

THE ANALYST

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

Obituary.

JOHN HOWARD BROWN JENKINS.

THE sudden death of John Howard Brown Jenkins at the Railway Clearing House on December 11th, while presiding at a meeting of railway officials, has deprived the chemical profession of a valued member, and those who had the privilege of his personal and intimate acquaintance of a very dear friend.

Mr. Jenkins was born in 1866, and in his early days was attracted to the science of engineering; in 1882 he commenced an apprenticeship at the Swindon works of the Great Western Railway under the late Mr. W. Dean, and in 1888 gained a Whitworth Exhibition. Later, he studied chemistry under the late F. W. Harris, F.I.C., in the Great Western Company's chemical laboratory; in 1892 he was appointed chemist to the Great Eastern Railway, and it was in this Company's laboratory at Stratford that he laid the foundations of the work which brought him to the notice of the industrial and engineering world. Subsequent to the grouping of the railways, he received the appointment of Chief Chemist to the London and North Eastern Railway, the position which he held at the time of his death.

His early engineering training assisted him in dealing with many of the problems he met with in the railway service, notably in the involved question of cylinders for compressed gases, on which he was regarded as an authority. In 1919 he became the Railway Companies' nominee to the Gas Cylinders Research Committee of the Department of Scientific and Industrial Research; on this Committee, he found his views in conflict with those of the majority, resulting in his issuing the Minority Report which accompanied the First Report of the Committee in 1921. Although his views were unacceptable to the majority of that

Committee, the sound basis and honesty of his opinions were freely admitted, and his report, even though it failed to gain support from any of his colleagues, was accepted by all as a valuable contribution to the knowledge of the subject. It is thought, by some, that certain aspects of the matter raised in the Minority Report may ultimately prove to be of much value in future developments. In further work he found his views to be in harmony with the whole Committee.

Mr. Jenkins' scientific and literary abilities found ample scope in the varied work of a railway chemist. He was a highly skilled metallographist, and contributed to the Society of Public Analysts in 1904, with Mr. D. G. Riddick, a valuable and beautifully illustrated paper on the Microscopic Examination of Metals. He also read before the Society in 1898 a paper on Japanese Wood Oil, and made many contributions to the discussions of papers on oils, waters, metals, etc. He was a Vice-President of the Society in 1915-16, and served on the Council during three separate periods. In 1897 he communicated to the Society of Chemical Industry papers on Hehner's Bromine Test for Oils and on Japanese Wood Oil, and in 1919 he wrote the Annual Report on Paints, Pigments, Varnishes and Resins for that Society. In 1923 he was elected a Fellow of the Institute of Chemistry.

His work as a railway chemist was well known to many chemists and manufacturers who had occasion to meet him at the Railway Clearing House and discuss matters connected with the packing and conveyance of ordinary and dangerous goods, but only those of his colleagues in close touch with his work know how valuable his help and advice were. All, however, who were associated with him in either capacity could not fail to recognise his unfailing courtesy and fairness. He was an indefatigable worker, a sound adviser, and a sincere friend, very modest and retiring, but tenacious to a degree of opinions he had formed and considered important. His kindly nature and humorous outlook, nevertheless, endeared him to those who differed from him, as well as those who agreed with him. He was a charming conversationalist and a remarkable correspondent. His death will be a great loss to the railway companies and the public for whom they cater. He leaves a widow and one son, who is a student at St. Thomas' Hospital Medical School.

L. ARCHBUTT.

Death.

WE greatly regret to announce the death, on February 6th, of Mr. James West Knights, one of the oldest members of the Society. He was for 50 years Public Analyst for the County and Borough of Cambridge, Hunts., Wisbech, and King's Lynn.

The Fatty Acids and Component Glycerides of some New Zealand Butters.

BY T. P. HILDITCH, D.Sc., F.I.C., AND EVELINE E. JONES, M.Sc.

(Read at the Meeting, February 6, 1929.)

WHEN a natural fat, composed of a mixture of neutral glycerides, is carefully oxidised in acetone solution with potassium permanganate, all the unsaturated groups present are broken down into mixtures of lower fatty acids and semi-acidic glyceride derivatives of azelaic acid, whilst fully-saturated triglycerides are left unaltered (Hilditch and Lea, *J. Chem. Soc.*, 1927, 3106; Collin and Hilditch, *J. Soc. Chem. Ind.*, 1928, 47, 261T). Consequently a natural mixture of fully-saturated and mixed saturated-unsaturated glycerides can be quantitatively resolved into a corresponding mixture of neutral and acidic products. This procedure not only leads to a simple method of ascertaining the proportion of fully-saturated glycerides in a fat, but, if they are present in reasonably large amounts, permits them to be isolated in quantity in the pure condition; analysis of the mixed fatty acids combined in the whole fat and in the fully-saturated portion by the methyl ester distillation method (*cf.* Collin and Hilditch, *loc. cit.*; Hilditch and Houlbrooke, *ANALYST*, 1928, 53, 246) then furnishes considerable information of a semi-quantitative nature with regard to the mode of union of the fatty acids with glycerol in the original fat.

This line of attack has been applied to the case of butter-fats, the materials selected for investigation being three bulk samples of ordinary deliveries of New Zealand butter. It was necessary, however, to devote considerable attention to the determination of the composition of the mixed fatty acids, since, as is well known, this is not an easy mixture to deal with on a quantitative basis. Indeed, the literature on the subject justifies this statement by its volume and also by the discrepant observations which it discloses. Whilst allowance should be made, especially in dealing with an animal fat (and perhaps above all with milk fats), for probable variations caused by differences in habit and feeding, it seems unlikely that these suffice always to account for the varying data which have been put forward from time to time; this view is perhaps confirmed by the fact that the characteristics which are the analyst's chief guide in the examination of butter (volatile soluble and insoluble acids, iodine value, saponification value, refractivity, etc.) have not been found subject to variations of by any means so wide an order.

THE DETERMINATION OF MIXED FATTY ACIDS BY FRACTIONAL DISTILLATION OF ESTERS.—It is probably unnecessary for the purpose of this communication to refer specifically to more than one or two of the more recent analyses of the mixed fatty acids of butter, commencing with that of Crowther and Hynd (*Biochem. J.*, 1917, 11, 139). These workers converted the mixed acids into methyl

esters and fractionally distilled the whole; the fact that even the lowest-boiling fractions contained some methyl oleate indicates that the fractionation method employed was not highly efficacious. Holland and Buckley (*J. Agric. Res.*, 1918, 12, 719), and subsequently Holland and co-workers (*ibid.*, 1923, 24, 365) have published data, obtained by somewhat similar methods, for a large number of butter-fats. The general results of both groups of workers were as follows:—

			Crowther and Hynd. Per Cent.	Holland and co-workers. Per Cent.
Butyric acid	4.6	2.2-4.2 ("by difference")
Caproic	"	..	1.7	1.3-2.4
Caprylic	"	..	1.3	0.5-1.0
Capric	"	..	1.3	1.2-2.0
Lauric	"	..	5.4	4.5-7.7
Myristic	"	..	17.7	15.6-22.6
Palmitic	"	..	16.0	5.8-22.9 ("by difference")
Stearic	"	..	3.7	7.8-20.4
Oleic	"	..	48.3	25.3-40.3

These results met with severe criticism from Channon, Drummond and Golding (*ANALYST*, 1924, 49, 311) and Elsdon (*ibid.*, 1924, 49, 423), mainly on the grounds that accuracy to the third place of decimals per cent. was apparently claimed and that the fractionation procedure had shown itself incapable of producing the binary mixtures of saturated esters necessary for arithmetical interpretation. Armstrong, Allan and Moore (*J. Soc. Chem. Ind.*, 1925, 44, 63T) emphasised the necessity for attention to certain vital points in connection with the preparation and fractionation of the esters (notably the isolation and characterisation of fractions of individual esters), and claimed that with due precautions an accuracy to within a unit per cent. was attainable.

We agree with the criticisms of the earlier attempts to apply the fractionation method quantitatively, and with Channon, Drummond and Golding in their statement that the procedure "can yield very valuable information if its limitations are recognised"; but we cannot go so far with these authors as to affirm that "as an exact quantitative method, it is of little value." Obviously, the strain upon the fractionation process should be lessened as much as possible by preliminary division of the acids into groups of varying character. Due attention to preliminary separations of this kind, coupled with precautions in the fractional distillation on the lines suggested by Armstrong, Allan and Moore, and subsequent workers, render an accuracy of within a unit per cent. quite attainable. This is shown by numerous results published from several independent sources within the past two or three years.

Whilst dealing with the fractionation method from a critical standpoint, two matters of detail may be mentioned:

(i) The isolation of absolutely individual esters, as postulated by Armstrong, Allan and Moore, is somewhat of a counsel of perfection when mixtures of small quantities of successive homologues are encountered; in such circumstances we prefer to collect a series of small fractions covering the region in which an individual member may be expected to predominate. This effectively minimises

any inaccuracy due to a possible error in the nature of the minor components of the mixture; the tables on pp. 80, 81 illustrate the general composition of final fractions as determined by our present method of operation.

(ii) It is our custom to collect the primary distillates in fractions as large as is consistent with the gradual rise in boiling-point and the general proportions of the mixture of esters known to be present. Each primary fraction is then submitted to refractionation as a separate entity; we do not in any circumstances add a subsequent primary fraction to the residue from the redistillation of its predecessor. This, in our opinion, makes the ultimate series of calculations more accurate, whilst it avoids the necessity for the employment of "corrected" weights in the latter.

In connection with the earlier data for the acids of butter-fat which we have discussed, we have a further criticism to make—all unsaturation seems to have been calculated on the assumption that oleic acid alone is present. The data which we give in this paper (*cf.* p. 84) show definitely that the unsaturated acids present contain small quantities of an acid less saturated than oleic. We have confirmed this by examination of the hydroxystearic acids prepared from the acids of the fractions concerned by alkaline permanganate oxidation, when, although we did not succeed in isolating any pure tetrahydroxystearic acid, the dihydroxystearic acid produced melted somewhat indefinitely about 5° below the true melting-point of 9, 10, dihydroxystearic acid from pure oleic acid. Further, a very small trace (insufficient for recrystallisation) of brominated acids insoluble in ether was obtained from the "liquid" fatty acids of the butter; this substance blackened, without definitely melting, at 170–180°. We conclude that the unsaturated acids of butter-fat (like those of most other fats) contain small proportions of linoleic acid and even traces of linolenic acid.

Allowance for this in the older analyses may reduce the proportion of unsaturated acids considerably in not a few cases (since, calculated on iodine value, two molecules of oleic acid appear as one of linoleic acid); consequently the amounts of some of the saturated acids will be correspondingly low.

In this connection, Holland's results with butter from cows fed on varying rations may be significant: the "oleic acid" figures, for example, for cows fed respectively on a general ration, on one including coconut oil, and on one including soya bean oil, were 30.8, 29.5 and 45.8 per cent., whilst the corresponding palmitic acid figures (determined "by difference") were 20.2, 17.1 and 8.7 per cent. It is quite possible that the apparent increase in "oleic acid" and the extremely low (differential) percentage of palmitic acid in the butter after diet including soya bean or other semi-drying oil, is really due in part to the presence of varying proportions of linoleic acid. We are proceeding with the investigation of butters from cows whose food has included different types of fatty material, and we hope thereby to throw further light on this matter.

As regards Holland and Buckley's figures for the volatile acids, it may be remarked that good agreement exists between these and the values obtained in

the present work, and also those arrived at recently by Virtanen and Pulkki (*Z. anal. Chem.*, 1928, 74, 321) in the case of ten samples of Finnish butter. The latter included the following range: butyric acid, 3.1–4.2 per cent., and caproic acid, 1.4–2.1 per cent. It is permissible to conclude that the general proportions of the four lowest acids of butter are now known within comparatively narrow limits.

It has been said that, in the fractionation analysis of mixed fatty acids, each fatty mixture should be considered on the lines of a separate problem from the point of view of general procedure. As a rule, it is sufficient to effect a preliminary resolution by the lead-salt method into two groups:

(i) "Solid" acids containing all the stearic or higher acids, all but traces of palmitic, and most of the myristic and lower saturated acids, with small proportions of oleic acid;

(ii) "Liquid" acids containing all C_{18} acids less saturated than oleic, most of the oleic, minor amounts of myristic and lower saturated acids, but only traces of palmitic and no stearic or higher saturated acid.

The "volatile" acids of butter render this particular case more complicated.

We studied Crowther and Hynd's method in the first instance, and satisfied ourselves that the volatility of methyl butyrate rendered its application unsuitable, apart from the tendency, already discussed, for methyl oleate to distil to some extent throughout the process.

We have found it best to adopt, in effect, an intensive "Reichert-Meissl" procedure on a somewhat large scale. The fatty acids from 300–600 grms. of butter-fat are subjected to careful steam distillation for about four or five hours, *i.e.* sufficiently to ensure that the whole of the butyric and caproic acids have been removed. The acids volatile in steam are extracted by ether and fractionated as such, mainly at atmospheric pressure; the difference in boiling-point between the successive homologues is sufficient to permit binary mixtures to be separated. The extracted aqueous liquors and the recovered distilled ether are titrated with alkali, any acid present being calculated as butyric acid. The acids non-volatile in steam are recovered and weighed, and a suitable portion is submitted to the lead-salt separation, the resulting "solid" and "liquid" acids being converted into methyl esters and fractionated quantitatively in the usual way.

The satisfactory concordance which we have obtained by this procedure is not likely to be due merely to repetition of the same sequence of processes on similar materials by the same manipulator, since the following differences have been introduced:

(i) The determination of mixed fatty acids in the whole butter-fats "A" and "B" was carried out on about 600 grms. of each fat, and in "C" on 300 grms., whilst those of the fully-saturated glycerides of "A" were made on 200 grms., and of "B" on 140 grms. of material.

(ii) In the lead-salt separations of the acids non-volatile in steam, the "solid" acids in the case of "A" (56.7 per cent.) had an iodine value of 10.1 of "B" (67.5 per cent.), 23.2, and of "C" (54.4 per cent.), 11.8. Similarly, whilst the "liquid" acids of "A" and "B" (iodine values respectively 82.7 and 80.9) consisted of the corresponding soluble lead salts from the original separation united with the mother-liquors from the recrystallisation of the separated lead salts, in the case of "C," the acids from the soluble salts (38.4 per cent., iodine value 83.1) and from the mother-liquors from recrystallisation (7.2 per cent., iodine value 62.1) were methylated and fractionated separately.

DETERMINATION OF THE COMPOSITION OF THE FATTY ACIDS OF BUTTER-FAT.—The method adopted in these investigations will be illustrated by a detailed account of the experimental work on the New Zealand butter-fat "A."

The fat (approx. 600 grms.) was hydrolysed by prolonged boiling with excess of alcoholic caustic soda, after which as much alcohol as possible was removed from the soap by distillation. The flask containing the residual soap was then connected to a condenser for steam-distillation, an efficient spray-trap being inserted between the exit from the flask and the inlet to the condenser. In order to effect smooth distillation without frothing, it was found best first of all to remove residual alcohol from the soap by cautious steam-distillation; the soap solution was then cooled in the flask until it commenced to set to a jelly. At this point sufficient aqueous sulphuric acid was added to provide a slight excess of mineral acid after all the alkaline base present had been neutralised, and the fatty acids were then submitted to steam-distillation for about five hours; during this time about 5 litres of aqueous condensate was collected.

After cooling, the contents of the flask were extracted with ether, and from the washed ethereal solution the mixed non-volatile fatty acids were carefully recovered and weighed (551.1 grms., mean equivalent 259.9, iodine value 42.2); the quantitative analysis of the non-volatile acids is described below.

COMPOSITION OF STEAM-VOLATILE ACIDS.—The aqueous condensates were extracted five times with ether; the united extracts were dried over anhydrous sodium sulphate (which was subsequently washed with fresh dry ether to remove any adherent fatty acids), and the bulk of the ether was removed by distillation on the steam-bath. Portions of the ether-extracted aqueous solution (5345 c.c.) and of the recovered distilled ether (2394 c.c.) were titrated with standard alkali, and the acidity found calculated as *butyric acid* (2.10 grms. and 0.24 grm. in the respective liquids).

The remaining volatile acids were slowly distilled from a Willstätter fractionation bulb and collected in small fractions: the greater part of the distillation was carried out at atmospheric pressure, but a moderate (water-pump) vacuum was used in the final stages. The first four fractions contained (diminishing) quantities of ether and came over below the boiling point of *n*-butyric acid; the acid present in these was calculated as butyric, and that in the remaining fractions was calculated

from the mean equivalents on the assumption that only binary mixtures were present:

No.	Grms.	B.pt. °C.	Pressure.	Mean equivalent.	Butyric. Grms.	Caproic. Grms.	Caprylic. Grm.	Capric acids. Grm.
	In aqueous solution				2.10			
	In recovered ether				0.24			
1	43.54	35/83	Atmospheric		0.97			
2	3.65	83/86	"		0.75			
3	1.96	88/156	"		1.59			
4	5.11	156/165	"		4.64			
5	6.30	165/170	"	95.4	4.27	2.03		
6	8.28	170/183	"	97.8	4.84	3.44		
7	1.47	142/155	Reduced	111.8	0.17	1.30		
8	4.23	155/167	"	119.0		3.68	0.55	
9	2.75	Residue		164.4			0.65	2.10
Totals:					19.57	10.45	1.20	2.10

COMPOSITION OF ACIDS NON-VOLATILE IN STEAM.—A portion of the non-volatile acids (303 grms.) was treated with lead acetate (212 grms.) in boiling alcohol (2430 c.c.). The deposited lead salts were separated and recrystallised from an equal volume of alcohol. The recrystallised lead salts were re-converted into fatty acids (solid acids S, 170.3 grms.), whilst the alcoholic solutions were united and the dissolved lead salts contained therein also converted back to fatty acids (liquid acids L, 130.3 grms.):

	Per Cent.	Present in 551.1 grms.	Mean equivalent.	Iodine value.
Solid acids S	56.7	312.2	259.2	101.5
Liquid acids L	43.3	238.9	257.3	82.7

Each group of acids was converted into neutral methyl esters, and these were fractionally distilled in the usual way from a Willstätter bulb under high vacuum; in both cases it was only necessary to re-fractionate the first fraction:

METHYL ESTERS OF SOLID ACIDS S.

Primary fractionation.					Refractionation.				
No.	Grms.	B.pt./ 1 mm. °C.	Saponi- fication equivalent.	Iodine value.	No.	Grms.	B.pt./ 1 mm. °C.	Saponi- fication equivalent.	Iodine value.
S1	52.71	70-130	254.2	2.6	S11	0.79	86-105	217.5	—
					S12	1.44	105-115	231.6	—
					S13	5.45	115-120	242.0	—
					S14	9.94	120-123	248.2	1.2
					S15	11.23	123-130	255.5	1.2
					S16	4.63	130-132	264.1	1.9
					S17	9.39	132-135	270.2	2.7
					S18	5.71	Residue	277.5	9.8
					48.58				
S2	48.07	130-132	273.2	6.2					
S3	13.01	132-135	280.6	12.0					
S4	13.22	135-136	287.6	18.0					
S5	21.14	136-144	294.6	22.0					
S6	8.84	144-148	296.4	21.5					
S7	7.62	Residue	305.0	20.6					
164.61									

METHYL ESTERS OF LIQUID ACIDS L.

Primary fractionation.					Refractionation.				
No.	Grms.	B.pt./ 1 mm. °C.	Saponi- fication equivalent.	Iodine value.	No.	Grms.	B.pt./ 1 mm. °C.	Saponi- fication equivalent.	Iodine value.
L1	33.51	44-130	232.8	34.1	L11	0.64	39-50	161.4	8.6
					L12	1.06	50-66	171.8	8.6
					L13	2.34	66-69	186.4	14.2
					L14	2.84	69-92	198.2	12.5
					L15	3.57	92-105	217.2	12.1
					L16	4.48	105-114	236.2	22.7
					L17	5.20	114-115	243.5	26.0
					L18	9.52	Residue	275.4	67.7
						29.65			
L2	16.55	130-140	288.0	76.2					
L3	40.91	140-150	296.3	96.8					
L4	12.67	150-152	297.7	98.0					
L5	11.44	152-155	296.6	99.8					
L6	8.99	Residue	325.5	108.4					
	124.07								

The primary residues were saponified, and any unsaponifiable matter present extracted by means of ether from the aqueous solution of the potassium salts. The fatty acids, freed from unsaponifiable matter, were recovered, and their equivalents and iodine values re-determined. These figures, which are more accurate than those for the small proportions of unsaponifiable matter present, have been employed in calculating the amount of the latter.

The amounts of each ester present can now be calculated (in the esters of liquid acids L the assumption is made that the unsaturated C_{18} esters present in L1 and L2 had the same relative composition as the first pure C_{18} ester fraction, *i.e.* possessed an iodine value of 96.8).

METHYL ESTERS OF SOLID ACIDS S.

	S11.	S12.	S13.	S14.	S15.	S16.	S17.	S18.	Total.	Per cent. as esters.	Per cent. as fatty acids.
	Grms.	Grms.	Grms.	Grms.	Grms.	Grms.	Grms.	Grms.	Grms.		
Laurate	0.68	0.49	—	—	—	—	—	—	1.17	2.4	
Myristate	0.11	0.95	5.45	7.66	5.63	0.97	0.15	—	20.92	43.1	
Palmitate	—	—	—	2.14	5.44	3.56	8.95	4.01	24.10	49.6	
Stearate	—	—	—	—	—	—	—	1.05	1.05	2.2	
Oleate	—	—	—	0.14	0.16	0.10	0.29	0.65	1.34	2.7	

	S1	S2.	S3.	S4.	S5.	S6.	S7.			
Laurate	1.27	—	—	—	—	—	—	1.27	0.8	0.8
Myristate	22.70	—	—	—	—	—	—	22.70	13.8	13.7]
Palmitate	26.15	41.63	7.64	4.45	2.01	0.30	—	82.18	49.9	49.9
Stearate	1.14	2.96	3.55	6.00	13.70	6.33	3.60	37.28	22.7	22.8
Arachidate	—	—	—	—	—	—	2.19	2.19	1.3	1.3
Oleate	1.45	3.48	1.82	2.77	5.43	2.21	1.83	18.99	11.5	11.5

METHYL ESTERS OF LIQUID ACIDS L.

	L11.	L12.	L13.	L14.	L15.	L16.	L17.	L18.	Total.	Per cent. as esters.	Per cent. as fatty acids.
	Grms.	Grms.	Grms.	Grms.	Grms.	Grms.	Grms.	Grms.	Grms.		
Caproate	0.08	—	—	—	—	—	—	—	0.08	0.3	
Caprylate	0.50	0.67	0.67	—	—	—	—	—	1.84	6.2	
Caprate	—	0.30	1.33	2.20	0.49	—	—	—	4.32	14.6	
Laurate	—	—	—	0.27	2.63	2.30	1.72	0.44	7.36	24.8	
Myristate	—	—	—	—	—	1.13	2.08	2.43	5.64	19.0	
Oleate	0.05	0.08	0.30	0.32	0.39	0.92	1.22	5.81	9.09	30.7	
Linoleate	0.01	0.01	0.04	0.05	0.06	0.13	0.18	0.84	1.32	4.4	

	L1.	L2.	L3.	L4.	L5.	L6.			
Caproate	0.09	—	—	—	—	—	0.09	0.1	0.1
Caprylate	2.08	—	—	—	—	—	2.08	1.7	1.6
Caprate	4.88	—	—	—	—	—	4.88	3.9	3.8
Laurate	8.32	—	—	—	—	—	8.32	6.7	6.6
Myristate	6.37	0.94	—	—	—	—	7.31	5.9	5.9
Palmitate	—	2.48	—	—	—	—	2.48	2.0	2.0
Oleate	10.28	11.38	35.74	10.89	9.60	6.87	84.76	68.3	68.6
Linoleate	1.49	1.65	5.17	1.78	1.84	1.31	13.24	10.7	10.7
Unsaponifiable	—	—	—	—	—	0.81	0.81	0.7	0.7

The whole of the experimental data for the original mixed fatty acids are then combined as follows:

NEW ZEALAND BUTTER-FAT "A."

Acid.	Volatile acids. Grms.	Acids non-volatile in steam.		Total. Grms.	Per cent. excluding unsaponi- fiable matter.
		Solid acids S. Grms.	Liquid acids L. Grms.		
	33.32	312.3	238.8	584.42	
Butyric ..	19.57	—	—	19.57	3.4
Caproic ..	10.45	—	0.16	10.61	1.8
Caprylic ..	1.20	—	3.85	5.05	0.9
Capric ..	2.10	—	9.16	11.26	1.9
Lauric ..	—	2.37	15.78	18.15	3.1
Myristic ..	—	42.77	13.97	56.74	9.7
Palmitic ..	—	155.80	4.77	160.57	27.6
Stearic ..	—	71.04	—	71.04	12.2
Arachidic (?) ..	—	4.14	—	4.14	0.7
Oleic ..	—	36.18	163.87	200.05	34.3
Linoleic ..	—	—	25.60	25.60	4.4
(Unsaponifiable) ..	—	—	1.64	1.64	—

The New Zealand butter-fats "B" and "C" were investigated by the same method, except that in the case of "C" the quantity of fat employed was only about half of that used in the other instances. The final results are given in the next tables:

NEW ZEALAND BUTTER-FAT "B."

Acid.	Volatile acids. Grms.	Acids non-volatile in steam.		Total. Grms.	Per cent. (excluding unsaponifiable matter).
		Solid acids S. Grms.	Liquid acids L. Grms.		
	32.71	369.2	178.2	580.11	
Butyric ..	18.09	—	—	18.09	3.1
Caproic ..	11.07	—	—	11.07	1.9
Caprylic ..	0.85	—	3.73	4.58	0.8
Capric ..	2.70	1.10	7.71	11.51	2.0
Lauric ..	—	7.82	14.93	22.75	3.9
Myristic ..	—	48.85	12.23	61.08	10.6
Palmitic ..	—	162.54	0.14	162.68	28.1
Stearic ..	—	49.12	—	49.12	8.5
Arachidic (?) ..	—	5.60	—	5.60	1.0
Oleic ..	—	94.17	116.73	210.90	36.4
Linoleic ..	—	—	21.39	21.39	3.7
(Unsaponifiable) ..	—	—	1.34	1.34	—

NEW ZEALAND BUTTER-FAT "C."

Acid.	Volatile acids. Grms.	Acids non-volatile in steam.		Total. Grms.	Per cent. (excluding unsaponifiable matter).
		Solid acids S. Grms.	Liquid acids L. Grms.		
	17.05	162.8	136.3	316.15	
Butyric ..	10.11	—	—	10.11	3.2
Caproic ..	5.24	—	—	5.24	1.7
Caprylic ..	0.93	—	1.73	2.66	0.8
Capric ..	0.32	—	6.86	7.18	2.3
Lauric ..	0.45	1.60	11.46	13.51	4.3
Myristic ..	—	20.10	13.83	33.93	10.8
Palmitic ..	—	88.95	0.53	89.49	28.4
Stearic ..	—	29.50	—	29.50	9.4
Arachidic (?) ..	—	2.50	—	2.50	0.5
Oleic ..	—	20.14	84.11	104.25	33.1
Linoleic ..	—	—	16.81	16.81	5.4
(Unsaponifiable) ..	—	—	0.97	0.97	—

In the next table are collected the characteristics of each of the butter-fats, together with the final results of the fractionation analyses, and the equivalents and iodine values of the mixed fatty acids and fats calculated from the latter figures:

MIXED FATTY ACIDS OF NEW ZEALAND BUTTERS.

BUTTER-FAT:	"A"	"B"	"C"
Sap. Equiv.	247.5	250.7	249.5
Iodine value	38.0	39.4	39.3
Reichert-Meissl value	28.4	25.8	25.4
Polenske value	1.9	2.3	2.1
Kirschner value	23.7	20.9	20.3
Butyric acid (calculated from Kirschner value) ..	4.2	3.7	3.6

ACIDS BY FRACTIONATION ANALYSIS:				"A"	"B"	"C"
				Per Cent.	Per Cent.	Per Cent.
Butyric	3.4	3.1	3.2
Caproic	1.8	1.9	1.7
Caprylic	0.9	0.8	0.8
Capric	1.9	2.0	2.3
Lauric	3.1	3.9	4.3
Myristic	9.7	10.6	10.8
Palmitic	27.6	28.1	28.4
Stearic	12.2	8.5	9.4
Arachidic (?)	0.7	1.0	0.5
Oleic	34.3	36.4	33.1
Linoleic	4.4	3.7	5.4

CALCULATED MEAN VALUES (from fractionation analyses):

Sap. equiv.	Fatty acids	238.2	238.0	237.8
	Glycerides	250.9	250.7	250.5
Iodine value.	Fatty acids	38.9	39.5	39.6
	Glycerides	36.9	37.5	37.6

Consideration of these figures should be prefaced by a word as to the limits of accuracy, qualitative and quantitative, of our experiments. Since the majority of the fatty acids of butter have been definitely recognised for many years, we have only formally identified our products in certain cases. We may point out, in passing, that individual esters, when predominating in the distillates, were readily recognised by their boiling-points at the pressure employed, and that, in particular, the greater part of the methyl palmitate present was obtained in fractions in which it was the main component and which crystallised in the well-defined form characteristic of this ester. Methyl stearate was also definitely recognised, both as ester and in the form of stearic acid isolated therefrom.

The small proportions of fatty acid calculated as "arachidic" represent increments of a fatty acid of higher molecular weight than stearic (calculated after elimination of any accompanying unsaponifiable matter); the amount present was so small that it was not possible to obtain any fraction rich in this material, and we have not therefore been able positively to identify it. In these circumstances we have calculated it in terms of the next even-member acid higher in the series than stearic, *i.e.* as arachidic acid. The error, if any, is small, owing to the minor amount present.

With reference to the linoleic acid content of butter-fat, we would point out that the presence of an acid more unsaturated than oleic is consistently borne out by the iodine values of the C_{18} unsaturated esters obtained in fractionation of the liquid acids (*cf.* pp. 81, 83). The major C_{18} liquid ester fractions from the respective butters possessed the following characteristics:

				Sap. equiv.	Iodine value.
"A"	296.3	96.8
"B"	293.6	99.3
"C"	293.5	97.2

We experienced more difficulty than had been anticipated in preserving the freshly-distilled unsaturated C_{18} esters from atmospheric oxidation, and it is consequently probable that the figures for linoleic acid are, if anything, on the low side

(especially in butter-fat "B"). Absorption of oxygen and diminution in iodine value set in rapidly when these esters are stored, and we have adopted the practice of placing such fractions in rubber-stoppered bottles in an atmosphere free from oxygen immediately they have been collected.

A comparison of the data obtained (*cf.* table, pp. 83, 84) shows that the mean equivalents of the mixed fatty acids, recalculated from the fractionation data, correspond fairly closely with those of the original fats, whilst the calculated iodine values are slightly, but consistently, low, probably for the reason just stated.

As regards the figures for individual acids, it appears probable from the results for "B" and "C" (two almost identical fats) that the mean error is only a few tenths per cent. except in the case of the saturated acids of highest molecular weight and the unsaturated acids. It may be repeated that we believe that this method of analysis is reliable to the nearest whole number (per cent.) (*cf.* ANALYST, 1927, 52, 253), and we are disinclined to lay much stress on fractional values. Nevertheless, in the case of butter-fats the small proportions of the lower acids present unfortunately render this course necessary to some extent.

The data for butyric-capric acids, however, are concordant in their relationship to the observed Kirschner values, and are also within the comparatively narrow range for each acid assigned by Holland and Buckley (*loc. cit.*).

The relation of the observed butyric acid content to that calculated on the assumption that the Kirschner value is a simple measure of butyric acid would seem to show that the latter registers, in terms of butyric acid, about 15–20 per cent. more than is actually present in the fat.

The unsaturated acids present are very similar in composition to those present in tallow, and consist of oleic acid admixed with about 12–15 per cent. of linoleic acid; the total unsaturated acid content of the mixed fatty acids now studied was 38.5–41 per cent.

The data for the higher saturated fatty acids, in which so much variation occurs in the earlier literature, are consistent in showing a content of 9.7–10.8 per cent. of myristic, and 27.6–28.4 per cent. of palmitic acid; whilst the observed values for stearic acid are 8.5, 9.4 and 12.2 per cent. We believe that the analyses establish palmitic acid as the predominating saturated component of these butter fatty acids, whilst they also show that myristic and stearic acids are each present to the extent of rather more than one-third of the weight of palmitic acid.

Mitchell (ANALYST, 1924, 49, 515) has recently obtained figures from which he concludes that the stearic acid content of butter-fats may range from practically nothing to 22 per cent. at least. It is noteworthy that in the three samples now examined, all otherwise closely similar, there is a wider variation in the stearic acid figures than in those of any of the other saturated acids.

From the fundamental standpoint it is perhaps more important to compare the *relative molecular quantities* of the acids combined in a natural fat, and therefore it is interesting to tabulate the number of equivalents of each fatty acid present in the three butter-fats and in tallow; for this purpose we have employed figures for a mutton tallow recently obtained in this laboratory, and for an Australian

beef tallow as recorded by Armstrong and Allan (*J. Soc. Chem. Ind.*, 1924, 43, 216T):

Acid.	Australian beef tallow.		Mutton tallow.
	Per Cent.		Per Cent.
Myristic	2.0	4.6
Palmitic	26.5	24.6
Stearic	22.5	30.5
Oleic	49.0	36.0
Linoleic	—	4.3

Composition of Fatty Acids in Equivalents (per cent.).

Acid.	New Zealand Butter-fats.			Tallows.	
	A.	B.	C.	Australian beef.	Mutton.
	Butyric	9.2	8.4	8.7	—
Caproic	3.7	3.9	3.4	—	—
Caprylic	1.4	1.3	1.4	—	—
Capric	2.7	2.8	3.1	—	—
Lauric	3.7	4.6	5.1	—	—
Myristic	10.2	11.0	11.2	2.4	5.4
Palmitic	25.7	26.2	26.3	28.3	26.3
Stearic	10.2	7.1	7.8	21.7	29.3
Arachidic	0.5	0.8	0.6	—	—
Oleic and linoleic	32.7	33.9	32.4	47.6	39.0

The following points may be noted:

(i) One hundred molecules of the mixed fatty acids include in all cases about 26 molecules of palmitic acid (how far this is a general rule in the case of tallows is of course uncertain; the average of four other available analyses of tallows, however, shows 27.6 mols. of palmitic acid per 100 mols. of mixed fatty acids).

(ii) The presence of the fatty acids lower than palmitic in butter-fat is balanced by a lower molecular content of C_{18} acids, as compared with the tallows—the palmitic acid figure remaining about the same.

(iii) About one-third of the molecules of the mixed fatty acids of the butters are those of oleic and linoleic acids.

(iv) Butyric, myristic and stearic acids are present in something approximating to equimolecular proportions in the butter-fats examined.

(v) The six lowest-molecular-weight acids of the butter-fats can be arranged as follows in pairs which correspond, roughly, in their respective molecular proportions:

Butyric (C_4), 8.4–9.2 per cent., and myristic (C_{14}), 10.2–11.2 per cent.

Caproic (C_6), 3.4–3.9 per cent., and lauric (C_{12}), 3.7–5.1 per cent.

Caprylic (C_8), 1.3–1.4 per cent., and capric (C_{10}), 2.7–3.1 per cent.

The circumstance that this approximate relationship may subsist between pairs of acids which respectively make up unit groups of 18 carbon atoms, together with the comparative deficiency of butter-fat in stearic acid as compared with

tallow, may not be without significance from a biochemical standpoint; but we hesitate, in the absence of a much wider series of analyses, to do more than draw attention to what is, perhaps, only a coincidence.

INVESTIGATION OF THE COMPONENT GLYCERIDES OF BUTTER-FAT.—As already stated, the procedure adopted has been to oxidise butter-fat until all unsaturated linkages have been converted into free acidic groups, leaving only the original fully-saturated glycerides in the form of neutral compounds. The latter have then been freed as completely as possible from acidic products of oxidation, and their weight noted; after which the composition of their mixed fatty acids has been determined precisely as in the case of the original butter-fat, except that, of course, the lead-salt separation has been omitted, unsaturated acids now being absent.

The distribution of the acids in the fully-saturated and the mixed saturated-unsaturated glycerides has then been arrived at by comparison with the analytical data for the original butter-fats: direct analysis of the fatty acids in the acidic oxidation products is impracticable owing to the impossibility at present of effecting quantitative removal of nonoic acid (one of the free acidic products) without concurrent decomposition of the acidic glyceride compounds.

Butter-fats "A" and "B" have been submitted to this treatment, which will be briefly described before summarising the analytical figures which have been obtained.

The fat (1 part) was dissolved in acetone (10 parts), and powdered potassium permanganate (4 parts) was added in small quantities at a time (with vigorous shaking at each addition), while the solution was gently boiled under a reflux condenser; boiling was continued for a short time after addition of the oxidant had been completed, after which as much acetone as possible was removed by distillation and treatment of the residue at 90–100° C. under reduced pressure. In order to minimise any chance of glyceride-hydrolysis in the alkaline solution (by potassium hydroxide possibly liberated from the permanganate), the resulting friable powder was mixed with powdered sodium bisulphite, and then with water; the aqueous mixture was warmed and cautiously decolorised by the gradual addition of dilute sulphuric acid with vigorous stirring; after cooling, the solution was thoroughly extracted with ether.

The united ethereal extracts, after washing with water, were shaken repeatedly with small quantities of dilute ammonia, followed (when the whole was definitely alkaline) by thorough washing with water to remove as much of the organic ammonium salts as possible. The ammoniacal and aqueous liquors were re-extracted with ether to remove any neutral glycerides present in the emulsified condition, and all the ethereal extracts were then concentrated and the residual neutral product dried.

If the iodine value of the neutral product was appreciable (*e.g.* above 0.5), the material was submitted to a repetition of the oxidation process. Eventually, crude neutral products with an iodine value of 0.3 or less were obtained; in this

condition, however, they still possessed a small acid value due to the retention of traces of the difficultly-removable acidic glyceride compounds (*e.g.* of the type $C_8H_5(O.CO.R)_2(O.CO.[CH_2]_7.COOH)$, where *R* represents a higher saturated fatty acid radicle).

They were therefore boiled in water to which dilute sodium carbonate was added until the whole was definitely alkaline to phenolphthalein. The aqueous layer (containing some emulsified neutral glycerides) was separated from the main fatty portion, which formed a clear upper layer; the latter was boiled several times with water until the washings remained neutral. By this means 80 to 90 per cent. of the crude neutral product was obtained in the form of material of negligible acid value, whilst ether-extraction of the united alkaline and aqueous wash-liquors furnished the remainder (10–15 per cent. of the whole) as a substance which still possessed a definite, though low, acid value. This acidity has been allowed for by assuming that it is due to the presence of acidic compounds of the same order as those removed by the sodium carbonate from the crude product (the free acidic compounds from the extracted alkaline wash-liquors having been isolated and their acid value determined).

The data thus obtained are sufficient to determine the proportion of fully-saturated glycerides present (to within one per cent.), and it then only remains to accumulate sufficient of these (a minimum of 150 grms. is desirable) for accurate determination of their fatty acid composition according to the scheme given on pp. 78–82.

FULLY-SATURATED GLYCERIDES OF BUTTER-FAT "A."—The fat yielded, as a result of complete oxidation, 33.6 per cent. of crude neutral products; oxidations were conducted on six batches of 100 grms. each, in order to provide sufficient material for detailed analysis.

On boiling the crude neutral product with dilute sodium carbonate, as described above, there were obtained:

- (a) 163.8 grms. completely neutral fat, sap. equiv. 229.3 (acid value 0.4);
- (b) 22.9 grms. fat extracted by ether, sap. equiv. 234.1 (acid value 6.4);
- (c) 12.5 grms. acidic material, sap. equiv. 167.9 (acid value 211.2).

Assuming that the acidic matter present in (b) has the same acid value as (c), the proportion of fully-saturated glycerides in the original fat is

$$\frac{33.6}{199.2} \left(163.8 + \frac{22.9 \times 204.8}{211.2} \right) = 31.3 \text{ per cent.}$$

The value 31 per cent. of fully-saturated glycerides in butter-fat "A" has been used in the subsequent calculations.

COMPOSITION OF THE FATTY ACIDS PRESENT IN THE FULLY-SATURATED GLYCERIDES OF BUTTER-FAT "A."

Analysis of the combined neutral products (a) and (b) gave results which are summed up in the following table :

Acid.	Volatile acids. Grms.	Acids non-volatile in steam. Grms.	Total. Grms.	Per cent. (excluding unsaponifiable matter).
	14.77	157.37	172.14	
Butyric	7.50	—	7.50	4.4
Caproic	5.79	—	5.79	3.4
Caprylic	0.75	1.37	2.12	1.2
Capric	0.62	3.83	4.45	2.6
Lauric	0.11	6.26	6.37	3.7
Myristic	—	31.76	31.76	18.5
Palmitic	—	78.78	78.78	45.9
Stearic	—	34.99	34.99	20.3
(Unsaponifiable) ..	—	0.38	0.38	—

FULLY-SATURATED GLYCERIDES OF BUTTER-FAT "B."—After complete oxidation the original fat yielded 33.5 per cent. of crude neutral products, which on boiling with dilute sodium carbonate gave:

- (a) 134.6 grms. completely neutral fat sap. equiv. 232.5 (acid value 0.3);
- (b) 9.5 grms. fat extracted by ether, sap. equiv. 232.2 (acid value 15.4);
- (c) 19.3 grms. acidic material, sap. equiv. 193.6 (acid value 104.9).

The proportion of fully-saturated glycerides is therefore

$$\frac{33.5}{163.4} \left(134.6 + \frac{9.5 \times 89.5}{104.9} \right) = 29.2 \text{ per cent.}$$

The value 29 per cent. of fully-saturated glycerides in butter-fat "B" has been used in subsequent calculations.

COMPOSITION OF THE FATTY ACIDS PRESENT IN THE FULLY-SATURATED GLYCERIDES OF BUTTER-FAT "B."

The final results of this analysis are summarised in the next table.

Acid.	Volatile acids. Grms.	Acids non-volatile in steam. Grms.	Total. Grms.	Per cent. (excluding unsaponifiable matter).
	12.2	115.3	127.5	
Butyric	5.35	—	5.35	4.2
Caproic	3.35	—	3.35	2.6
Caprylic	3.50*	0.77	4.27	3.3
Capric	—	3.04	3.04	2.4
Lauric	—	5.54	5.54	4.3
Myristic	—	22.44	22.44	17.6
Palmitic	—	58.25	58.25	45.7
Stearic	—	24.93	24.93	19.6
Arachidic	—	0.33	0.33	0.3

* This figure is almost certainly high at the expense of correspondingly low values for caproic and capric acids, owing to an unfortunately incomplete separation at the close of the fractional distillation of the volatile acids in this analysis.

DISTRIBUTION OF THE FATTY ACIDS IN THE GLYCERIDES OF BUTTER-FAT.—
The next tables show the general composition of 100 parts of the glycerides of butter-fats "A" and "B," as indicated by the foregoing analyses.

BUTTER-FAT "A."

			Original fat.	Fully- saturated glycerides.	Mixed saturated- unsaturated glycerides (by difference).	
			100	31	69	
Glycerol residue	5.1	1.7	3.4	
						(Molecular ratios).
Butyric acid	3.2	1.3	1.9	22
Caproic	1.7	1.0	0.7	6
Caprylic	0.9	0.3	0.6	3
Capric	1.8	0.8	1.0	6
Lauric	2.9	1.1	1.8	9
Myristic	9.2	5.4	3.8	17
Palmitic	26.2	13.4	12.8	50
Stearic	11.6	6.0	5.6	20
Arachidic	0.7	—	0.7	2
Oleic	32.5	—	32.5	115
Linoleic	4.2	—	4.2	15

BUTTER-FAT "B."

			Original fat.	Fully- saturated glycerides.	Mixed saturated- unsaturated glycerides (by difference).	
			100	29	71	
Glycerol residue	5.1	1.6	3.5	
						(Molecular ratios).
Butyric acid	2.9	1.1	1.8	20
Caproic	1.8	0.7	1.1	9
Caprylic	0.8	0.9*	(-0.1)	—
Capric	1.9	0.7	1.2	7
Lauric	3.7	1.2	2.5	13
Myristic	10.0	4.8	5.2	23
Palmitic	26.6	12.5	14.1	55
Stearic	8.1	5.4	2.7	9
Arachidic	1.0	0.1	0.9	3
Oleic	34.6	—	34.6	123
Linoleic	3.5	—	3.5	12

* This figure is almost certainly high, *cf.* footnote to Table, p. 89.

On the whole, the series of analyses for the two fats (which, it will be remembered, differ mainly in that "A" has somewhat higher Reichert-Meissl and Kirschner values than "B") are in fair accordance and show clearly:

(a) That the proportion of fully-saturated glycerides in these fats is of the order of 30 per cent.;

(b) That all the saturated acids are distributed more or less evenly throughout both the fully-saturated and the mixed saturated-unsaturated parts of the fat. Recent work on the Reichert-Meissl, Kirschner, iodine and other values of fractions of Irish butter-fat separated by chilling at various temperatures has led Arup (ANALYST, 1928, 53, 641) to a similar conclusion.

The general distribution of the saturated fatty acids is made clearer by comparing the proportions of these acids present in the whole fat, the fully-saturated part, and the mixed saturated-unsaturated part. The tables which follow give these data for each fat both in the form of weight-percentages and of molecular percentages (the proportionate numbers of *equivalents* of each acid present).

RELATIVE COMPOSITION OF THE SATURATED FATTY ACIDS.

(i) WEIGHT PERCENTAGES.

Acid.	Butter-fat "A."			Butter-fat "B."		
	Whole fat.	Fully-saturated part.	Mixed saturated-unsaturated part.	Whole fat.	Fully-saturated part.	Mixed saturated-unsaturated part.
Butyric ..	5.5	4.4	6.7	5.2	4.2	6.1
Caproic ..	2.9	3.4	2.5	3.2	2.6	3.7
Caprylic ..	1.5	1.2	1.7	1.3	3.3*	—*
Capric ..	3.1	2.6	3.6	3.3	2.4	4.2
Lauric ..	5.1	3.7	6.4	6.5	4.3	8.5
Myristic ..	15.8	18.5	13.1	17.7	17.6	17.7
Palmitic ..	45.0	45.9	44.3	46.9	45.7	47.8
Stearic ..	19.9	20.3	19.4	14.2	19.6	9.0
Arachidic ..	1.2	—	2.3	1.7	0.3	3.0

(ii) MOLECULAR PERCENTAGES.

Acid.	Butter-fat "A."			Butter-fat "B."		
	Whole fat.	Fully-saturated part.	Mixed saturated-unsaturated part.	Whole fat.	Fully-saturated part.	Mixed saturated-unsaturated part.
Butyric ..	13.6	11.0	16.2	12.7	10.5	14.6
Caproic ..	5.4	6.5	4.6	6.0	4.9	6.7
Caprylic ..	2.3	1.8	2.5	1.9	5.0*	—*
Capric ..	3.9	3.3	4.5	4.1	3.1	5.2
Lauric ..	5.5	4.1	6.8	7.0	4.7	9.0
Myristic ..	15.0	17.9	12.3	16.8	17.0	16.4
Palmitic ..	38.2	39.6	36.9	39.5	39.3	39.4
Stearic ..	15.2	15.8	14.6	10.8	15.2	6.7
Arachidic ..	0.9	—	1.6	1.2	0.3	2.0

* Cf. footnotes to Tables on pp. 89, 90.

Bearing in mind the undesirability of attaching too great importance to the values for caproic-lauric acids (which are present in amounts too small for accurate determination by the methods employed), it nevertheless appears that there is a definite, though slight, tendency for the lower fatty acids to associate with the unsaturated fatty acids more than with the higher saturated acids. This is compensated for by a slight corresponding concentration of myristic, palmitic

and stearic acids in the fully-saturated glycerides; but it is noteworthy that the relative proportions of palmitic acid vary less than those of the other acids. Palmitic acid, indeed, appears to stand somewhat apart in its general relationships from the other acids throughout the whole series of analyses—a feature which we believe is characteristic of this acid in many other fats of vegetable, as well as animal origin.

We have not yet attempted to separate the fully-saturated glycerides by selective crystallisation from an appropriate solvent, but their free solubility in ether and acetone leads us to believe that they consist of a complex system of mixed glycerides, and to share the view of other workers that simple triglycerides are not present; from the general characteristics and properties of the material it does not appear probable, moreover, that glycerides containing only palmitic and stearic acids are present in any notable proportion.

COMPOSITION OF THE MIXED SATURATED AND UNSATURATED GLYCERIDES.—The numerical data which we have obtained permit us to estimate the relative molecular proportions of saturated and unsaturated fatty acids combined in this section of the fat, and therefrom to give certain limiting figures for the amounts of mono-oleo- and di-oleo- glycerides and of triolein which may be present (in discussing this aspect of the results, the unsaturated matter is referred to for simplicity as though it were all made up of oleic acid). It would be possible to assign definite values to each of these three groups if, for example, we were able to obtain an independent figure for the percentage of triolein present; but our attempts to devise a procedure to this end have hitherto been unsuccessful.

We have two almost independent methods available for determining the molecular ratio of saturated and unsaturated acids combined in the mixed saturated-unsaturated glycerides of the fat:

(i) Given the percentage of fully-saturated glycerides present, the mean equivalents of these and of the original fat, and the proportion of saturated acids in the original fat, the mean equivalent, α , of the saturated acids linked with unsaturated acids in mixed glycerides can be directly obtained, and hence the molecular ratio of the saturated and unsaturated acids:

$$\begin{array}{c} \text{Acids in 100 grms. fat} \\ \hline \text{Mean equivalent of} \\ \text{total fatty acids.} \end{array} = \begin{array}{c} \text{Acids in fully-saturated part} \\ \hline \text{Mean equivalent of these} \\ \text{acids.} \\ \begin{array}{ccc} \text{oleic} & & \text{linoleic} \\ \text{acid} & & \text{acid} \\ + & & + \\ \hline 282 & & 280 \end{array} \\ + \frac{\text{saturated acids}}{\text{in mixed part}} \\ \hline \alpha \end{array}$$

This gave the following results when applied to the experimental data:

	Mean equivalent of saturated acids linked with unsaturated acids.	Ratio of mols. saturated acids per 100 mols. unsaturated acids.
Butter-fat "A"	207.9	106 : 100
" " "B"	211.8	103 : 100

(ii) From the differential determinations of each individual acid recorded on p. 90, the molecular ratios of the saturated and unsaturated acids can be directly derived (the ratios for each acid have been inserted in the fifth column of the tables indicated); these give the following values:

					Ratio of mols. saturated acids per 100 mols. unsaturated acid.
Butter-fat "A"	104 : 100
" " "B"	104 : 100

The agreement between the respective estimates for each fat is satisfactorily close, and the mean ratios 105: 100 for butter-fat "A" and 103.5 : 100 for butter-fat "B" have been employed in the calculations which follow. It is readily possible, knowing this ratio, to deduce the general composition of the fats on the successive hypotheses that either dioleo-glycerides or triolein are completely absent (absence of mono-oleo-glycerides is inconsistent with the observed figures); the resulting data are as follows:

Molecular percentages.			Weight percentages.		
Mono-"oleo"- glycerides. Per Cent.	Di-"oleo"- glycerides. Per Cent.	Tri- "olein." Per Cent.	Mono-"oleo"- glycerides. Per Cent.	Di-"oleo"- glycerides. Per Cent.	Tri- "olein." Per Cent.
<i>Butter-fat "A."</i>					
54	46	—	51	49	—
77	—	23	74	—	26
<i>Butter-fat "B."</i>					
53	47	—	50	50	—
76	—	24	73	—	27

Therefore, in each of the original butter-fats, there cannot be *less* than about 36 per cent. of mono-oleo-disaturated glycerides, nor can there be *more* than 18 per cent. of tri-olein (or 35 to 36 per cent. of di-oleo-monosaturated glyceride). The actual values lie somewhere between the limiting figures given; in the absence of any direct method for determining triolein, this is as far as the analytical data take us.

We venture to predict, however, that the actual values are not widely removed from those for a mixture of mono-oleo-disaturated and dioleo-monosaturated glycerides. Our belief that triolein is not likely to be present in any large proportion is based (i) on the argument that, since it is now tolerably evident that simple triglycerides of the saturated acids are either absent from, or present in only minute amounts in butter and most other solid fats, there is no reason to suppose that oleic acid will tend to form triolein in large proportions, and (ii) on the absence of any positive indication of its occurrence in any fat hitherto investigated which contains sufficient saturated acid to provide mixed glycerides with all the oleic acid present.

SUMMARY.—Methods have been developed for the determination of the proportions of each of the fatty acids contained in butter fat, and for a semi-quantitative determination of the manner in which the acids are combined to form the component glycerides of the natural fat. The investigation has been carried out in connection with three samples of New Zealand butter, all of which gave results of a similar order.

The composition of the mixed fatty acids has been obtained by:

(i) Removing as much of the lower fatty acids as possible by prolonged distillation in steam, the steam-volatile acids being recovered and fractionally distilled in the form of free acids;

(ii) Separating the fatty acids non-volatile in steam into two groups by means of the lead salt and alcohol method, followed by conversion of the acids from the soluble and insoluble lead salts into methyl esters, which were quantitatively fractionated at low pressure in the usual manner.

The approximate composition of the fatty acids was: Butyric, 3; caproic, 2; caprylic, 1; capric, 2; lauric, 4; myristic, 11; palmitic, 28; stearic, 9; oleic, 33–34; and linoleic, 4–5 per cent. The values for the volatile fatty acids accord with recent determinations by other workers; the values for myristic, palmitic and stearic acids differ from many previously recorded, whilst there is consistent evidence of the presence of a small percentage of acids less saturated than oleic.

The procedure for the study of the component glycerides consisted in oxidising the butter-fat under conditions in which all unsaturated components were transformed into acidic products, whilst glycerides containing only saturated fatty acids remained unaltered. The latter were recovered, their proportion noted, and the composition of the mixed fatty acids contained therein determined as above. From the resulting data the following general conclusions were drawn:

(i) The butter-fats examined contained about 30 per cent. of fully-saturated glycerides. The fatty acids therein were the same as those in the whole fat, and in proportions not widely different from those of the latter; but a tendency was noted for the occurrence of somewhat less of the volatile fatty acids in the fully-saturated part, coupled with a correspondingly slight concentration of the higher saturated fatty acids in this group of the glycerides. It is probable that all these glycerides are of the complex mixed type.

(ii) The remainder of the fat (about 70 per cent.) consisted of mixed glycerides of saturated and unsaturated acids, the molecular proportions of the acids being about 104 mols. of saturated to 100 mols. of unsaturated acid. The amount of mono-oleo-disaturated glycerides in the original fat is *at least* 36 per cent., and there cannot be *more* than 18 per cent. of triolein (or 36 per cent. of dioleo-mono-saturated glycerides); although no positive data are available, it is quite probable

that but little tri-olein is present, and that the approximate composition (in round numbers) of the butter-fats examined is:—

	Per Cent.
Mixed fully-saturated glycerides	ca. 30
Mixed mono-oleo-disaturated glycerides	ca. 36
Mixed di-oleo-monosaturated	ca. 34

It may be noted that all, except the unsaturated acids, are comparatively evenly distributed throughout the whole fat.

These results, of course, do not necessarily hold in detail for butters produced from widely varying sources. The investigation is being extended to butters made from the milk of cows feeding on a variety of diets, including (if possible) cases in which hard and soft oilcakes have been respectively employed in the diet.

We desire to express our most cordial thanks to Messrs. Lever Bros., Ltd., who have assisted us by obtaining supplies of the butters which we have studied, by carrying out some of the determinations of the technical "constants" of the fats, and, especially, by the provision of a Research Studentship in the Department of Industrial Chemistry of this University, which has enabled one of us to pursue the research.

THE UNIVERSITY,
LIVERPOOL.

DISCUSSION.

The PRESIDENT said that in this remarkable and valuable paper (not the first which the Society had had from Professor Hilditch) the authors had dealt in a fundamental manner with a fat which many had to examine daily. He had a suggestion to make on one small point: Professor Hilditch seemed to wonder whether the accuracy of some of his results was not due to coincidence. Would it not be possible to apply this method of fractionation to known mixtures of fatty acids, so as to substantiate the percentage accuracy of his separation?

Mr. E. R. BOLTON remarked that he was struck by the dramatic way in which the paper opened, by pointing out that the data available were in a chaotic condition. Professor Hilditch had gone forward, step by step, and had given us absolutely new information. One of the most striking points was the fact that the butyric acid, as determined by the Kirschner method, was rather in excess of that actually present; this was interesting, as it had always been understood that the Kirschner method afforded a good measure of the butyric acid. Mr. Bolton then referred to the problem of the detection of carcase fat in butter, and mentioned that the problem had become a very live one in India, where certain castes were not allowed to eat any butter containing carcase fat. Government chemists, therefore, had to face this problem, and had considerable difficulty in proving a butter to be free from this fat. If it were possible to bring the stearic acid of pure butter fat within narrower limits than the 0 to 22 per cent. recorded by Mr. Mitchell, to evolve a method for its accurate and rapid determination, and to state that pure butter should not contain more than a certain amount of that acid, it might afford a means of settling the problem of the Indian chemists.

Mr. C. A. MITCHELL said the method devised by Professor Hilditch and his co-worker marked a great advance on the original oxidation method of Hazura

and Grüssner, which gave results of qualitative rather than quantitative value. With regard to Mr. Bolton's suggestion of using the proportion of stearic acid as a means of differentiating between butter fat and animal body fat, this was the goal to which Hehner and he (the speaker) had worked for months, but the idea had had to be abandoned owing to the very wide variations in the stearic acid content of butter fat. Most of the early experiments on the crystallisation of butter fatty acids from a solvent saturated with pure stearic acid gave deposits ranging from a mere trace up to about 6 per cent., but subsequently several undoubtedly pure samples gave deposits of 12 to 22 per cent., and subsequently this had been repeatedly confirmed. It was also an interesting confirmation that the amount of stearic acid found by Holland and Buckley in American butter fat was over 20 per cent.

Dr. H. E. Cox remarked on the fact that the Kirschner value was shown to give an apparent butyric acid content about 20 per cent. too high, and asked for information as to exactly what acids were represented by the Reichert-Meissl, Polenske and Kirschner values, respectively. There was sometimes observed in genuine butter an unusually large spread between the Reichert-Meissl and the Kirschner values, amounting to as much as 25 per cent. or more of the Reichert-Meissl value, which was a larger variation than appeared in Prof. Hilditch's figures for the sum of the capric, caproic and caprylic acids, part of which, with the butyric acid, accounted for the Reichert-Meissl value.

Professor HILDITCH, replying, said that with regard to the President's question as to whether it were not possible to test the accuracy of the results: this had been done in the case of comparatively simple mixtures, and the results could be relied upon, but it had not been done with more complicated mixtures, such as the particularly difficult mixture which resulted when C_4 and C_{10} acids were present. It might be a very good idea to try this.

Mr. Bolton and Dr. Cox had both mentioned the Kirschner number. Professor Hilditch did not think he could add much on this point to what had been said in the reading of the paper. As far as his figures were concerned, the butyric acid figure was more likely to be on the high side than on the low side, and probably the Kirschner value included caproic acid. What he was really trying to get at was the constitution of natural fats, as far as this method would take him, and the examination of the composition of the glycerides contained in the fats; butter fats were one section of the work.

With regard to the percentage of stearic acid in butter fat, on the whole he agreed with Mr. Mitchell. He had found that there was considerable variation.

Referring to the length of time taken by this test, Professor Hilditch stated that at the present time he and his co-workers were trying to see if they could work out a modified test occupying not more than two days. They were also studying the method from the point of view of analytical application. Regarding Dr. Cox's question with reference to the Reichert-Meissl and Polenske values, he really hesitated to say, but it was quite possible that the "spread" which was mentioned existed, but he did not see that this could really be correlated with the results recorded in this paper.

Electrolytic Determination of Lead in Urine.

By T. COOKSEY, Ph.D., B.Sc., F.I.C., AND S. G. WALTON.

NUMEROUS methods, both chemical and electrolytic, have been proposed for the determination of the small amounts of lead occurring in pathological urine, but those who attempt it appreciate the difficulty of determining this substance when present in minute quantity only and mixed with the comparatively large number of other substances, organic and inorganic, which form the normal constituents of urine. Under the legislation of the present day, by which employees who are proved to be suffering from occupational diseases are granted compensation, the accuracy of the determination of lead in urine is of considerable importance in the diagnosis of cases of plumbism due to conditions of employment. A large quantity of literature is available to chemists, describing various methods for the determination of lead, and suggesting modifications of such methods, with a view to obtaining more reliable results. But in all, difficulties more or less considerable are encountered, due to the fact that the lead to be determined must be separated in some form from the large quantity of material present.

In attempting to minimise these difficulties, the following electrolytic method, based on the precipitation of the lead as metal, has been worked out. It possesses certain advantages:—(1) It can be applied to the urine without previous treatment; (2) A very small addition of chemical reagents is required, and these can be very easily freed from lead. The length of time taken to obtain the final result is not appreciably shortened, but the actual time occupied in manipulation is small.

In our hands very satisfactory results have been obtained, and we are now asking that the method may be published in *THE ANALYST*, in order that it may be subjected to wider criticism.

The following are the details of the method:—The acidity of the urine is ascertained, methyl red being used as indicator, and therefrom is determined the amount of acetic acid that it is necessary to add to 500 c.c. of the urine (contained in a suitably shaped beaker) so that the total acid present is approximately equivalent to 3 grms. of acetic acid.

The solution is now electrolysed, the strength of current used being 0.3–0.4 amp. The platinum electrodes, consisting of cone and wire spiral (see Baird and Tatlock's Catalogue, 1928, C.7070) were those used for this purpose. They should be so connected that the lead is deposited on the spiral, which should reach to within 1 cm. of the bottom of the beaker. It is advisable to select a narrow shaped beaker, of 500 c.c. capacity, and 11 cm. high, in order to promote an efficient circulation of the liquid.

When the electrolysis (which can proceed overnight) is finished—after 16 or 17 hours—a small amount of phosphate is sometimes found adhering to the cathode. The beaker containing the urine is carefully removed while the current is still passing, and immediately replaced by one of the same height but half the capacity, containing 250 c.c. of distilled water and 1 c.c. of strong hydrochloric acid. The passage of the current is allowed to continue for 2 hours. At the end of this time it will be found that all the phosphate has been dissolved. This beaker is now replaced (with the current still flowing) by one of the same size containing 250 c.c. of distilled water, which is allowed to remain for half-an-hour. It is then removed and the current switched off.

After that part of the cathode which remained above the level of the liquid has been wiped clean to remove adhering spray, the cathode is washed with alcohol and allowed to dry. The lead is dissolved in 4 c.c. of strong hot nitric acid by pouring the acid over the cathode several times. This solution is transferred to a small glass crystallising dish. The cathode is again treated in the same manner with a hot mixture of 1 c.c. of strong nitric acid and 9 c.c. of distilled water, and finally washed, 5 c.c. of hot water being used.

The whole is evaporated to dryness over the water bath, 1 c.c. strong hydrochloric acid added, and the solution again evaporated to complete dryness. The residue is warmed with 0.1 to 0.2 c.c. of hydrochloric acid and a few drops of distilled water, and 4 c.c. of distilled water added to the solution, which is again warmed on the water bath to ensure complete solution.

The liquid is transferred to a graduated measure and made up to 6 c.c., and 3 c.c. of this solution are transferred to one of a number of small test tubes which are marked at 3 c.c. A standard solution containing 0.00001 grm. of lead per c.c. is prepared and added to a series of comparison tubes (of the same bore and colour) in the following amounts:—0.0 c.c., 0.5 c.c., 1.0 c.c., 1.5 c.c., 2.0 c.c., 2.5 c.c., and 3.0 c.c. To each tube is then added the same amount of hydrochloric acid as that contained in the solution to be tested, and the volume made up to the 3 c.c. mark with distilled water. Two c.c. of a *freshly prepared* saturated solution of potassium metabisulphite are then added to each, and the contents of the tubes are well mixed, and the tubes corked and allowed to stand for 1–2 hours, after which the tube contents are again well mixed, and the turbidity of the sample tubes compared with that of the standards.

If the lead content is heavy, a smaller quantity than 3 c.c. should be taken for the determination, and made up to 3 c.c. with distilled water and sufficient hydrochloric acid to preserve the same acidity as in the standards.

As it may occasionally happen that a small proportion (usually, however, not more than one-tenth) of the lead present is not removed in the first electrolysis, it is advisable to submit the sample and washings to a second treatment. After electrolysis the urine contained in the beaker is heated on the water bath for one hour, cooled, and made up to the original volume with distilled water. In the event of a deposit of phosphate forming in the cathode during the first electrolysis,

0.5 c.c. of glacial acetic acid is added, and the urine again electrolysed overnight. The hydrochloric acid and the water washings from the first electrolysis are combined and heated on the water bath for one hour, cooled, 1 c.c. of strong hydrochloric acid added, and the volume made up to 500 c.c. This solution is used for the first washing of the electrode in the second electrolysis, the current being passed for 2 hours. The wash is immediately replaced by one containing 250 c.c. of distilled water. The cathode is again treated in the same way as in the first electrolysis. Any lead present is added to that found previously.

It is of advantage to have a considerable reserve of voltage, which is made use of during the washing stages. For the purposes of checking materials, a blank experiment should be carried out with the glassware, electrodes and reagents made use of in the analysis.

Normal urine examined by this method was found to possess a lead content varying from 0.02 to 0.05 mgrm. per litre, averaging 0.04 mgrm. per litre. The method, as described, is intended for the determination of the very small amounts of lead occurring in urine, and the accuracy of its results compares very favourably with those obtained by other methods in use. Any small error made in the comparison of the lead sulphite precipitates will necessarily be multiplied in the determination of amounts considerably larger than those usually occurring, but in such cases the method of determination of the amount of lead deposited on the cathode may be varied as found necessary.

OFFICE OF THE DIRECTOR GENERAL OF
PUBLIC HEALTH, SYDNEY, N.S.W.

The Solubility of Reinsch Antimony Films in Water.

BY S. G. CLARKE, B.Sc., A.I.C.

IN almost all of the papers on the quantitative application of the Reinsch test for antimony (deposition on copper from a halide solution) special mention has been made of the necessity, in stripping the deposited film, of allowing as little time as possible to elapse between removing the antimony-coated coil from the boiling solution at the end of the deposition and immersing it in the stripping solution. The reason given for this is that the coating is liable to undergo a change which renders it partly insoluble in the stripping reagent. My experience is that this is more likely to happen with films containing more than 0.0010 gm. of antimony than with smaller amounts; which is perhaps the reason why I have rarely encountered this troublesome feature in dealing with a very large number of determinations of minute amounts of antimony.

There is another, and perhaps more important, reason why no delay should take place between removing the coil after deposition and stripping it. It is, of course, necessary to wash or rinse the coil with water as an intermediate step between the above two operations. The purpose of this note is to direct attention to the fact that if this washing is unduly prolonged, or, indeed, not carried out as rapidly as possible, distinct amounts of antimony are removed from the coil when ordinary distilled water is used. Series of experiments which have been carried out on the following lines show that this solubility of the deposited antimony film is due to the presence of dissolved oxygen in the distilled water.

A number of strips of pure electrolytic copper foil, 20 cm. \times 2.4 cm., were coiled into flat, rather open, spirals, cleaned by immersion in dilute nitric acid (sp. gr. 1.2) and well washed; 0.0005 gm. of antimony was then deposited on each by the Reinsch reaction. Each coil was withdrawn from the boiling solution by means of a hooked glass rod, washed free from acid by dipping into a large volume of distilled water (the time taken in this washing was, on the average, approximately two seconds), and then at once immersed for a definite time in 50 c.c. of ordinary distilled water contained in a 100 c.c. beaker. The water was poured off promptly at the end of the time, and the antimony which had passed into solution was determined as follows:

Five c.c. of 20 per cent. sodium hydroxide solution were added, and hydrogen sulphide passed through the liquid for 15 seconds; a trace of copper accompanying the antimony was thereby precipitated and was allowed to settle out on a water bath and finally filtered off. Five c.c. of concentrated sulphuric acid were added to the filtrate, which was then evaporated until it just fumed; after cooling, this was taken up in 15 c.c. of water, heated to boiling and cooled, and the antimony determined colorimetrically by the pyridine and iodide method described fully in an earlier paper (Clarke, *ANALYST*, 1928, **53**, 373).

Results were obtained as follows:

	Antimony on copper. Grm.	Time of immersion.* Minutes.	Standard antimony solution required.† c.c.	Antimony dissolved. Grm.
(1)	0.0005	1	0.15	0.000015
(2)	0.0005	2	0.3	0.00003
(3)	0.0005	5	1.2	0.00012
(4)	0.0005	10	1.5	0.00015
(5)	0.0005	15	1.7	0.00017
(6)	0.0005	25	1.9	0.00019

* The water was not stirred during the immersion. It was neutral in its reaction to litmus.

† Standard antimony solution = 0.0001 gm. per c.c.

With the exception of (3), the above figures, when presented graphically, produce a smooth curve which becomes almost parallel with the time axis as the time of immersion increases, indicating removal, to a great extent, of the factor causing solution as more antimony enters solution. This lends support to the view that the solution of the antimony is due to dissolved oxygen. The following

experiments afford confirmation of this. In these tests distilled water, which had been boiled and cooled rapidly immediately before use, was used for the immersion of the coils. The coils were rinsed, as before, with ordinary distilled water.

Antimony on copper, Grm.	Time of immersion, Minutes.	Antimony dissolved. Grm.
0.0005	1	nil
0.0005	2	nil
0.0005	5	nil
0.0005	15	0.000015

The trace of antimony dissolved in the last test is doubtless due to absorption of oxygen during the immersion.

The main fact which emerges is that it is not necessary to use boiled-out water for washing Reinsch coils after deposition; ordinary distilled water may be used, provided that washing does not occupy more than a few seconds.

RESEARCH DEPARTMENT,
WOOLWICH.

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.

MEASUREMENT OF THE STRENGTH OF SUNLIGHT.

THE following description of a method which has been in use at Salford for the past three years, for the purpose of comparing the relative *strength* of sunlight in various districts (as distinct from the actual *amounts*), may be of interest to those interested in the smoke-pollution problem. It is well known that the blanket of smoke which hangs over most large towns is instrumental in robbing the sunlight of a large part of its hygienic value, to the detriment of the health of the inhabitants of the district. The ultra-violet rays are the first to be cut off by the smoke-polluted atmosphere, and the absence of these is partly responsible for the development of rickets, anaemia, and tuberculosis, particularly in children.

It is claimed that by the method given here the comparative chemical activity of the light received at different stations can be measured with reasonable accuracy, provided that the tests are carried out at each of the places under conditions as similar as possible.

The method, which was described by the Manchester Air Analysis Committee in 1924, consists in exposing in glass bottles a solution of potassium iodide acidified with sulphuric acid, in the presence of air, to the action of the light. Free iodine is liberated, and the amount is proportional to the chemical activity of the light received. The percentage of ultra-violet light transmitted by ordinary glass is very small, but sufficient for the purpose of giving comparative results.

The strengths of the solutions found to be most convenient are as follows:— Potassium iodide, 20 grms. per litre; sulphuric acid, *N*/4 approx.; sodium thio-sulphate, 1 c.c. equals 0.001 grm. of iodine.

Two-ounce stoppered bottles are used for the actual tests. Ten c.c. of potassium iodide solution and 10 c.c. of the sulphuric acid are pipetted into a bottle, which is then placed on a 6-inch white tile in an open position where no shadow will fall on it during the day. It is left exposed for 24 hours, and its contents then titrated with the thiosulphate solution. If any delay is likely to occur between the removal of the bottle and its titration, the bottle may be kept in a small closed tin until ready, so as to prevent any further action of the light.

The result of the titration is recorded as mgrms. of iodine. A fresh bottle should be exposed at the end of every 24 hours. The change should, of course, be made at the same time each day, particularly in the summer.

Any blank on the iodide solution is allowed for by making up a second bottle at the same time as the one to be exposed and keeping the former in a closed tin until the exposed solution is titrated.

The seasonal variations in the activity of the sun's rays are illustrated by the following monthly tables for the Regent Road station for 1927. This station is in a congested quarter of Salford :

		Mgrms.			Mgrms.
January	41.2	July 208.0
February	68.5	August 178.6
March	159.6	September 118.7
April	148.7	October 65.5
May	188.6	November 68.5
June	179.4	December 42.0

The totals for the two half years of 1926 and 1927 for all the four stations at which tests have been carried out are as follows:

1926.				
	Regent Road.	Nab Top Sanatorium	Ladywell Sanatorium.	Drinkwater Park.
	Mgrms.	Marple. Mgrms.	Mgrms.	Mgrms.
First half year	.. 744.5	885.8	842.9	876.9
Second ,, ,,	.. 869.2	860.8	812.0	870.3
1927.				
First half year	.. 786.0	911.8	952.5	1036.2
Second ,, ,,	.. 681.2	766.7	792.9	809.9

It will be remembered that 1926 was marked, during the latter half of the year, by the coal stoppage. This affected the amounts of dust and smoke in the atmosphere to a considerable degree, and the figures given above for 1926 reflect the unusual conditions then prevailing.

The station at Nab Top is several miles from Salford, in Cheshire; Ladywell Sanatorium is on the outskirts of the City, and Drinkwater Park is outside the City boundary in the neighbouring district of Prestwich. None of these three stations is affected to anything like the same degree as the Regent Road station by the effects of the combustion of coal, and the atmosphere, in comparison to that of the latter, is relatively free from smoke. It will be seen that the total for Regent Road for the first half year is considerably lower than those for the

other three stations, but that during the second half year, whereas the totals for the outlying stations were approximately the same as for the first half year, the total for Regent Road was some 17 per cent. higher, and when it is remembered that the atmosphere during these six months was marked by a cleanliness hitherto unknown, due to the enforced restricted use of raw coal, the connection between the fact and the increased activity of the sunlight, will be appreciated.

The figures for 1927 show no such striking results, and the Regent Road station, for both the half yearly periods, received less active sunlight than the other three.

Another significant fact with regard to the monthly figures is that during the summer months the figures for Regent Road more nearly approached those for the outer stations, and on two or three occasions actually exceeded the figures for one or two of the latter. This is probably due to the fact that less coal is burnt in the summer months, and also to the absence of part of the population on holiday.

The method described is obviously of value in emphasising the need for some form of smokeless fuel for use in the domestic grate, which has been proved to be the chief offender in rendering the atmosphere so unfit to breathe.

H. H. BAGNALL.

MUNICIPAL LABORATORY,
SALFORD.

Notes from the Reports of Public Analysts.

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

CITY OF LEEDS.

REPORT OF THE CITY ANALYST FOR THE THIRD QUARTER, 1928.

ANALYTICAL work was commenced in the newly-equipped laboratories at No. 1, Swinegate, in the early part of July.

Of the 377 samples examined, 332 (326 formal and 6 informal) were taken under the Sale of Food and Drugs Acts, 1875-1927, 8 (4 formal and 4 informal) under the Fertilisers and Feeding Stuffs Act, 1926, and 5 (formal) under the Rag Flock Acts, 1911 and 1928. Of the food and drugs samples, 59 (57 formal and 2 informal), or 17·8 per cent., were adulterated.

MILK.—Fifty-one of 237 samples (234 formal and 3 informal) examined were below standard.

It has transpired that some farmers producing Grade A milk make a practice of bottling the milk from individual cows instead of the mixed product of the whole herd. This may explain in part the low fat content of some of these milks, though it is not suggested that fat deficiencies of 18 to 20 per cent. are thus wholly accounted for.

POTTED MEAT.—Of 13 samples examined, 3 contained 6·0, 7·5 and 0·8 per cent. starch, respectively; the remaining samples were free from starch. In the first case the manufacturer is now selling the article under a different name.

SWEET NITRE.—Of 4 samples examined, 3 were deficient in ethyl nitrite to the extent of 9·9, 31·0 and 100·0 per cent. respectively, calculated on the minimum of 1·52 per cent. of the active principle. Proceedings were instituted in the second and third cases, the retailers concerned being ordered to pay costs. In the 100 per cent. deficiency case the article sold proved to consist of an aqueous alcoholic solution of 3·7 per cent. ammonium acetate and 15 per cent. of sucrose; it has been supplied to the retailer as “Nitre Sweating Mixture.”

TOXICOLOGICAL ANALYSIS.—The organs of an elderly man, suspected of having died from sheep-dip poisoning, were submitted for analysis. This case, following closely upon the Pace trial, excited considerable interest. No poison was found, and the sheep-dip, moreover, proved to be a non-arsenical preparation possessing a creosote acid basis.

C. H. MANLEY.

GIBRALTAR.

ANNUAL REPORT OF THE CITY ANALYST AND BACTERIOLOGIST FOR 1927.

THE number of samples of foods and drugs examined under the Public Health Ordinance during the year was 168, of which 24 were below the official standards.

GOATS' MILK.—Of the 56 samples examined, 10 were deficient in fat, 4 contained added water, 1 showed both abstraction of fat and added water, and 4 contained unboiled milk. The statutory limits for goats' milk are 3·5 per cent. of fat and 8·0 per cent. of solids-not-fat. As pointed out in previous reports, some milk vendors habitually remove the scum which rises to the surface on boiling, to improve the appearance, thereby robbing the milk of a proportion of its fat. In view of the fact that vendors of boiled goats' milk declare the milk “skimmed,” thus evading the law, amendments to the Public Health Ordinance are under consideration, and include the following:—“No sample of boiled *scummed* goats' milk shall contain less than 3·5 per cent. of milk fat.” It is expected that this will become operative in the near future.

Goats' Unboiled Milk.—While no sample of raw imported milk was discovered there were, however, four samples showing some contamination with unboiled milk. By law no imported milk may be offered for sale to the public unless it has been boiled in Gibraltar. This is a necessary precaution against milk-borne diseases, the Council having no control over the source of production.

CONDENSED AND DRIED MILK.—An Ordinance has been prepared, based on the standards required in England, and is expected to come in force within the year.

BACTERIOLOGY AND HEALTH WORK.—During the year the number of samples and specimens examined was 4188, including drinking waters, swabs, blood, faeces, and human milk. The serological agglutination test was carried out on the 209 goats living on “The Rock.” All were found free from undulant fever. One hundred and twenty-one rats, caught on the quays or in Gibraltar, were examined for the plague bacillus; all were free.

A. G. HOLBOROW.

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.

"EGG FLOUR."

ON November 28, a grocer was summoned at Lichfield for selling "egg flour" containing no eggs.

According to the prosecution an inspector purchased a packet of egg flour at the defendant's shop for 5½d. The packet was advertised as "——'s, Beat All Egg Flour. Requires no eggs, egg powder, or baking powder." The cost of the best self-raising flour should not exceed 3d. per packet.

The County Analyst (Mr. E. V. Jones), giving evidence, said that the flour was devoid of egg, and consisted of self-raising flour to which had been added a small percentage of coloured maize, prepared in such a manner as to resemble flour containing small lumps of dried egg, which was very misleading to the public and most unfair to manufacturers of the genuine article.

The defendant said that he did not buy the colour, but that he had bought the maize as an egg substitute. The article was originally known as "Tingle's Egg Flour"; it had been made in Lichfield for 30 years, and he himself had been making it for 12 years. He had omitted to change the name to "egg substitute."

A fine of 10s., with £6 15s. 6d. costs, was imposed.

"HOME-MADE" LEMON CHEESE.

ON December 5th a shopkeeper was summoned before the Stipendiary Magistrate of Salford (Mr. P. W. Atkin), for selling home-made lemon cheese not of the nature, substance and quality demanded, and the makers were also summoned under Sec. 27 of the Sale of Food and Drugs Act, 1875, for giving a label falsely describing the article.

Mr. H. H. Tomson, Deputy Town Clerk, prosecuting on behalf of the Health Committee, said that the Food and Drugs Inspector, by means of his deputy, purchased two small jars of home-made lemon cheese at the defendant's shop for which he paid 1s. 1d.

The sample was analysed by the City Analyst (Mr. H. H. Bagnall), and his certificate stated that "Home-made lemon cheese should consist of butter, sugar, eggs and lemon juice and rind. The fat in this sample consists of margarine, cane sugar is replaced to the extent of 39 per cent. by glucose syrup, and the protein content indicates the presence of not more than the merest trace of eggs. It contains about 8 per cent. of water in excess of that associated with fat, protein, glucose, starch, and lemon products, and is artificially coloured. This is not home-made lemon cheese, as two of the principal constituents, butter and eggs, are practically entirely absent, and sugar is replaced to a large extent by glucose syrup. The starch is a foreign ingredient and should not be present at all." Mr. Tomson said that it was not submitted that the article was unwholesome, but it was not home-made. There were many articles on the market equal to this at half the price.

In pleading guilty, one of the partners of the firm stated that they sold the article as they received it. In the case against the makers, Mr. Tomson contended that the glass jars were made up in a way that was calculated to deceive the ordinary housewife. The lithographed label bore the words "Home made" in imitation script.

"There is no statutory standard for lemon cheese," Mr. Tomson proceeded, "but my submission is that an article which is described as home-made should be made of materials which one would expect to be used in an ordinary domestic household. This is not a home-made lemon cheese because it is made in a factory and sold wholesale throughout the country. I am not, however, challenging the wholesomeness of the commodity."

Evidence was given by the Public Analyst (Mr. H. H. Bagnall), by the Lancashire County Analyst (Mr. G. D. Elsdon) and by the Food Inspector, to the effect that, while there was no legal standard for lemon cheese, custom fixed the standard that it should be made from butter, sugar, eggs and lemons. There was no objection to the article in question if it was not sold as home-made.

Mr. V. Parker, for the defence, suggested that a purchaser would not be deceived by the label, and said that if it had been intended to imply that the article was made on the shopkeeper's premises, the words "Our own make" would have been used. The label did not necessarily mean that the commodity was made on the premises at which it was sold. The firm was giving people value for their money, and the purchaser was getting a perfectly pure and wholesome article.

On the summons against the shopkeeper a fine of £5 was imposed, and the makers were fined £5, with £15 special costs, the Stipendiary remarking that a home-made article should, in his opinion, be made from ingredients that the ordinary housewife would use.

BORIC ACID SOLD AS A FOOD PRESERVATIVE.

ON December 6, a druggist was summoned at Barnsley, under the Public Health (Preservatives, etc., in Food) Regulations for selling boric acid with a recommendation that it was a food preservative.

Mr. N. P. Lester (Assistant Town Clerk) explained that only two articles could be sold as food preservatives, and that in this case boric acid (which was not one of the approved articles) had been sold in a container which recommended it as a preservative of milk.

Mr. N. Goodyear, for the defence, pleaded guilty to a technical offence, but contended that, although there was nothing to prevent anyone buying boric acid and using it as a food preservative, yet an offence was committed when the seller recommended its use for that purpose. The Regulations under which the proceedings were taken had only come into force at the beginning of the year, and it was difficult for druggists' assistants to keep pace with the numerous regulations that were being made.

The Magistrates decided that the offence was due to a misunderstanding, and dismissed the summons on payment of costs.

Department of Scientific and Industrial Research.

REPORT OF THE WATER POLLUTION RESEARCH BOARD FOR THE YEAR 1927-28.*

THE objects of the Water Pollution Board are to collect and collate all pertinent scientific and technical information, so that it may be readily available for practical application by those who are concerned with water supply and the disposal of polluting liquids; to encourage and co-ordinate relevant scientific research in this country; and to undertake such investigations as are necessary in the public interest and not otherwise provided for. A survey of the River Tees is in view in conjunction with the Tees Fishery Board.

In considering water-supply it is proposed particularly to investigate the base exchange or zeolite treatment of water, with special reference to the rate and extent of the base exchange; wastage of material; possibility of contamination of the softened water by silica and alumina; how far the action is a surface one, depending upon size and texture of particles; and the process of regeneration. With regard to sewage disposal, the activated sludge process is to be investigated on biological lines, and in the case of the treatment of industrial effluents a beginning has been made with beet sugar factories effluent, and the experiments, though not yet complete, suggest that a practical solution will be found in biological filtration.

The ideal solution of the problem would be such an alteration of processes that no waste water would be discharged, and, as a rule, less drastic treatment should be needed to render the waste waters fit for re-use than for discharge. The removal of suspended solids by means of detritus tanks, reasonably small; a graded series of mechanically operated and cleaned screens, and finally sedimentation tanks are suggested. Fine screening is not advocated, and punched metal screens would obviate the difficulty of leaves, beet debris, etc., getting entangled in the mesh of woven screens. The sugar and dissolved organic material not removed in such a plant, by gradual accumulation would be liable to fermentation, so that a proportion of treated water would need to be discharged daily and the loss made up with fresh water.

D. G. H.

* Obtainable at H.M. Stationery Office, Adastral House, Kingsway, W.C.2. Price 6d. net.

U.S.A. Department of Agriculture.

STANDARD FOR MAYONNAISE SALAD DRESSING.

ACTING upon the recommendation of a joint Committee, including representatives of the Association of Dairy, Food and Drug Officials of the United States, the Association of Official Agricultural Chemists, and of the United States Department of Agriculture, the Secretary of Agriculture has adopted the following definition and standard for mayonnaise salad dressing.

MAYONNAISE, MAYONNAISE DRESSING, MAYONNAISE SALAD DRESSING is the clean, sound, semi-solid emulsion of edible vegetable oil and egg yolk or whole egg, with vinegar or lemon juice, and with one or more of the following: Salt, spice, sugar. The finished product contains not less than 50 per cent. of edible vegetable oil, and the sum of the percentages of oil and egg yolk is not less than 78.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Determination of Honey in Honey Cake. R. T. A. Mees. (*Chem. Weekblad*, 1928, 25, 674-676.)—It is shown that since the composition of the sugars used in the manufacture of honey cake is almost unaffected by the baking process, the percentage of fructose in an extract of the cake may be taken as a measure of the amount of honey used. The fructose is determined on an extract prepared by grinding 30 grms. of cake with water in a mortar, the volume being made up to 200 c.c. and the mixture centrifuged and filtered. The same solution may be used for the determination of the extract from the specific gravity. To 2.5 c.c. of a *N* solution of iodine and 3 c.c. of *N* sodium hydroxide solution are added 20 c.c. of the ten-fold diluted extract. After 5 minutes in the dark the solution is acidified with 1 c.c. of 4 *N* hydrochloric acid and then titrated with a 10 per cent. solution of sodium sulphite, the exact end-point being finally obtained by means of a 5 per cent. solution as in the method of Kolthoff (*ANALYST*, 1922, 47, 301; 1923, 48, 386). If 20 c.c. of mixed Fehling's solution are then added to the mixture (about 30 c.c.) the reducing powers of the solution may be determined, and the result calculated as percentage of fructose. The method has been tested on pure sugars and on sugar mixtures and cakes of known composition, and gives results which are higher and nearer the true values than those obtained by Kolthoff's method or by the usual indirect polarimetric method. Of 77 commercial samples of cake, about half were considered to contain natural honey, and the fructose content varied from 4 to 37 per cent. J. G.

Quantitative Determination of Oxymethylfurfural in Honey. J. Fiehe. (*Z. Unters. Lebensm.*, 1928, 56, 200-203.)—In Troje's method (*Z. Ver. deut. Zuckerind.*, 1925, 75, 635) an extract of the honey in anhydrous ethyl acetate is evaporated *in vacuo* at 45° C., and the residue oxidised by an alkaline solution of iodine, the excess of which is back-titrated with sodium thiosulphate solution. It is shown that the method is affected by the presence of sugars, organic acids, aromatic compounds, colouring matters and other substances in the honey, and may therefore give high values indicating an oxymethylfurfural content, even when this substance is absent. J. G.

Occurrence, Detection and Determination of Lauric Acid in Alcoholic Beverages. J. Grossfeld and A. Miermeister. (*Z. Unters. Lebensm.*, 1928, 56, 167-187.)—Lauric acid (0.2 mgrm.) may be detected in the presence of myristic acid or 3 mgrms. of caproic acid by the extraction of 50 c.c. of the sample with 50 c.c. of ether, the residue after evaporation of the ether being dissolved in 3 c.c. of alcohol, and boiled under a reflux condenser for 10 minutes with

0.5 c.c. of 0.5 *N* alcoholic potassium hydroxide solution. The residue left on evaporating the solution is then dissolved in 3 c.c. of water and heated on the water-bath for 5 minutes with 0.2 c.c. of glycerin. The addition of 0.25 c.c. of 15 per cent. magnesium sulphate solution, followed by filtration while hot through a tightly packed asbestos filter, gradually produces a precipitate of magnesium laurate. For the determination of the lauric acid, 500 c.c. of the sample and 300 c.c. of water are distilled, and the distillate (450 c.c.), together with the 50 c.c. of alcohol used to wash the condenser, is boiled under a reflux condenser for 30 minutes with 5 c.c. of a 75 per cent. solution of potassium hydroxide. The solution is evaporated, and the residue is dissolved in 25 c.c. of a solution of 25 grms. of sodium acetate, 5 c.c. of glacial acetic acid and 1 c.c. of 1 per cent. phenolphthalein solution in 250 c.c. of water, and 30 per cent. acetic acid is added till only a faint red colour remains, which is then removed by one drop of the acetate solution and restored by addition of 0.1 *N* sodium hydroxide solution. After filtration in the presence of kieselguhr the solution is boiled, 5 c.c. of the magnesium sulphate solution added, and any red colour again dispersed by the acetate solution. After 24 hours the precipitate is filtered on a Gooch crucible previously washed with a little magnesium laurate solution and dried in the steam-oven, dried, washed, gently heated and the crucible re-weighed. From the loss in weight, which gives the lauric anhydride, the factor 1.105 is used to obtain the magnesium laurate, and after a correction for the solubility of that salt (0.5 or 0.6 mgrm., according to whether the volume is 40 to 43 c.c. or 44 to 50 c.c., respectively), the factor 0.9472 gives lauric acid. The caprylic acid value is determined by the addition of 1 c.c. of a 50 per cent. solution of potassium hydroxide to the distillate (95 c.c.) obtained from 100 c.c. of sample. The mixture is boiled under a reflux condenser for 30 minutes, evaporated to dryness, the residue dissolved in 10 c.c. of water, neutralised to phenolphthalein with 30 per cent. acetic acid, diluted with 50 c.c. of water, and 25 c.c. of a 1.5 per cent. solution of magnesium sulphate added. After 24 hours the solution is filtered, 10 c.c. of a solution of 50 grms. of sodium acetate, 3.12 grms. of copper sulphate and 5 c.c. of 20 per cent. acetic acid per litre added, and the precipitate filtered off, dried and weighed on a Gooch crucible. The solubility correction is 0.7 mgrm., calculated as copper laurate, and the factor 0.869 then gives the caprylic acid value in c.c. of 0.01 *N* acid, whilst the factor 1.44 gives its weight in mgrms. It is concluded that the principal constituent of the so-called grape or cognac oils is lauric, and not capric acid, in the form of esters. The lauric acid content of a beverage does not appear to depend on the alcohol content. The acid can be removed from fusel oils by rectification, and is obtained in the "second runnings" during distillation.

J. G.

Sesamin and Sesamol. W. Adriani. (*Z. Unters. Lebensm.*, 1928, 56, 187-194.)—The author distinguishes the following constituents of sesame oil, and complains of their confusion in the literature (*cf.* Hönig, *Chem. Weekblad*, 1925, 22, 509; Malagnini and Armanni, *ANALYST*, 1907, 32, 391):—*Sesamin*,

$C_{20}H_{18}O_6(?)$, may occur to the extent of 1 per cent., and has been isolated by crystallisation from alcohol as long colourless needles, m.pt. $122.5^\circ C.$, $[\alpha]_D^{20} + 68.23^\circ$ (chloroform), sparingly soluble in ether or petroleum spirit and easily soluble in acetone or chloroform. It differs from phytosterol in that it gives Bömer's reactions (a green colour turning to red and then to reddish-blue when shaken with equal parts of concentrated sulphuric acid and acetic anhydride, and a cherry-red colour turning to blue when a solution in chloroform is shaken with one drop of concentrated sulphuric acid), but it does not give Baudouin's reaction. *Sesamol*, $C_7H_6O_3$, m.pt. $65.5^\circ C.$, occurs to the extent of 0.1 per cent., has a phenolic odour and gives the Baudouin and Kreis tests (a pine chip dipped in the sesamol and then in concentrated hydrochloric acid gives a green colour). *Sesamolín*, m.pt. $93.6^\circ C.$, $[\alpha]_D^{20} + 218.4^\circ$ (chloroform), also gives Baudouin's test, since hydrochloric acid converts it into sesamol and samin, $C_{20}H_{18}O_7 + H_2O = C_7H_6O_3 + C_{13}H_{14}O_5$. It constitutes 0.3 per cent. of the oil. *Samin*, $C_{13}H_{14}O_5$, was isolated as long colourless needles, m.pt. $103^\circ C.$, $[\alpha]_D^{20} + 103^\circ$ (chloroform). J. G.

Luminescence of Oils and Fats. A. van Raalte. (*Z. Unters. Lebensm.*, 1928, 56, 195-198.)—The author provides further evidence in favour of his hypothesis that the appearance of luminescence in refined oils and fats is due to the removal of vitamins which inhibit luminescence in the crude product. For example, vitamins may be produced in certain fats by exposure to sunlight, and their powers of luminescence then disappear (see, however, Carrière, *Chem. Weekblad*, 1928, 25, 632). The conclusions of Feder and Rath (*Z. Unters. Lebensm.*, 1927, 54, 321) are also criticised. (Cf. ANALYST, 1928, 53, 617.) J. G.

Volumetric Method for the Determination of Tin in Preserves and other Foodstuffs. B. Glassmann and S. Barsutzkaja. (*Z. Unters. Lebensm.*, 1928, 36, 208-212.)—The mixed sample (50 grms.) is dried, ignited, the ash extracted with 30 c.c. of dilute nitric acid (1 : 2) to remove iron, copper and lead, and after filtration the residue and paper again ignited. The tin is then reduced to metal by fusion at low red-heat with 1 gm. of potassium cyanide, the melt extracted with 150 c.c. of water, and the residue (and filter), after filtration of the extract, digested with 25 c.c. of concentrated hydrochloric acid in a flask fitted with a Bunsen valve. One gm. of zinc is added to complete the reduction, the solution cooled in a stream of carbon dioxide, and titrated with a 0.02 *N* solution of potassium dichromate in the presence of a little potassium iodide with starch indicator. The dichromate solution is standardised against a solution containing a known amount of pure tin prepared in a similar way. The method, which takes about 6 hours, has been applied to fish products, a maximum error of -7 mgrms. being recorded for a tin content of 50 mgrms. (-2.5 mgrms. for 10 mgrms. tin). The tin content of a $2\frac{1}{2}$ years old sample, kept in the open tin container for 12 days, rose from 154.6 to 420 mgrms. per kilo. J. G.

Determination of Caffeine in Tea. S. Gobert. (*Ann. Falsif.*, 1928, 21, 517-518.)—Three grms. of the very finely powdered sample of tea are moistened

with 4 c.c. of ammonia, which causes the cells to swell and admit solvent, and also sets free combined caffeine. After half an hour four ethyl acetate extractions are made, each with 25 c.c., and the dried extract is purified by mixing with 4 c.c. of ammonia (22° Bé), and, after standing, is extracted 4 times with 25 c.c. portions of ethyl acetate. After centrifuging for 5-7 minutes and decanting, the ethyl acetate is distilled off, and the residue dried. It is then twice extracted with 50 c.c. and once with 25 c.c. of boiling water, 15 c.c. of 1 per cent. potassium permanganate solution are added to the extract, and after 15 minutes the manganese is precipitated by 12 volume hydrogen peroxide containing 1 per cent. of glacial acetic acid. The filtrate is then evaporated, and the residue dried, extracted 3 times with chloroform, and weighed. A moisture determination is made at the same time. Very closely agreeing duplicate results were obtained on samples of Ceylon tea.

D. G. H.

Determination of Morphine. A. K. Balls and W. A. Wolff. (*J. Biol. Chem.*, 1928, 80, 379-402.)—The determination of morphine in biological material is difficult and tedious. The existing methods have been subjected to a critical study, and their sources of error are pointed out. All the methods consist of two parts: first, isolation of the alkaloid in a state of relative purity; second, measurement of its amount. When morphine is isolated by the precipitation of accompanying impurities, there is apt to be a retention of morphine by the precipitate, which cannot always be removed by exhaustive washing. Methods which avoid precipitation frequently require evaporation of neutral or alkaline morphine solutions, and this decomposes a considerable amount of morphine. The decomposition products may follow the morphine through the analysis, and may finally be determined with it, because of the similarity in their reactions, but this precludes any distinction between morphine and its oxidation products in the original material. Control determinations on tissues which contain known amounts of morphine may give satisfactory results, but may not indicate that the method is suitable for the determination of unchanged morphine in the presence of oxidised morphine. A series of methods for the determination of morphine in biological material is proposed which, it is hoped, eliminates many of these errors. The methods are unsuitable for amounts of morphine less than 20 mgrms. per 100 grms. of material, but, where applicable, they possess the advantages of rapidity, simplicity, exclusion of the oxidation products of morphine, and inclusion of a desirable check on the final result. Methods are described for morphine determination in muscle, urine and blood. Provided the isolation process has yielded pure morphine, almost any of the methods of final determination are satisfactory, and the authors have selected from among the gravimetric determinations the silicotungstic acid precipitation of Bertrand (*Compt. rend.*, 1899, 128, 743), given in detail for morphine determination by Balls (*J. Biol. Chem.*, 1926-27, 71, 543), and chosen on account of the valuable check which may be made on the validity of the results.

P. H. P.

Action of Schiff's Reagent on Pyramidone. A. Valdiguié. (*J. Pharm. Chim.*, 1928, **120**, 506-510.)—The addition of a few drops of Schiff's reagent (made up according to any of the formulae, provided an excess of sulphur dioxide is avoided) to an alcoholic or water solution of pyramidone or its salts, such as the camphorate or salicylate, provided the solution is not too acid, produces a red coloration. In the absence of other substances giving a reaction with Schiff's reagent, the reagent may be used for a colorimetric determination, and the limit of sensibility is about 1 part in 10,000. The colour is not affected by air or light, and is more red than violet. A critical study of the reaction suggests that a molecular combination of the sulphited rosaniline and pyramidone takes place.

D. G. H.

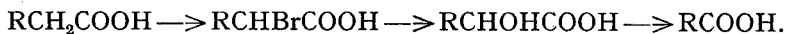
Biochemical.

Distribution of Unsaturated Fatty Acids in Tissues. III. Vital Organs of Beef. W. R. Bloor. (*J. Biol. Chem.*, 1928, **80**, 443-454.)—Data have previously been presented by Bloor on the unsaturated fatty acids of the heart (*J. Biol. Chem.*, 1926, **68**, 33) and other muscles (*J. Biol. Chem.*, 1927, **72**, 327) of the ox, and similar data are now given as to the more important vital organs of the same animal. A table of results shows that the lipid content of the organs varied greatly both for the same organ and for different organs. The constituent which varied least for each organ (not more than 30 per cent. above or below the average value) was the total phospholipid, which was relatively constant, and may therefore be regarded as a tissue constant characteristic of the particular organ. The arrangement of the organs in the order of their phospholipid content gave a series which represented the order of their functional activity, namely, brain (highest), liver, pancreas, kidney, and lung. The similarity in the weights of lecithin and cephalin in the various samples of all tissues points either to an equimolecular equilibrium, or to an equimolecular combination, between the two. The average iodine values of lecithin and cephalin strongly indicated a close similarity between the two compounds. Certain organs showed higher iodine values for phospholipid than others, but the difference was not great, and this similarity points to a considerable basic content of the same phospholipids in all organs and tissues. The content of fatty acids found indicated that the lecithin fraction was quite pure, but that the cephalin and fat fractions contained considerable admixtures of other substances. The mixture of fatty acids obtained by saponification of the various fractions consisted, as in the muscles, of about one-half liquid or unsaturated acids and about one-quarter solid acids, the remaining quarter being unaccounted for, and thus constituting a problem which must be solved. The unsaturated acids were examined for acids of the higher degree of unsaturation, those with three and four double bonds. They contained relatively large amounts of a 4-bond acid, probably arachidonic, but no appreciable amount of 3-bond acid. The amount of 4-bond acid was greatest in the brain, next in the liver and kidney, then lung, then pancreas.

P. H. P.

Highly Unsaturated Fatty Acid of Liver Lipids. Preparation of Arachidonic Acid. J. B. Brown. (*J. Biol. Chem.*, 1928, 80, 455-460.)—Experiments have been carried out with two objects, (1) to verify the presence of arachidonic acid in the lipids of liver, and (2) to isolate a specimen of the pure acid. As regards the first, the methyl esters of the fatty acids of liver lipids were prepared and fractionated into four fractions, and each fraction was analysed. The bromides of each fraction were found to contain practically the same amount of bromine, the results in each case being very close to the theoretical for methyl octobromoarachidate. As regards the second, the combined bromides of the several fractions were reduced with zinc in neutral alcohol, and practically pure methyl arachidonate was prepared. From this ester the acid was obtained by saponification. Some oxidation took place during the saponification, and the final product of iodine number 316 (theory 334) represents the purest specimen so far obtained. The results indicate therefore that arachidonic acid is the sole highly unsaturated fatty acid present in appreciable quantities in the lipids of pig liver. It occurs to the extent of 2.0 to 7.7 per cent. of the total fatty acids, depending on the method of calculation. The arachidonic acid obtained was a light amber oil, less mobile than the ester, and with a distinct fishy odour. The properties of methyl octobromoarachidate, methyl arachidonate and arachidonic acid are described. P. H. P.

Oxidation of Lignoceric Acid. F. A. Taylor and P. A. Levene. (*J. Biol. Chem.*, 1928, 80, 609-613.)—When cerebronic acid was oxidised to the next lower unsubstituted fatty acid, instead of pure lignoceric acid being obtained, a product was isolated that appeared to be a mixture of acids. It was therefore decided to determine whether an α -hydroxy acid of known purity related structurally to cerebronic acid could be degraded by oxidation, under similar conditions to those applied to cerebronic acid, to the next lower acid of the series, in good yield and uncontaminated by substances that could not be separated easily. Lignoceric acid was converted into its next lower homologue by passing it through the following steps (Levene and West (*J. Biol. Chem.*, 1913-14, 16, 475)):



These reactions were partly carried out by Meyer, Brod and Soyka (*Monatsh. Chem.*, 1913, 34, 1113), and by Levene and Taylor (*J. Biol. Chem.*, 1922, 52, 227), but since it has recently become known that some higher aliphatic substances change on standing and cannot be brought back to their original melting points, and since the materials used by Levene and Taylor had stood for some time, it was decided to check their figures. The new experiments show that the melting points recorded by each group of workers should be increased, as shown by the following table:

	Meyer, Brod and Soyka. °C.	Levene and Taylor. °C.	Taylor and Levene. °C.
α -Bromolignoceric acid	68-69	68.5	69.5-70.5
α -Hydroxylignoceric acid	91-92	91-92	94-95
Isotricosanoic acid	—	73.5	76.5-77.5

Oxidation of α -hydroxylignoceric acid evidently gives rise to a single substance, isotricosanoic acid. The crude acid melted at 75–77° C., and the best specimens at 76.5–77.5° C. Further evidence is given in favour of the view of Meyer, Brod and Soyka that lignoceric acid is not a normal acid, for in this series the acid with an odd number of carbon atoms melts at a point between the melting points of the two adjacent even-numbered acids. P. H. P.

Loosely-Bound Sulphur in Egg Albumin. W. D. Treadwell and W. Eppenberger. (*Helv. Chim. Acta*, 1928, 11, 1035–1042.)—Fresh egg-white was extracted with boiled water and filtered, and 20 c.c. of the extract heated with 20 c.c. of a 0.1 *N* solution of sodium hydroxide in a thermostat maintained at 78° C. by means of boiling alcohol. Air was excluded to avoid oxidation, the apparatus being arranged so that the operations were carried out in an atmosphere of nitrogen. The excess of alkali was then neutralised with 0.1 *N* hydrochloric acid, and the sulphide ions produced in the partly hydrolysed solution were titrated electrometrically with a 0.002 *N* solution of lead nitrate against a silver and silver chloride electrode. A 0.01 *N* standardised solution of sodium sulphide was used for comparison purposes, and was found to keep well in an atmosphere of nitrogen. This controlled hydrolysis gave hydrolysis-curves which indicate that the reaction is bimolecular and is finished after 3 hours, 0.266 per cent. of sulphide sulphur being found in the egg albumin. If this constitutes one-sixth of the total sulphur, the latter would comprise 1.616 to 1.520 per cent. of the protein molecule, the molecular weight of which would then be about 12,000. J. G.

Volumetric Method for the Determination of Protein Solutions. W. D. Treadwell and W. Eppenberger. (*Helv. Chim. Acta*, 1928, 11, 1053–1062.)—A volume of the solution corresponding with about 30 mgrms. of protein is precipitated with about 200 mgrms. of the purest tannin (free from gallic acid) in the form of a 0.5 or 1 per cent. solution, and the mixture diluted to 100 c.c., shaken, and allowed to settle. A solution of Prussian blue (prepared by the addition of 25 c.c. of 0.2 *N* potassium ferrocyanide solution to 18.8 c.c. of 0.2 *N* ferric chloride solution, and dilution to 500 c.c.) is then added from a burette, the end-point being reached when a faint blue colour is imparted to the supernatant liquid after it has been centrifuged for 5 minutes (2,000 revolutions). At this point the albumin, which is a positive colloid, has completely adsorbed the negatively-charged Prussian blue, and 1 gm. of gelatin and egg-albumin may be taken as equivalent to 2.3 and 1.1 millimols of ferrocyanogen, respectively, the proportions of Prussian blue (*B*) and egg-albumin (*E*) or gelatin (*G*) being related by the equations $B=0.915 \times E^{0.688}$ and $B=1.92 \times G^{0.688}$ respectively. The end-point is unaffected by the presence of amino-acids, but inorganic salts should be removed by dialysis, and the acidity of the medium should be adjusted approximately to the P_H value corresponding with the iso-electric point of the substance concerned (gelatin 4.7, egg-albumin 4.8). The adsorbed colour is deepest in the case of gelatin, but egg-albumin gives more reproducible results, particularly if a standard of known strength is used for comparison purposes. J. G.

Further Application of the Vanillin and Hydrochloric Acid Reaction in the Determination of Tryptophane in Proteins. I. Kraus Ragins.

(*J. Biol. Chem.*, 1928, **80**, 543-550.)—The author criticises the attack made by Looney (*J. Biol. Chem.*, 1926, **69**, 519; *ANALYST*, 1926, **51**, 588) on a previous paper by her (Kraus (*J. Biol. Chem.*, 1925, **63**, 157; *ANALYST*, 1925, **50**, 246)) on the vanillin and hydrochloric acid reaction for tryptophane, and states that it is "quite clear that he has not read the paper sufficiently carefully to warrant his generalised conclusions." A slight modification in the procedure of the reaction is now described, which has been devised to save time and material. The reaction is allowed to take place in the centrifuge tube instead of the precipitate being transferred to a flask, and a 0.2 mgrm. standard is used in place of the 0.4 mgrm. standard described previously. For concentrations of tryptophane of less than 0.2 mgrm., or for blanks, a known amount of tryptophane is added in order to insure quantitative precipitation. The vanillin and hydrochloric acid reaction applied directly to sixteen highly purified proteins gave very unsatisfactory results, as previously, but when the same proteins were first hydrolysed by trypsin and the indirect vanillin and hydrochloric acid reaction applied (which means precipitation of tryptophane by mercuric sulphate under definite conditions) good results were obtained. The filtrate from the mercury-tryptophane precipitate was shown definitely not to contain tryptophane. If peptide tryptophane is present in the mercury-tryptophane precipitate it reacts in the same way as free tryptophane with the vanillin and hydrochloric acid reaction. Proline or proline-containing proteins, such as gelatin, in the concentrations used by Komm (*Z. physiol. Chem.*, 1926, **156**, 161) have no effect on the vanillin and hydrochloric acid colour reaction, but in higher concentrations a secondary colour forms which interferes with the true tryptophane colour. Chloride ion concentrations of 0.3 per cent. or higher interfere with the quantitative precipitation of tryptophane by mercuric sulphate, whilst sodium ion concentrations up to 2 per cent. have no effect. Thus sodium is not a factor in retarding the precipitation of tryptophane by mercuric sulphate in a pepsin-hydrochloric acid medium.

P. H. P.

Colorimetric Determination of Inorganic Sulphate in Small Amounts of Urine. B. S. Kahn and S. L. Leiboff. (*J. Biol. Chem.*, 1928, **80**, 623-629.)—

A colorimetric method is described for the determination of inorganic sulphate in small amounts of urine. The inorganic sulphate is precipitated as benzidine sulphate. The precipitate is then diazotised and coupled with phenol in an alkaline medium to produce a yellow colour which is proportional to the amount of benzidine. This is compared in a colorimeter with a similarly treated standard sulphate solution. Phenol was found to be the ideal reagent for colour development. It forms a dye which is soluble in aqueous solutions, highly stable, and whose tinctorial power does not demand excessive dilutions. An excess of phenol has no effect upon the colour development, but an excess of alkali retards it. When known amounts of sulphur in the form of sulphate were added to urine very good recoveries were obtained. The method was checked against a gravimetric method, and the results obtained are tabulated.

P. H. P.

Highly Accurate Method for the Analysis of Urea. M. Taylor. (*J. Amer. Chem. Soc.*, 1928, **50**, 3261-3265.)—Being a non-electrolyte of low equivalent weight and chemically indifferent, urea forms an excellent reference substance for use in physico-chemical and colloidal problems. It may be determined with an accuracy of 0.02 per cent. by conversion into ammonia and carbon dioxide by the action of hydrochloric acid in an autoclave. This reaction proceeds to completion in presence of a very slight excess of the acid, and, as the latter does not vaporise under the conditions used, distillation of the resulting ammonia with soda is not necessary. The digestion is carried out in a 500 c.c. stoppered conical Pyrex or silica flask provided with an exit tube of inverted U-shape sealed in or near the top of the flask. The flask is weighed, and the urea solution and standard acid are pipetted into it, the weight being noted after each addition. A Pyrex test-tube, containing a little water into which the side-tube dips, acts as scrubber to the escaping carbon dioxide, this being absorbed by the distilled water, containing a few c.c. of soda solution, in the autoclave. Subsequent expulsion of the carbon dioxide by aeration or boiling of the reaction mixture is then unnecessary. The flask and scrubber are covered with tinfoil while in the autoclave, and, after the air has been displaced and the heating continued for 30 minutes at 2 atmos., and for 4 hours at 4 atmos., the autoclave is allowed to cool. The glass stopper of the flask is then removed and replaced by a rubber bung carrying a tube, by means of which the contents of the scrubber are sucked back, and the scrubber rinsed several times into the flask. The excess of acid is titrated with soda solution after addition of two drops of 0.02 per cent. methyl red solution. T. H. P.

Urobilin Content of Normal Human Blood. M. A. Blankenhorn. (*J. Biol. Chem.*, 1928, **80**, 477-485.)—Although the physiology of urobilin deals indirectly with blood urobilin, the normal urobilin content of human blood has not before been described, and there has been no method for its determination. Most urobilin studies have been made either with the Schlesinger fluorescence method or with the Ehrlich test. Of these, the latter is not very sensitive whilst the Schlesinger test is very sensitive (0.0048 mgrm. per 100 c.c.) and highly specific; Elman and McMaster (*J. Exp. Med.*, 1925, **41**, 503) have made it quantitative by the development of a standard. A method for blood urobilin is now described by the author. It consists in the application of the fluorescence test to blood serum modified to make it more sensitive by refinements in two main directions; namely, to provide absolutely clear supernatant solutions in which to develop fluorescence, and to examine these solutions in a dark room with an intense beam of light. The fluorescence is then measured quantitatively by means of the Elman and McMaster standard prepared by a simplified technique. Details of the method are given, together with illustrations of the lamp and apparatus used for comparison of the solutions with the standards. The standard used is a dilute aqueous solution of neutral acriflavine in a series of concentrations, the strongest of which is a solution of 1 part in 10,000,000, and the weakest a solution of 1 part in 200,000,000. A chart is given which is arranged for direct determination of urobilin values. With

this method it has been possible to detect fluorescence in practically every normal human blood. Of 128 specimens of human blood presumably normal, but two were negative, and in twelve instances no test could be made owing to inability to clear the specimens. Probably with greater experience none will be found negative, and with good technique there should be very few with which no test can be made. The average of a series of 107 normals gives 0.28 mgrms. of urobilin per 100 c.c.; this may be slightly greater than is justifiable, as a few may have been slightly abnormal specimens. Numerous pathological specimens were measured which gave results as high as 33 mgrms. per 100 c.c., the highest values being in patients with nephritis, malaria, pneumonia, and tuberculosis with fever; one patient with complete obstruction of the gall duct and with complete intestinal acholia was clearly negative. This agrees with current ideas of urobilin metabolism.

P. H. P.

Organic Analysis.

General Method for the Micro-Determination of Carbon by the Use of Chromic Acid Oxidation. A. Boivin. (*Comptes rend.*, 1928, **187**, 1076-1079).—The carbon content of an organic substance may be accurately determined by subjecting a portion, weighed on the microbalance, to chromic acid oxidation in the presence of sulphuric acid and silver dichromate in a boiling water bath for half an hour. Nicloux's apparatus (*Compt. rend.*, 1927, **184**, 890) is used, modified so that any carbon monoxide that may be produced is burnt by an electrolytically heated platinum wire in the bulb containing the potassium hydroxide. The method is universally applicable, and, by weighing the precipitates on special small Pregl filters, the proportion of carbon may be calculated without using a microbalance. The method is regarded as nearly as accurate as Pregl's method. Such substances as graphites, osazones, picrates, alkaloids, silicotungstates, and mercury compounds were satisfactorily analysed.

D. G. H.

Physical Properties of Pure Triglycerides. R. B. Joglekar and H. E. Watson. (*J. Soc. Chem. Ind.*, 1928, **47**, 365-368T).—Various pure triglycerides have been prepared by a modification of Bellucci's process, a mixture of three molecules of glycerol and one of the acid being heated to 180° C. and the temperature gradually raised to 215° C. during 2 hours at 30-40 mm. pressure, then to 250° C. in 1 hour; this temperature was maintained for 1 hour, while the pressure was reduced to 6 mm. The crude products were crystallised six or seven times from concentrated alcohol and recrystallised from light petroleum and ether, the last traces of solvent being expelled at 100° C. under reduced pressure. The melting point and refractive index are of little value as criteria of purity, and the densities, viscosities, and solidification points vary appreciably even when a fair degree of purity is attained. The viscosity appears to be the most sensitive test for impurity, but its determination requires very careful temperature adjustment, and for practical testing the solidifying point is almost as accurate, and far easier to measure. As regards the complex nature of the solidification of the

triglycerides, it is sufficient to regard these as existing in two forms, the β -form obtained by crystallisation from solvents, and the α -form by heating the β -form to its melting point, which appears to be a transition temperature. When a triglyceride is cooled, crystallisation does not occur without seeding until the solidification point of the α -form is reached. In the neighbourhood of this temperature, once crystallisation has started, the transformation $\alpha \rightarrow \beta$ takes place in a few minutes, and results in resolidification. Consistent values are obtainable if 3 grms. or more are introduced into a 2 cm. tube surrounded by an air-jacket immersed in a bath, the temperature of which is raised during the experiment so as to be not more than 4° C. below that of the substance. Stirring has no effect with tristearin, but should be adopted with the lower members, as it reduces the time required for the temperature rise. The lower melting point (2) is taken as the temperature of initial softening in a capillary tube with a temperature rise of about 1° C. per minute, and the higher (1) as the temperature of complete liquefaction under similar conditions. The results obtained are summarised in the following table:

	Caprin.	Laurin.	Myristin.	Palmitin.	Stearin.
Melting point (1)	31·6	46·2°	56·5°	65·6°	71·8° C.
" " (2)	—	18·0°	33·0°	46·2°	55·0° C.
Solidifying point	30·3°	45·3°	56·1°	65·2°	71·3° C.
Viscosity at 70° C.	0·0688	0·1030	0·1342	0·1679	0·1850(75°C)
	60° C.	60° C.	60° C.	80° C.	80° C.
Refractive index at °	1·4370	1·4402	1·4428	1·4376	1·4395
Density at °	0·9059	0·8943	0·8860	0·8663	0·8632
Surface tension	27·3	27·9	28·7	27·6	28·1

For tristearin-tripalmitin mixtures containing from 25 to 50 per cent. of tristearin double solidification points may be observed, but this has not been found with other mixtures. The complete higher solidification curve, unlike that for the corresponding acids, is characteristic of a simple mixture. The refractive index of tristearin-tripalmitin mixtures is a linear function of the composition at both 70° and 80° C. The density, viscosity and surface tension of these mixtures present no unusual features.

T. H. P.

Determination of Neutral Oil in Sulphonated Oils. Committee Report. G. W. Priest. (*J. Amer. Leather Chem. Assoc.*, 1928, 23, 599.)—Three samples of sulphonated oil prepared in the laboratory were analysed in three laboratories for their neutral oil content by five different methods:—(1) The official method of the American Leather Chemists' Association which necessitates a complete analysis (*J. Amer. Leather Chem. Assoc.*, 1920, 283). (2) Lewkowitch's method, in which 30 grms. of the oil are dissolved in 50 c.c. of water, and 20 c.c. of ammonia and 30 c.c. of glycerin are added, the mixture being extracted with two portions of 100 c.c. each of ether. The ethereal extract is washed, evaporated and dried to constant weight. (3) The same method as No. 2, but with the use of only 10 grms. of the oil. (4) The same method as No. 3, but with petroleum spirit used instead of ether. (5) Ten grms. of the oil are dissolved in 50 c.c. of water. Fifty c.c.

of alcohol are added, and the mixture rendered neutral to phenolphthalein with $N/2$ potassium hydroxide solution. The mixture is extracted three times with petroleum spirit, the mixed extracts being washed, evaporated and dried to constant weight.

All three laboratories condemn method No. 1 as giving excessively high results in neutral oil content. One laboratory recommends method No. 2 until further research shows which solvent is the best. The other laboratories recommend method No. 5 as giving by far the best separation of the emulsion and the clearest solutions, but not necessarily the most accurate results. There was difficulty in drying the neutral oils to constant weight, as they did not become constant after 23 weighings covering a period of 30 days, the losses in weight varying between 25 and 5 per cent.

R. F. I.

The Cold Test for Neatsfoot Oils. A. C. Orthmann and W. J. Arner. (*J. Amer. Leather Chem. Assoc.*, 1928, 23, 595.)—The test described was devised to overcome the inaccuracies of existing methods, in which the low temperature obtained by the use of the usual freezing methods is uncontrollable. In this new method the low temperature is obtained by passing dry air through ether contained in a Dewar flask. A test-tube, 23 cm. long \times 1.5 cm. internal diam., is filled to a depth of 3 cm. with the oil. The tube is provided with a rubber stopper through which a special long-stemmed thermometer passes, reaching to just below the surface of the oil, the scale being wholly above the rubber stopper. The long test tube is inserted in another rubber stopper (with 3 holes) fitting the 3-walled Dewar flask, and provided with two tubes for the aspiration of dry air through the ether with which the Dewar flask is three-quarters filled. The initial temperature of the oil is that of the laboratory, and when the temperature has fallen to about 10° C. above the expected pouring point, observations are made at each degree, the whole apparatus being tilted to see whether or not the oil still flows. The "pour-point" is taken as 1° C. above the point at which the oil ceases to flow. The cloud-point is also easily obtained, since the whole apparatus is transparent.

Tests carried out on a number of oils by this method and by that proposed by the American Society of Testing Materials showed good uniformity, but the new method is much simpler and more rapid. The apparatus is obtainable from Messrs. Carl Stelling, Hamburg.

R. F. I.

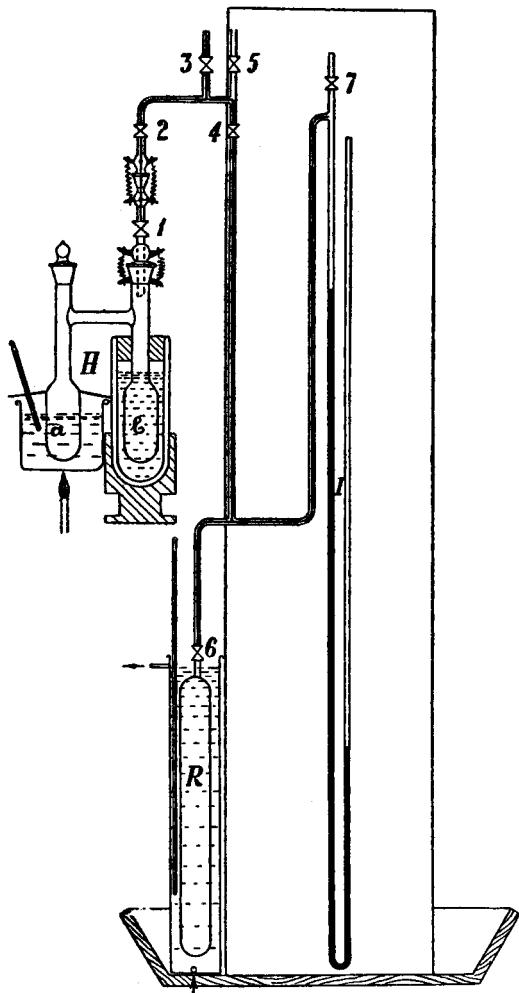
Note by Abstractor.—In the discussion which followed the paper several speakers expressed the view that the Cold Test for Neatsfoot Oils is obsolete.

Determination of the Hydrogen Value of Unsaturated Compounds. H. I. Waterman, J. N. J. Perquin and H. A. van Westen. (*J. Soc. Chem. Ind.*, 1928, 47, 363–365T.)—The procedure described represents an attempt to devise a more widely applicable method than that of Grün (*Die Analyse der Fette und Wachse* 1925, p. 188) for determining the hydrogen value of unsaturated compounds, including those with high vapour pressures at the ordinary temperature. The catalyst used consists of active palladium on norit or carboraffin as support,

and is prepared by Kaffer's method (*Ber.*, 1924, 57, 1263), the reduction being effected by hydrazine sulphate. The catalyst is prepared to contain 10 per cent. of palladium, calculated on the air-dry carbon. The hydrogen used is freed from oxygen by passage over a red-hot platinum star, then dried by concentrated sulphuric acid, and finally led through

a vessel placed in liquid air. The gas is brought, *via* the taps 5, 4 and 6, into the evacuated vessel R (about 1600 c.c.), which is carefully calibrated beforehand by weighing full of water. This calibration may be controlled by removing part of the hydrogen with a Töpler high-vacuum pump working automatically, the pressure and temperature of the gas in R, surrounded by a water-jacket, being determined before and after the suction. This method gave 1586.5 c.c., and the weighing method 1588.2 c.c., for the capacity of R at 0° C. The hydrogenation apparatus H, evacuated beforehand, is now filled with a known volume of hydrogen by opening taps 1 and 2, reading the pressure on the manometer, and then closing 1 and 2. The catalyst (2 grms.), freed from gas by being heated in an oil-bath at about 200° C. under constant pumping (1 hour usually sufficient), and previously introduced into tube *a*, and tube *b*, containing a weighed amount of the unsaturated substance, having been placed in liquid air, vessel H, including the tubes up to tap 6, is pumped wholly free from air.

Before the commencement of the hydrogenation the vessel *a* is cooled with liquid air, so that the substance in *b* distils from *b* to *a*, *b* not being cooled during this operation. Then, after introduction of a known volume of hydrogen, the hydrogenation starts and may be accelerated by shaking vessel H, which is separated from the apparatus after taps 1 and 2 have been closed. After hydrogenation, the tube *a* is cooled with liquid air, and the vessel H is completely evacuated after being reconnected, the residual hydrogen, including that between taps 1, 2, 3, 5, and 6, being pumped through a coil cooled with liquid air into a special measuring tube by the Töpler pump.



The reaction product, if volatile, is now passed into *b* by placing the liquid air cooling vessel round *b* and no longer cooling *a*, which is finally heated in an oil-bath to about 200° C., in order to add any hydrogen adsorbed by the catalyst to the quantity in the measuring tube. The weight of the volume of the hydrogen used in the hydrogenation is calculated and expressed as per cent. of the unsaturated compound taken. The maximum error seems to be about 5 c.c. of hydrogen, or ± 0.5 mgrm. The duration of the contact between catalyst, substance and hydrogen has been taken at about 15 mins., but this may possibly be lessened. The following hydrogen values were obtained by this method: cyclohexane, practically nil; benzene, 0; amylene, a value equal to that calculated from the known bromine value.

T. H. P.

Two New Methods for Determining Phenol in Waste Liquors. H. Dehe. (*Chem. Ztg.*, 1928, 52, 983-985).—The phenol in waste liquors from gas-works, etc., may be determined by the two following methods:—To 200 c.c. of the well-mixed liquor are added 10 c.c. of saturated zinc acetate solution, 40 c.c. of 5 per cent. silver nitrate solution (if much hydrocyanic or thiocyanic acid is present, a greater volume of the silver nitrate or a more concentrated solution must be used), and 10 c.c. of dilute sulphuric acid (1 : 3), the containing flask being left for 3-5 hours with occasional vigorous shaking. One-half of the total volume is then filtered, and the filtrate acidified with dilute sulphuric acid, treated with a few drops of 3 per cent. hydrogen peroxide solution, and distilled until the distillate fails to respond to the test for phenol with Millon's reagent and nitric acid. The total distillate is rendered strongly alkaline with concentrated sodium hydroxide and evaporated, the cold residue being transferred to a 500 c.c. flask and made up to volume.

(1) *Iodimetric determination.*—Ten c.c. (or more if little phenol is present) of this solution are heated to 50-60° C. with water and treated with 15 c.c. (more if the phenol-content is high) of 0.1 *N* iodine solution, allowed to cool, acidified with about 15 c.c. of diluted sulphuric acid (1 : 3), and titrated with 0.1 *N* thio-sulphate solution (1 c.c. of 0.1 *N* iodine corresponds with 1.567 grm. of phenol).

(2) *Fractional titration.*—About 55-60 c.c. of the distillate made up, as above, to 500 c.c., are left for a few minutes in a closed Erlenmeyer flask with as much solid baryta as will lie on the point of a knife, the flask being shaken occasionally. After removal of the precipitate by filtration, 25 c.c. of the filtrate are treated with about 10 drops of dilute sulphuric acid (1 : 3), the barium sulphate being filtered off and washed two or three times with as little carbon dioxide-free water as possible. If any barium sulphate passes the filter, the filtrate is returned to the filter before the latter is washed. Exactly 0.2 c.c. of 0.1 per cent. alizarine yellow solution is then added, and the P_{11} of the solution adjusted to 11.04 by means first of strong sodium hydroxide solution and dilute sulphuric acid and afterwards of 0.1 *N* sodium hydroxide and 0.1 *N* acid, use being made of a buffer solution consisting of 50 c.c. of 0.1 *N* sodium hydroxide and 3 c.c. of 0.1 *N* hydrochloric acid. Exactly 0.1 c.c. of 1 per cent. phenolphthalein solution is next added, the solution being

titrated to $P_H = 8.4$, with the help of a buffer solution containing 62 c.c. of borate solution (12.404 grms. H_3BO_3 and 10 c.c. of 0.1 *N* sodium hydroxide, made up to 1 litre with water free from carbon dioxide) and 38 c.c. of 0.1 *N* hydrochloric acid. From the volume of 0.1 *N* acid used, the correction for pure water is subtracted; 1 c.c. of 0.1 *N* acid corresponds with 9.4 mgrms. of phenol or, if the above conditions are observed, the number of c.c. of 0.1 *N* acid, when multiplied by 2.09, gives the number of grms. of phenol per litre of the waste liquor. If the latter contains less than 4 grms. of phenol per litre, the 500 c.c. of distillate is evaporated to 250 c.c., the factor then becoming 1.045. T. H. P.

Determination of Ionone. R. D. Hendriksz and A. Reclaire. (*Perf. and Ess. Oil Rec.*, 1928, 19, 493.)—Five c.c. of the ionone are heated under a reflux condenser for two hours with a mixture obtained by dissolving 15 grms. of hydroxylamine hydrochloride in 37.5 grms. of water, adding 18 grms. of potassium hydroxide dissolved in 37.5 grms. of water, and, if necessary, filtering. After the boiling, the liquid, as hot as possible, is poured into a separating funnel, the aqueous layer being run off and the oximated oil washed thrice with hot brine and filtered as hot as possible (in a little drying oven at 100° C.). The nitrogen in about 0.5 to 1 gm. of the oximated oil is determined by the Kjeldahl-Gunning method (see *ibid.*, 1927, 18, 130; 1928, 19, 143), the oxidation being usually complete in 2–3 hours. If *a* is the number of c.c. of 0.2 *N* sulphuric acid required per 1 gm. of the oil, the percentage of ionone in the sample is given by $53.82 a / (14 - 0.042 a)$.

T. P. H.

Inorganic Analysis.

Precipitation of Lead by *o*-Oxyquinoline. V. Marsson and L. W. Haase. (*Chem. Zeit.*, 1928, 52, 993–995.)—Lead is precipitated as flocculent, yellow oxyquinolate by addition of a cold saturated aqueous solution of the reagent to the feebly ammoniacal solution. The excess of ammonia is expelled by boiling after the precipitation, the precipitate collected after 12 hours' standing in the cold, washed with a minimum of water, dried, and weighed; lead factor, 0.4185. The precipitate has a certain solubility, which is decreased to 0.004 gm. per litre by the employment of 10 times the quantity of reagent required for precipitation. W. R. S.

Determination of Vanadium in Steel. K. Swoboda. (*Chem. Zeit.*, 1928, 52, 1014–1015.)—The sample (2 grms.) in a 500 c.c. flask is dissolved in warm sulphuric acid (1 : 6; 50 c.c.), the loss of water being made good. The boiling solution is oxidised with nitric acid, drop by drop, and an excess of 5 c.c. added; the red fumes are boiled off. Fifty c.c. of 10 per cent. ammonium persulphate are added; boiling is continued for some minutes, and again after addition of ammonia in excess. After the heat is removed, 50 c.c. of ammonium persulphate, and 100 of ammonium molybdate solution, as used for the phosphorus determination, are added. The precipitate is dissolved in strong nitric acid, tungstic

acid remaining in solution ; the clear boiling solution is precipitated by portions of 3 to 4 drops of 10 per cent. sodium phosphate solution at intervals of 20 seconds, until the colour of the precipitate changes from dark orange to pale yellow-orange (10 to 40 drops in all). The change marks complete vanadium precipitation by the formation of the yellow phosphomolybdate. Boiling is continued until the liquid is very concentrated ; the precipitate is then collected and washed with a solution of 20 c.c. of ammonia (0.91) and 25 c.c. of strong sulphuric acid per litre. Filter and precipitate are returned to the flask and heated with 50 c.c. of nitric, 5 c.c. of phosphoric, and 50 c.c. of sulphuric acid (all strong). When the paper has been oxidised, the solution is evaporated until it fumes. After cooling, dilution with water, oxidation with permanganate, and renewed cooling, 50 c.c. of hydrochloric acid (1:1) are added, and the solution boiled down until it fumes copiously. The cold mass is taken up in 250 c.c. of water, and the blue solution is heated to 80° C. and titrated with permanganate. $V=0.915 \text{ Fe}$.

W. R. S.

Precipitation of Tungsten as Mercurous Tungstate. V. Spitzin. (*Z. anal. Chem.*, 1928, 75, 433-440.)—The precipitation of sodium tungstate solutions by mercurous nitrate was investigated. The solution is made neutral to methyl orange with nitric acid, boiled, and precipitated with an excess of a solution of mercurous nitrate (the salt dissolved in water without addition of nitric acid ; solution kept over metallic mercury). The precipitate is washed as usual and ignited in porcelain without the use of a blast burner. Quantitative results are thus obtained without subsequent addition of alkali. If, however, the solution is acid before precipitation and afterwards neutralised by alkali, the recovery is not quantitative ; the explanation advanced is that mercurous tungstate is decomposed by nitric acid into free tungstic acid and mercurous metatungstate, which is soluble.

W. R. S.

Use of Potassium Iodate in Back Titration for the Determination of the Hypochlorite Content of Solutions. J. R. Lewis and R. F. Klockow. (*J. Amer. Chem. Soc.*, 1928, 50, 3243-3244.)—In the determination of hypochlorite by means of arsenite, thiosulphate, or iodide solution, the excess of these reagents may be determined by titration with potassium iodate. (1) A measured volume of the hypochlorite is added to a known volume in excess of standard arsenite solution, the unused arsenite being then titrated with 0.1 N iodate solution in presence of at least 12 per cent. hydrochloric acid ; the results thus obtained are accurate in presence of small proportions of nitrate or chlorate, (2) The hypochlorite is added to the acid or neutral thiosulphate solution, the liquid being then cooled in an ice-bath and titrated with iodate solution ; in this case the results are not concordant if chlorate is present. (3) When the hypochlorite is reduced by potassium iodide the mixture is cooled in ice-water before titration with iodate ; here, too, chlorate vitiates the results.

T. H. P.

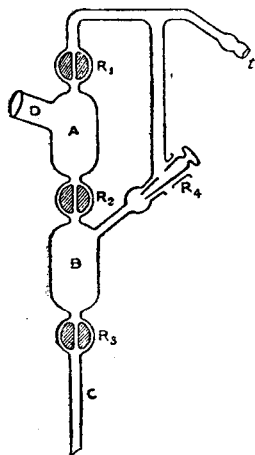
Physical Methods, Apparatus, etc.

Colour-Measurement of Tanning Extracts. M. A. de la Bruere. (*J. Int. Soc. Leather Trades Chem.*, 1928, 12, 485).—The tintometer usually employed for the colour-measurement of tannin extracts (that of Lovibond) is subject to a large personal error with different operators, and with the same operator under different conditions of light. The author finds that by photographing their absorption spectra the red and yellow glasses used allow rays of very varying wave-length to pass. For determining the colour of a solution he suggests the employment of a photo-electric cell coupled to a galvanometer. The rays from an iron arc lamp after passing through a condenser, a colour filter, and the solution under examination, act on a photo-electric cell forming part of a circuit embracing a battery and a galvanometer. Successive readings are taken with the test solution and with distilled water by means of various colour-filters, and the differences obtained are noted. A curve is then constructed, representing the colour of the solution examined, having for abscissae the colours in spectroscopical order, and for ordinates the ratio between the deviations obtained with the test solution and with water.

R. F. I.

Calorimetric Investigations. Benzoic Acid as a Standard for the Standardisation of Combustion Calorimeters. P. E. Verkade. (*Chem. Weekblad*, 1928, 25, 666–667).—The author discusses the decision in 1922 of the International Union of Pure and Applied Chemistry to adopt as standard in the calibration of bomb calorimeters the value 6319.0 15° cal. for the heat of combustion of benzoic acid, corresponding with the isothermal heat of combustion of 1 gm. weighed *in vacuo* and burnt at 20° C. He concludes that this value differs but little from the true figure, though the results of other workers indicate that some difficulty may be experienced in maintaining a constant temperature of 20° C. during the experiment, and it is desirable that such experiments be arranged so that the mechanical equivalent of heat is not required for the calculation of the result in calories. The temperature coefficient is -0.238 cal./°C., and the factors 1.00084 and 1.00075 may be used to obtain the values when the benzoic acid is weighed in air with platinum and brass weights, respectively. The values in 15° cal. are tabulated for temperatures between 0° and 21° C.

J. G.



Separator for Fractional Distillation under Reduced Pressure. R. Delaby and R. Charonnat. (*Bull. Soc. Chim.*, 1928, 43–44, 1287–1288).—The apparatus shown in the diagram consists of 2 bulbs, A and B, of about 60 c.c. capacity for ordinary laboratory work, the upper bulb being connected at D with the side tube of the distilling flask or the refrigerator. R_1 , R_2 , and R_3 are stop-cocks, R_4 is a stopcock allowing for the entrance

of air, and t connects with the vacuum pump. At the beginning of the distillation R_1 and R_2 are open, R_3 and R_4 shut, and the first fraction collects in A and runs into B. R_2 is then closed, air allowed to enter by R_4 , and the fraction run out through R_3 . During this time the second fraction is collecting in A. R_3 is then shut, followed by R_1 , to allow a vacuum to re-form in B, R_4 being turned to its original position, and immediately after R_1 is again opened. D. G. H.

Automatic Pipette. M. Hyman. (*J. Soc. Chem. Ind.*, 1928, 47, 3681.)—

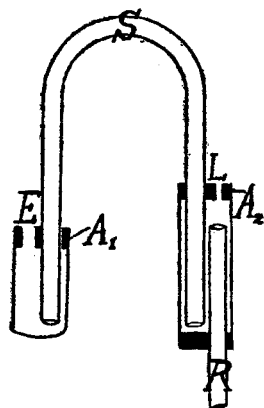
The arrangement shown allows a large number of equal portions of a liquid to be measured out quickly and accurately. The stem of the pipette is cut off at the mark and is fitted, by means of a rubber stopper, into the wide tube D about 3 inches long. This tube is constricted at A, and is connected by rubber tubing ($1\frac{1}{2}$ inches long) with the glass tube C which serves as mouthpiece. The pipette is filled by suction until the liquid overflows into D. The rubber tubing is then compressed, the level of the liquid falling somewhat below the graduation mark. The mouthpiece is then closed with the forefinger and the rubber tubing released, with the result that the liquid rises again in the pipette and overflows. The pipette, held by the mouthpiece only, is now raised from the liquid and the contents are delivered into the receiving vessel, any necessary drainage time being allowed as usual. When the trap D becomes full of overflowing liquid, the rubber tubing and mouthpiece are removed, and the liquid is poured out.



T. H. P.

Practical Siphon. H. Wentzel. (*Chem.*

Zig., 1927, 52, 898.)—The siphon, which is particularly suitable when the rate of flow of liquid is to be controlled (*e.g.* in cooling vessels), consists of an inverted U-tube S, the ends of which are held in wider tubes by the stoppers A_1 and A_2 . These are provided with holes E and L for liquid and air inlets, respectively, and R is an adjustable outlet tube.



J. G.

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A summary of the interim report of the British Association Research Committee on the sources of early Sumerian copper—Analyses of bronze from Ur—Egyptian bronze—Ancient ores.

THE PROTECTION OF ANIMAL FIBRES AGAINST CLOTHES MOTHS AND DERMESTID BEETLES. By C. O. CLARKE. *J. Text. Inst.*, 1928, 19, 295-320 (December).

Species of clothes moths—Life history—The Dermestidae—Methods of control—Sterilisation and fumigation—"Moth balls," etc.—Outline of patents—Methods of rendering the fibre moth-proof—List and outline of patents—References to the literature—Illustrative plates.

RECENT DEVICES FOR MEASURING THE FLOW OF AIR. By R. A. H. FLUGGE-DE-SMID. *J. Chem. Met. & Mining Soc., S. Africa*, 1928, 29, 82-95 (October).

Measurement of low velocities in mines—Measurement of high velocities—Pitot tube and manometer—Micromanometer—Measurement of the flow of compressed air—Correction for temperature.

Reviews.

LABORATORY MANUAL FOR THE DETECTION OF POISONS AND POWERFUL DRUGS.

By Dr. WILHELM AUTENRIETH, Professor in the University of Freiburg I.B.

Authorised translation by WILLIAM H. WARREN, Ph.D., Professor of Organic Chemistry in Clark University, Worcester, Mass. Pp. xxvi and 698. London: J. & A. Churchill. Price 30s.

Autenrieth's manual has now reached its sixth American edition, the translator being Dr. W. H. Warren. This work, we venture to suggest, should find a home on the shelves of every analyst who may be called upon to make toxicological investigations, and those who have to make assays of pharmaceutical preparations and liquids containing alkaloids will find many useful hints. The examination of blood stains is also included.

The manual is essentially practical, and is chiefly concerned with the chemical nature, isolation and estimation of poisonous substances, so that if detailed knowledge of symptomatology and lethal dose of a poisonous substance is required, other works will have to be consulted.

An examination of analytical methods described reveals that they are for the main part sound, and the worker following the technique advised should bring his investigation to a satisfactory termination. Full references are given to original papers, so that the bibliography may be looked up. The chief line of criticism that may be directed to this work is in the form of errors of omission rather than commission. To mention a few, there is no reference to the Hartridge reversion spectroscope for the estimation of carbon monoxide in blood, and we should have thought that the estimation of ethyl alcohol in blood and urine had assumed sufficient forensic importance to warrant inclusion. In the section on blood stains the valuable haemochromogen crystal test, as worked out by Takayama, should be mentioned.

The book contains two indexes, author and subject, both of which are extremely thorough.

G. ROCHE LYNCH.

HANDBUCH DER BIOLOGISCHEN ARBEITSMETHODEN, E. ABDERHALDEN. Abt. IV, ANGEWANDTE CHEMISCHE UND PHYSIKALISCHE METHODEN. Teil 8, Heft 9. DIE REFRAKTOMETRISCHE UNTERSUCHUNG DER MILCH. By E. REISS. Berlin: Urban and Schwarzenberg, 1928.

Those workers who, interested in the attention which has recently been given to the use of the refractometer in milk analysis in this country, turn to this book for full information on the subject will be disappointed. It is a small pamphlet, containing in all some twelve pages of text, although the title-pages, tables of contents, and index of the whole volume, of which this forms a part, are included. The index extends to forty pages, each of three columns, so that this section will be necessary for all those who have the remainder of the volume.

Of the twelve pages of text, six and a half are taken up with figures and tables. One page contains a description of the instrument and introductory remarks, whilst an adequate description of the determination of fat in milk and cream occupies three pages, although this is a process which is not likely, in view of the accurate and convenient mechanical methods now available, to be used to any extent in this country. One page contains an outline of the methods used for the determination of the refraction of milk serum, whilst the determination of lactose by similar means completes the story in about half a page.

It is a little difficult to see of what use such an outline can be to anyone. To those experienced in such methods the almost complete absence of guides to interpretation and the sketchy method of treatment throughout will prove particularly irritating, whilst those seeking their first knowledge of the subject in these pages may easily be misled into thinking that the various processes described yield far more definite information than they actually do.

As so much work has been done on this subject in Germany, it seems strange that the opportunity afforded to the writer, of collecting together the available information, has not been utilised to a much greater extent. This section will be of no assistance to those who wish to decide for themselves what legitimate value the refractometer has in milk analysis.

G. D. ELSDON.

PHOTOCHEMICAL PROCESSES. By GEORGE B. KISTIAKOWSKI, Research Associate in Chemistry, Princeton University. American Chemical Society Monograph Series. New York: The Chemical Catalog Company, Inc. 1928. Price \$5.50.

The kinetics of photochemical reactions has been treated in this book from the standpoint of the quantum theory. In an introductory chapter the author briefly examines the theories of the primary process accompanying the absorption of light by molecules, and the mechanism of the resulting chemical change. Then follow chapters on the Einstein Equivalence Law, Chain Reactions, Photosensitisation, Catalysis and Inhibition, and the Effect of Temperature and the Frequency of Radiation on the Rate of Photochemical Reactions.

The author has accomplished the very difficult task of co-ordinating a mass of heterogeneous information into a highly readable book. This has been done without the sacrifice of detail, which makes it still more remarkable. The experimental data have been subjected to a detailed examination, and the author has dealt with contradictory evidence in a critical manner. The skill with which he has analysed the information available on photochemical processes has added very largely to the value of the book.

If there be any point of criticism that can be raised, it is that the introductory first chapter is too condensed for a student reading the book for the first time. This chapter might with advantage be treated at more length, and in a more elementary manner.

The author is to be congratulated on a good, clear account of the basis of photochemistry which takes the subject further than any previous text-book on the subject.

W. E. GARNER.

GLYCEROL AND THE GLYCOLS. By J. W. LAWRIE, Ph.D. Pp. 447. New York: The Chemical Catalog Co., Inc. 1928. Price \$9.50.

This volume is one of the American Chemical Society's series of monographs; its author is a research chemist with the Du Pont Co., and he obviously has experience of the glycerin industry. There is no other volume, so far as the reviewer is aware, which concentrates so much information on glycerin; it is almost an encyclopaedia of the published work on either glycerin or ethylene glycol; there are but few important papers which are not noted. This fact is a distinctive merit, but also has disadvantages, for one finds that it is so encyclopaedic that it is not critical. On each subject there are described many methods, whether it be of manufacture, distillation, or analysis, but the critical faculty is lacking; there is no indication which process is to be recommended, and some are of no real value or importance.

After a short historical chapter, there follow about 100 pages describing processes of saponification, evaporation and distillation; not much is novel, but all the more modern types of multiple effect evaporators are carefully described. Then there is a 50-page chapter on fermentation glycerin; this is particularly interesting, because it is less well known; all the patented processes are described, but again without sufficient criticism, as some are not of any great merit. The difficulties of purification and the need of extra distillation are not quite sufficiently brought out here; the optimistic statements of inventors or patentees are cited, often without comment. To read all these one would almost think that fermentation glycerin, instead of being unable to compete with the product of the soap works, was an established manufacture; it certainly is not in Europe, despite its importance to Germany during the War. Chapter VI collects the published data on physical properties, both the common and uncommon ones.

Chapters VII and VIII give the chemical reactions of glycerol, and IX and X its quantitative determination. These chapters seem to the reviewer the weakest, for they give methods without comment and end with tables showing erroneous results; these are to illustrate the difficulties of glycerin analysis, but really they only show bad working; analyses of crude glycerin which only add up to 96 to 99 per cent. are manifestly wrong, and could only be passed out by a blunder; they are not representative. It is possible to check the results of glycerin analyses by addition and specific gravity calculations so as to eliminate such errors; so, apart from human fallibility, which makes occasional blunders escape the most systematic checks, these should not appear. The main causes of differences in glycerin analyses when they do arise, which is not frequently, is the sampling in the presence of settled salt. The method for water estimation (p. 296) is older than stated; it was worked out many years ago by R. G. Grimwood.

Chapters follow on commercial utilisation, production and prices, and on nitroglycerin; perhaps the last is better dealt with in books on explosive manufacture. Chapter XIV is of interest; it treats of the manufacture of ethylene glycol and its properties, advantages and disadvantages. Lastly, there is a brief prophecy (Chap. XV) of the probable future of the industry which is of particular interest, as the publication coincides with a period of acute depression in the glycerin trade. Dr. Lawrie does not make a very cheerful prophet, but we shall not all agree with his views.

The book is a really valuable one, and is carefully written and free from serious errors. In a future edition Rayner's papers on the formation of trimethylene glycol in crude glycerin ought to be noted.

H. E. Cox.

LES PLANTES À PARFUMS DES COLONIES FRANCAISES. Report by M. E. Maunier to the Congres du Comite National des Conseillers du Commerce Exterieur, Nice, Janvier, 1928. Pp. 134. Marseilles: Institut Colonial. 1928. Price, post free, France 10 fr., abroad 12 fr.

This address, which has been reprinted by the Institut Colonial de Marseille, gives details for each colony as to its present output of perfumery materials, and of its possibilities as regards soil and climate for their further development. It is claimed that the French colonies, comprising nearly 11,000,000 sq. kilometres, and having the most varied soil and climate, should be able to produce practically all the natural raw materials required by the perfumer, instead of France importing from foreign countries, as in 1926, more than 15,000 quintals of essential oils, valued at more than 153,000,000 francs.

W. H. SIMMONS.

Publications Received.

ASPECTS OF AGE, LIFE AND DISEASE. By SIR HUMPHREY ROLLESTON, M.D.
London: Kegan Paul, Trench, Trubner & Co., Ltd. 1928. Price 10s. 6d.
net.

A collection of papers on subjects allied to medicine, including: Concerning old age—Some medical aspects of holidays—The medical aspect of tobacco—Professional careers—Poetry and physic—Medical aspects of Samuel Johnson, etc.

EXPERIMENTS WITH HANDWRITING. By ROBERT SAUDEK. Pp. 395. London:
George Allen and Unwin Ltd. 1928. Price 10s. net.

The development of the graphic faculty—Relative speed of the act of writing—Authentic and spurious expression in handwriting—The central nervous system and the act of writing—Individual features of handwriting and their symptomatic significance.

ANNUAL SURVEY OF AMERICAN CHEMISTRY. Vol. III. Edited by CLARENCE J.
WEST. New York: Chemical Catalog Co. 1928. Price \$3.00.

SIMPLE QUALITATIVE ANALYSIS. (Practical Chemistry, Part III.) E. J. HOLM-
YARD. London: G. Bell & Sons. Price 1s.

Elementary text-book.

QUALITATIVE ANALYSIS. By W. WARDLAW, D.Sc., and F. G. PINKARD, M.Sc.
Pp. 166. London: Longmans, Green & Co. 1928. Price 3s. 6d.

Elementary text-book. Scheme of analysis used in laboratories of Birmingham University.

FOOD PRODUCTS. THEIR SOURCE, CHEMISTRY AND USE. By E. H. BAILEY and
H. S. BAILEY. Pp. 563. Philadelphia: P. Blakiston's Son & Co. 1928.

HERMES OR THE FUTURE OF CHEMISTRY. By T. W. JONES. Pp. 88. London:
Kegan Paul, Trench, Trubner & Co., Ltd. Price 2s. 6d. net.

A volume in the "To-day and To-morrow" Series.

ALKALINE ACCUMULATORS. By J. T. CRENNELL and F. M. LEA. Pp. 132.
London: Longmans, Green & Co. 1928. Price 10s. 6d. net.

A HANDBOOK OF CLINICAL CHEMICAL PATHOLOGY. By F. SCOTT FOWWEATHER,
M.D., M.Sc. Pp. 216. London: J. & A. Churchill. 1929. Price 8s. 6d.

THE STRUCTURE OF AN ORGANIC CRYSTAL. By SIR W. H. BRAGG, F.R.S. (Fison
Memorial Lecture, 1928.) London: Longmans, Green & Co. Price 1s. 6d.
net.

VOLUMETRIC GLASSWARE. By VERNEY STOTT. Pp. 232. London: Witherby.
Price 20s. net.

CATALYTIC PROCESSES IN APPLIED CHEMISTRY. By T. P. HILDITCH, D.Sc., F.I.C.
Pp. 360. London: Chapman & Hall. Price 16s. net.