *sesquioxide* would be eliminated.

WRITING OF FORMULAE.—The unification of current modes of naming inorganic compounds is hindered by fundamental differences in the languages, and often formulae are written and compounds spoken of differently. The Latin nations propose to make their language conform to the formulae in the same way that Anglo-Saxons, Germans, etc., do. The Dutch Committee has proposed that for compounds with pronounced polarity the positive element should be placed first, and it suggested that the order in which the elements become more negative should be as follows:—Si, C, Sb, As, P, N, Te, Se, S, I, Br, Cl, O (*e.g.* Cl_2O , ICl_3). For combinations of metals among themselves the metals should be either in order of atomic weight (French proposal), or in that of their electro potential or, more simply, in their alphabetical order (Dutch proposal).

NAMES OF OXYGENATED ACIDS DERIVED FROM NON-METALS (OR FROM METALS OF A SIMILAR CHARACTER).—France, Denmark and U.S.A. have presented lists which all proceed from the original method of nomenclature by the use of the terminations *-ique*, *-ic*, or *-syre*, with *-eux*, *-ous* or *-syrling* for compounds lower in oxidation, and the prefixes *meta*, *ortho* and *pyro* for other degrees of oxidation. It is agreed to suppose that *ortho* indicates the acid richest in water, *meta* the poorest, and *pyro* an intermediate acid, but this is vague. It has been suggested that *meta* should denote the loss of one molecule of water. If certain acids are agreed upon as fundamental and their actual names preserved, a number of others might then lose their prefixes *ortho*, *meta* or *pyro*. A more hydrated acid than the fundamental one would be called *aquo* (instead of *ortho*), and a less hydrated one *anhydro*, and numerical prefixes would indicate composition, no explanation being required when they equal unity. Thus H_2CO_3 would represent carbonic acid; H_4CO_4 , aquocarbonic (instead of orthocarbonic acid); H_2SiO_3 , silicic (metasilicic); H_4SiO_4 , aquosilicic (orthosilicic); H_3PO_4 , phosphoric (orthophosphoric); HPO_3 ,

* *Chem. Weekblad*, 1926, 23, 86–93.

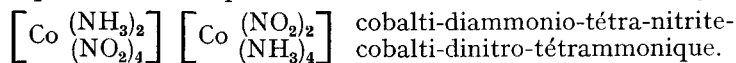
anhydrophosphoric (metaphosphoric); $H_4P_2O_7$, anhydrodiphosphoric (pyrophosphoric); H_3BO_3 , boric (orthoboric); HBO_2 , anhydroboric (metaboric); $H_2B_4O_7$, pentahydrotetraboric (pyroboric); H_2WO_4 , tungstic; $H_2W_4O_{13}$, trianhydro-tetratungstic (metatungstic); H_2MoO_4 , molybdic; $H_6Mo_7O_{14}$, tetranhydroheptamol-ybdic (heptamol-ybdic) acid. The following list shows that generalisation is possible for the iodic and periodic acids:— HIO_3 , iodic acid; HI_3O , $(3HIO_3-H_2O)$, anhydrotriiodic acid; $H_2I_4O_{11}$ $4(HIO_3-H_2O)$, anhydrotetraiodic; HIO_4 , periodic (metaperiodic); H_3IO_5 , aquoperiodic (paraperiodic); H_5IO_6 , diaquoperiodic (periodic); $H_4I_2O_9$, aquodiperiodic (paradiperiodic) acid. H_5IO_6 might be taken as the fundamental acid, and the names would then become:— H_5IO_6 , periodic acid; H_3IO_5 , anhydroperiodic; HIO_4 , dianhydroperiodic; $H_4I_2O_9$, trianhydrodi-periodic acid, etc. However, the analogy with perchloric acid is then lost. An indication of the acid value of little known acids could be made by a numerical prefix. The names of anhydrides should be reserved for oxides forming acids with water, such as sulphurous anhydride; SO_2 ; sulphuric, SO_3 ; silicic, SiO_2 ; and phosphoric, P_2O_5 . Where all oxygen is replaced by sulphur the term sulphanh-ydride might be used. Anhydrides of bases should be anhydroxides.

NAMES OF SALTS.—The terms *-ate*, *-ite* (or their equivalents) for oxygenated acids are agreed upon. Acid salts can be precisely indicated by adding a numerical prefix before the word "acid" or "hydrogen," e.g. SO_4HK , sulfate acide de potassium, or potassium hydrogen sulphate; PO_4H_2K , phosphate diacide de potassium, or potassium dihydrogen phosphate. The number of acidic hydrogens replaced is calculated on the formula of the acid, not on the total formula of the salt (e.g. F_2HK , difluorure acide de potassium, potassium hydrogen difluoride).

WORDS DENOTING ACIDITY AND BASICITY.—It is proposed to replace the terms *mono*, *bi*, etc., by *monoacidic*, etc., used either as adjectives or substantives, so that oxides would be classed not as *mono-acidic*, but as *monobasic*, etc. An objection was raised at the Cambridge Conference to the sound in English of the expression "acidic," etc., and a consideration of the applicability of *monohydric*, etc., not only to acids, but also to bases and alcohols is requested.

WATER IN COMPOUNDS.—The U.S.A. Committee suggests the restriction of the word *hydroxide* to compounds containing OH, and hydrate for those containing water. Successive dehydration might be indicated (Kollo) by putting the water fixed most firmly in brackets (thus $[Na_2HPO_4(H_2O)_7]$, $5H_2O$), but, in view of the small amount of exact data at present available as to the composition of such products, the proposal seems premature.

COMPLEX COMPOUNDS (NOT DOUBLE SALTS).—If the complex ion is negative the heavy metal is put first, then the entire molecules terminated by the letter *-o*, and arranged in order of relative mass, then the negative radicle in the same order, also terminated by the letter *-o* (*nitro*, *chloro*, etc.), except that which comes last, which would have the same termination as a salt (e.g. $[Cr(NH_3)_2(NO_2)_4]K$, chromi-diammonio-tétranitrite mono-potassique). If the complex ion is positive the metal would come first, then the negative radicles in order of relative mass, then the entire molecules in the same order, with the termination of *-o*, except that water is terminated by *aque* (e.g. $Cl_2[CrCl(NH_3)_5]$ dichlorure chromi-chloro-pentammonique. With complexes the names of the two ions are joined, e.g.



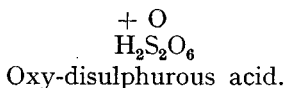
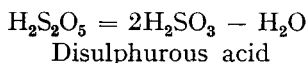
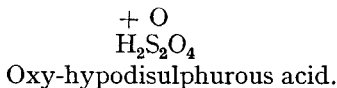
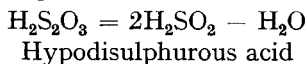
Non-electrolytes follow the order: metal, radicles in order of relative mass, whole molecules (e.g. $Cr \begin{array}{c} (SCN)_3 \\ (NH_3)_3 \end{array}$ chromi-trisulphocyano-triammoniac). The termination

-aque might be replaced by *hydrine* or *hydrique* (corresponding to *ammonique*). Del Campo suggests that the positive ions should be placed in the inverse order to that of negative ions. An indication of the degree of acidity in Roman figures would eliminate the terms *-eux* and *-ique* (e.g. $[\text{Cr}(\text{NH}_3)_2(\text{NO}_2)_4]\text{K}$, chrome III-diammonio-tetranitrite de potassium (or monopotassique), or in English or German, etc. (on account of the inverse order of the ions) $\text{K}[\text{Cr}(\text{NH}_3)_2(\text{NO}_2)_4]$, potassium chromium-III-diammonio-tetranitrite, or kalium-chrom-III-diammonio-tetranitrite. For slightly stable compounds an appropriate adjective could be added to the name of the salt (e.g. Cl_2Zn , $2\text{C}_5\text{H}_5\text{N}$, *clorure de zinc dipyridiné*; SO_4Cu , 4NH_3 , *sulfate de cuivre-II-tetrammonié*).

INORGANIC SULPHUR COMPOUNDS.—There is unanimous objection to the term *hyposulphurous acid* for $\text{H}_2\text{S}_2\text{O}_3$, and *thiosulphuric acid* is proposed, so that $\text{H}_2\text{S}_2\text{O}_4$, long called *hydrosulphuric acid*, would become *hyposulphurous acid*. Ludwig proposes the use of the prefix "thio" in a general way for partial replacement of oxygen by sulphur, and "sulpho" for complete substitution. The method of the Chemical Society and of the American Chemical Society of designating the group SO_3H as "sulpho" is criticised. In France doubly linked sulphur is called "sulpho," and singly linked sulphur "thio." This would be suitable for derivatives of phosphoric acid, arsenic acid, etc. If *thio* is reserved for the replacement of oxygen by sulphur, the difference between $\text{CH}_3\text{.CS.OH}$ and $\text{CH}_3\text{.CO.SH}$ cannot be indicated. "Thion" and "thiol" have been proposed at Geneva to meet this

difficulty, so that $\text{S:P} \begin{cases} \text{SH} \\ \text{SH} \\ \text{OH} \end{cases}$ would be thiondithiolphosphoric acid. In this case

"sulpho" should be used for sulphonated acids. It is recognised as essential that agreement should be reached over the names of the oxygenated acids. Del Campo proposes names based on the actual names of the chloric and nitric series, e.g.



BASIC COMPLEX SALTS.—In basic salts, such as $\text{PbCl}_2\text{.PbO}(\text{NO}_3)_2$, etc., the word *basic* might be replaced by *oxy* or *oxide*, to make the denomination of oxygenated salts conform to those of the halogen series (e.g. mercuric sulphate and oxide, $\text{HgSO}_4\text{.2HgO}$, as in potassium and aluminium sulphate). To specify the formula the notation of the complexes could be generalised (e.g. mercuric dioxide sulphate or mercuric sulphato dioxide, $\left. \begin{matrix} \text{O}_2 \\ \text{SO}_4 \end{matrix} \right\} \text{Hg}_3$).

II. REPORT OF THE OCTOBER MEETING OF THE COMMISSION FOR THE REFORM OF NOMENCLATURE AT PARIS.*

The foregoing report was taken as a basis of discussion, and a preliminary report of a German Commission (*Zeitsch. angew. Chem.*, 1925, 38, 713) was also considered.

I. CLASSIFICATION FOR TABLES AND INDEXES.—The method suggested by Prof. Delépine was adopted (with a slight difference in the German system).

* *Chem. Weekblad*, 1926, 23, 96-99.

In order to settle the symbols for the elements Ar-A; Xe-X, Em-Rn, I-J, Cp-Lu, Tu-Tm, Ct-Hf, Gl-Be, Cb-Nb, a committee (with power to take any necessary evidence and to decide the symbol to be adopted) was appointed, consisting of Messrs. Greenaway, Crane, Urbain, Fichter, Jorissen, and Parravano.

II. VALENCY.—Roman figures were adopted, placed between two hyphens for interpolation in words, or one hyphen at the end (*e.g.* eisen-III-chlorid, chlorure d'or-III. For alloys the proportion of atoms of each constituent should be indicated as, *e.g.* bismuth-2-magnesium-3, or more simply by their formulae.

III. WRITING OF FORMULAE.—The proposals were adopted. Fluorine is added to the Dutch list of polarity, being placed between Cl and O.

IV. OXYGENATED ACIDS.—The proposed list was, in the main, adopted. The suggestions concerning *ortho*, *meta* and *pyro* were suspended pending further information as to the constitution of the isopolyacids. Anhydrides and sulphanhidrides can be designated as such or by the proportions of their constituents.

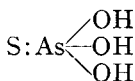
V. SALTS.—The terminations *-ite*, *-ate* and *-ure* (or their equivalents in other languages) were adopted. The suggestions for acid salts were accepted, except that *hydro* is to be used for *hydrogen* in English. It is best to refer to super acid salts by their formulae.

VI. WORDS DENOTING ACIDITY AND BASICITY.—In addition to the recommendations of Prof. Delépine's report, it is suggested that a word at once adjectival and substantival is useful for acids and bases. The adjectives *mono*, *di*, etc., are accepted in the sense of the report, and Messrs. Patterson and Greenaway would, in addition, add *hydric*.

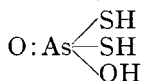
VII. WATER OF COMBINATION.—The term *hydroxide* should be adopted for combinations with metals such as HOK, (HO)₂Ba, and *hydrate* for such combinations as Cl₂ + *n*H₂O, where the water is not so intimately combined.

VIII. COMPLEX COMBINATIONS.—The principle in the suggested order of radicles and molecules was agreed to, as was also the proposal for designating valencies. *Hydrine* was regarded as preferable to *aque*.

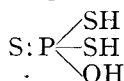
IX. SULPHUR COMPOUNDS.—The Commission for Organic Nomenclature decided to designate singly linked sulphur as *thiol*, and doubly linked as *thiono*, and the same principle has been adopted for inorganic compounds. If all the oxygen atoms are replaced by sulphur the prefix *thiol* is used, *e.g.*



Acide thiono-arsenique.



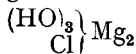
Acide dithiol-arsenique.



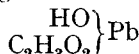
Acide thiono-dithiol-phosphorique.

The terms *thiosulphuric* and *thiosulphate* should replace *hyposulphurous* and *hyposulphite* for S₂O₃H₂ and S₂O₃M₂, and the attention of manufacturers should be drawn to the arrangement.

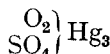
X. BASIC SALTS AND ACID COMPLEXES.—The proposed terms for basic salts are regarded as convenient, *e.g.*



Trihydroxy-chlorure
de magnésium.



Hydroxy-acétate
de plomb.



Dioxy-sulfate de
mercure.

Further light is required on the composition of acid complexes before their nomenclature can be decided.

D. G. H.

The Biological Testing of Therapeutic Substances.*

THE Pharmacological Laboratories of the Pharmaceutical Society have been established to provide facilities for manufacturers and others to have drugs (other than sera and bacteriological preparations) tested by biological means. The methods of testing and the standards are those laid down in the unanimous recommendations in the recent report of the Second International Conference on the Biological Standardisation of Certain Remedies, convened by the Health Committee of the League of Nations at Geneva on August 31, 1925.

(1) **AQUEOUS EXTRACT OF THE POSTERIOR LOBE OF THE PITUITARY GLAND.**—The standard to be adopted will be the dried substance of the posterior lobe prepared as described in U.S.P. X. (p. 20, *Standard Powdered Pituitary*). The test will consist in comparing the stimulant action of the unknown sample with that of the standard on the isolated muscle of the uterus of the virgin guinea-pig. The strength of the sample will be stated in units, 1 unit being defined as the amount of activity in 0.5 mgrm. of the standard powder. A test for pressor activity will be carried out if desired. A test to detect histamine will be made in each case.

There is at present no pharmacopoeial guidance in this country on the strength of the extract that should be prepared. The U.S.P. X. requirement is that 1 c.c. of extract should contain the activity of 5 mgrms. of the standard powder, or 10 units. This is a weaker extract than some now commonly sold in this country.

The Geneva Conference recommended the prescription of a hydrogen ion concentration for the extract between the limits represented by P_{H4} and P_{H5} , and that the sterile extract should be sealed in ampoules of hard glass.

Certificates for approved products will be issued by the Society.

(2) **DIGITALIS, STROPHANTHUS, AND SQUILL.**—The Geneva Conference recommended as a standard for digitalis a sample of dried leaves to be prepared in Holland under the supervision of Prof. R. Magnus of Utrecht. This standard is not yet available, but a sample of a strength closely approximating to that which will finally be adopted as the standard has been placed at the disposal of the Laboratories of the Department of Biological Standards, Medical Research Council, and will be used as the standard for the present.

The method to be adopted will be Magnus's modification of Hatcher's method on the cat, as described in the report of the Geneva Conference. The investigations mentioned (not yet published) show that in this country, where only the small *Rana temporaria* is available, the frog test is much less accurate (unless large numbers of frogs are used) than in some other countries.

A sample of leaves sent to be tested will be considered satisfactory if it has not less than 75 per cent., and not more than 125 per cent. of the activity of the sample to be used as standard, when tested by the cat method. Tinctures will be tested in comparison with tincture prepared from the standard sample; permissible deviation from the standard sample ± 25 per cent.

Strophanthus.—Tinctures will be tested by the cat method, a sample of ouabain being used as the standard of reference, as recommended by the Conference.

Squill will be tested by the cat method in the form of tinctures.

Certificates confined to the words "Tested physiologically and approved by the Pharmaceutical Society of Great Britain," and accompanied by an identification number, may be quoted on the labels of products issued for sale.

(3) **ERGOT.**—Samples of ergot will be tested in the form of powder. It will be assumed that the therapeutically active substance is the specific alkaloid ergotamine, and a test of its amount will be applied to the acid alcoholic extract of the powdered drug.

As standard a sample of ergotamine tartrate prepared by the Sandoz Chemical Works, Basle, is available.

The test applied will be the capacity of an acid alcoholic extract prepared from the sample to reverse the action of adrenaline either on the blood pressure of a pithed cat or on the isolated uterus of the rabbit. After consultation with the Director of the Department of Biological Standards, Medical Research Council, it has been decided to approve as good samples of ergot those containing not less than 1 mgrm. per grm. of the specific alkaloid.

* *Pharm. J.*, 1926, 116, 205. A pamphlet will be forwarded to enquirers by the Secretary of the Pharmaceutical Society, 17, Bloomsbury Square, London, W.C.1.

The present liquid extract of the B.P. is so made that the active alkaloid is not present in appreciable quantities. The Society, however, will permit the use of the following formula in the case of extracts of ergot made from samples of the powdered ergot which has passed the test: "Prepared from powdered ergot physiologically tested and approved by the Pharmaceutical Society of Great Britain." This statement must be accompanied by the batch number of the ergot from which the extract was prepared.

Milk and Dairies Order 1926

AND

Public Health (Imported Milk) Regulations 1926.

Drafts of the above documents entitled, respectively, "Draft, dated March 5th, 1926, of the Milk and Dairies Order, 1926, proposed to be made under the Milk and Dairies (Consolidation) Act, 1915," and "Draft dated March 5th, 1926, of the Public Health (Imported Milk) Regulations, 1926, proposed to be made by the Minister of Health," are being issued, and copies of the drafts may be purchased through any Bookseller, or directly from H.M. Stationery Office, Adastral House, Kingsway, London, W.C.2.

Any representations on the draft Order or Regulations should be sent to the Secretary to the Ministry of Health within a period of 40 days from this date.

MINISTRY OF HEALTH, WHITEHALL, S.W.1.
9th March, 1926.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Alkaline Milk and its Detection by the Brom-Cresol Purple Test. I. F. Procter and A. T. R. Mattick. (*J. Agric. Sci.*, 1926, 16, 145-148.)—In an attempt to explain unsatisfactory coagulation of milk by rennet in cheese-making, P_H determinations of freshly drawn milk have been made. Brom-cresol purple papers were used in the cowshed, standard solutions of various P_H values (6.5 to 7.13), containing this indicator, being employed as checks. Of the 3473 samples examined, 93 per cent. of the reactions were normal (P_H 6.5 to 6.75), 3.3 per cent. definitely abnormal (P_H 6.9 to 7.13), and the remainder were classified as doubtful. It is claimed that Sheather's objections to the use of brom-cresol purple (*J. Comp. Path.*, 1924, 4) do not hold when the samples are taken fresh from the cow.

J. G.

Fresh and Preserved Eggs. G. Filandeau. (*Ann. Falsificat.*, 1925, 18, 515-517.)—When boiled hard, fresh eggs are easy to shell and show very small air-spaces; the white is opalescent, homogeneous and elastic, and the yolk mostly well centred and rarely visible and never in contact with the shell. Eggs which have been preserved by means of lime or silicate usually crack at about 70° C., the crack being always lengthwise; the shell is difficult to remove, the air-space mostly reduced, the yolk visible and displaced towards the point, and the

white flabby and often separated into two or three layers. Eggs preserved by freezing remain upright, and sometimes float, in water; when boiled hard, they show an air-space often 20 to 25 per cent. of the total volume, an eccentric yolk, and a white of poor appearance and odour. Eggs preserved by the Lescardé process are, when cooked, difficult to shell, and if this is done carefully, the hyaline membrane is found to be detached from the shell, the chamber thus formed containing a drop of water; the white is of normal appearance, but forms concentric layers, and the yolk is displaced towards the point. When broken on a plate, the fresh egg gives a thick homogeneous white with a narrow, more liquid edge, whereas with preserved eggs two distinct layers of white are formed, the outer one being far less liquid than the one surrounding it.

T. H. P.

Avgotarachon. C. Pyriki. (*Zeitsch. Nahr. Genussm.*, 1925, 50, 366–371.)—

This fish-roe delicacy is prepared in Greece, Turkey and Egypt from the roe of fish of two species, the roe being salted, dried for three or four days in the sun, and immersed for a time, in the form of U-shaped rolls, in melted beeswax, to give it a protective coating. It is quite free from bacteria, and appears to be identical with the fish-roe cheese made in the Dardanelles district. Analysis of two different qualities of avgotarachon (I, II), fish-roe cheese (III), and granular (IV), and pressed caviar (V), gave the following mean percentage results:—

	I.	II.	III.	IV.	V.
Water	31·86	25·87	19·38	48·86	37·79
Protein	38·84	48·24	38·81	29·34	38·01
Fat	22·98	20·99	28·87	13·98	15·52
Nitrogen-free extractives	0	0	(6·33)	1·30	1·08
Ash	6·39	4·45	10·61	7·42	7·60
Sodium chloride ..	4·60	1·42	—	6·18	6·22

The constants of the brownish-yellow fat from the two qualities of avgotarachon were as follows:—Sp. gr. at 15° C, 0·9059, —; refractometer reading at 40° C., 56·5, 61·0; m.pt., 25·0, 24·8° C.; solidifying point, 23·8, 23·2° C.; iodine value, 83·0, 91·0; saponification value, 141·0, 137·0; Reichert-Meissl value, 1·75, —; Polenske value, 0·50, —; acid value, 50·0, —. The ash of the material consists in the main of soda, phosphoric acid, and chlorine. The calculated food-value is given.

T. H. P.

Identification of Creatine. R. J. Williams and P. A. Lasselle. (*J. Amer. Chem. Soc.*, 1926, 48, 536–537.)—In view of the conflicting descriptions of the physical properties of creatine, the authors have examined samples obtained from salmon and other sources. These were first identified by their chemical properties and analyses, and then shown to crystallise in thin monoclinic prisms, which on cursory examination might appear orthorhombic. To most individuals they are tasteless and produce a burning after-sensation in the back of the mouth, but to some they appear bitter. The samples examined decomposed with marked effervescence at temperatures ranging from 290° to 293° C. (corr.), according to purity.

J. G.

Effects of Fine Grinding upon Flour. C. L. Alsberg and E. P. Griffing. (*Cereal Chemistry*, 1925, 2, 325-344.)—When flour is ground too fine, the starch granules are injured, so that part of the starch swells and becomes dispersed when the flour is doughed. This is accompanied by increase in the cold water extract, in the diastatic conversion of the starch, and in the initial rate of fermentation. Pronounced over-grinding damages flour for baking purposes, despite increased absorption due to swelling of starch granules, and also injures the gluten.

T. H. P.

Composition of Preserved Peas in Relation to their Diameter. E. Lasausse. (*Ann. Falsif.*, 1926, 19, 28-40.)—Muttelet (*Ann. Falsif.*, 1925, 18, 5) proposed a chemical method of detecting the fraudulent substitution of old dry peas for the young green peas which are alone supposed to be used in the preparation of products labelled, e.g. "Petits pois très fins." He found that as the pea matures and increases in diameter the ratio of the cellulose, and also that of the nitrogenous substances, to the dry extract diminishes, whereas the ratio of the soluble hydrolysable substances to the dry extract increases, and the ratio of the hydrolysable insoluble substances to the dry extract diminishes (*cf.* ANALYST, 1926, 150). In the preparation of preserved peas a process of screening by hand or by machinery is used, and in judging of the value of a particular product the uniformity of the size of the peas is one of the criteria. As this judgment is largely subjective, the author has studied and extended Muttelet's observations on the relationship between the chemical composition and the diameter of peas of various origin. He shows that yellow peas of the same mean diameter as younger green peas contain different proportions of starch and cellulose, and will absorb different amounts of water when boiled (e.g. yellow peas, 285 per cent.; green peas, 35 per cent.). On the other hand, peas passing through a sieve of the same size may show analytical values close to those of peas passing through a sieve of another size. Analyses of fresh peas sorted into different sizes, and subjected to a usual method of preserving showed distinct chemical differences between the sizes, but the figures varied with the district of origin. Hence, chemical methods, including that devised by Muttelet, will give only an approximate idea of the original sizes of the peas, and a more direct method of determining this has still to be found.

D: G. H.

Examination of Cacao Butter for Alkali and Alkaline Earth. J. Prescher and R. Claus. (*Zeitsch. Nahr. Genussm.*, 1925, 50, 429-430.)—When steam is passed for 30 minutes into 30 grms. of melted cacao butter, the aqueous extract obtained yields no fatty acid when cleared, evaporated to a volume of 25 c.c., and acidified with dilute hydrochloric acid; thus the absence of alkali may be presumed. If the residual fat from the above treatment, mixed with 5 c.c. of hydrochloric acid, is again subjected to the action of steam, the aqueous extract obtained gives a precipitate with ammonia and ammonium carbonate or oxalate, lime being present to the extent of 0.01 gm. per 100 grms. of the fat. Whether

this lime is derived from the natural salts of the fat or from the water used for washing the material is not known. Animal fats, such as pork dripping, show at most a turbidity when tested as above.

T. H. P.

Sperm Oil and Whale Oil, and Spermaceti. E. André and T. François. (*Compt. Rend.*, 1926, 182, 497-499.)—Samples of sperm oil, whale "lard," and oil from the muscular tissues of the whale have been analysed, the last-named for the first time. The amounts of glycerin determined by the acetin method in the three saponified oils were 8.1, 1.3 and 5.5 per cent. respectively. The corresponding quantities of alcohols of high molecular weight were 36.4, 40.0 and 17.5 per cent. The oil from the muscular tissues thus contains more than half its weight of glycerides, the remainder consisting of esters of the higher alcohols. Samples of spermaceti similarly treated were found to contain 0.7 per cent. of glycerin. It is concluded that the products of the sperm whale are midway between true oils and liquid waxes.

J. G.

Tannins for Use in Wine Making. F. Levallois. (*Ann. Falsif.*, 1926, 19, 15-28.)—Since tannins for wine making are not subject to any regulations in France, they are liable to considerable adulteration, and it is suggested that definitions of the various types, together with some analytical limits, should be enforced, particularly with regard to the content of tannic acid. The various methods for the determination of tannin have been critically reviewed, and many have been found only applicable to nearly pure samples. Two methods for commercial tannins depending on the extraction of the tannin from concentrated extracts by means of ethyl acetate or acetone have proved useful in many cases when the results have been considered in conjunction with other data. Extraction should be at the boiling point of the solvent, and the weight of solvent taken should be five or six times that of the extract, and at least three extractions should be made. The results are, in general, on the high side, owing to a tendency for the solvent to form very stable molecular complexes with the tannin, but ethyl acetate does not dissolve more than small proportions of the tannin of chestnut or valonia. The official method of the International Leather Trades Chemists still appears the most satisfactory.

D. G. H.

Examination of Commercial Tannic Acid. W. B. Forbes. (*Pharm. J.*, 1926, 116, 225-229.)—The British Pharmacopoeia and the British Pharmaceutical Codex describe *Acidum Tannicum*. B.P., as a pure substance having the formula of Schiff's digallic acid, but the author's work shows that the commercial drug contains gallotannin, gallic acid and water. A comparative study of various methods has been made. Precipitation by tartar emetic (Richards and Palmer) gave results much too low, the sodium tungstate method gave very irregular results, and Dreaper's copper sulphate method gave results about 10 per cent. too low, whilst Löwenthal's permanganate method, with different and untrustworthy factors by different workers, was found to be, at best, only a comparative value. The most trustworthy results were those obtained by Hooper's modification of Chapman's

cinchonine method (ANALYST, 1925, 50, 162) and Crouzel's phenazone (antipyrin) method (*Chem. Zeit. Rep.*, 1902, 174), with which it was found to be best to add the tannic acid solution to an excess of phenazone, to filter off the precipitate in a Jena glass filter, and to dry the precipitate at 100° C., half of the weight being due to the tannic acid.

Ware's method of precipitation with ferric ammonium citrate (ANALYST, 1924, 49, 467) was made quantitative by collecting the precipitate in an alundum crucible, washing it with water, drying and igniting it, and weighing the ferric oxide. The corresponding amount of tannic acid was then found by the use of a factor obtained by the results of determinations of tannins by the methods of Chapman and Crouzel. The results thus obtained were fairly constant.

In the following table of results given by four typical samples of tannic acid, B.P., the gallic acid was determined by Dreaper's copper method (*Chem. News*, 1904, 90, 111), and the water by drying over sulphuric acid *in vacuo*.

Sample.	Tannic Acid.			Gallic acid. Per Cent.	Water. Per Cent.	Total. Per Cent.
	Cinchonine method.	Crouzel's method.	Mean.			
	Per Cent.	Per Cent.	Per Cent.			
A	85.9	85.03	85.46	6.9	8.0	100.36
B	83.4	83.3	83.35	6.4	8.8	98.58
C	81.7	83.6	83.65	6.4	9.38	98.43
D	85.2	85.26	85.23	6.9	8.2	100.33

The Codex states that "many commercial samples (of tannic acid) contain gallic acid," and "are used in dyeing, in the manufacture of ink, etc., and are not suitable for medicinal use." The percentage of gallic acid which makes a sample of tannic acid unsuitable for medicinal use is not stated, but it would appear that none of the four samples of tannic acid, B.P., examined was suitable for medicinal use on this basis.

Lead and Arsenic in Tartar Emetic. E. Griffiths-Jones. (*Lancet*, 1926, 210, 194.)—Last year in Egypt 54,316 patients were treated for bilharzia, the total number of injections of 6 per cent. tartar emetic solution being 311,656, so that each patient received in the whole treatment about 1.5 grms. of tartar emetic. As it was found that severe symptoms occasionally resulted, search was made for possible impurities in the drug, and several samples were found to contain lead or arsenic or both. According to the B.P., tartar emetic "yields no characteristic reactions for lead, copper, arsenic, iron, sodium, ammonium, chlorides, or sulphates." This paragraph was quoted in their favour by both British and German manufacturers, who admitted the presence of lead (0.09 to 0.12 per cent.) in their products, but claimed that if tartar emetic were tested for lead by the methods given on p. 492 of the B.P., none would be found. This contention is extraordinary, in view of the obvious fact that the antimony would interfere with these tests.

With the colorimetric test given in *Squire's Companion to the British Pharmacopoeia*, 20 parts per million of lead will give an appreciable coloration. To make certain that this coloration was due to lead, and not to any other impurity, the following gravimetric process has been devised:—Twenty grms. of the tartar emetic are dissolved in the minimum quantity of water, and treated with sodium hydroxide solution, until the white precipitate first formed re-dissolves, after which a further excess of sodium hydroxide is added. Hydrogen sulphide is then passed through the liquid for several hours, the beaker allowed to stand for several hours, and the black precipitate filtered off through a Gooch crucible, washed with ammonium sulphide and then with water. The precipitate is next dissolved in boiling dilute nitric acid, and the solution treated with sulphuric acid, boiled until fumes appear, cooled, diluted and allowed to stand, and the precipitate of lead sulphate collected on a Gooch crucible, washed with 10 per cent. sulphuric acid, and then with water, dried and weighed. One sample, found to contain 200 parts of lead per million by the colorimetric method, yielded 198.7 parts by this gravimetric method. Another sample showed about 1200 parts per million colorimetrically, and 1266 parts gravimetrically.

For the detection of arsenic 0.5 grm. of the tartar emetic is dissolved in 5 c.c. of water, the solution treated with 17 c.c. of strong hydrochloric acid and distilled after the addition of 1 or 2 drops of a strong solution of stannous chloride, until only a few c.c. remain in the flask. An adapter from the condenser dips into a small amount of water to absorb the hydrogen chloride gas which first passes over. The distillate is redistilled, the last few c.c. being rejected, and the second distillate is used for testing for arsenic by the Gutzeit process. The limit adopted for arsenic is 10 parts per million as determined by this process.

Determination of the Strength of Liquid Hydrocyanic Acid by Specific Gravity. M. Walker and C. J. Martin. (*Ind. Eng. Chem.*, 1926, 18, 139–142.)—Tables are given showing the specific gravities of aqueous solutions containing from 80 to 100 per cent. of pure hydrocyanic acid and at temperatures varying from 0° C. to 25° C. W. P. S.

Alkaloid Content of Belladonna Root. J. J. Blackie. (*Pharm. J.*, 1926, 116, 231).—Experiments were carried out to ascertain the content in alkaloid of the first, second, third, and fourth year roots. The method of determination used was that of the U.S. Pharmacopoeia:—Ten grms. of the powdered root were covered with a mixture of 3 parts of ether and 1 part of chloroform. The menstruum was thoroughly mixed with 5 c.c. of ammonia, percolation was carried out till the percolate gave no precipitate with Mayer's reagent. The percolate was extracted with dilute acid, and the extract made alkaline with ammonia and extracted with chloroform, the solvent removed by evaporation and the alkaloids titrated with *N*/50 sodium hydroxide. The results were as follows:—1st year, 0.72; 2nd year, 0.65; 3rd year, 0.66; and 4th year, 0.60 per cent. The 3rd year roots are the most profitable to collect, as they are the largest in size. Roots of plants grown from seed from U.S.A., collected after four years, showed an

alkaloid content of 0.45 per cent., the acid-insoluble ash being 1.5 per cent. The figure for ash is well below that required by the U.S. Pharmacopoeia, which specifies not more than 4 per cent. It is recommended that the British Pharmacopoeia should include standards for acid-insoluble ash and alkaloid, and also for dimensions of belladonna root.

R. F. I.

Characteristic Reactions of Luminal. F. Ranwez. (*J. Pharm. Belg.*, 1924, 6, 501-505; *Chem. Abstr.*, 1925, 19, 3561.)—The nitroluminal is reduced by adding to the mixture granulated zinc and dilute sulphuric acid, and when solution is complete the clear liquid is decanted, cooled to 5° C., and treated with an aqueous solution of 0.1 grm. of potassium nitrite. Diazoluminal is produced, and at this dilution remains in solution. It may be identified by the following colour reactions:—(1) On the addition of a few particles of β -naphthol an intense, blood-red coloration is produced. (2) The addition of α -naphthol followed by sodium hydroxide to alkalinity gives an intense orange-red colour. (3) Sodium β -naphthol-3,6-disulphonate gives a cherry-red colour. (4) Phenol gives a deep brown colour. (5) Resorcinol gives an intense orange-brown colour. (6) Phloroglucinol gives a brownish red colour. (7) Guaiacol, a yellow-brown colour. (8) Thymol, a brown-yellow colour. (9) Eugenol, a bright cherry-red colour. Pyrogallol and gallic acid do not produce characteristic colorations.

Biochemical, Bacteriological, etc.

Odour-Intensity and Odour-Quality. J. Missenden. (*Perfum. and Ess. Oil Record*, 1926, 17, 62-64.)—The lipin theory of odour is that all substances capable of being detected by the olfactory sense must be volatile, and must also be soluble in lipin (the fatty substance in the nasal membranes), and that the quality of the odour is determined by the degree of chemical reaction between the substance and lipin. There is also justification for concluding that the *intensity* of odour is determined by the quantity of a substance which is enabled to react chemically with lipin, and that the *quality* of odour is determined by the force of molecular exchange. If both force of molecular interchange and quantity of substance are great, the effect is likely to be overpowering, as in the case of strong ammonia. Lower aldehydes (*e.g.* propionic aldehyde) with less than 5 carbon atoms in their molecule have an acrid odour, medium aldehydes (*e.g.* citral) with 10 carbon atoms have an agreeable odour, and higher aldehydes with more than 10 carbon atoms have no odour. Actual experiments with lipin have shown that with the first class of aldehyde the interchange is rapid; with the second, slow, whilst with the third, little or no interchange takes place. It is probable that with substances which smell somewhat alike differences in molecular valency regulate the rate of exchange. Thus benzaldehyde ($C_6H_5.COH$), nitrobenzene ($C_6H_5.NO_2$), benzonitrile ($C_6H_5.CN$), and phenyl azoimide ($C_6H_5.N_3$), all smell very much alike. This may be attributed to the action of the governing benzene radical upon lipin, the slight variations being caused by subordinate reactions due to the respective

aldehyde, nitro, nitrile, and azoimide groups. The effect of the combined reactions is communicated as one co-ordinated impulse to the olfactory nerve-endings. An experimental comparison between the odours of citronellal and menthol goes far to support the lipin theory. By depriving menthol of two atoms of hydrogen it is converted into menthone. When menthol vapour is taken into the nasal passages it is absorbed by the lipin, menthone being produced and the lipin itself hydrogenated. The partial paralysis of the olfactory sense on smelling menthol for some time is to be attributed to the hydrogenation of the lipin, whereby a thick, heavy, non-sensitive fat is formed. The inhalation of ether quickly reduces the paralysis; methylated spirit has a similar but slower effect. Citronellal, however, has no spare hydrogen in the ring, a ketone group taking the place of an aldehyde group; therefore its smell is not "biting." The differences between pleasant and unpleasant or peculiar smells are satisfactorily accounted for by the obvious differences in the univalent groups beneath the rings. A convenient classification of odours seems possible, provided that the rate of chemical action on lipin is studied, and also provided that a standard lipin can be obtained synthetically.

Quantitative Determination of Tyrosine and Histidine in Protein. A Method for Determining Tyramine in Protein-containing Mixtures. M. T. Hanke. (*J. Biol. Chem.*, 1925, 66, 475-488.)—When tyrosine is boiled with mercuric acetate under suitable conditions, and the resulting clear solution treated with sodium chloride, a white solid is obtained that has the formula $C_9H_9Hg_2Cl_2O_3N$, and is very insoluble in water and in a mixture of amino acids. It can thus serve for the separation of tyrosine from other amino acids formed by the acid hydrolysis of a protein. It can be dissolved in dilute sulphuric acid or in 20 per cent. hydrochloric acid. Hydrogen sulphide passed into its suspension in water, or its solution in acids removes the mercury quantitatively, leaving the original tyrosine, which can be determined according to the method of Hanke and Koessler (*J. Biol. Chem.*, 1922, 50, 235, 271). The amino acids precipitated with the tyrosine from protein hydrolysates do not interfere with the colorimetric determination of tyrosine, with the exception of histidine. The complete analytical process, which is described in detail, is, briefly, as follows:—The protein is hydrolysed with sulphuric acid. Histidine is precipitated as the silver compound, and its amount determined colorimetrically. The filtrate from the histidine silver is boiled with mercuric acetate and treated with sodium chloride, which precipitates tyrosino-mercuric chloride. The precipitate is dissolved in 20 per cent. hydrochloric acid, freed from mercury with hydrogen sulphide, and the tyrosine determined in the filtrate from the mercuric sulphide. A method for determining tyramine in protein-containing matter, with the use of a mercury compound of tyramine formed under identical conditions, is briefly outlined. P. H. P.

Histidine and Tyrosine Content of a Number of Proteins. M. T. Hanke. (*J. Biol. Chem.*, 1925, 66, 489-493.)—By means of the method described in the preceding abstract the histidine and tyrosine contents of a number of proteins

have been determined. The mean values obtained are as follows: (The first figure is the per cent. of histidine and the second the per cent. of tyrosine):—Gelatin, 0.53, 0.25; casein, 2.61, 4.5; crystalline egg albumin, 2.3, 2.35; squash seed globulin, 2.26, 3.05; gliadin, 2.1, 2.35; hordein, 0.98, 2.43; zein, 1.25, 3.66; secalin, 1.23, 1.37; sativin, 0.74, 1.56; sorghumin, 0.51, 2.3; fibrin (sheep), 2.18, 3.3; fibrin (swine), 2.27, 3.45; fibrin (cattle), 2.05, 3.5. Other methods for the determination of these compounds are discussed, and a table shows that the tyrosine figures obtained by the new method are in fairly close agreement with those obtained by the gravimetric method. For casein, squash seed globulin, gliadin and zein, they are identical, but for gelatin, egg albumin, hordein and secalin, the values are slightly higher than those obtained gravimetrically, which was to be expected. Most colorimetric processes give higher results than gravimetric processes.

P. H. P.

An Alcohol-Soluble Protein isolated from Polished Rice. W. F. Hoffman. (*J. Biol. Chem.*, 1925, 66, 501–504.)—The alcohol-soluble protein of rice, obtained in small quantities by Osborne, Van Slyke, Leavenworth and Vinograd, (*J. Biol. Chem.*, 1915, 22, 259), has been prepared in a sufficient quantity for analysis, in order to determine whether or not it possesses the physical and chemical characteristics of the prolamines. From 6.5 kilos of commercial polished rice the yield of protein was 7.5 grms. of dried product, or 0.12 per cent. of the polished rice. An elementary analysis showed it to contain less nitrogen and more carbon than is found in wheat prolamine, but not to differ essentially from the prolamines of maize, kafir corn, etc. An analysis (by the Van Slyke method) of nitrogen distribution, the results of which are recorded, showed that it differs from wheat gliadin and rice glutenin. It does not contain the high percentage of ammoniacal nitrogen which is typical of the prolamines; it contains more basic nitrogen than any prolamine heretofore isolated, but only a small amount of the basic nitrogen is lysine. The arginine content is much higher than that of any known prolamine, but is lower than the values reported for the associated protein, oryzenin. Thus rice contains a trace of a protein soluble in hot alcohol which resembles the prolamines of kafir corn and sorghum in solubility, but does not contain the large amount of amide nitrogen which has been considered characteristic of prolamines.

P. H. P.

Colorimetric Determination of Phosphorus. C. H. Fiske and Y. Subbarow. (*J. Biol. Chem.*, 1925, 66, 375–400.)—The disadvantages of the method of Bell and Doisy (*J. Biol. Chem.*, 1920, 44, 55), and of the modifications of this method by Briggs (*J. Biol. Chem.*, 1922, 53, 13; 1924, 59, 255) for the determination of phosphorus in blood and urine are discussed in detail. A table shows how the development of colour by hydroquinone is affected by various interfering substances. The authors have now placed the colorimetric method upon a sounder basis, and it may be applied with safety to the analysis of any sort of biological material if the precautions stated are taken. They use, in place of the reducing agent hydroquinone, 1, 2, 4-aminonaphtholsulphonic acid. This

gives the maximum amount of colour in about 5 minutes, and accurate results in the presence of at least ten times the amount of inhibiting material that would be permissible with hydroquinone. The isomer 1, 2, 6-aminonaphtholsulphonic acid acted equally well, but it is not so easily obtained as the other compound. Details are given for the preparation of the necessary solutions and for the determination. The applications of the method are also discussed. A special problem which was always difficult to handle by the older phosphate methods is the determination of traces of phosphorus in the presence of a large excess of carbonaceous matter.

P. H. P.

Presence in various Fungi of an unknown Oxidase. J. Wolff. (*Compt. Rend.*, 1926, **182**, 343-344.)—The property shown by glycerin extracts of various fungi of enabling atmospheric oxygen to oxidise ferrous salts obtained from wines (*cf.* ANALYST, 1925, **50**, 77), and hitherto attributed to laccase (*Compt. Rend.*, 1925, **181**, 939), has been found to be due to a new oxidase (ferrase). By warming an extract poor in laccase to 80° C. for 5 minutes the other characteristic properties of the laccase were completely destroyed, but the oxidising power was reduced only by about one-half. It is concluded that, like laccase and tyrosinase, ferrase should occur principally in the vegetable kingdom.

J. G.

Vitamin Studies. XI. Inorganic Blood Phosphorus and Bone Ash in Rats fed on Normal, Rachitic and Irradiated Rachitic Diets. R. A. Dutcher, M. Creighton and H. A. Rothrock. (*J. Biol. Chem.*, 1925, **66**, 401-407.)—Experiments were carried out on rats to determine the composition of blood and bone at different ages. The bones were extracted according to Steenbock's method, and the results of the analyses are shown in charts. The inorganic blood phosphorus in normal rats, weighing between 35 and 40 grms., was 10 mgrms. per 100 c.c. of serum, and during an 8-week feeding period, tended to stay at this level, falling slowly to 8 mgrms. per 100 c.c. of serum at the end of that time. During the 8 weeks the percentage of ash in the dry extracted femurs of these rats increased steadily from 40 to 62 per cent. When rats received Steenbock's rachitic ration, inorganic blood phosphorus dropped from 10 mgrms. to 1.6 mgrms. in a period of 3 weeks, after which it rose slightly, due to fasting. The ash of the dry extracted femurs of the rachitic rats fell from 40 to 26.5 per cent. in 3 weeks, and finally to 24 per cent. When Steenbock's rachitic ration was irradiated with ultra-violet light the inorganic blood phosphorus and the percentage of bone ash fell midway between the normal and rachitic groups. Apparently the rat cannot store the antirachitic factor in significant amounts, for the reason that calcium and phosphorus deposition is hampered from the beginning of the experimental feeding period. The method outlined offers an opportunity of making a quantitative study of the antirachitic properties of food materials with the use as a standard of the least amount of any food which will prevent a fall in the percentage of bone ash, below that possessed by the young normal animals at the beginning of the experiment, or the minimum quantity of food required to bring about the optimum deposition of mineral matter.

P. H. P.

Relation between the amount of Ultra-Violet Light received by Hens and the amount of Antirachitic Vitamin in the Eggs produced. J. S. Hughes, L. F. Payne, R. W. Titus and J. M. Moore. (*J. Biol. Chem.*, 1925, 66, 595-600.)—Eggs from four pens of hens receiving various amounts of ultra-violet light were tested by feeding growing chicks with the eggs. Pen I received direct sunshine +30 minutes of ultra-violet light per day from the mercury arc lamp; Pen II received sunshine filtered through glass +30 minutes of light per day from the mercury arc lamp; Pen III received direct sunshine; and Pen IV received only glass-filtered sunshine. Each lot of chicks received a basal ration and one egg a day from one of the pens. The inorganic blood phosphorus and bone ash content of the chicks was determined. Eggs from Pens I and III had the highest antirachitic vitamin content, those from Pen IV the lowest. Whether those from Pen I were richer in the vitamin than those from Pen III is not known. Results show that the amount of ultra-violet irradiation which a hen receives is an important factor in determining the antirachitic vitamin content of the eggs which she produces when her feed is low in the antirachitic vitamin. A lower percentage of eggs with a low antirachitic vitamin content is hatched than of eggs having a high antirachitic vitamin content, other influencing factors being constant. Chicks a day old hatched from the eggs low in the vitamin contained less calcium in their bodies than day-old chicks hatched from eggs high in the vitamin. Thus, disturbances in the mineral metabolism may begin before the chick is hatched.

P. H. P.

Vitamins in Canned Foods. IV. Green Peas. W. H. Eddy, E. F. Kohman and V. Carlsson. (*Ind. Eng. Chem.*, 1926, 18, 85-89.)—Experiments on vitamins A and B were conducted on the principle of withholding the vitamin in question until a definite decline in weight was shown by the animal, and then feeding it with the peas. The increase in weight was then compared with that obtained with a standard. Canned peas were found to be of at least half the value of butter as a source of vitamin A, but the value appears to decrease as the peas mature. Peas are richer than either milk or tomato juice in vitamin B, the richness increasing with the maturing of the peas and not being appreciably affected by "processing," although the duration and method of "blanching" is a factor. Peas rank as one of the most valuable antiscorbutics, being very rich in vitamin C, but no evidence of the presence of vitamin D, the antirachitic factor, could be found.

D. G. H.

Effect of Vitamin Potency of Cold-pressed Cod-Liver Oils. A. D. Holmes and M. G. Pigott. (*Ind. Eng. Chem.*, 1926, 18, 188-189.)—Six typical crude cod-liver oils were pressed under commercial conditions by refrigeration in a brine-cooled tank, and the solidified stearine was separated by filtration. Analyses of the crude and pressed oils showed that there was but little difference between the chemical and physical characters of the two classes of oils, and biological tests did not indicate any significant difference in vitamin potency.

W. P. S.

Determination of Calcium in Tissues, Faeces and Milk. R. C. Corley and W. Denis. (*J. Biol. Chem.*, 1925, **66**, 601-608.)—Determinations of the inorganic constituents of tissues are almost invariably carried out on the ash, and ashing is laborious time-consuming and expensive. The authors use autoclave digestion as a substitute for ashing and report results obtained by its application in the determination of calcium in tissues, faeces and milk. They have also used it for the determination of calcium in the bone. The method, which is described in detail, consists in treatment with sodium hydroxide in an autoclave, followed by a double precipitation with ammonium oxalate, with intermediate oxidation by means of potassium permanganate. The permanganate destroys any traces of organic matter that may have been carried down with the oxalate. There must be a rough parallelism between the amount of protein present and the amount of sodium hydroxide used, otherwise the large excess of alkali at the high temperature used attacks the glass, with the formation of silicates that precipitate as silicic acids in acid solution. For this reason 0.01 *N* sodium hydroxide is used for the autoclave digestion of milk or faeces in place of 0.1 *N* sodium hydroxide which is used for the digestion of tissues or bone. Tables are given of the results obtained, and the results from the ashing and autoclave methods are compared.

P. H. P.

Relation between the Bacterial Count of whole Milk and that of the Cream and Skim Milk separated from it. C. S. Leete. (*J. Agric. Res.*, 1925, **31**, 695-699.)—If a clean separator is used, cream obtained by centrifugal separation will not have a greatly higher bacterial count than the original whole milk, but the reverse is the case with cream separated by gravity. The latter method, however, is not used to any great extent for making commercial cream, and high bacterial counts in such cream may be attributed to the poor quality of the milk used and to inefficient sterilisation of apparatus.

T. H. P.

Toxicological and Forensic.

A Chemical Test for Alcoholic Intoxication. H. W. Southgate. (*Medico-Legal Society*, Jan. 1926; *Lancet*, 1926, **210**, 207-209.)—Four years' investigation on human subjects has shown that the concentration of alcohol in the blood is proportional to the toxic effect produced, that there is a close relationship between the concentration of alcohol in the blood and in the urine, and that one can be deduced from the other. In the tests described the blood samples were taken from a vein, and the alcohol was determined by distillation and oxidation with dichromate and expressed as mgrms. per 100 grms. of blood. The concentration curve of blood and alcohol rose very rapidly (in about 1 hour) to its maximum, and slowly came down, taking about 12 hours to return to normal, which was probably zero. The rate of disappearance was practically a straight line, and this held good for a wide range of concentration and for all the persons tested. The glucose curve rose with equal rapidity, but fell within about 1½ hours to the normal of about 80 mgrms. per 100 grms. The kidneys could keep back glucose

until it reached quite a high percentage in the blood, but had not this power for alcohol, and even half a glass of beer caused alcohol excretion. The actual maximum concentration varied with the dose. When alcohol was taken with food the concentration rose more slowly and never reached such a high maximum as when the dose was taken on an empty stomach. Some of the alcohol was held between the interstices of the food, but not enough to account for the difference, and what became of it was not known. The descending curves of concentration with and without food were parallel when the subject took exercise, as well as when he rested. The tolerance of individuals varied very much according to their habits. For example, it was shown by Schweisheimer in 1913 that an abstainer showed a high concentration, and too heavy drinkers relatively low ones. Yet the factor of personal idiosyncrasy was very great, and it was impossible to be certain from the percentage in the urine how much alcohol had been taken. Gréhant was the first to show that the concentration of alcohol in the blood was the measure of its toxic effect. When the body's maximum was reached the curve turned over and ran flat for a while (Gréhant's plateau). The subject was then dead drunk, and the alcohol escaped by any pore it could find. There was no practical difference between the toxicity of a rising and a falling concentration measured on the same level. Comparison of the relative effects of whiskey and stout, containing the same amount of alcohol, showed that with whiskey the concentration reached a higher maximum much more quickly than with stout, had a greater effect on the subject, and passed off more quickly. This was because the stout took much longer to drink. Toxicity was measured by the subjects' ability to draw a square, with its diagonals, inside a circle.

Miles has shown that the concentration of alcohol in the urine passes that of the blood almost at once, and maintains a fairly constant ratio towards it (1.35 to 1.45), whatever food is taken and whatever urine is passed. This point is important, as the evidential value of the test depends upon it. A motorist who was arrested for drunkenness had 257 mgrms. of alcohol per 100 grms. in his urine 15 minutes after the arrest, but 40 minutes later was released after being found sober by the police surgeon. A sample of urine taken some time after arrest would naturally not show the same concentration as at the time of arrest. To find this, the test might be standardised, another sample of urine being taken at a measured time afterwards, and the concentration at the time of arrest plotted on the resulting curve, the time between arrest and the taking of the first sample being known. Any standard devised should be based on behaviour tests made with individuals of varying tolerance, each of whom had a dose that would make his concentration the same as that of the others. This test would show what was the average concentration beyond which a person was not fit to be in charge of a car.

Poisoning by Lead Tetra-Ethyl. C. Norris and A. O. Gettler. (*J. Amer. Med. Assoc.*, 1925, 85, 818-820; *Chem. Abstr.*, 1925, 19, 3544.)—A volatile lead compound was isolated from the brain tissue in two of the four cases studied, the amount found being 4.25 and 2.27 mgrms. In the two cases in which the

volatile compound could not be isolated the patients had lived 27 and 49 hours, respectively, after exposure to the lead tetra-ethyl, which had apparently in the interval been hydrolysed into a non-volatile form. No volatile lead compound could be isolated from any other tissues. Contrary to the usual findings in lead poisoning, the bones in these cases did not contain as much lead as the brain and liver. The large amounts in the brain suggest a special attraction by brain tissue for lead tetraethyl. Absorption of the poison through the lungs was indicated by the large amount of lead present in them. The blood and all the organs contained lead, the liver and kidneys yielding large amounts. A small quantity of mercury was found in the liver and kidneys in all four cases.

Report of Committee on Lead Tetra-ethyl. (*Ind. Eng. Chem.*, 1926, 18, 193-196.)—A committee appointed by the United States Public Health Service to inquire into the risks to health involved in the retail distribution and general use of petrol motor fuel containing lead tetraethyl (*cf. ANALYST*, 1925, 50, 84) has reported that drivers of cars using this fuel, and in which the concentration of lead tetraethyl was not greater than 1 part to 1300 parts by volume of petrol, showed no definite signs of lead absorption after exposures approximating to two years. In garages and stations in which the fuel was used, some absorption of lead was noticed, but the effect was slight in comparison with that shown by workers in other industries where there was known to be a serious exposure to lead dust. In regions where lead tetraethyl petrol had been used to the greatest extent as a motor fuel for periods of two to three years, no definite cases were discovered of recognisable lead poisoning or other diseases resulting from the use of lead tetraethyl petrol. Apart from the question of the fuel, it would seem that wherever motors are housed together there is an accumulation of lead dust which may prove to be a source of danger to the workers. During the investigation, the effects of the fuel on two hundred and fifty-two individuals (adult males) were studied, and particular attention was directed to the determination of lead in the faeces as an index of lead ingestion, to the determination of the number of stippled red blood cells (*cf. ANALYST*, 1925, 50, 83), and to measurement of the strength of the extensor muscle of the forearm. The first two determinations yielded results of positive value.

W. P. S.

Water Analysis.

Identification of Urine in Water. W. Austen. (*Wasser u. Gas*, 1925, 15, 484-492; *Chem. Abstr.*, 1925, 19, 3553.)—The method of Jolles for the detection of urine has been tested on various waters and found to be uniformly successful. The test is applied as follows:—Ten c.c. of the water are shaken with 1 c.c. of a 5 per cent. alcoholic solution of thymol, then treated with 10 c.c. of fuming hydrochloric acid (sp. gr. 1.19) containing 5 grms. of ferric chloride per litre, left for 5 minutes, and shaken with 4 c.c. of chloroform. A violet coloration indicates the presence of urine in the water. The reaction is not hindered by chlorine or nitrates, but nitrites interfere with it. This is prevented by adding ferrous ammonium

sulphate salt. The test is sensitive in a dilution of 1 : 100, but cannot be applied to filtered water, since filtration removes the indican, the substance in urine upon which the test is based.

Agricultural Analysis.

Mechanical Analysis of Soils. Report and Recommendations by a Sub-Committee of the Agricultural Education Association. (*J. Agric. Sci.*, 1926, 16, 123–144.)—The methods of mechanical analysis of soils at present available are compared and criticised, and it is concluded that the one employed in this country does not give absolute results. In the method recommended more complete dispersion of the soil is obtained by oxidation with hydrogen peroxide, followed by treatment with hydrochloric acid and dispersion in ammoniacal water; this gives higher figures for clay and less erratic results. The subsequent separation of the soil into fractions is carried out by Robinson's pipette method, which yields results comparable with those obtained by the present beaker sedimentation method, but is more controllable and convenient to carry out. It is emphasised, however, that with particular types of soil, the ordinary methods may break down. The omission of the "fine gravel" group is recommended, owing to the difficulty in obtaining representative samples, particles of this size being determined with the stones and rocks. No change in the names assigned to the fractions is proposed, but if the logarithms of their settling velocities are plotted against the summation percentages, analyses might be transferred from one scale to another (the actual settling rate being known), by interpolation. The 100-mesh sieve for the separation of coarse and fine sand is also retained in spite of its limitations. Experimental details of the methods recommended are given in an appendix.

J. G.

Relation between Effectiveness and Composition of Petroleum Insecticides. G. P. Gray and E. R. de Ong. (*Ind. Eng. Chem.*, 1926, 18, 175–180.)—Laboratory and field tests showed that certain petroleum distillates are very injurious to vegetation, even when applied (sprayed) as 5 per cent. emulsions. The toxicity of thirty-five oils used in the experiments appeared to increase approximately in proportion to the amount of unsaturated compounds present, and it is suggested that a simple sulphonation test, in which the amount of the oil soluble in 37 *N* sulphuric acid (fuming) is determined, would give information as to the safety of an oil. Of the oils examined, seven which were very injurious to vegetation contained over 40 per cent. of compounds soluble in sulphuric acid, whilst oils having no effect on vegetation, and therefore classed as safe, contained less than 20 per cent. of such compounds.

W. P. S.

Organic Analysis.

Portable Combustion Apparatus for the Detection and Measurement of Small Quantities of Petrol Vapour. C. A. Neusbaum, P. L. De Verter and E. W. Dean. (*Ind. Eng. Chem.*, 1926, 18, 183–185.)—The apparatus consists

of a glass combustion chamber and a measuring burette, combined in the form of a U-tube; this part of the apparatus is provided with a water jacket. The top of the combustion chamber is fitted with a ground-joint stopper carrying a three-way tap which connects with the sampling tube. The current for heating the platinum wire in the combustion chamber is supplied by three dry cells through a rheostat and ammeter. The combustion chamber and burette are first filled with mercury, the gas under test is then drawn into the apparatus, its volume measured, and the platinum wire is heated for two minutes. The residual gas is then cooled, measured, and the decrease in volume noted. The apparatus is standardised with air containing a known quantity of hydrocarbon vapour, as determined in a Haldane apparatus, and the results are expressed in terms of pentane.

W. P. S.

Determination of the Water Content of Liquid Glue. W. A. Kingman.

(*Ind. Eng. Chem.*, 1926, 18, 93-94.)—The determination is made in a special burette-condenser, which consists of a condenser at the top, a side arm connecting it with the distilling flask, and below this the burette, with a bulb just above the stopcock holding 5 c.c. to the lowest graduation of the burette. About 3 c.c. of tetrachlorethane are put into the burette, and the mixture, in the proportion of 50 c.c. of tetrachlorethane to 15 grms. of liquid glue, is heated in the distilling flask, which is immersed in an oil bath. When the water has evaporated from the sample the burette side arm will become dry and clear. Any drops of water are washed down the burette with naphtha. Tetrachlorethane is drawn off from the bottom of the burette, and the volume of water is read after cooling. D. G. H.

Determination of Alkali and Acid in Wool. H. R. Hirst and A. T. King.

(*J. Textile Inst.*, 1926, 17, 94-103.)—If wool is treated with either acid or alkali, it holds them so tenaciously that it cannot practically be freed from them by washing with water. The problem of determining this acid or alkali is further complicated by the presence in commercial wools of soaps which are hydrolysed in the wool into an acid soap and sodium hydroxide. In view of the fact that present methods are unsatisfactory, the following methods have been devised:—*Alkali*.—Ten grms. of the wool are left in 250 c.c. of water for one hour, after which about 1 gm. of terephthalic acid is added, and the beaker kept at 50° C. for 2 hours, with occasional stirring. The wool is then well pressed and rinsed, and the united solutions filtered from the excess of terephthalic acid. The filtrate is treated with a known excess of 0.1 N-sulphuric acid, again filtered and back-titrated with 0.1 N-sodium hydroxide solution, brom-phenol blue being used as indicator. The amount of excess of sulphuric acid found is a measure of the alkali combined with the terephthalic acid, and thus the percentage of alkali in the wool can be determined. Terephthalic acid was chosen because of its very low solubility in water and the solubility of its sodium salt in water with practically no hydrolysis. Although only very slightly soluble in water (0.168 gm. per litre at 20° C.), it is stronger than the soap acids and decomposes soap. Its solution has a P_H value of about 4.5. If the end-point is obscured by dyestuff dissolving from the wool, a

gravimetric method must be employed. The filtered sodium terephthalate solution, instead of being titrated, is treated with excess of sulphuric acid, and the precipitated terephthalic acid collected and weighed. This method, however, is not quite so accurate, and gives high results. For example, in an experiment in which 0.20 per cent. of sodium hydroxide had been used 0.226 per cent. was found. The alkali from calcium soaps is also determined by these methods, since calcium terephthalate is soluble in water to the extent of 0.484 grm. per litre at 60° C. When such soaps are present a separate determination of the calcium would be necessary.

Sulphuric acid.—Fairly satisfactory results are obtained by placing the wool or cloth in water at 60° C., and adding 1 grm. of magnesium carbonate. After standing overnight the sulphuric acid is determined gravimetrically with barium chloride. This is especially useful when the cloth readily colours the solution and obscures the end-point. But the objection is that any calcium sulphate in the wool would be included as sulphuric acid. The method recommended is to leave the cloth for 12 hours with an excess of sodium terephthalate solution in the cold. Double decomposition takes place, and all the sulphuric acid will be found in the solution, and can be calculated on a basis similar to that used in the above method for alkali. Sodium terephthalate in solution is not affected by soap acids. The method can also be applied to the determination of acid in spent dye-bath solutions.

R. F. I.

Miscellaneous Reactions for Artificial Silk. (*Textile Colourist*, 1925, 47, 177; *J. Textile Inst.*, 1926, 17, A53.)—Ruthenium oxychloride with ammonia ($\text{Ru}_2(\text{OH})_2\text{Cl}_4(\text{NH}_3)_7 \cdot 3\text{H}_2\text{O}$) colours cuprammonium silk pink, whereas viscose remains white. De-nitrated nitro-cellulose silk is coloured red, changing to violet, on standing. Naphthylamine Black 4B colours cuprammonium silk dark blue, and viscose silk light blue in a neutral hot bath. Acetate silk dissolves in glacial acetic acid; the others do not. Diphenylamine is coloured blue by nitro-cellulose fibres, but is not affected by the other products. Methylene blue dyes viscose a deep shade, nitro-cellulose silk a distinct blue, cuprammonium silk a faint tint, and acetate silk irregularly.

R. F. I.

Inorganic Analysis.

Colorimetric Method for the Determination of Oxygen. V. V. Efimov. (*Biochem. Zeitsch.*, 1925, 155, 371–375; *Chem. Abstr.*, 1925, 19, 3504.)—One per cent. of glucose and 1 per cent. of potassium carbonate are dissolved in a 0.1 per cent. aqueous solution of indigo carmine. After 24 hours the solution will be decolorised, owing to the formation of the leuco base, and it is then protected from the air by pouring a layer of vaseline oil over it and sealing the flask. By exposing various amounts of the leuco base (removed from the flask by means of special capillary pipettes) in the same volumes of water to the air a range of colour standards is obtained. The solution to be tested for oxygen is placed, with as little shaking as

possible, in a tube and covered with oil, and a known amount of the leuco base is added. The colour developed is compared with the standards against a potassium dichromate solution as background. To convert the results into mgrms. of oxygen a comparison is made with the results obtained by Winckler's method.

Oxygen Absorption by Anthraquinone- β -sulphonic Acid in Alkaline Solution. T. K. Kruse. (*J. Pharmacol. Proc.*, 1925, 25, 151; *Chem. Abstr.*, 1925, 19, 3232.)—Alkaline solutions of sodium dithionate ($\text{Na}_2\text{S}_2\text{O}_4$) absorb the last fraction of oxygen very slowly, but if anthraquinone- β -sulphonic acid is added to such a solution, the rate of absorption increases and becomes more nearly constant. The most suitable mixture is: Sodium dithionate, 15 to 20 per cent.; anthraquinone- β -sulphonate, 1 to 2 per cent.; and potassium hydroxide, 10 per cent. A solution of this composition absorbed the oxygen in air in 10 to 15 displacements. The reagent with potassium hydroxide indicates complete absorption by a colour change, the solution wetting the capillary of the absorber then remaining red. The absorptive power of the reagent becomes sluggish after being kept for 14 days.

The Kjeldahl-Pregl Method applied to Nitro-compounds. A. Elek and H. Sobotka. (*J. Amer. Chem. Soc.*, 1926, 48, 501-503.)—In the Kjeldahl-Pregl micro-method for the determination of nitrogen it has been found an advantage to add 50 to 100 mgrms. of glucose to 3 to 10 mgrms. of sample. One gm. of potassium sulphate, a crystal of copper sulphate, and 3 c.c. of sulphuric acid are also added, and digestion and distillation carried out as usual, an indicator being used to ensure the addition of a sufficient amount of alkali. An hour is required for one determination, and satisfactory results have been obtained with aliphatic, heterocyclic and isocyclic nitro-compounds. J. G.

Rapid Detection of Small Amounts of Aluminium in certain Non-Ferrous Materials. G. E. F. Lundell and H. B. Knowles. (*Ind. Eng. Chem.*, 1926, 18, 60-61.)—The method depends on the formation of a bright red lake when aurin tricarboxylic acid is added to an aluminium salt under certain conditions. One gm. of alloy is dissolved in 5 c.c. of concentrated nitric acid, and, after the addition of 30 c.c. of an 8 per cent. solution of sodium hydroxide, is boiled for 1 minute, when 20 c.c. are added of an 8 per cent. solution of sodium sulphide, made from the sodium hydroxide solution by saturating a given volume with hydrogen sulphide and adding a like volume. After filtering, the filtrate is acidified with an excess of 2 c.c. of dilute hydrochloric acid (1:1), and digested at 40-60° C. until the precipitate settles; it is then filtered, hydrogen sulphide expelled by boiling the filtrate, and nitric acid added, if necessary, to clarify it. After evaporating the liquid to 20-30 c.c. and filtering it, if necessary, 10 c.c. of 36 per cent. of acetic acid are added, then 5 c.c. of a 0.2 per cent. solution of aurin tricarboxylic acid, and a 10 per cent. solution of ammonium carbonate in dilute ammonium hydroxide (1:2), slowly and with stirring, until an excess of 5 to 10 per cent. is present. The colour is compared with those given by known amounts of aluminium. This method removes all the usual elements except aluminium

and phosphorus. If less sodium sulphide is added and the second filtration omitted, time is saved, satisfactory results are usually obtained, and sulphide need not be used at all with brass and such other alloys as give large precipitates with sodium hydroxide.

D. G. H.

Determination of Cerium in Alloy Steel. K. Swoboda and R. Horny. (*Zeitsch. anal. Chem.*, 1926, **67**, 386-398.)—Two grms. of drillings are dissolved in 60 c.c. of hot hydrochloric acid (1:1) in a 500 c.c. beaker. The solution is treated, drop by drop, with enough nitric acid to oxidise iron and tungsten, then with 60 c.c. of 25 per cent. tartaric acid and 30 to 35 c.c. of 10 per cent. stannous chloride solution. A slight excess of strong sodium hydroxide solution is added, and the liquid transferred to a graduated 500 c.c. flask. After cooling, and addition of 10 c.c. of alcohol to prevent oxidation of the ferrous hydroxide, the volume is made up to 500 c.c., and 250 c.c. filtered quickly through a double paper into a 500 c.c. beaker. Hydrochloric acid is added, drop by drop, till the reaction is distinctly acid, the solution boiled, and the cerium precipitated with 2 grms. of solid ammonium fluoride. The liquid is now neutralised with ammonia, 1 to 2 drops excess of hydrochloric acid added, and the precipitate left to settle for an hour. It is filtered on pulp, washed with a solution containing 3 grms. of ammonium fluoride per litre till free from sodium salts, ignited in a platinum crucible, and weighed as CeO_2 . The precipitation as oxalate gave low results.

W. R. S.

Separation of Tantalum and Niobium. G. W. Sears. (*J. Amer. Chem. Soc.*, 1926, **48**, 343-348.)—The following method of separation is based on the observation that the earth acids show differential solubility towards sulphuric acid after fusion with pyrosulphate at a temperature of 835° to 875° C. The mineral (tantallite or columbite) is ground to pass through a 150-mesh sieve and fused with 9 parts of sodium pyrosulphate; a platinum-nichrome thermo-couple dipping in the fused mass is used for controlling the temperature. The time factor seems to have little or no effect. The cooled mass is leached with water (1 to 2 hours), and the residual earth acids washed by decantation with hot 3 *N* hydrochloric acid until free from iron. The white residue is transferred to a beaker covered with a watch-glass, and heated with sulphuric acid (1:1) "until all evidence of boiling ceases." The cooled liquid is filtered by suction through an asbestos filter, and the residue washed with cold 6 *N* sulphuric acid until the washings give no precipitate with ammonia. It is stated that, with a temperature range of 840° to 850° C., the niobium is completely dissolved, whilst the tantalum is left in the residue; with lower fusion temperatures some of the tantalum dissolves. These conclusions are based on qualitative tests only (*cf.* ANALYST, 1925, **50**, 494), the earth acid residue being dissolved in hydrofluoric acid, the solution concentrated by evaporation, and treated with potassium chloride. If no precipitate was formed, the solution was evaporated nearly to dryness, and the residue examined under the microscope for needles of potassium fluotantalate. Niobium was tested for in the same residue, or in the filtrate from the fluotantalate, by reduction with tin and hydrochloric acid (2:1 water).

W. R. S.

Influence of Citrates on the Precipitation of Barium Sulphate. M. L. Nichols and O. J. Thies, Jr. (*J. Amer. Chem. Soc.*, 1926, 48, 302-309.)—When a barium salt solution is added to a solution of sodium sulphate and citrate, a gelatinous precipitate of barium citrate is first formed, which, on shaking, changes to a milky precipitate. This quickly disappears, and the liquor appears clear or slightly opalescent. It is a dispersion of negatively charged colloidal barium sulphate. The sol is coagulated by acids and salts, but the precipitated barium sulphate cannot be filtered and washed. The determination of sulphate as barium sulphate in the presence of citrate is quite satisfactory if slightly more than enough hydrochloric acid to convert the citrate into citric acid is added before the precipitation with barium chloride.

W. R. S.

Physical Methods, Apparatus, etc.

Use of Methyl Salicylate in a Flowmeter. R. H. K. Foster. (*Ind. Eng. Chem.*, 1926, 18, 82.)—Methyl salicylate (oil of wintergreen), sp. gr. 1.186, may advantageously be used for measuring the differential pressure through the capillary of a flowmeter, since the meniscus is not affected by any grease film present, the vapour pressure and rate of diffusion into air or gas is very low, and air bubbles are more easily removable from the inverted U-tube than with water. Methyl salicylate dissolves rubber, and soap is the best lubricant for the stopcock.

D. G. H.

New Method for the Determination of the Molecular Weight of Proteins. T. Svedberg and R. Fåhræus. (*J. Amer. Chem. Soc.*, 1926, 48, 430-438.)—The protein solution is centrifuged in a closed cell until the sedimentation and diffusion of the heavy molecules balance one another. If the expressions defining sedimentation and diffusion are then equated, a relation is obtained connecting the molecular weight (M), temperature, concentration (c), speed of the centrifuge, partial specific volume, density of the solvent, and the distance from the centre of rotation (x). By integrating between two values of x , M may then be found. The cell containing solutions of different concentrations is photographed during the process and at equilibrium, and by means of a self-registering microphotometer curves are obtained which enable the relation between c and x to be found. The source of light used is an incandescent or mercury vapour lamp, with suitable filters, according to whether the protein has light absorption in the visible or in the ultra-violet spectrum. The results obtained are independent of variations in the working conditions. Determinations were carried out on carefully prepared solutions of carbon-monoxide-methaemoglobin, purified by electro dialysis. Two determinations in each case gave the value $M=4 \times 16,700$, the minimum value obtained from the iron-content being 16,700. The fact that no systematic variation of M with x was observed, indicates that in one and the same solution there is only one kind of haemoglobin molecule.

J. G.

Reviews.

THE LABORATORY BOOK OF MINERAL OIL TESTING. By JAS. A. HICKS. Fourth edition, revised by ARTHUR W. COX. Pp. 108, including Appendix and Indexes, and 56 Illustrations. London: Chas. Griffin & Co., Ltd. Price 5s. net.

Mr. Hicks's well-known little book has been out of print for some years, and the issue of a new edition has been delayed pending the completion and publication, by the Institution of Petroleum Technologists, of Standard Methods for the testing of petroleum and its products.

The present edition has been thoroughly revised, and includes references to all these standard methods, with their serial numbers and a separate index to them, as well as descriptions of many other methods and appliances employed for the testing of asphalt, crude petroleum, fuel oil, lubricating and gas oils, transformer oil, kerosene, white spirit, motor spirit, paraffin scale and wax.

The large number of tests included in this small volume has necessitated very brief descriptions of many of the methods and pieces of apparatus used, but the descriptions are clear, and, as a handy book of reference, the volume will be found very useful by all analysts who undertake this kind of work. The book is very well printed and illustrated and is well bound.

To those who are not familiar with the subject, the references to the standard methods may be a little puzzling, and it would be as well to make clear, when the book is reprinted, that they refer to the serial designations given in the booklet of Standard Methods published by the I.P.T. at their address in Bedford Street, W.C.2.

L. ARCHBUTT.

INORGANIC PHYSICAL CHEMISTRY. By G. H. CARTLEDGE. Pp. xv. + 463. Boston, U.S.A.: Ginn & Co. Price \$4.80.

This textbook is based on the curriculum which has been adopted at the Johns Hopkins University; in consequence, it is perhaps best fitted to be read in conjunction with a course such as that for which it has been designed, where what is known as "physical" chemistry is taught, not as a separate subject, but in connection with the respective courses of general, inorganic and organic chemistry. There is something to be said for and against this system; from the point of view of the readers of *Inorganic Physical Chemistry*, it has the defect of depriving them of the benefit of Professor Cartledge's lucidity on the subjects of colloids, the physical properties of solids and liquids, and the vapour pressure of volatile mixtures, these items being deemed outside the scope indicated by the title.

Part I, entitled "The Nature of Matter," which comprises roughly a quarter of the text, contains an excellent account of the history of theoretical chemistry, from the atomic speculations of Democritus, through the early quantitative work of Rey and Boyle, down to the discovery of the isotopes of the common elements, the artificial disintegration of the atom, and the technique of modern atomic weight determinations, a typical example of which is quoted in much detail. Part I also includes a most clear elementary exposition of the Kinetic Theory.

The other three parts include, with the exceptions mentioned, the usual subjects covered in a second year course of physical chemistry. Analysts will appreciate the treatment in Part IV ("Applications") of the theoretical aspects of inorganic analysis. This section also contains a useful chapter on complex ions.

Mention must be made of an admirable introduction in which the methods of science in general and of chemistry in particular are briefly discussed. The whole book is written in a concise but thoroughly readable style, and the problems given at the ends of chapters seem particularly stimulating and well designed. Although the gap that it seems intended to fill for American students hardly exists in a country which has had for several years the advantage of Senter's *Outlines of Physical Chemistry*, the book will undoubtedly be found of value.

H. R. AMBLER.

L'EMPLOI DES INDICATEURS COLORÉS. LA DÉTERMINATION COLORIMÉTRIQUE DE LA CONCENTRATION DES IONS HYDROGÈNE. Par I. M. KOLTHOFF. Traduit sur la troisième édition Allemande par EDMOND VELLINGER. Pp. 250. Paris: Gauthier-Villars et Cie.

In recent years the study of the hydrogen ion concentration of solutions has assumed great importance. The simple process of the titration of an acid by an alkali involves the necessity of following to some extent the change in the hydrion concentration, and the separation of certain metals in the ordinary "group" analysis by precipitation depends on a control of the same factor.

In industry the rigid control of the hydrion concentration, or, in other words, the acidity, of electroplating solutions has made it possible to obtain smooth deposits of both iron and nickel by electro-deposition; in connection with the manufacture of leather the work of Procter and Wilson has thrown light on the relation between hydrion concentration and tanning processes. Since the classical investigations of Sorensen, who introduced the term P_H , much important work has been done on the influence of acidity on the behaviour of enzymes, and in recent years the importance of hydrion concentration determinations on soils has been realised by students of ecology and agriculture.

These few examples will serve to show the importance of a rapid and simple method for the determination of the P_H of a solution; of the possible methods, there is no doubt that the one in which "indicators"—that is, substances which change their colour in accordance with changes in the hydrion concentration of their environment—are used is by far the most important, and hence any book dealing with this subject merits consideration.

The book under review, *La Détermination Colorimétrique de la Concentration des Ions Hydrogène*, is a French translation of the third edition of Kolthoff's German text; it deals in a very able and clear manner with the theory and application of indicators for the determination of hydrogen ion concentrations under a variety of conditions.

The following subjects, among others, are discussed: The principles of neutralisation, hydrolysis of salts, the theory of buffer solutions and the neutralisation of mixtures of acids. The colour change of indicators is dealt with, and the

use of a series of indicators for the purpose of determining hydron concentrations within wide limits is described.

In a subsequent chapter the use of indicators for the purpose of following the neutralisation of acids and alkalis is discussed, and numerous examples are given which show the application of indicator methods. Directions are given for the preparation of various indicator and buffer solutions, and brief details are given of the methods available for the determination of the P_H of a solution by the use of indicators. The subject of the influence of neutral salts on indicators, as well as other sources of error, is discussed.

The book contains a section dealing with hydron measurements in various analytical and technical processes, and also one dealing with the use of indicator papers; these papers may prove very useful in the control of industrial processes. An account of the theory of the structure of indicators concludes a most interesting and readable, as well as useful, book; it will appeal, however, more to the laboratory and research chemist than to the "practical" man.

The book contains a number of references to the original literature which are well chosen and adequate, and which should enable those interested in any particular branch of the subject of indicators to obtain further information.

It is somewhat unfair to criticise a book, which deals so well with so many subjects, for errors of omission, but the present writer would have liked to see a table of Walpole's acetate buffer mixtures, which are only mentioned in a literature reference, on account of their ease of preparation and of their usefulness when dealing with reducible substances. From the point of view of commission the reviewer would have preferred a somewhat different arrangement of the subject matter, the use of the symbol HIn for an acid indicator instead of the rather confusing symbol HI , and, lastly, as a matter of individual fancy, the placing of literature references at the bottom of each page instead of at the end of the chapter.

The translator appears to have done his work well, although there are a large number of minor and obvious misprints, particularly in the spelling of authors' names; in addition to translating the book, he has written two appendices describing the determination of the P_H of intra-cellular liquids, and the recent work on the spectrophotometric method for following changes in an indicator.

The absence of an index detracts somewhat from the usefulness of the book, but, in spite of this, it is one which can be recommended very strongly.

S. GLASSTONE.

INTERMEDIATES FOR DYESTUFFS. By A. DAVIDSON, B.Sc., A.I.C. Pp. ix. +256.
London: Ernest Benn, Ltd. 1926. Price 36s. net.

This is an excellent book written by a man who knows his subject and who knows how to distinguish between the important and the minor processes.

The short introduction explains the importance and the application of dyes in general and the nature of the chromophores which are met with in commercial dyestuffs. The arrangement of the subject seems practical, but, as the author himself points out, presents certain difficulties, because many compounds belong to different classes and it is a matter of choice where to describe them.

First of all the chlorination products of benzene are dealt with, and the different methods of preparing them are given, the different possibilities being shown on an instructive chart, as are also the derivatives of the chlorobenzenes (mono- and di-).

Short but sufficient details are given in each case; the author does not attempt to give everything that has been published, but leaves it to the reader to consult the literature of each subject, which is carefully selected. This is very important, because in most books of this type the author gives every possible reference, and leaves it to the unfortunate reader to find out which method is actually used in technical works. It is evident that in some cases the data given are not absolutely correct. The reviewer considers it preferable to make *p*-chlorophenol from phenol and sulphuryl chloride rather than from *p*-dichlorobenzene, because he has not been able to obtain the excellent yields cited by the Badische Company in their German Patent No. 281175. These, however, are very slight drawbacks when one considers the large amount of excellent information on almost every subject in the wide field of intermediates.

After a description of the derivatives obtained from benzene, including nitrobenzenes, toluenes and amines, the sulphonic acids, chloro-derivatives of toluene and xylene, naphthalene is dealt with, and a very careful selection of the most recent literature on this subject is added. Accurate information on the various derivatives of the many sulphonic acids of naphthalene is given, and the different amidonaphtholsulphonic acids and their properties are described.

The derivatives of β -naphthol follow, and methods for the preparation of phthalic anhydride, including the newest methods (*e.g.* oxidation with air), are given. In this connection the reviewer would like to point out that the catalytic process was not first published by Harry Gibbs, as is stated and almost universally believed in the United States. The original patent was taken out almost two years before by Wohl, in Danzig, and was assigned to the Badische Anilin and Sodafabrik. It seems important to rectify this widespread error which is known to Mr. Gibbs, as he mentioned it to the reviewer as early as 1920 in Wilmington (Del.). A similar error, not made in this book, is often met with in American literature on the synthetic production of anthraquinone by Heller's process, as Mr. Davidson rightly points out on page 209.

The description of the derivatives of anthraquinone is the best the reviewer so far has met with. He would like to point out that, for the first time, the actual method for the preparation of 4·8-diamidoanthranufin is given (p. 234). It is a pity that the author was not aware of the latest investigation on the quantitative aspect of the sulphonation of anthraquinone, with and without mercuric sulphate, but that work was not published until just after this book left the press.

The volume ends with the stabilised diazo compounds and miscellaneous intermediates.

The author is to be warmly congratulated upon his publication. The printing and paper leave nothing to be desired.

H. E. FIERZ.

PRACTICAL PHYSICAL AND COLLOID CHEMISTRY FOR STUDENTS OF MEDICINE AND BIOLOGY. By L. MICHAELIS. Authorised Translation from the 2nd German Edition by T. R. PARSONS. Pp. x.+194. Cambridge: Heffer & Sons, Ltd. 1925. Price 7s. 6d. net.

Professor Michaelis's text-book is to be cordially recommended to "students" of medicine and biology in both the narrower and the wider meaning of the term. To the student who is reading for his degree examination the book will be invaluable, because of the series of admirably planned experiments, which not only teach the methods of physical-chemistry, such as the technique for the measurement of osmotic pressure, viscosity, surface-tension, conductivity, electrical potentials, etc., but also make these methods of living interest by showing how they can be applied to the study of biological problems.

The practical instructions are concisely given and the object of the exercise is clearly indicated. All the exercises involve the measurement of a quantity and, in most cases, the co-relation between two quantities, as, for example, the relation between the precipitating action of tannin on gelatin and the hydrogen ion concentration of the solution, (p. 71), or that between the concentration of tributyrin in a solution and the surface tension (p. 83), a method that is ingeniously adapted for the determination of the concentration of the fat-splitting enzyme in physiological fluids such as serum (p. 85), or the digestive juices (p. 87).

To the advanced student engaged in research, the book gives an admirable review of the physical methods now available for biological research. References to the original papers in which the methods are first described or adapted add greatly to its value. All the well-recognised standard methods are clearly described, and among the less well known are included the technique for the determination of the chlorine ion—a useful extension of the method of electrometric analysis, which although it has been available for some years, has not received the attention that might have been anticipated. Another useful and novel method included for the first time in a text book is the use of surface active indicators for titration, a method that might prove particularly convenient for coloured or turbid fluids.

The English version of the book is to be welcomed, since it will make it available to a wider public, but it is a pity that the author's preface has been omitted. The translation by Mr. T. R. Parsons is satisfactory. A somewhat unorthodox use of the word "whose" as a relative pronoun of neuter gender occurs on p. 8, but otherwise the translation is free from grammatical structure belonging more appropriately to the German language. The use of the letter "h" in ordinary small print to symbolise the concentration of the hydrogen ion does not harmonise with the conventions of English script, and is an innovation that seems to have nothing in particular to recommend it. The more usual form, C_H or $[H\cdot]$ does at least stand out clearly in the text.

The book is printed in a clear and pleasing type, and the reproduction of the illustrations is good. Minor printers' errors occur on pp. 9, 24, 164, and 173, where a name which became eminent as "Tyndall" has been allowed to appear as "Tindall."

DOROTHY JORDAN LLOYD.

SOME NEWLY-DISCOVERED STANZAS WRITTEN BY JOHN MILTON. By H. C. H. CANDY, B.A., B.Sc., F.I.C. Pp. viii. + 192. London: Nisbet & Co. Price 7s. 6d. net.

The name of Mr. Candy will be familiar to the readers of *THE ANALYST* as the joint author of a successful treatise on chemistry, but to the literary world he has become known through his discovery of a whole series of poems by Milton.

The story of the discovery is a remarkable one, and one's first instinct is to discount its value, since it hardly seems credible that a set of stanzas by a poet so much studied as Milton has been should not only have lain hidden for over three centuries, but should also not even have been heard of by any of his earlier commentators. Indeed, such a strange thing could hardly have happened unless the poet himself had been anxious to disown the poems. Yet, in view of their subject, he had good reason for wishing to suppress them, for they would have been a strong weapon against him in his controversy on divorce. Thus there is a reasonable explanation of the poems not being known, and, apart from this, no one who studies the internal evidence presented in this book can come to any other conclusion than that they were written by Milton.

The stanzas, which number 166 of eight lines each, are written on the blank spaces on the pages of a German edition of Ovid's *Metamorphoses*, published in 1563 at Frankfort, and although each deals with the subject of the accompanying woodcut, it is not merely a translation of the text.

To prove that the stanzas are by Milton and are in his handwriting, Mr. Candy has made an exhaustive literary comparison of them with his published poems, and has also made a minute examination of the letters in the writing itself compared with admitted writing of Milton of the same period (about 1623). The evidence thus gathered is presented in a form which is scientific in character, and is essentially based on a balancing of points of resemblance and difference, just as is that obtained by a chemical analysis. The only criticism to be made is that the arrangement of the selected specimens of writing might have been better, since it is not always easy to see which is the writing from the Ovid and which the admitted writing.

Each stanza is separately discussed, and reduced facsimiles of some of them and of the scenes to which they refer are given. Authentic specimens of Milton's earlier and later styles of writing are also reproduced, and as frontispiece there is an interesting portrait by Cornelius Jansen of the poet as a boy.

Members of our Society will be glad to congratulate a fellow member both on the luck which brought this discovery within his reach, and on the skill and patience he has shown in establishing the authorship of these stanzas. His work must link his name with that of the poet.

EDITOR.