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The Examination of Lard

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THE term "lard" is now applied to the fat from any part of the pig, and only relatively small quantities of the product originally limited to the leaf fat are still sold as "butchers' lard" or "home-rendered lard." There is therefore a much wider variation than formerly in the composition of lard, and this involves greater possibilities of adulteration. A considerable amount of fat produced by fat-renderers from the waste from butchers' shops, pigs' bones, etc., is refined and sold, either alone or in admixture with lard. Beef products may readily occur in these mixtures, and during the last two years we have met with numerous samples undoubtedly containing beef fat—in one instance as much as 50 per cent. The difficulty of detecting adulteration is still further increased by the availability of hydrogenated oils of almost any desired consistence. Hence, routine tests such as the determination of the iodine value, refractive index, specific gravity, and melting and solidifying points are no longer sufficient criteria of genuine lard. Other tests for particular types of adulterants must be made, and we recommend in particular that the Bömer value of all samples should be determined.

It has been shown that extraordinary variations may occur in the major component fatty acids of lard (palmitic, stearic, oleic and linolic acids) according to the age of the pig^{1,2} and the character of the ration, and such variations, of course, result in some highly abnormal figures for the analytical constants usually determined.

Thus, König and Schluckebier¹ observed a rise in the melting-point of the body fat and a fall in its iodine value as pigs grow older. Ellis and Hankins² confirmed these results and concluded that ingested fat is deposited to some extent with only slight modification of its glyceride structure. In young pigs, ingested fat forms a high proportion of the body fat; in older animals, an increasing quantity of the body fat is derived by synthesis from carbohydrates and protein in the feed, and this fat is usually of a harder type. Callow³ reports a correlation between the iodine value and the rate of deposition of the fat.

Ellis and Isbell^{3,4} found that, with a surplus of fatty food, ingested fat tends to be deposited in the body fat. Their analytical results related to samples of back fat from hogs on different rations, ranging from brewers' rice with protein supplement (which resulted in a very hard lard particularly low in linolic acid)

to soya beans and peanuts (which resulted in very oily carcasses). Some of their results are given in Table I.

TABLE I

	Iodine value	Satd. acids Per Cent.	Oleic acid Per Cent.	Linolic acid Per Cent.
1. Brewers' rice and protein supplement ..	54.7	37.5	56.1	1.9
2. Corn with protein supplement	60.8	37.4	49.5	8.2
3. Peanut meal 1 part, corn meal 2 parts ..	72.6	30.2	52.6	13.0
4. Soya beans + 2.5 per cent. of corn ..	78.3	30.8	44.1	20.0
5. Peanuts	89.6	19.5	55.0	20.3
6. Soya beans	93.2	26.0	39.1	30.6

In a later paper Ellis and Zeller⁵ refer to lard with iodine values of 30 and 100. These were the result of different rations—copra cake on the one hand, and peanuts or soya beans on the other.

Battacharya and Hilditch⁶ also found that the presence of arachis oil in the diet of pigs led to an increase of unsaturation in the stored fat and to an increase in the proportion of linolic acid in the unsaturated acids.

IODINE VALUE.—Various limits have been proposed for the iodine values of average commercial lards. Our results suggest that in some instances the upper limit should be increased. Whereas years ago an iodine value of 66 was rarely exceeded, figures as high as 69 and 70 are by no means uncommon to-day. Table II gives the values obtained on 113 samples examined since 1933. Many lards with high iodine values have been fully investigated but without yielding evidence of adulteration.

TABLE II

IODINE VALUES OF LARDS

Iodine value ..	Below 57	57.0-58.9	59.0-60.9	61.0-62.9	63.0-64.9
No. of samples ..	2	3	13	16	24
Iodine value ..	65.0-66.9	67.0-68.9	69.0-70.9	71.0-72.9	Above 72.9
No. of samples ..	18	21	8	5	3

Of the 113 samples examined, 108 (96 per cent.) fall within the range of 57 to 73.

REFRACTIVE INDEX.—Although the figures recorded by Ellis and Isbell^{3,4} range from 1.4582 to 1.4636 at 40° C. (equiv. to Zeiss butyrometer readings of 48.3 to 56.5), most commercial samples have values falling within a much narrower range. Our results on 105 samples examined since 1933 are given in Table III. The figures tend to be concentrated between 50 and 52.

TABLE III

ZEISS BUTYROMETER READINGS OF LARDS

Zeiss reading ..	Below 49.7	49.7-50.0	50.1-50.4	50.5-50.8	50.9-51.2
No. of samples ..	0	4	9	20	32
Zeiss reading ..	51.3-51.6	51.7-52.0	52.1-52.4	52.5-52.8	52.9-53.2
No. of samples ..	21	16	1	1	1

THE BÖMER VALUE.—The glycerides which separate from an ethereal solution of lard, etc., include most of the higher fully saturated and probably a proportion of the mono-unsaturated glycerides. Since lard normally contains only about 5 per cent. of fully saturated glycerides,⁷ whereas beef fat contains about 17 per cent.,⁸ considerable differences in the amounts of deposits are to be expected. The amount obtained from mixtures, however, varies considerably with the particular samples of lard and beef fat, and no test (including that of Stock⁹) is capable of giving satisfactory quantitative results.

Emery,^{10,11} examining the m.p. of the crystals, concluded that "when the melting-point of the separating glycerides is below 63.4° C. the presence of beef

fat should be suspected, while a melting-point of 63° C. or below can be regarded as positive evidence that the sample is not pure lard." His procedure is substantially reproduced in the British Pharmacopoeia, 1932.

Bömer¹² investigated the saturated glycerides of lard by repeated fractional precipitation and crystallisation from ether. He concluded that tri-stearin was not present and that the glycerides consisted of palmito-distearin (m.p. 68.5° C.) and stearo-dipalmitin (m.p. 58.2° C.). He also found that the palmito-distearin derived from lard differed in m.p. and crystalline form from the palmito-distearin derived from mutton tallow, and concluded that the former was α -palmito-distearin and that the latter contained the palmitic residue in the β -position.

These were based on comparison with synthetic glycerides, prepared by methods which were later discredited, and they were superseded by configurations given by Amberger and Wiesehahn.¹³ The palmito-distearin (m.p. 68° C.) separating from lard was therefore accepted as the β -palmito- $\alpha\alpha'$ -distearin, and the glyceride of beef tallow (m.p. 63.3° C.) was thought to be the α -palmito- $\alpha'\beta$ -distearin. Whilst the palmito-distearin with m.p. 68° C. is doubtless β -palmito-distearin, the compound with m.p. 63.3° C. is not necessarily α -palmito-distearin, but quite possibly β -palmito-distearin containing a little myristo-palmito-stearin or dipalmito-stearin. Many slightly impure palmito-stearins melt at 63–65° C.¹⁴

Bömer¹⁵ subsequently extended the test to the fatty acids of the separated glycerides. He found that the difference between the m.p. of the glycerides and of the fatty acids was about 5.2° C., and in some lards reached 6.9° C. With beef and mutton fats the differences ranged from 0.1° to 2.6° C. He concluded that with glycerides melting between 60° and 61° C. the presence of beef fat is indicated when the difference between the m.p. of the glycerides and of the fatty acids is less than 5.0° C., and with glycerides melting between 65° and 68.5° C., when the difference is less than 3° C.

Sprinkmeyer and Diedrichs¹⁶ applied the method to numerous samples of genuine lard, beef and mutton fats, and also to mixtures. With lards the difference between the m.p. of the least soluble glycerides and their constituent fatty acids ranged from 4.4 to 7.4° C.; with beef and mutton fats the difference ranged from 0.8° to 1.2° C. They suggested the factor $Mg + 2d$, which is the m.p. of the glycerides plus twice the difference, and found this value for lard to range from 73.1 to 76.5 and for beef and mutton fats from 65.2 to 67.3. With mixtures containing 5 to 10 per cent. of beef or mutton fat the value $Mg + 2d$ was always below 72 and usually below 70. The presence of hydrogenated oils or fats in lard affected the factor $Mg + 2d$ to the same extent as the presence of beef fat. Bömer¹⁷ found that with lards the values for $Mg + 2d$ always exceeded 71, whilst the values with beef fat, mutton fat and horse and goat fats were well below this figure.

Jesser¹⁸ obtained results for $Mg + 2d$ ranging from 73.3 to 76.8. Prescher¹⁹ examined 58 fats of known character by the method of Bömer, and also using the difference value formulated by Polenske.^{20,21} Of 25 samples of adulterated lard, in only three, containing respectively 10, 20 and 30 per cent. of beef tallow, could adulteration be detected by the Polenske method; the others, some containing as much as 15 per cent. of beef tallow, gave negative results. The Bömer method failed with only 2 fats—in which 5 and 10 per cent. of beef tallow were present. Eighteen samples of pure lard were passed as satisfactory by the Bömer method, whilst the Polenske procedure gave false indications of adulteration with two.

Vitoux and Muttelet,^{22,23} used acetone for the first crystallisation of the glycerides, and suggested the use of a factor equivalent to $Mg + d$ (in place of $Mg + 2d$); the introduction of this variation does not appear to offer any advantage.

Details of the test as elaborated by Bömer and his co-workers are published in "*Einheitliche Untersuchungen für die Fett- und Wachsendustrie*," issued by the German Commission for Fat Analysis.

In 1920 Kerr²⁴ published details of a test in which only 5 g. of fat were dissolved

in acetone, and the crystallisation was allowed to take place at 30° C. The melting-points of the separating glycerides and of their constituent fatty acids were determined, and the factor $Mg + 2d$ was calculated. Using these conditions Kerr proposed 73° C. as the minimum figure for genuine lards. This test was described in detail in the "*Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists*," and is still included as a "tentative" method in the 4th Edition, published in 1935. It is stated that "when the melting-point of the glycerides obtained by this method is below 63.6° C. the presence of beef fat or other fat containing tristearin should be suspected, and a melting-point of 63.2° C. or lower is evidence that the sample is not pure lard." In the errata to this edition²⁵ is the direction that "if the melting-point of the glycerides + twice the difference between the melting-point of the glycerides and the melting-point of the fatty acids is less than 73° C. the lard is regarded as adulterated."*

In most of our work we have used the procedure specified by the A.O.A.C., and we have found no reason to dispute the standards adopted. In the examination of 15 genuine lards, most of which had been purchased from small pork-butchers' shops as "home-rendered," the lowest figure for the m.p. of the separating glycerides was 64.5° C.; the "difference" figures ranged from 4.2 to 6.1° C., and the factor $Mg + 2d$ from 73.4 to 76.9. Various specimens from a pig's carcass were obtained from a local pork butcher and the fat was rendered in the laboratory. Our results, given in Table IV, also include the figures for two commercial articles described as "Refined lard—prepared from commercial hog fat" and "ham bone fat."

TABLE IV

Source of fat	Iodine value	Butyro-ref. reading 40° C.	Glycerides from acetone (A.O.A.C.)			
			M.p. °C.	M.p. of fatty acids °C.	Diff. °C.	Mg + 2d
Back	61.8	50.4	64.7	59.6	5.1	74.9
Leaf	56.4	49.5	64.9	60.6	4.3	73.5
Midrum	50.2	48.4	64.4	58.3	6.1	76.6
*Collar	70.8	51.7	65.3	60.2	5.1	75.5
Leg bones ..	67.1	51.2	64.7	60.0	4.7	74.1
H-bones	63.9	51.9	64.8	60.4	4.4	73.6
Refined lard ..	—	—	65.0	61.0	4.0	73.0
Ham bone fat ..	—	—	64.7	59.6	5.1	74.9

*Very soft consistence. Ten g. required to give glyceride separation.

Our results on various beef products and some hydrogenated oils and fats, given in Table V, are in agreement with the published work. It will be seen that hydrogenated products usually, but not invariably, cause a lowering of the Bömer value. The hydrogenated sesamé oil had a Bömer value of 74.5, but it should be noted that the m.p. of the glycerides was as high as 68.7° C. In particularly hard fats—beef suet and a hydrogenated cotton-seed oil with iodine value 42—the m.p. of the fatty acids was slightly higher than that of the corresponding glycerides.

In attempts to obtain approximately quantitative results by the Bömer test standard mixtures of genuine lard and beef fat were made and the Bömer values were determined. Table VI contains our results. A regular gradation in the figures is to be observed, and this is shown more clearly by the continuous line in Fig. 1.

With a harder type of fat, such as beef suet, a greater variation in the Bömer figures was observed. The results were somewhat irregular for mixtures containing more than 40 per cent. of suet. A heavily hydrogenated cotton-seed oil (iodine value 42) in admixture with lard also caused a rapid fall in the Bömer values. The results were uniform up to about 30 per cent. admixture and thereafter irregular.

* For a recent report on the Bömer method by a Committee of the American Chemical Society see ANALYST, 1940, 65, 508.

In Fig. 1 the central discontinuous line refers to mixtures of lard and beef suet, and the lowest curve to the mixtures of lard and hydrogenated cotton-seed oil. Two mixtures of lard with 20 and 40 per cent. of beef dripping gave Bömer figures similar to those obtained from the corresponding mixtures with beef suet.

TABLE V

Source of fat	Iodine value	Glycerides from acetone (A.O.A.C.)			
		M.p. °C.	M.p. of fatty acids °C.	Diff. °C.	Mg + 2d
Beef fat	—	61.6	60.9	0.7	63.0
„ dripping	—	60.6	59.8	0.8	62.2
„ suet	—	61.4	61.6	-0.2	—
Edible beef fat	—	61.0	60.6	0.4	61.8
Crude tallow	—	61.5	61.5	0	61.5
Beef tallow, No. 7	—	60.5	60.5	0	60.5
„ „ „ 8	—	60.7	60.6	0.1	60.9
* „ marrow fat	—	59.2	56.0	3.2	65.6
<i>Hydrogenated oils and fats</i>					
Ground nut	73.5	66.2	63.2	3.0	72.2
Lard	57.4	61.8	57.6	4.2	70.2
Palm	40	60.4	54.6	5.8	72.0
Pig fat	43	61.9	57.9	4.0	69.9
Sesamé	56	68.7	65.8	2.9	74.5
Cotton-seed	42	59.8	60.0	-0.2	—
„	68	54.5	49.7	4.8	64.1
Whale	—	56.9	52.7	4.2	65.3

* Very soft fat. Fifteen g. used and glycerides separated only after about 8 days.

TABLE VI

	Melting-points, °C.		Diff. °C.	Mg + 2d
	Glycerides	Fatty acids		
Lard	65.0	59.7	5.3	75.6
Lard + 2 per cent. of beef fat ..	64.4	58.8	5.6	75.6
5 „ „ „ „ „ ..	64.0	59.3	4.7	73.4
10 „ „ „ „ „ ..	63.9	60.3	3.6	71.1
20 „ „ „ „ „ ..	63.3	60.5	2.8	68.9
40 „ „ „ „ „ ..	62.6	61.1	1.5	65.6
60 „ „ „ „ „ ..	61.8	61.2	0.6	63.0
80 „ „ „ „ „ ..	61.4	61.0	0.4	62.2
Beef fat	61.6	60.9	0.7	63.0

The variation in the Bömer values obtained by different workers using the same standardised procedure on the same sample is not unduly large when one considers that differences of only 0.1° C. in each of the two melting-points may result in a difference of 0.5° C. in the value Mg + 2d. With genuine lards or mixtures containing not more than 30 per cent. of adulterant it is unusual for two workers to record values for Mg + 2d differing by as much as 1° C., but with mixtures containing large proportions of adulterant a somewhat greater variation must be admitted.

We have some evidence that with such mixtures some variation results when different quantities of fat are employed. In the method given by the A.O.A.C. there is the direction that "should the volume of crystals materially exceed 3 c.c., take a smaller quantity of lard (3-4 grams) for a new test." From a few experiments we have made it would appear to be equally important to avoid reducing the amount of fat so far that a very small amount of deposit is obtained. This is

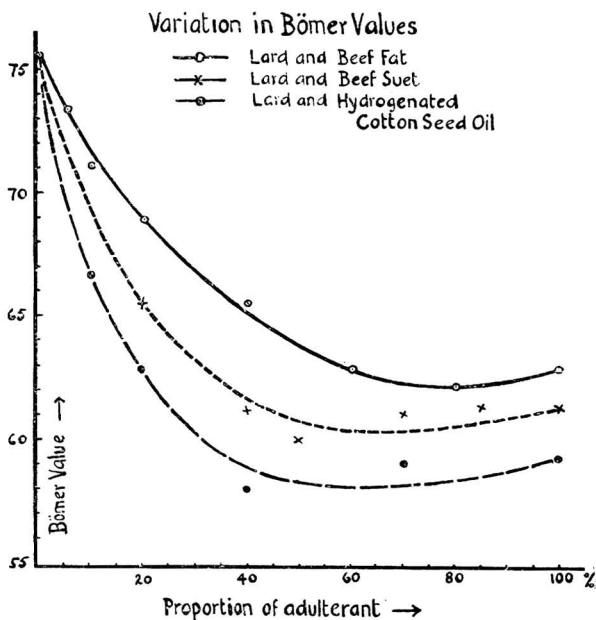
not surprising when one considers the different glycerides which may be deposited from an acetone solution of beef fat. Hilditch and Paul,⁸ in work on the component glycerides of ox depot fat, found that the following glycerides may be deposited from an acetone solution:

Most of the tripalmitin ..	2.8	from a total of 3.4 per cent.
Most of the dipalmito-stearin	5.4	" " " " 7.8 " "
All the palmito-distearin ..	5.8	" " " " 5.8 " "
All the tristearin (if any) ..	0.4	" " " " 0.4 " "
Some of the "oleo" dipalmitin	4.9	" " " " 14.7 " "
Some of the "oleo" palmito-stearin	0.8	" " " " 32.0 " "
All the "oleo" distearin ..	2.3	" " " " 2.3 " "
None of the palmito-di"oleins"	0	" " " " 23 " "
Some of the stearo-di"oleins" ..	1.5	" " " " 10.9 " "

Note.—In the above grouping, "palmito" and "palmitin" include the minor component glycerides of myristic, tetradecenoic and hexadecenoic acids.

It seems reasonable to assume that, even with a constant temperature, different concentrations of fat in the solvent will result in a deposition of variable proportions of the different glycerides. With lard, the glyceride structure of which is given by Hilditch and Stainsby,⁷ it would appear that the deposit will consist almost entirely of the palmito-distearin and the dipalmito-stearin.

FIG. I.



Differences between the melting-points obtained in two laboratories may occur. With certain glycerides and fatty acids a few of the crystals will melt only when the temperature is raised. However, in the method specified by the A.O.A.C. the temperature is recorded when the whole is clear, and we have adopted this procedure throughout. For the determinations we have used the apparatus specified in *Methods of Analysis* (A.O.A.C.), with the addition of a small electric lamp which is shaded and arranged to direct the light through the water in which the tubes are immersed. Duplicate determinations of melting-points in tubes filled from the same batch of material rarely differ by more than 0.1 or 0.2° C.

If a low figure is obtained in the Bömer test a search for iso-oleic acid must be made and the m.p. of the sterol acetates determined. If these tests preclude the presence of hydrogenated oils and fats, the adulterant is beef fat (or mutton or horse fat), and an approximate measure of the proportion of beef fat can be obtained by comparison with known mixtures.

We do not suggest that these curves are exactly reproducible. They will obviously vary to some extent with the particular lard or beef product in the mixtures, and quite clearly only the first portion of the curves may be used. We submit, however, that if the details specified by the A.O.A.C. are closely followed, a Bömer value of 70 indicates the presence of between 5 and 15 per cent. of a beef product, and a value of 65 indicates between 15 and 40 per cent.

THE DETECTION OF HYDROGENATED OILS AND FATS.—Three procedures for determining iso-oleic acid are available, *viz.* separation of the lead salts with ether, the method of Twitchell,²⁶ and the method of Cocks, Christian and Harding.²⁷ For the examination of lard we much prefer the method of Twitchell, since it effects more complete separation of the liquid unsaturated acids. In fact, with genuine lards the iodine values of the "solid" acids separated by Twitchell's procedure are so uniformly low that the test may be used to obtain confirmatory evidence of the presence of substantial amounts of beef fat.

In Twitchell's process two precipitations of the lead salts are made in 95 per cent. alcohol, and this seems to be desirable. It obviously reduces the possibility of obtaining mixed salts containing saturated and unsaturated acids in combination with the same lead atom. In his original paper Twitchell states that with one precipitation the "solid" acids prepared from the insoluble lead salts will have iodine values ranging from 3 to 10, and that no amount of washing of the precipitate will lower this figure. With a second precipitation, however, the iodine value of the "solid" fatty acids is reduced to small proportions. Our results on lard samples are in agreement with this. Hilditch and Priestman²⁸ find that Twitchell's separation results in somewhat low figures for the saturated fatty acids, appreciable quantities of myristic acid (if present), and smaller quantities of palmitic acid, finding their way into the soluble lead salt or "liquid" acid portion. This, of course, is unimportant in the examination of lard for hydrogenated products.

Cocks, Christian and Harding²⁷ state that sometimes they were unable to get consistent results by Twitchell's method, and that the figures for iso-oleic acid were considerably below the truth when the method was applied to certain hydrogenated oils. They recommend an alternative procedure in which there is only one precipitation of the lead salts in 92-93 per cent. alcohol, the insoluble lead salts being filtered off and washed with petroleum spirit. In our hands this method has given unsatisfactory results with lard. On several occasions with known genuine samples the iodine value of the "solid" acids has been as high as 10, and with one lard a value of 14 was obtained. We can only conclude that the "solid" acid fraction is usually contaminated with a substantial amount of "liquid" acids.

For the lead salt and ether method we have followed the details given in Bolton's description²⁹ of the test, with the exception that dilute nitric acid has been used for liberating the fatty acids from the lead salts. Generally speaking, the results have been satisfactory, although we have occasionally encountered explosive boiling (with some loss of the acids) in "refluxing" the lead salts of the "solid" acids with ether. With lard the method has given slightly higher proportions of saturated fatty acids, but the iodine value of the "solid" fraction has tended to be somewhat greater than with Twitchell's method. In a few results with hydrogenated products the percentage of iso-oleic acid obtained after separation by the lead salt and ether method has been slightly less than in the Twitchell method. So far as our experience goes, therefore, we consider that, for our purpose, the greater accuracy in the determination of iso-oleic acid (should it be

present) which may be afforded by other methods must be sacrificed in favour of the more complete removal of the unsaturated "liquid" acids by the Twitchell method. In the examination of lard for the possible presence of small amounts of hydrogenated oil it is our opinion that the method chosen should be the one in which the usual unsaturated acids are most completely removed, so that, if the iodine value of the "solid" acids is appreciable, this shall not indicate an imperfect separation of "liquid" acids, but shall prove the presence of unsaturated solid fatty acids.

Using Twitchell's method on known genuine lards and pig fat, the highest figure we have obtained for the iodine value of the "solid" acids is 2.3, and this was equivalent to an indication of 0.9 per cent. of iso-oleic acid (expressed as a percentage of the total fatty acids). This result was obtained on the specimen of fat rendered from H-bones. More frequently with genuine lards the iodine value was very close to 1.0.

With beef products the iodine value of the "solid" acids is always greater than with lard. Our results have varied from 4.4 to 7.6, and these figures are in agreement with those obtained by other workers.^{26,28} The difference in the figures obtained for genuine lard and beef products appears to be quite significant and, in our opinion, valuable confirmatory evidence can be obtained by using Twitchell's method in the examination of lards which are adulterated with substantial proportions of beef fat.

Table VII contains our results on the specimens of fat rendered from different portions of the pig's carcass (*vide supra*), on a few beef products, and on two samples of "lard" with Bömer values of 63 and 67. The proportions of the acids are expressed as percentages of the total fatty acids.

TABLE VII

				Solid acids Per Cent.	Iodine value of solid acids	Solid saturated acids Per Cent.	Iso-oleic acid Per Cent.
<i>Pig fat</i>							
Back	40.8	0.9	40.4	0.4
Leaf	43.0	0.7	42.7	0.3
Midrum	46.6	0.7	46.2	0.4
Collar	30.3	1.3	29.9	0.4
Leg bones	36.2	2.0	35.4	0.8
H-bones	37.0	2.3	36.1	0.9
<i>Beef products</i>							
Beef fat	43.2	6.9	39.9	3.3
„ dripping	47.1	4.9	44.5	2.6
„ suet	57.5	4.9	54.4	3.1
Edible beef fat	55.9	7.5	51.2	4.7
Crude tallow	50.1	7.6	45.9	4.2
Beef	„	No. 7	..	54.4	6.0	50.8	3.6
„	„	„ 8	..	57.4	6.9	53.0	4.4
„	„	marrow fat	..	38.8	4.4	36.9	1.9
„Lard”	(1)	43.9	5.0	41.5	2.4
„	(2)	39.0	3.1	37.7	1.3

The high results for beef products may presumably be due in part to the presence of some iso-oleic acid, but, probably to a large extent, also to the presence of oleic acid contaminating the "solid" fraction. Grossfeld³⁰ reports the presence of iso-oleic acid in beef fat (1-1.8 per cent.) and in one sample of mutton fat (5.35 per cent.). It is certainly not easy to reduce the iodine value of the solid acids to low dimensions by further solution and reprecipitation. In one of our experiments the solid fatty acids were separated from four samples of the same beef suet. With the first sample two precipitations were made, as in the standard Twitchell method, and with the others, three, four, and five precipitations were made. The iodine values of the "solid" acids so prepared were 5.0, 3.8, 3.0 and 2.6.

In Table VIII the results obtained by Twitchell's method and by the lead salt and ether method are compared, the proportions of acids again being expressed as percentages of the total fatty acids.

TABLE VIII

				Method	Solid acids Per Cent.	Iodine value of solid acids	Solid saturd. acids Per Cent.	Iso-oleic acid Per Cent.
Leaf fat	Twitchell	43.0	0.7	42.7	0.3
				Lead salt and ether	45.7	3.0	44.2	1.5
				Do.	44.4	2.0	43.4	1.0
Pig leg bone	Twitchell	36.2	2.0	35.4	0.8
				Lead salt and ether	*—	3.1	—	—
,, caul fat	Twitchell	46.6	0.7	46.2	0.4
				Lead salt and ether	48.2	1.4	47.4	0.8
Beef fat	Twitchell	43.2	6.9	39.9	3.3
				Lead salt and ether	47.2	9.6	42.2	5.0
"Lard" (1)	Twitchell	43.9	5.0	41.5	2.4
				Lead salt and ether	47.0	4.1	44.9	2.1
,, (2)	Twitchell	39.0	3.1	37.7	1.3
				Lead salt and ether	42.2	5.8	39.5	2.7
Hydrogenated pig fat	Twitchell	58.6	16.6	47.8	10.8
				Lead salt and ether	56.6	15.7	46.7	9.9
,, sesamé oil	Twitchell	60.4	29.5	40.6	19.8
				Lead salt and ether	58.8	28.2	40.4	18.4
,, cotton-seed oil (I.V. 42)	Twitchell	71.5	25.2	51.5	20.0
				Lead salt and ether	65.9	23.0	49.1	16.8
,, cotton-seed oil (I.V. 68)	Twitchell	42.4	34.1	26.3	16.1
				Lead salt and ether	*—	33.6	—	—

* Some loss of solid acids.

The figures in Table IX indicate that the results obtained in Twitchell's method are reproducible. The acids are expressed as percentages of the total fatty acids. For the first and third samples the duplicate determinations were made by different workers.

TABLE IX

	Wt. of fatty acids used g.	Solid acids Per Cent.	Iodine value of solid acids	Solid saturated acids Per Cent.	Iso-oleic acid Per Cent.
Hog fat (commercial sample)	2.919	38.0	2.2	37.1	0.9
	3.276	39.1	2.5	38.0	1.1
Pig leg bones	4.533	36.2	2.0	35.4	0.8
	3.118	36.1	1.9	35.3	0.8
Hydrogenated arachis oil	2.996	43.5	47.2	20.7	22.8
	1.939	45.3	47.4	21.4	23.9
Beef suet	2.820	56.9	5.0	53.7	3.2
	3.035	57.5	4.9	54.4	3.1
	3.009	56.0	5.0	52.9	3.1
,, fat	3.135	43.2	6.9	39.9	3.3
	3.352	42.8	6.6	39.7	3.1

OTHER TESTS

EXAMINATION OF STEROLS.—A convenient procedure is given by More,³¹ while Hawley³² describes a different technique, in which the digitonide is precipitated in a chloroform solution of the fat. We prefer the former method.

It has been our practice to acetylate the digitonides obtained by More's procedure in a small bottle under pressure, although, according to some workers, boiling with acetic anhydride in an open tube is adequate. It is not necessary to use an oil-bath at 140° C. to remove the excess of acetic anhydride, and in fact such treatment is objectionable. It frequently results in a darkening of the material, and this necessitates treatment with charcoal. Our procedure is to remove the glass stopper from the acetylation bottle, and to substitute a small wash-bottle fitting, one tube of which extends well into the bottle. The other tube, which projects only just below the cork, is connected to a water pump and the bottle is then supported in a small bath of boiling water. The excess acid is soon removed and a very clean acetate is obtained. This is subjected to the usual crystallisation process. We find the Jena micro filter (12 G3—capacity 2 ml.), recommended by Hawley, to be very useful, and with quite small amounts of material it is easily possible to make four or even five recrystallisations.

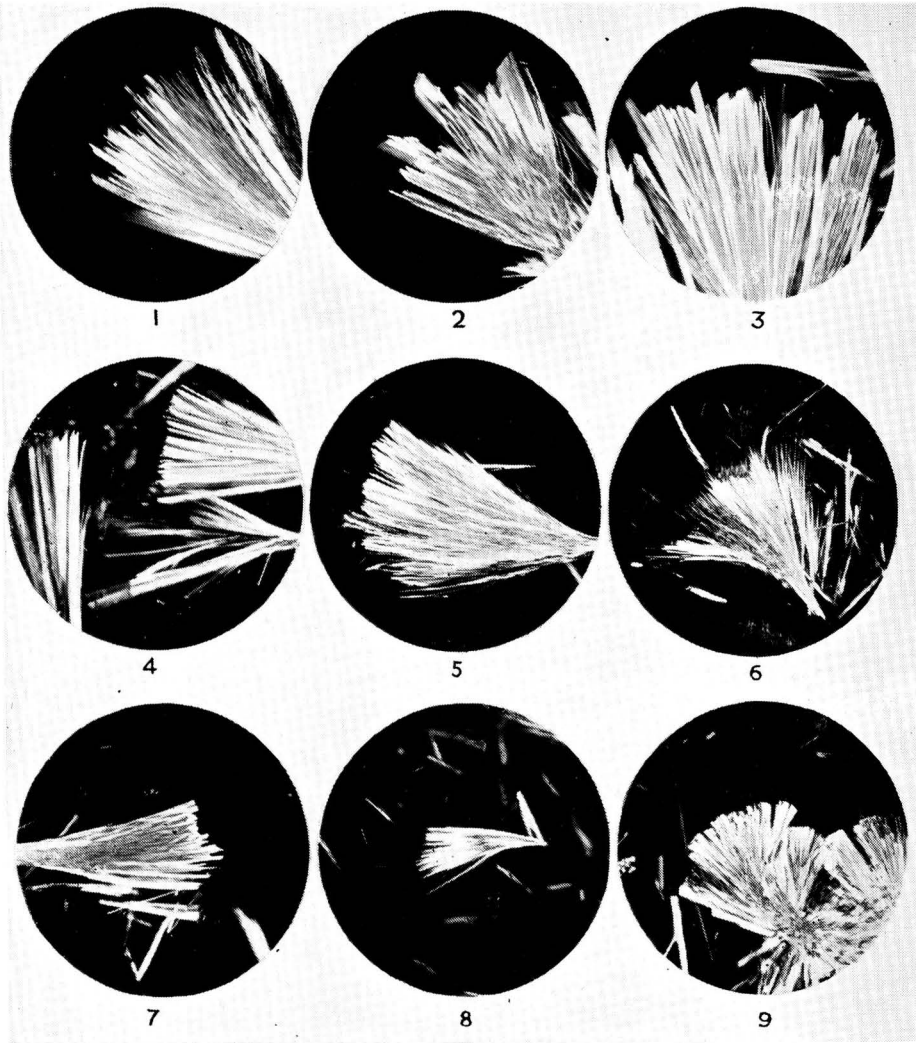
MICROSCOPIC EXAMINATION OF CRYSTALS.—A microscopical examination of the crystals that separate from ether should always be made. The method adopted by some workers of cooling in water or ice water an ethereal solution of the fat leads to poor results. Our procedure is to dissolve 40 drops of the melted fat in 10 ml. of ether in a test-tube, which is then lightly plugged with cotton wool and left for the solution to crystallise at room temperature overnight. Should there be a large deposit it is desirable to begin again with a smaller quantity of fat. Some of the deposit is withdrawn by means of a dipping tube, allowed to fall into a drop of clove oil on a microscope slide, and then covered with a cover-slip. These conditions promote the formation of large crystals, and with genuine lards there is no difficulty in obtaining large well-formed crystals with typical chisel ends.

In the crystals produced under these conditions from known mixtures of beef fat and genuine lard we find a gradual transition from the large chisel-shaped crystals to the typical beef-type crystals and no suggestion of a mixture of both forms. The proportion of beef fat, etc., required to effect the change in crystal form varies with the product used. With beef fat we found that a 40 per cent. admixture was necessary before the chisel ends disappeared completely and typical beef-type crystals were to be observed. With beef suet a 10 per cent. admixture caused some alteration from the typical lard crystals, with 20 per cent. the chisel ends were still just visible, and probably the typical beef crystals would have been obtained with about 30 per cent. admixture. With mixtures made from one sample of beef dripping 10 per cent. caused noticeable alteration from the typical lard crystal, and crystals of the beef type were obtained with a 20 per cent. admixture. These results are illustrated in Plate I.

Certain mixtures of lard with 10 or 20 per cent. of hydrogenated oils gave crystals which could not be distinguished from beef fat, others yielded crystals unlike those from either beef fat or lard. Fortunately such adulterants would usually be detected in the sterol acetate test or in the determination of iso-oleic acid.

Our examination of such mixtures suggests that the genuineness of a sample of lard is not necessarily established when it is possible to observe chisel ends to the crystals. If to find such features a careful search of the slide has to be made and, as is sometimes suggested, the ends of the crystals focussed carefully with the $\frac{1}{8}$ -inch objective, it is almost certain that the lard is adulterated. We have on occasion heard the opinion expressed that if the beef-type crystal is to be found the lard may be adulterated, but that if the crystals have chisel ends the lard is undoubtedly genuine. We incline to the view that when the crystals have chisel ends the lard may be genuine, but that when the beef-type crystal is obtained the lard is undoubtedly adulterated.

PLATE I. CRYSTALS OF LARD AND OF MIXTURES OF LARD WITH BEEF FATS



1. Lard. 2. Lard + 5 per cent. of beef fat. 3. Lard + 10 per cent. of beef fat.
4. Lard + 20 per cent. of beef fat. 5. Lard + 40 per cent. of beef fat. 6. Beef
fat. 7. Lard + 20 per cent. of beef suet. 8. Lard + 40 per cent. of beef suet.
9. Lard + 10 per cent. of beef dripping. Magnification, $\times 70$.

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DISCUSSION

The PRESIDENT observed that he had experienced great difficulty in the microscopical examination of the crystals separating from ether, because different forms could be obtained according to the way in which the slide was made and the amount of pressure applied to the cover-glass. He thought that a fair standard would be that the melting-point of the crystals should not be below 63° C.

Mr. K. A. WILLIAMS remarked that the problem before the meeting was one of the most difficult in the analysis of fats. Mr. Sutton had given an admirable account of the methods by which the authors had attacked it, but it was doubtful whether the problem was yet solved. Allegations of adulteration could not generally be refuted by strict evidence of the origin of the fat, and any form of "appeal to the pig" was out of the question. The manufacturer often bought his supplies in the form of crude fat, and whilst he attempted to control his suppliers by carefully-drawn contracts, he could not rely on a warranty, since he refined the material before selling it. For this reason he depended on analysis to check the quality of his raw material, and Mr. Williams quoted one case in which it proved necessary to sample and analyse separately every barrel received in a factory during a period of several months. This placed a heavy burden on the manufacturer, and ruled out the possibility of using any such elaborate investigation as could be made by a local authority on single samples. The manufacturer's difficulties were aggravated by a notable divergence between the views of different experts on the relative merits of various tests. At the date of the meeting it seemed quite sufficient to rely on the B.P. 1932 tests, as presumably these were good enough for the pharmaceutical product and should therefore be satisfactory for a commercial article. If circumstances rendered other testing necessary, Mr. Williams hoped that any method decided upon would be such as could reasonably be applied in factory control. At present there was a "no man's land" in which a pure lard could appear adulterated and an impure lard might be passed. The authors proposed to remove the "no man's

land" by making the standards so stringent that any material falling in it would appear adulterated. This was obviously unfair to the manufacturer, and Mr. Williams trusted that no such principle would be adopted.

He wished to emphasise the point that the manufacturers with whom he had come in contact were concerned to produce a pure product, and worked to what appeared to him to be high standards; but their position was impossible if different authorities required different standards without giving any notice of their preference to the manufacturer.

Mr. Williams showed how the standards suggested in the paper differed radically from those of well-known authorities, although the latter were based on a far wider range of samples than those now proposed. He pointed out how dependent the B.P. crystallisation test was on the exact technique employed, and showed the effect of slight departures from a given form of the test on the melting-point. The latter could be raised by nearly 2 degrees by alteration in the washing technique, and an amplified description was therefore most necessary.

Dealing with the form of the crystals deposited from ether, Mr. Williams demonstrated by lantern slides how both pure and adulterated lards often gave crystals of typically beef-like appearance; but these could usually be resolved into the well-known forms of chisel-shaped or pointed nature by gentle rubbing of the cover-glass on the microscope slide. The false appearance of a beef-like form from pure lard was due to crystallisation in rosettes in which the crystals lay on their edges, and the broad faces of the pure lard crystals could be observed only when the rosettes were broken up. Beef crystals were needle-shaped and softer than lard crystals, so that the rubbing could not make them appear like the latter.

There seemed to be insufficient evidence as yet to prove that a Bömer number of 70 necessarily indicated the presence of 5 to 15 per cent. of beef fat, especially when it was remembered that pigs received a huge variety of diet, the oily constituents of which were often later found in the lard. There were as yet no published data for the effects of many diets on the analytical figures. From the analytical point of view it was also clear that the divergence of 0.2° C. between duplicate melting-points, noted by Mr. Sutton as a reasonable experimental error, led to a possible difference of 1.0° C. in the Bömer number—itsself enough to render the method unsatisfactory.

Mr. Williams therefore preferred, until such time as general agreement could be reached, to use the B.P. standards as being more satisfactory than the authors', and as giving the purchaser all the protection that could reasonably be expected.

Mr. W. M. SEABER mentioned that with certain fats he had been unable to obtain any crystals at all.

Dr. J. H. HAMENCE said that this difficulty in obtaining crystals was a common occurrence with hardened oils. Mixtures consisting entirely of hardened fats crystallised out very slowly—indeed, some would not yield any deposit when crystallisation from ether was attempted under the conditions described for lard in the British Pharmacopoeia. Had Mr. Sutton any experience with the Bömer test when applied to lards with very high iodine values? He (the speaker) had been unable to find anything wrong with some samples which had had iodine values as high as 90, and had reported them to be genuine lards.

Mr. H. E. MONK said that he had not been able to follow how the authors would discover whether an adulterant was hydrogenated fat or beef-fat; the iodine value would not afford much help. Could Mr. Sutton indicate what were the smallest amounts of hydrogenated fat and beef fat that could be detected with reasonable certainty in lard? The photomicrographs shown by the authors were excellent, but he had been astonished to hear that the crystals had been obtained at "room temperature." The slightest variation in technique might produce remarkable differences and rigid control of the temperature and other conditions was necessary if deductions were to be drawn from the form of the crystals.

Dr. C. A. MITCHELL recalled that when Hehner and he were working on flare lard they found that if it were repeatedly recrystallised from ether, beef-like clusters of crystals were obtained, while at the same time the stearic acid content rose with each recrystallisation. He suggested that it might be possible to fix a minimum standard for the proportion of stearic acid in the crystals first obtained.

Mr. R. C. CHIRNSIDE suggested that X-ray patterns of the crystals might afford useful information.

Mr. J. R. NICHOLLS, referring to the question as to which was the best test, said that the Bömer value was essentially an arbitrary accentuation of the result given by the B.P. test and was subject to the equivalent of three experimental errors. He thought that the one value given by the B.P. test was sufficient for deductive purposes. Some years ago a considerable amount of work had been done in the Government Laboratory on the melting-points of the crystals from beef and lard and he had looked up the working sheets. The lowest melting-point for pure lard was 62.7° C. and the highest for pure beef fat was also 62.7° C. obtained after 10 crystallisations. Referring to the crystal form, Mr. Nicholls said that lard crystals should not be mistaken for beef fat crystals; it was well known that, with repeated recrystallisation, one could get beef fat crystals similar to those of lard. Crystals from lard were straight, whilst those from beef fat were usually curved.

Mr. SUTTON, replying, said that in the first place he did not wish to be quoted as suggesting that 57 was the limit for the iodine value. He merely recorded that of the 113 samples examined recently in his laboratory, the iodine value of 96 per cent. of them fell within the range of 57 to 73. Both Professor Hilditch and Mr. Williams had raised the question why the factor Mg + 2d

should be chosen. He had not seen any reason for this stated in the literature, but might suggest two points for consideration. First, lard and beef fat differed in both the glyceride melting-point and the "difference" figure, and it therefore seemed proper to take both into consideration in formulating any factor as a criterion of genuineness. Secondly, the figures for d were relatively more divergent, so that it seemed reasonable to use as high a multiple of d as possible. On the other hand, the greater the multiple of d incorporated in the factor the greater was the possible experimental error. $Mg + 2d$ was presumably a compromise giving a factor which indicated most quickly the presence of beef products and at the same time could be duplicated with reasonable agreement.

He could not agree with Mr. Williams about the beef-type crystals which presented a different appearance if rolled over by moving the cover slip. It was not reasonable to assume that all the lard crystals on a slide would be appropriately placed so that only the edges of the crystals would be viewed through the microscope. No manipulation of the cover-slip was necessary or desirable. If the slide were prepared as suggested, one always found the chisel end with genuine lard—in fact, there was no difficulty in recognising a genuine lard crystal. If one did exert pressure on the cover-slip one stood a chance of flattening the crystals. In reply to Dr. Mitchell's statement, he had no experience of the effect of several crystallisations from ether, and his remarks on crystal form referred solely to the standard procedure given in the paper.

He had had several samples with high iodine values and some of these gave insufficient glycerides for the Bömer test. If no glycerides at all were obtained from acetone and adulteration was suspected because of a high iodine value, it could be very certain that beef fat was not present, and he would at once have recourse to the iso-oleic acid test. In deciding on the nature of the adulterant one had to consider all the results. As to the proportion of beef fat that could be detected, he believed that as little as 5 per cent. would usually lower the Bömer figure below the value suggested for genuine lard.

Professor T. P. HILDITCH sent the following written communication:

The Bömer values for differences between melting-points of the glycerides separated from acetone and of their mixed fatty acids depend upon a number of factors which make the interpretation complex and render necessary very close control in the technique employed in carrying out a determination. In the first place, each of the individual mixed triglycerides separated by crystallisation can exist in four polymorphic forms (one vitreous and three— α , β' , β —crystalline), so that it is of the greatest importance to use conditions of crystallisation that will lead to the production of the most stable (β), or at all events the β' , form. Complete melting-point data for simple saturated triglycerides, and for symmetrical and unsymmetrical mixed triglycerides containing two saturated fatty acids, have been given by Malkin with Clarkson, Meara and Carter (*J. Chem. Soc.*, 1934, 666; 1939, 103, 577, 1141, 1512), and these authors also define the necessary crystallisation conditions for production of the stable forms.

Secondly, the presence of minor component acids in the crystallised aggregates from acetone (*e.g.* myristic, oleic, isooleic acids) is probably the determining factor in the glyceride and fatty acid melting-points obtained from different fats. This is appreciated by the authors, who have illustrated the point by details from the only study of an animal (ox) depôt fat* which has yet been published (similar data for two pig depôt fats have now been published; *Biochem. J.*, 1940, 34, 971; *ANALYST*, 1940, 569). In a genuine pig fat there is only about 1 per cent. of combined myristic acid and, owing to the relatively high proportion of oleic acid in the fat, less oleo-disaturated glycerides are present to separate in the fraction least soluble in acetone than in ox depôt and many hydrogenated vegetable fats. Hence the separated glycerides consist, to a greater extent than in other cases, of palmitostearins; the twofold result is a high melting-point for these glycerides and a melting-point for their mixed fatty acids controlled by the proportions of palmitic and stearic (oleic, myristic) acids present. Beef fats contain about 4 per cent. of combined myristic acid and much higher proportions of oleo-disaturated glycerides than pig fat; the result is a lower melting-point of the glyceride conglomerate separated from acetone, although the melting-point of the mixed fatty acids therefrom is much the same as, or may be even slightly higher than, in the corresponding material of a pig fat. This is because the presence of more oleic and myristic acid is compensated for by the higher proportions of stearic to palmitic acid in these beef mixed acid fractions, as compared with those from a pig fat.

Similar complications underly the data for the various hydrogenated fats shown in Table V. In all these, according to the degree of hydrogenation in any one fat, a certain amount of sparingly soluble mono-*iso*-oleodisaturated glycerides may be expected to be separated in the "Bömer fraction," the melting-point of which it will tend to lower. Groundnut oil, which contains saturated C_{20} , C_{22} and C_{24} acids, and sesame (also, *e.g.* linseed or soya-bean) oil, all of which contain very little palmitic acid, produce fractions least soluble in acetone which will include a high proportion of tristearin (m.p. $71^\circ C.$), formed by hydrogenation of triunsaturated C_{18} glycerides. On the other hand, palm and cottonseed oils, rich in palmitic acid, contain large proportions of palmito-glycerides, and whale oil contains complex mixed glycerides of acids with 16, 18, 20 and 22 carbon

* Even in this detailed study, the small amount of myristic acid has to be included with palmitic acid in considering the component glycerides.

atoms; in these cases, therefore, the melting-point of the least soluble glyceride fraction of the hydrogenated fats is lower, owing to the substantial absence of tristearin and the presence of a greater number of mixed saturated glycerides.

The significance of the expression " $Mg + 2d$ " for the Bömer value appears uncertain to the writer. Clearly it is an attempt to combine in one fraction both the high melting-point of the pig fat glyceride fraction and the difference between the melting-points of the glycerides and the fatty acids. But why $2d$? Why not simply d or, if a multiple be desired, why is double the difference chosen? Since the combination of both the glyceride melting-point and the difference between the melting-points is needed to define the genuineness of a pig fat, would not the end be more clearly attained by recording each separately and assessing the fat by consideration of each value? (For instance, in Table VI, the values in columns 2 and 4 illustrate the effect of the addition of beef fat to lard, in the writer's opinion, much more effectively and rationally than the " $Mg + 2d$ " in column 5.)

Quinaldinic Acid as a Reagent for the Separation of Copper and Cadmium

BY A. J. LINDSEY, PH.D., M.Sc., F.I.C., AND R. J. SHENNAN, M.Sc., A.I.C.

RÂY and Bose¹ introduced quinaldinic acid as a reagent for the separation of copper from cadmium, but under the conditions specified we failed to obtain satisfactory results,² and we could not effect a separation by more rigorous pH control with sodium acetate as our buffering agent.³ Recently, Majumdar⁴ has criticised our work on the ground that our procedure differed from that of Rây and Bose, whose results he claims to have substantiated when precipitating from sulphuric acid solutions. Majumdar also states that "the separation of copper from cadmium by free quinaldinic acid in presence of acetic acid has always been found to give high results, owing to co-precipitation of cadmium." This was precisely our contention,³ and is in direct contradiction to Rây and Bose, who claim that "the separation may also be carried out in presence of 2 to 3 ml. of glacial acetic acid."

We have shown that, over the pH range 2.5 to 6.86, copper can be completely precipitated from acetic acid—sodium acetate solutions, and have also observed that the pH range can be extended to pH 1.5 if acetic acid is used alone. We have already stated that the separation of copper from cadmium is impossible in acetic acid solution; Majumdar agrees with this, yet considers it curious that we did not attempt such a separation.⁴

In view of Majumdar's insistence that a successful separation can be made in sulphuric acid solution, we have done further work to explain the divergence between our results and those of Rây and his co-workers.

The determinations of copper in pure copper sulphate solutions were made under the conditions set out by Rây and Bose. A 1 per cent. solution of sodium quinaldinate was used as precipitant, and in every instance the solution was made up to a total volume of 180 ml. A typical selection of the results is given in Table I.

TABLE I

Copper taken mg.	2N H ₂ SO ₄ added ml.	Reagent solution ml.	Wt. of ppt. mg.	Copper found mg.	Error mg.
26.0	2	20	171.2	25.62	-0.38
26.0	5	20	164.9	24.67	-1.33
26.0	10	20	133.0	19.90	-6.1
26.0	2	30	174.1	26.02	+0.02
26.0	5	30	174.2	26.04	+0.04
26.0	5	30	174.1	26.02	+0.02
26.0	10	30	159.6	23.89	-2.11
26.0	10	40	174.1	26.02	+0.02
26.0	10	40	173.9	26.01	+0.01
26.0	10	50	174.2	26.04	+0.04

These results indicate that for complete precipitation of copper from solutions containing up to 5 ml. of 2 *N* sulphuric acid a twofold excess of reagent is necessary, while for solutions containing 10 ml. of 2 *N* sulphuric acid a threefold excess is essential. Rây and Bose state that a "slight excess" of reagent is necessary, but give no data, while Majumdar, who does not stress the point, used quantities of reagent conforming approximately with the requirements that we find necessary.

Having established precisely the conditions for complete precipitation of copper, we attempted to separate copper from cadmium on the lines suggested by Rây and Bose. The cadmium used was a solution of the pure sulphate, of which the cadmium content had been determined by electrolysis and by precipitation as quinaldinate. The same reagent was used as in the experiments above. In certain instances the cadmium in the filtrate was determined as quinaldinate. Results are given in Table II.

TABLE II

Metals taken mg.	2N H ₂ SO ₄ added ml.	Reagent solution ml.	Wt. of ppt. mg.	Metals found mg.	Error mg.
Cu. 26·00 } Cd. 31·22 }	2	30	{ 178·9 —	26·77 —	+0·77 —
Cu. 26·00 } Cd. 31·22 }	5	30	{ 178·7 —	26·74 —	+0·74 —
Cu. 26·00 } Cd. 62·44 }	10	40	{ 176·7 —	26·43 —	+0·43 —
Cu. 26·00 } Cd. 31·22 }	2	30	{ 179·1 121·9	26·80 30·01	+0·80 -1·21
Cu. 26·00 } Cd. 93·66 }	10	40	{ 176·9 376·8	26·46 93·05	+0·46 -0·55
Cu. 26·00 } Cd. 31·22 }	5	30	{ 176·4 124·0	26·39 30·54	+0·39 -0·68
Cu. 26·00 } Cd. 93·66 }	2	30	{ 176·8 377·4	26·45 92·87	+0·45 -0·79
Cu. 26·00 } Cd. 93·66 }	10	40	{ 176·5 377·2	26·40 92·87	+0·40 -0·79
Cu. 26·00 } Cd. 62·44 }	5	30	{ 176·5 250·1	26·40 61·59	+0·40 -0·85
Cu. 26·00 } Cd. 62·44 }	10	40	{ 177·6 249·7	26·58 61·55	+0·58 -0·89
Cu. 52·00 } Cd. 31·22 }	2	60	{ 352·2 —	53·34 —	+1·34 —
Cu. 26·00 } Cd. 93·66 }	10	40	{ 176·8 —	26·45 —	+0·45 —

In every instance the result for copper was high and that for cadmium correspondingly low. Care was taken in the washing of the copper precipitate to avoid retention of soluble cadmium salts. Tests on the copper quinaldinate precipitates, with "Cadion" as the reagent, showed traces of cadmium. These results completely endorse our previous statement and do not agree with the findings either of Rây and Bose or of Majumdar.

Majumdar (*loc. cit.*) states that copper quinaldinate is appreciably soluble in acetic acid and that copper is completely precipitated from sulphuric acid at *pH* 1·22; from this he concludes that the solubility of copper quinaldinate in acetic acid is a specific effect and has nothing to do with *pH*. This conclusion is erroneous. Table I shows that copper quinaldinate is somewhat soluble in sulphuric acid of *pH* 1·22 (10 ml. of 2 *N* sulphuric acid in 180 ml. solution), but evidently solution can be prevented by employing excess of reagent. In this connection we made the following experiment: An excess of copper quinaldinate

was shaken with sulphuric and acetic acids of various concentrations for a period of 24 hours. The undissolved salt was filtered off, and the copper in solution was determined either electrolytically or colorimetrically.

TABLE III

2N H ₂ SO ₄ in 180 ml. of solution ml.	pH	Copper found in solution mg.	Copper quinaldinate dissolved mg.
180	0 (approx.)	101.9	680.8
10	1.22	3.6	24.1
0.9	2.05	0.2	1.3
Glacial acetic acid in 180 ml. of soln. ml.			
45	2.05	2.0	13.4
35	2.2	0.4	2.7
21	2.5	0.3	2.0

The results given in Table III show that copper quinaldinate is soluble in sulphuric acid solutions and that the solubility is related to the pH.

From Shennan's paper it will be noted that whereas copper is completely precipitated at pH 2.2 when no sodium acetate is present, it is only about half precipitated when this salt is used. It is obvious therefore that Majumdar has misunderstood the reference to the effect of sodium acetate upon the solubility of the precipitate.

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The Micro-Titrimetric Determination of Carbon Dioxide in Carbonates

BY A. F. COLSON, B.Sc., F.I.C.

THE titrimetric method of determining carbon dioxide in carbonates, known as the "shaker" method, has been found to give satisfactory results on the macro-scale, and it has now been adapted to the determination of micro-quantities of carbon dioxide when only a few mg. of material are available.

MACRO METHOD.—The sample is put into a Kjeldahl flask fitted with a rubber stopper carrying (1) a tap funnel, and (2) a glass tube bent at right-angles and connected with a narrow cylindrical vessel containing a mixture of sodium hydroxide and barium chloride solutions. This absorption vessel is provided with a glass tap by means of which the assembled apparatus may be joined to a water pump and evacuated. When this has been done an excess of dilute hydrochloric acid is admitted to the sample, and the reaction vessel is carefully heated until the absorption vessel becomes distinctly hot. During this period the whole apparatus is shaken by hand to facilitate absorption of the liberated carbon dioxide. The absorption vessel is then detached, and the residual alkali is neutralised with

hydrochloric acid after addition of phenolphthalein indicator. Finally, the precipitate of barium carbonate is dissolved in a measured excess of standard hydrochloric acid, and the excess is titrated with standard sodium hydroxide solution, methyl orange being used as indicator.

MICRO METHOD.—Preliminary experiments indicated that the successful adaptation of this method to the requirements of micro-analysis must depend primarily upon the fulfilment of the following conditions:—(1) In contrast to the macro procedure, the contents of the absorption vessel must be protected from atmospheric carbon dioxide until most of the residual alkali has been neutralised. (2) The amount of "dead" space in the apparatus must be reduced to a minimum. (3) Suitable precautions must be taken to ensure that "blank" values given by the reagents, etc., are small in comparison with the amounts of carbon dioxide to be determined.

Of the many types of apparatus constructed the compact form shown in Fig. I most nearly satisfies the specified conditions. The apparatus is made of Pyrex glass and consists essentially of a reaction vessel, A, and an absorption vessel, B, each provided with a suitable tap funnel for the introduction of reagents. It is supported at a convenient height by the three glass rods R_1 , R_2 , R_3 , sealed to the base of the flask B.

Reagents.—The following solutions are required:— N and $N/100$ sodium hydroxide solutions; 10 per cent. barium chloride solution; N , $N/15$ and $N/50$ solutions of hydrochloric acid; methyl red indicator (0.05 per cent. aqueous solution); phenolphthalein indicator (0.05 per cent. solution in 50 per cent. alcohol).

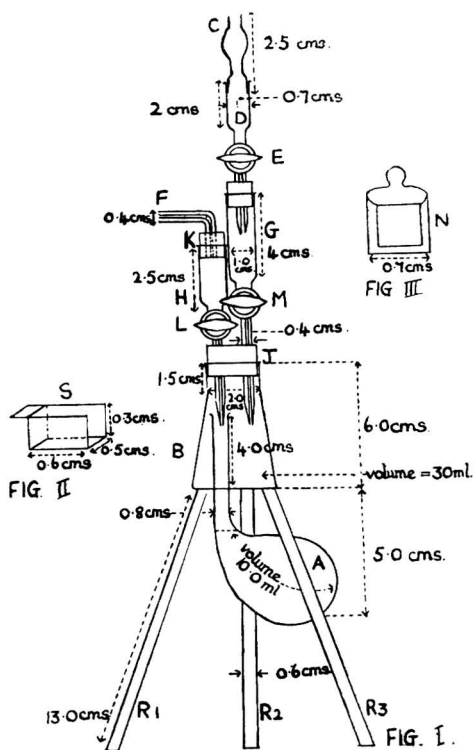
To reduce the "blank" value of the reagents to a minimum the following simple precautions must be taken:—(1) The distilled water used in the preparation of the solutions, and for all other relevant operations, must be boiled in a stream of air free from carbon dioxide and allowed to cool without interruption of the air flow. (2) The N sodium hydroxide solution must be treated with about 0.5 per cent. of barium chloride crystals and allowed to stand for about one day before use.

The strength of the $N/100$ sodium hydroxide solution is best expressed in terms of mg. of the N hydrochloric acid equivalent to 1.0 ml. of the alkali. The N hydrochloric acid solution is standardised against pure sodium carbonate solution and its strength expressed in terms of mg. of carbon dioxide equivalent to 1.0 mg. of the acid.

Procedure.—In making a determination of carbon dioxide the following operations must be carried out in the order given:

(1) The small guard tube C, containing "Carbosorb," is removed from the funnel D, and a little glass wool is introduced into the funnel and pressed down firmly against the tap E. (2) The rubber stopper, which carries the two small funnels G and H, is detached from the neck of the flask B, and the weighed sample, together with its platinum container S (Fig. II) is carefully introduced into the reaction vessel A, and followed by 1.0 ml. of distilled water. (3) The stopper is replaced in such a manner that the tip of the funnel H lies just above the mouth of the reaction vessel. The apparatus must then be rendered air-tight by running a little melted paraffin wax round the rim of the flask B where it meets the rubber stopper. The junctions between the funnel stems and the stopper are similarly sealed. (4) The tube F is now joined to a glass tap through which connection may be made to a water suction-pump, or to a source of air free from carbon dioxide, as required. (5) With the tap E closed, the apparatus is evacuated, filled with air free from carbon dioxide, and again evacuated. (6) 0.25 ml. of 10 per cent. barium chloride solution is introduced into the funnel D and drawn through the glass wool filter pad into the funnel G. It is followed by 0.25 ml. of N sodium hydroxide solution and about four drops of phenolphthalein indicator solution. The perfectly clear solution is then allowed to enter the flask B, and the funnels D and G are rinsed through with about 0.5 ml. of water, applied in several small

portions. The funnel G is finally rinsed out with *N* hydrochloric acid and water, the rinsings being rejected. (7) 0.3 ml. of *N* hydrochloric acid and one or two drops of methyl red indicator solution are admitted from the funnel H to the sample in the reaction vessel A. This volume of acid is sufficient for any 5 mg. sample.



(8) The reaction vessel, A, is now carefully heated over the flame of a micro burner until the solution boils, and heating is continued until the temperature of the absorption vessel, B, has risen to about 70° C. The apparatus is then allowed to cool for a few seconds, and alternate heating and cooling are continued until only one or two drops of liquid remain in the reaction vessel. During this process the contents of the vessels A and B must be almost continuously agitated to facilitate absorption of the liberated carbon dioxide and to prevent loss of solution from the reaction vessel by sparging. Agitation of the solutions is conveniently effected by holding the supports R_1 , R_2 , R_3 in the right hand, and imparting a gentle circular movement to the apparatus, while pressing the three supports firmly against a smooth bench surface.

(9) The reaction vessel is next filled with distilled water from the funnel H, to a point about two-thirds up the narrow neck. (10) A stream of air free from carbon dioxide is passed through the tube F attached to the rubber stopper K, and, without interruption of the air flow, the stopper K is inserted into the funnel H, which has previously been completely filled with water. By opening the tap L the apparatus is filled with the purified air. The tap L is then closed and the apparatus is shaken gently for two or three minutes. (11) The apparatus is next partially evacuated, and the residual alkali in the absorption vessel is nearly neutralised by dropwise addition of *N*/15 hydrochloric acid, from the funnel G.

(12) The tap M is closed, and the apparatus is filled with air free from carbon dioxide by opening the tap L after connecting the tube F with the supply of air. (13) The rubber stopper is then removed, and the wall of the absorption vessel is rinsed down three or four times with small quantities of water from a small wash-bottle with a fine jet. The solution is then exactly neutralised with $N/50$ hydrochloric acid. (14) A weighed excess of N hydrochloric acid (about 160 mg.) contained in the small stoppered glass vessel N (Fig. III) is introduced into the vessel B, and followed by two drops of methyl red indicator solution, and the solution is boiled gently for five minutes over the flame of a micro burner. (15) Finally, the flask is closed with a rubber stopper and cooled by standing the apparatus in a large beaker of cold water, and the excess of hydrochloric acid is titrated with $N/100$ sodium hydroxide solution. From the volume of alkali required, the weight of N acid used to react with the barium carbonate is obtained, and the carbon dioxide content of the sample is calculated. The time taken to carry out a complete determination is about one hour. Before the actual determinations a "blank" test must be made under exactly the same conditions. It is convenient for the calculation to express the value of the "blank" in terms of mg. of N hydrochloric acid.

Results.—Selections from a large number of results obtained in the determination of approximately 1.0, 0.5 and 0.25 mg. of carbon dioxide in specially prepared pure barium carbonate are given in Table I. By the macro "shaker" method this pure barium carbonate was found to contain 99.72 per cent. of carbon dioxide, and this value was used throughout in calculating the weights of carbon dioxide recorded in the third column of the table.

TABLE I

DETERMINATION OF CARBON DIOXIDE IN BARIUM CARBONATE

Expt. No.	BaCO ₃ taken mg.	CO ₂ present mg.	CO ₂ found mg.	Error mg. CO ₂	Error Per Cent.
A1	4.814	1.070	1.057	-0.013	-1.20
A2	5.058	1.124	1.136	+0.012	+1.10
A3	4.825	1.073	1.045	-0.028	-2.60
A4	4.782	1.063	1.107	+0.044	+4.10
A5	5.063	1.126	1.124	-0.002	-0.20
B1	2.607	0.580	0.575	-0.005	-0.90
B2	2.470	0.549	0.553	+0.004	+0.70
B3	2.588	0.575	0.554	-0.021	-3.70
B4	2.444	0.543	0.520	-0.023	-4.20
C1	1.250	0.278	0.266	-0.012	-4.30
C2	1.293	0.288	0.278	-0.010	-3.50
C3	1.190	0.265	0.250	-0.015	-5.70
C4	1.220	0.271	0.257	-0.014	-5.20

DISCUSSION OF SOURCES OF ERROR.—Some of the operations described above were designed to eliminate or reduce the errors derived from various sources. The filtration of the N sodium hydroxide solution through the glass wool pad contained in the funnel D, serves to remove any suspended barium carbonate that may be present in the reagent. Any traces of barium carbonate deposited in the funnel G would subsequently be dissolved by the $N/15$ hydrochloric acid used for the neutralisation of the residual alkali in the absorption vessel, and in consequence this acid would contain a little dissolved carbon dioxide, which would give rise to a positive error if it were allowed to enter the alkaline solution in the absorption vessel. The funnel G must, therefore, be rinsed out with dilute acid and water before the introduction of the $N/15$ hydrochloric acid.

The importance of excluding air from the interior of the apparatus until nearly

the whole of the residual alkali has been neutralised is clearly illustrated by the following "blank" determinations. In Expt. No. 1 the neutralisation of the alkali was carried out after the absorption flask B had been opened by removal of the stopper. The two values obtained were high and in poor agreement. In Expt. No. 2 the neutralisation was effected without opening the absorption vessel. The values thus obtained were lower and much more concordant. In Expt. No. 3 the apparatus was kept closed and also sealed with paraffin wax as described above. The two "blank" values obtained were lower than those in Expts. Nos. 1 and 2, and were in close agreement. (See Table II.)

TABLE II

Expt. No.	"Blank" value	
	N HCl mg.	CO ₂ mg.
1	11.90	0.262
	14.80	0.326
2	3.48	0.077
	3.36	0.074
3	1.60	0.035
	1.40	0.031

For several reasons it would be preferable to use a ground-glass joint in place of the rubber stopper J, but in practice it was found that such joints possess the serious disadvantage of frequently "seizing up." Diffusion of carbon dioxide into the rubber stopper J is a possible source of error, to prevent which the rubber stopper is impregnated with a mixture of paraffin wax and vaseline in the following manner: The stopper is immersed in a melted mixture of paraffin wax and vaseline, contained in a thick-walled round-bottomed flask attached to a good water pump. The flask is evacuated and then air is admitted. Alternate evacuation and admission of air are repeated until air bubbles no longer rise from the surface of the rubber. Finally, the stopper is removed from the flask and the excess of wax is carefully wiped off (*cf.* Pregl, "*Quantitative Organic Microanalysis*," 3rd English Ed., p. 26). In the course of a determination this stopper is subject to the action of the steam which enters the absorption flask B, and a little paraffin wax is carried down into the sodium hydroxide solution. Experiments to determine the effect of this impurity indicate that it does not increase the "blank" values by more than 0.01 mg. of carbon dioxide. The solubility of carbon dioxide in the acid solution contained in the reaction vessel A, may give rise to a small negative error, and that solution is therefore distilled almost to dryness to expel as much as possible of the dissolved gas.

It is desirable to reduce the amount of "dead" space in the apparatus as much as possible. For this reason the reaction vessel is partly filled with water at a suitable stage in the determination. After the residual alkali in the absorption flask has been almost completely neutralised by *N*/15 hydrochloric acid, the flask is opened and rinsed down with water. Any hydrochloric acid adhering to the sides of the flask is therefore carried down into the slightly alkaline solution. Experiments indicated that the amount of hydrochloric acid thus introduced is not likely to exceed 0.1 ml. of *N*/100 hydrochloric acid. Since the neutralisation of the residual alkali is always interrupted when at least 0.05 ml. of *N*/15 hydrochloric acid is still required, this additional 0.1 ml. of *N*/100 acid can have no harmful effect. The shape of the vessel employed to contain the weighed sample appears to be of some importance. A small circular glass dish, 4 mm. deep and 4 mm. in internal diameter, was formerly used, but more accurate results are obtained with the platinum scoop S, illustrated in Fig. II. Apparently, when the small glass dish is employed, the whole of the sample does not always come into contact with the acid in the reaction vessel.

The concentrations of the acid and alkali solutions used in this method were chosen with the object of reducing the volume of the apparatus without undue sacrifice of accuracy. For this reason, also, weighed amounts of *N* hydrochloric acid solution are used in preference to larger measured volumes of more dilute acid. The error involved in weighing out the required amount of standard acid does not exceed 0.1 mg., which corresponds to approximately 0.002 mg. of carbon dioxide. The exact neutralisation of the residual alkali in the absorption vessel is facilitated by the following simple device employed by A. Benedetti-Pichler (*Z. anal. Chem.*, 1928, p. 200) and others. By applying a thin film of paraffin wax to the tip of the micro-burette, very small drops of solution can be collected at the orifice, and transferred to the titration vessel by allowing them to touch the wall of that vessel. Additions of 0.01 ml. to 0.03 ml. of 0.02 *N* hydrochloric acid can thus be made. The same technique is employed in the final titration with *N*/100 sodium hydroxide solution. In this titration it is desirable to work with the same portion of the burette for the actual determination of carbon dioxide and for the "blank." It is also advisable to allow a constant drainage time of about two minutes before the final burette readings are taken.

SUMMARY.—A method is described for the micro-titrimetric determination of carbon dioxide in carbonates. Various sources of error are discussed in detail, and a number of results are given which indicate that quantities of 1.0, 0.5 and 0.25 mg. of carbon dioxide may be determined with a deviation of not more than about 1.5, 2 and 5 per cent. respectively from the correct values.

I wish to express my thanks to the Directors of Imperial Chemical Industries, Ltd., for permission to publish this work, which was carried out in the Research Laboratory of their subsidiary Company, Imperial Chemical Industries (Alkali), Ltd., Northwich.

RESEARCH DEPARTMENT
I.C.I., WINNINGTON
NORTHWICH, CHESHIRE

December, 1939

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.

THE CYANOMETRIC DETERMINATION OF COBALT AND NICKEL*

A CYANOMETRIC process for determining cobalt and nickel in presence of each other has been published by B. S. Evans (*ANALYST*, 1937, **62**, 363). This method, although excellent, has one or two disadvantages: first, ammonia cannot be used in the initial stage of the titration; secondly, there is a fairly large interval between the first and second stages, which necessitates the burettes being left during that time.

It has been found that when the cyanide is added to the cobalt solution, previously acidified with dilute nitric acid, and the liquid is allowed to stand for at least four minutes with occasional shaking, the cobalt proceeds to the Co:5KCN stage, whereas nickel remains at the Ni:4KCN stage. The following conditions have been worked out for determining cobalt, or cobalt and nickel together: The test sample should contain not more than 0.04 g. of cobalt and nickel, and the volume of liquid should be 150 ml. and approximately neutral. The solution is treated with 10 ml. of nitric acid (sp.gr. 1.2), and with potassium cyanide in excess and allowed to stand for four minutes with occasional shaking. Twenty ml. of dilute ammonia (1/1) (sufficient to neutralise the nitric acid and to leave an excess of about 10 ml.) and 10 ml. of potassium iodide solution (4 per cent.) are added, and the titration is carried out in the ordinary way.

The following results were obtained for cobalt alone:

Cobalt taken g.	Titration ml.	Cobalt found g.
0.0200	35.00 — 9.90 = 25.10	0.0202
0.0200	34.80 — 9.60 = 25.20	0.0202
0.0200	34.30 — 9.10 = 25.20	0.0202

* Communication from the Research Department, Royal Arsenal, Woolwich.

The following results were obtained for cobalt and nickel together:

Cobalt taken g.	Nickel taken g.	Total titration ml.	Titration for nickel ml.	Titre for cobalt ml.	Nickel found g.	Cobalt found g.
0.0100	0.0100	34.90—12.10=22.80	15.80—5.70=10.10	22.80—10.10	0.0101	0.0102
0.0100	0.0100	35.00—12.50=22.50	16.30—6.20=10.10	22.50—10.10	0.0101	0.0100

In these tests the total cyanide used was measured, and then to an exactly similar quantity of cobalt and nickel, acidified with nitric acid, an excess of cyanide was added to bring cobalt to the Co : 5KCN stage. Excess of cyanide was then destroyed by boiling with 15 ml. of ammonia (1/1) and 25 ml. of hydrogen peroxide (20 vols.) for about 20 minutes. Under these conditions the cobalt proceeds to the Co : 6KCN stage, forming a stable compound. Nickel is then precipitated, free from cobalt, with solid dimethyl-glyoxime, and determined as usual. In all the determinations the factors were 0.001 for nickel, and 0.000803 for cobalt.

The concentration of the nitric acid in the initial stage of transforming cobalt to the Co : 5KCN condition has some importance. For example, with volume 250 ml. instead of 150 ml. the following results were obtained:

Cobalt taken g.	Cobalt found g.
0.0200	0.0197
0.0200	0.0196
0.0200	0.0198

The inference is that the concentration of the nitric acid was too low for transformation of the whole of the cobalt to the Co : 5KCN stage in the time stated.

W. J. AGNEW
October, 1940

THE USE OF ANHYDRITE AS A DESICCATING AGENT

Two papers by Hammond and Withrow (*Ind. Eng. Chem.*, 1930, 653 and 1112) have not received, in England, the attention that they deserve; they were not abstracted in THE ANALYST. These authors claim that anhydrite is the cheapest and most satisfactory chemical for laboratory use as a desiccating agent. It has been in regular use in this laboratory for over a year, and these claims have been found to be fully warranted.

To prepare anhydrite, commercial gypsum—selected white lumps—is broken up and sieved. The portion that passes an I.M.M. size-5 sieve and is retained by size-10 is used. This is spread out to a depth of about 1 inch in shallow metal trays, which are heated to 230–250° C. (Hammond and Withrow say that 250° C. must not be exceeded) in an electric oven with automatic temperature control. The heating is continued for one hour after a test of the oven vent with a cold watchglass shows that no more moisture is being evolved, and the product is then transferred while hot to tins with well-fitting lids and subsequently bottled. Fresh gypsum requires about 4 hours' heating; it does not undergo any visible change in the process. The anhydrite will absorb about 6 per cent. of its weight of water, and when exhausted can be reactivated by again heating to 230–250° C.

By weighing at intervals a small quantity exposed to the air in a flat dish, it has been found that the absorption of moisture is rapid and even; there is no tailing off in the rate of absorption until over 5 per cent. of the weight of the anhydrite has been absorbed. Anhydrite is a more powerful dehydrating agent than fused calcium chloride. Ten g. of calcium chloride were exposed to the air until it had taken up water equal to about 5 per cent. of its weight. It was then put in a vacuum desiccator over anhydrite for 24 hours; more than half the absorbed moisture was removed. Hammond and Withrow claim that anhydrite will also remove water from sulphuric acid, but this could not be confirmed. Possibly, the vapour pressure of anhydrite is higher than that of moist sulphuric acid at tropical temperatures but lower in more temperate climates.

Anhydrite is very clean in use. Desiccators and gas drying tubes can be emptied and refilled in a moment without the necessity for washing out and drying that the use of calcium chloride or sulphuric acid involves. As with sulphuric acid, it is not possible to tell by appearance when the absorbing power is falling off, but the preparation of a new supply or the reactivation of the old is so cheap and simple that there is little temptation to keep it in use when it may be approaching exhaustion. If necessary, the activity is easily tested by placing a little in a test-tube surrounding a thermometer and adding a few drops of water. If the material is still active, there is an immediate rise in temperature.

Anhydrite is an ideal agent for removing the last trace of water from organic liquids and can be used for drying alcohol. Three hundred ml. of commercial absolute alcohol (about 98 per cent.) were added to 300 g. of anhydrite. There was an immediate rise in temperature. The alcohol was distilled off, and the distillate was found to contain 99.8 per cent. of alcohol.

The authors state that anhydrite owes its rapid action to the fact that it is denser than gypsum. There is no shrinkage during the change to anhydrite, and accordingly the grains are porous.

GOVERNMENT ANALYST'S LABORATORY
MADRAS

HERBERT HAWLEY

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ALUMINA FOR THE ESTIMATION OF CAROTENE

In my paper on Carotene and Allied Substances, published in THE ANALYST (1940, 65, 266), when describing my "3 per cent. acetone" chromatographic process, I mentioned that I had had difficulty in obtaining an alumina that would give results similar to those obtained with Merck's alumina.

Since then I have tried one recently put on the market by Messrs. Savory & Moore. This appears to act very similarly to Merck's. The following are typical comparative results:

	<i>Dried Grasses</i>		Carotene (Savory & Moore's alumina)
	"Crude carotene" parts per million	Carotene (Merck's alumina)	
(1)	250	155	165
(2)	230	190	185
(3)	410	360	360
		<i>Dried Tomatoes</i>	
	250	40	40
	(most of this is lycopene)		
		<i>Dried spent Tea Leaves</i>	
	105	62	66

It should be pointed out that the alumina readily takes up moisture, and it is desirable to heat it over a very low flame shortly before the tests are made.

W. M. SEABER

ANALYTICAL LABORATORY
OAK AVENUE

HAMPTON, MIDDLESEX

October 28th, 1940

Erratum. November issue, p. 596 (Detection of Beef Fat in Lard), last line but two of the Note. For " $\pm 0^{\circ}\text{C.}$ " read " $\pm 0.1^{\circ}\text{C.}$ "

Ministry of Food

Emergency Powers (Defence)

STATUTORY RULES AND ORDERS. 1940. No. 1748

Food (Compound and Mixed Feeding Stuffs)

Order, dated September 27, 1940, amending the Compound and Mixed Feeding Stuffs (Control) (No. 2) Order, 1940*

THIS Order (which came into force on October 7th) amends "the Principal Order"^a as follows:

- (a) by inserting in Article 1 thereof immediately after the definition of "Licensed concentrate" the following definition:

"Licensed molassed feeding stuff" means a molassed feeding stuff which a person is authorised to manufacture for sale or prepare for sale in accordance with the terms of a licence granted under this Order."

- (b) by inserting in such Article immediately after the definition of "Low protein oilseed cake or meal" the following definition:

"Molassed feeding stuff" means a feeding stuff (other than a compound or concentrate as they are defined in the Feeding Stuffs (Maximum Prices) Order, 1940,^b or a concentrate, as therein defined) which contains not less than 20 per cent. of molasses."

* H.M. Stationery Office, York House, Kingsway, London, W.C.2. Price 4d. net.

^a S.R. & O., 1940, No. 1119 (*cf.* ANALYST, 1940, 505). ^b S.R. & O., 1940, No. 11.

- (c) by substituting for paragraph (c) of Article 2 of that Order the following paragraph:
 "(c) Use in the manufacture or preparation of any compound or mixed feeding stuff described in the first column of Part A of the First Schedule hereto any ingredient other than an ingredient specified in Part A of the Second Schedule hereto or use as aforesaid any ingredient mentioned in the first column of Part B of the said Second Schedule except as specified opposite to such ingredient in the second column thereof."
- (d) by substituting for the First and Second Schedules to that Order the First and Second Schedules to this Order.

THE FIRST SCHEDULE

PART A.—COMPOUND CAKES AND MEALS

The Compound or Mixed Feeding Stuff must comply with the following conditions as to percentage contents (Third Column of Table).

CATTLE AND SHEEP FOODS.

National Cattle Food, No. 1 (Dairy ration or cattle or sheep fattening ration).—Oil, min. 4, max. 6; albuminoid, min. 19·5, max. 21; fibre, max. 9·5.

National Cattle Food, No. 2 (Ration for grass feeding or fattening).—Oil, min. 4, max. 6; albuminoid, min. 14, max. 17; fibre, max. 12.

National Cattle Food, No. 3 (Rearing) (Ration for rearing young stock).—Oil, min. 4·5, max. 6; albuminoid, min. 20, max. 22; fibre, max. 8.

National Calf Gruel.—Oil, min. 6, max. 12; albuminoid, min. 15, max. 24; fibre, max. 7.

Each of the four preceding compounds shall contain not more than 2·5 per cent. of lime (calc. as CaCO₃) exclusive of lime in the form of bone flour; not more than 1·5 per cent. of lime in the form of bone flour, and not more than 1·5 per cent. of salt (calc. as NaCl).

PIG FOODS.

National Pig Food, No. 1 (Pig nuts or pig meal for sows or weaners).—Oil, min. —, max. 4; albuminoid, min. 16, max. 18; fibre, max. 7.

The compound shall contain the following percentages of ingredients (Fourth Column of Table):—Maize meal and/or barley meal and/or dried potato products, min. 35, max. —; wheat feed (other than bran), min. 20, max. —; oats, min. —, max. 10; bran, min. —, max. 10; low protein oilseed cake and/or meal, min. —, max. 20; *fish and/or animal protein rich substances, min. 5, max. —; molasses, min. —, max. 5; *sundries including vitamin potent substances and mineral matter at maker's discretion provided that the salt-content of the compound shall not exceed 0·5 per cent., min. —, max. 10; together with sufficient *vegetable protein to ensure that the albuminoid-content agrees with the requirements given above.

National Pig Food, No. 2 (Pig nuts or pig meal for fattening).—Oil, min. —, max. 4; albuminoid, min. 13, max. 15; fibre, max. 7. Percentage ingredients as follows:—Maize meal and/or barley meal and/or dried potato products, min. 35, max. —; wheat feed, min. 25, max. —; ground oats and/or bran, min. —, max. 20; low protein oilseed cakes and/or meals, min. —, max. 25; *fish and/or animal protein rich substances, min. 2·5, max. —; molasses, min. —, max. 5; *sundries (as for No. 1, *supra*), plus *vegetable protein.

POULTRY FOODS (Mash or Pellets).

National Poultry Food, No. 1 (Summer Laying) (Laying mash or pellets).—Oil, min. 3, max. —; albuminoid, min. 17, max. 19; fibre, max. 8. Percentage composition:—Wheat feed and/or low protein oilseed cake and/or meal, min. 40, max. 65; cereals, min. 25, max. 50; *fish and/or animal protein rich substances, min. 5, max. 10; molasses (for pellets only), min. —, max. 5; *sundries including vitamin potent substances and mineral matter at maker's discretion, provided that the salt-content of the compound (calc. as NaCl) does not exceed 1 per cent., min. —, max. 7·5; plus *vegetable proteins to ensure specified albuminoid-content. No decorticated or un-decorticated cotton-seed cake or meal may be used.

National Poultry Food, No. 1A (Winter Laying).—Oil, min. 3, max. —; albuminoid, min. 17, max. 19; fibre, max. 8. Percentage composition:—Wheat feed and/or low protein oilseed cake and/or meal, min. 40, max. 65; cereals, min. 25, max. 50; *fish and/or animal protein rich substances, min. 5, max. 10; molasses (for pellets only), min. —, max. 5; *cod-liver oil equiv. to not less than 1 per cent. (on the weight of the compound) of cod-liver oil containing 85 international units of vitamin D per g.; *sundries (as for National Poultry Food, No. 1), min. —, max. 7·5; plus *vegetable protein as necessary for albuminoid-content. No decorticated or un-decorticated cotton-seed cake or meal may be used.

National Poultry Food, No. 2 (Summer Growing).—Oil, min. 3, max. —; albuminoid, min. 14, max. 17; fibre, max. 8. Percentage composition:—Wheat feed and/or low protein oil seed cake and/or meal, min. 40, max. 65; cereals, min. 25; max. 50; *fish and/or animal protein rich substances, min. 5, max. 10; molasses (for pellets only), min. —, max. 5; *sundries (as for National Poultry Food, No. 1), min. —, max. 7·5; plus *vegetable protein as necessary for albuminoid-content. No decorticated or un-decorticated cotton-seed cake or meal may be used.

* These ingredients may be in the form of licensed concentrates.

National Poultry Food, No. 2A (Winter Growing).—Oil, min. 3, max. —; albuminoid, min. 14 max. 17; fibre, max. 8. Percentage composition:—Wheat feed and/or low protein oil seed cake and/or meal, min. 40, max. 65; cereals, min. 25, max. 50; *fish and/or animal protein rich substances, min. 5, max. 10; molasses (for pellets only), min. —, max. 5; *cod-liver oil equiv. to 1 per cent. (on weight of compound) of cod-liver oil containing 85 international units of vitamin D per g.; *sundries (as for National Poultry Food, No. 1), min. —, max. 7.5; plus *vegetable protein as necessary for albuminoid-content. No decorticated or undecorticated cotton-seed cake or meal may be used.

National Poultry Food, No. 3 (Battery).—Oil, min. 3, max. —; albuminoid, min. 15, max. 18; fibre, max. 8. Percentage composition:—Wheat meal and/or low protein oilseed cake and/or meal, min. 35, max. 65; cereals, min. 30, max. 50; *fish and/or animal protein rich substance, min. 5, max. 10; molasses (for pellets only), min. —, max. 5; *cod-liver oil (as for National Poultry Food, No. 2A, *supra*); *sundries (as for National Poultry Food, No. 1), min. —, max. 15; plus *vegetable protein as necessary for albuminoid-content. No decorticated or undecorticated cotton-seed cake or meal may be used.

National Baby Chick Food.—Oil, min. 3, max. —; albuminoid, min. 16, max. 18; fibre, max. 6.5. Percentage composition:—Wheat feed and/or low protein oilseed cake and/or meal, min. 35, max. 65; cereals, min. 30, max. 50; *fish and/or animal protein rich substances, min. 5, max. 10; molasses (for pellets only), min. —, max. 5; *cod-liver oil (as for National Poultry Food, No. 2A, *supra*); *sundries (as for National Poultry Food, No. 1, except that salt must not exceed 0.75 per cent.), min. —, max. 20; *vegetable protein as necessary for albuminoid-content. Rye or rye products may not be used. No decorticated or undecorticated cotton-seed cake or meal may be used.

NOTE.—(1) The conditions as to oil, albuminoid and fibre contents found on analysis are subject to the limits of variation specified in the Fertilisers and Feeding Stuffs Regulations, 1932.

(2) Where cod-liver oil is prescribed, substances other than cod-liver oil may be used, provided that : (a) the vitamin D content of such substances in the aggregate is not less than that prescribed for cod-liver oil; (b) such substances are warranted in writing by the maker thereof as fully effective for poultry in accordance with the Chick Test of the British Standards Institution.

PART B. CEREAL MIXTURES. PERCENTAGE COMPOSITION (Third Column of Table)

National Cereal Mixture, No. 1.—Barley meal, min. 15, max. 30; maize meal, min. 45, max. 60; fine wheat feed, and/or imported middlings and/or imported pollards, min. 15, max. 25.

National Cereal Mixture, No. 2.—Maize meal, min. 35, max. 55; cattle ground oats, min. 30, max. —; fine wheat feed, and/or imported middlings and/or imported pollards, min. 10, max. 15.

National Poultry Corn, No. 1A.—Wheat and/or barley, min. 15, max. 30; oats, min. 20, max. 45; cut or kibbled maize, min. 40, max. 60.

National Poultry Corn, No. 1B.—Wheat and/or barley, min. 15, max. 30; clipped oats, min. 20, max. —; cut or kibbled maize, min. 40, max. 60.

National Poultry Corn, No. 2A.—Wheat and/or barley, min. 15, max. 30; oats, min. 20, max. —; whole maize, min. 40, max. 60.

National Poultry Corn, No. 2B.—Wheat and/or barley, min. 15, max. 30; clipped oats, min. 20, max. —; whole maize, min. 40, max. 60.

National Chick Feed, No. 1.—Fine cut wheat, min. 20, max. 45; No. 4 maize grits, min. 45, max. 70; cut groats, min. 10, max. —.

National Chick Feed, No. 2.—Cut wheat, min. 20, max. 40; No. 3 maize grits, min. 30, max. 60; whole groats, min. 10, max. —; dari and/or millet, min. —, max. 30.

National Chick Feed, No. 3.—Fine cut wheat, min. 50, max. —; No. 4 maize grits, min. 30, max. —.

National Chick Feed, No. 4.—Cut wheat, min. 50; No. 3 maize grits, min. 25, max. 45; whole groats, min. 10, max. —.

THE SECOND SCHEDULE

PART A

Any feeding stuff contained in the First Schedule to the Feeding Stuffs (Maximum Prices) Order, 1940, as amended, and any of the following feeding stuffs:

Alfalfa meal, biscuit meal, buckwheat (whole, crushed or ground), cod-liver oil or other vitamin potent substances, dari (whole, crushed or ground), distillery dreg meal, dredge corn (whole, crushed or ground), dried butter milk, dried clover meal, dried grass meal, dried liver meal, dried milk, dried yeast, feeding dried blood, groats, herbs, home-grown barley, oats, peas or wheat (whole, crushed or ground), Kaffir corn (whole, crushed or ground), licensed concentrates, licensed molassed feeding stuffs, liquorice-root, lucerne meal, malt, millet (whole, crushed

* These ingredients may be in the form of licensed concentrates.

or ground), mineral matter, rye (whole, crushed or ground), spices, sugar cane molasses, tares (whole, crushed or ground), wheat germ, whey paste, whey powder.

PART B

First Column Ingredient	Second Column Permitted use
1. Cocoa shell, husk, bran or waste.	Use in cattle or sheep foods (in quantities not exceeding 2.5 per cent. of the completed compound or mixed feeding stuff).
2. Dried milk, dried butter milk or whey powder.	Use in calf gruels and baby chick foods.
3. Wheat, whole, crushed or ground.	Use in poultry and chick foods.

STATUTORY RULES AND ORDERS. 1940. No. 1898

Raw Materials (Fertilisers)*

THIS Order revokes the Control of Fertilisers (No. 1) Order, 1939, Direction No. 5,^a and fixes the basic prices of superphosphate of lime, ground phosphate and compound fertilisers, for the months of November and December, 1940, and the first six months of 1941.

STATUTORY RULES AND ORDERS. 1940. No. 1899

Raw Materials (Fertilisers)†

THE CONTROL OF FERTILISERS (No. 7) ORDER, 1940, DATED OCTOBER 28, 1940, MADE BY THE MINISTER OF SUPPLY UNDER REGULATIONS 55 AND 98 OF THE DEFENCE (GENERAL) REGULATIONS, 1939.

THIS Order specifies further conditions for the purchase or sale of sulphate of ammonia.

Department of Scientific and Industrial Research

METHODS OF ANALYSIS OF COAL AND COKE‡

THIS Paper supersedes Survey Paper No. 7, which was published in 1927. It embodies the results of further investigation of details of the earlier methods, and includes methods for the examination of coke. The principal changes in the existing methods relate to those for the determination of volatile matter, nitrogen, phosphorus, and agglutinating value; methods included for the first time are for the determination of chlorine, arsenic and the forms of sulphur; additional methods are given for the determination of carbon dioxide and sulphur; and the section on the Gray-King carbonisation assay, previously dealing only with the low-temperature form, now includes a description of the apparatus and method for the high-temperature modification (at 900° C.), which is intended for the rapid evaluation of coals for the carbonisation industries.

VOLATILE MATTER.—The standard method described (heating for 7 minutes at 925° C.) should be used for all coals that give a coherent coke button and do not decrepitate. A modification, in which a slower rate of heating is employed, is used for coals that decrepitate, and another modification (admixture with non-caking coal) to prevent losses due to entrainment and decrepitation. An alternative method, requiring a more elaborate apparatus, may be used for anthracites and non-caking coals containing up to 12 per cent. of volatile matter. For coal the maximum furnace temperature should be 925° C., whilst for coke 950° C. is specified.

NITROGEN.—The method specified is a modification of the Kjeldahl process in which selenium is used as catalyst. The use of mixtures of selenium and mercury compounds is not recommended, for, although there is a slight saving in time during the combustion, subsequent addition of sodium sulphide becomes necessary.

PHOSPHORUS.—In the method previously specified several fusions of the ash with sodium carbonate were required, with extraction of each melt with water until all phosphorus had been removed. The new method consists in removing silicon from the ash by evaporation with mixed hydrofluoric and nitric acids, precipitating the phosphorus as phosphomolybdate, and titrating the precipitate. It was found by the Northern Coke Research Committee that comparatively

* H.M. Stationery Office, York House, Kingsway, London, W.C.2. Price 1d. net.

^a S.R. & O., 1940, No. 1712.

† H.M. Stationery Office. Price 1d. net.

‡ Fuel Research Paper No. 44. Physical and Chemical Survey of the National Coal Resources, October 28th, 1940. H.M. Stationery Office, York House, Kingsway, London, W.C.2. Price 2s. net.

excessive amounts of arsenic or titanium did not affect the results materially, but a special procedure may be necessary for ashes containing much vanadium.

AGGLUTINATING VALUE.—In the empirical test described, the agglutinating value is the maximum whole-number ratio of sand to unit coal in a mixture of sand and coal which, after carbonisation under specified conditions, gives a coherent mass capable of supporting a 500-g. weight, with less than 5 per cent. of the mixture remaining as a loose powder.

CHLORINE.—No practical method for the determination of water-soluble chlorine can be recommended. For the determination of total chlorine the coal is mixed with anhydrous sodium carbonate, and the mixture is heated at $475^{\circ} \pm 25^{\circ} \text{C.}$, in a muffle furnace until combustion is complete (about 20 hours). The chlorine is extracted with dilute nitric acid, the solution is treated with excess of standard silver nitrate solution, and the excess of silver nitrate is titrated with potassium or ammonium thiocyanate solution.

The amount of chlorine in coal usually ranges from 0.05 to 0.20 per cent., but some coals may contain as much as 1.0 per cent. and others as little as 0.01 per cent.

ARSENIC.—Arsenic is present in most coals, but usually in traces—less than 3 or 4 p.p.m. (as arsenious oxide), though in exceptional instances 40 to 50 p.p.m. may occur. In the method described the coal (ground to pass a 72-mesh B.S. sieve) is incinerated with a mixture of magnesium oxide, sodium carbonate and potassium nitrate, similar to that proposed by Hertzog (*Ind. Eng. Chem., Anal. Ed.*, 1935, 7, 163). The bases retain the arsenic, whilst the potassium nitrate maintains oxidising conditions and prevents the arsenic compounds being reduced to more readily volatilised arsenious compounds. After the incineration the arsenates are reduced to arsenites by means of sulphur dioxide, to ensure the subsequent complete reduction to arsine.

The arsenic is determined by a modification of the Gutzeit method, the arsenic being reduced to arsine, which is brought into contact with a paper strip impregnated with mercuric bromide. The length of the stain formed on the strip is a measure of the amount of arsenic. The apparatus described and illustrated is a modification of that devised by Crossley (*J. Soc. Chem. Ind.*, 1936, 55, 272r). The bottom section of the delivery tube contains filter-paper impregnated with lead acetate, and the middle section is packed with glass wool moistened with lead acetate solution. Any hydrogen sulphide is removed in these sections, whilst the arsine passes on to the mercuric bromide paper in the top section.

SULPHUR.—"Sulphate" sulphur is determined by extracting the coal with dilute hydrochloric acid and determining the sulphur in the extract. "Pyritic" sulphur is insoluble in dilute hydrochloric acid, but is quantitatively dissolved by dilute nitric acid. The "pyritic" iron is determined in the nitric acid extract, and the amount of sulphur corresponding to FeS_2 is calculated from the result. The two acid extractions may be carried out on the same portion of the coal sample. If they are made simultaneously, it is necessary to determine the iron in the hydrochloric acid extract to obtain the correction for the "pyritic" iron determination.

REPRODUCIBILITY OF ANALYTICAL RESULTS.—The degree of divergence to be expected between determinations made in the same laboratory or in different laboratories is discussed in an appendix. Only for volatile matter was the divergence in experimental results large enough to be serious. This is attributed partly to the fact that the apparatus used had not been fully standardised in different laboratories. Another appendix deals with the methods of reporting results, and particularly the method of calculating them on the basis of mineral-free matter or pure coal substance.

New South Wales

ANNUAL REPORT OF THE GOVERNMENT ANALYST FOR THE YEAR 1938

The total number of samples examined in the Chemical Laboratory is reported by Mr. S. G. Walton (Government Analyst) to be 33,655, including 31,079 under the Pure Food and Milk Acts, and 2576 for the Public Services. Of the 21,947 samples of milk examined, 1.36 per cent. of those from the metropolitan area were adulterated and 7.23 per cent. of the country samples.

BROWN BREAD.—The trade asked that the standard for fibre content should be lowered from the present figure (1.2 per cent.) to 0.75 per cent. As this would be equivalent to permitting brown bread to be made from a mixture of about 1 part of whole wheat flour with 2 parts of white flour, it was decided not to alter the present standard.

WHOLEMEAL BREAD.—Deficiencies in the crude fibre content of wholemeal bread led to several prosecutions during the year. In fixing the standard a generous tolerance was allowed, the minimum figure (2 per cent.) being less than that usually found. Thus the proportion of crude fibre in wholemeal milled in the Department of Agriculture ranged from 2.38 to 2.55 per

cent. Analyses were also made of wholemeal breads, prepared and baked in the Department of Agriculture, and the results were as follows:

Proportion of wholemeal used Per Cent.	Maximum moisture Per Cent.	Crude fibre (calculated on dry substance)	
		Determined Per Cent.	Calculated Per Cent.
100	45.7	2.57	2.57
80	45.4	2.12	2.06
60	45.0	1.66	1.54
40	45.0	1.12	1.03
100+5 per cent. of gluten	46.4	2.50	2.45

BAKING POWDER AND SELF-RAISING FLOUR.—A large manufacturing company requested the deletion of the Regulation requiring the declaration, on the label of packages containing self-raising flour and baking powder, of the presence of acid phosphate, where either sodium or calcium phosphate was used as the acid constituent of the aerating medium. An investigation of the baking and keeping properties of different types of self-raising flour and baking powder was therefore undertaken. The acid constituent of the samples prepared was (1) cream of tartar, (2) cream of tartar and tartaric acid, (3) calcium acid phosphate, and (4) sodium acid pyrophosphate. A self-raising flour was also prepared containing a mixture of calcium acid phosphate and sodium acid pyrophosphate (No. 6), a cream of tartar self-raising flour (No. 5) being used for comparison. The samples of baking powders and self-raising flours were prepared to give a theoretical yield of 15 per cent. by weight, and 52 grains per lb., respectively, of available carbon dioxide. In the tartrate powders and flours the acid ingredient and sodium bicarbonate were mixed to give an exact chemical balance, whilst the acid phosphate preparations were made according to formulae supplied, the acid ingredient being in slight excess. The actual available carbon dioxide of each sample was determined at the time of making the baking tests. Each powder and flour was stored under normal conditions for about 3 months, to ascertain its keeping qualities, the baking powders being packed in lever-lid tins and the flours in paper one side of which was glazed. After 3 months' storage the available carbon dioxide was again determined. The following percentage results, showing (a) original available carbon dioxide and (b) available carbon dioxide after 3 months, were obtained:—No. 1 (a) 14.58, (b) 13.09; No. 2 (a) 14.74, (b) 13.09; No. 3 (a) 11.9, (b) 4.68; No. 4 (a) 14.3, (b) 13.82.

These and other experiments showed that powders and flours prepared with acid calcium phosphate were inferior to tartrate preparations in keeping properties, but that the latter were inferior to acid sodium pyrophosphate preparations.

The baking tests were made by an expert at the East Sydney Technical College, scones and sponges being baked under standardised conditions. For the scones the grading of the powders by the expert in order of merit was 1, 2, 4, 3 and for the sponges it was 1, 2, 3, 4. In every instance the tartrate baking powder and self-raising flour yielded products superior in texture, flavour and volume. The articles baked with acid phosphate invariably had a marked flavour to which the Australian palate would have to become accustomed.

As a result of this investigation it was decided not to accede to the request mentioned above, but permission was given to alter the declaration required by deletion of the word "acid."

STANDARDS FOR CHEESE.—The Department of Agriculture concurred in alterations recommended, and these were given effect to by the Pure Food Advisory Committee. The revised standards are as follows:

- (1) *Cheese* is the solid or semi-solid product obtained by coagulating milk, cream or skim-milk with rennet or acid. It may contain ripening ferments, seasonings, salt (sodium chloride), flavourings, and permitted colouring matter. It shall not contain any foreign fat. For the purpose of this Regulation, milk shall be deemed to be the milk of any domestic animal.
- (2) *Cream cheese* shall contain not less than 70 per cent. of milk-fat in its water-free substance, and not more than 50 per cent. of water.
- (3) *Full or whole milk cheese* shall contain not less than 50 per cent. of milk-fat in its water-free substance, and not more than 40 per cent. of water.
- (4) *Skim-milk cheese* is cheese which contains less than 30 per cent. of milk-fat in its water-free substance.

There shall be written in the statement or label attached to every packet which contains skim-milk cheese or on the cheese itself in bold-faced sans-serif capital letters of not less than 18 points face measurement in such colours as to afford a distinct colour contrast to the ground, the words "Skim-milk Cheese."

- (5) Cheese sold under the name of Edam, Gruyère, Gouda, Stilton, or any other generally known name, shall correspond thereto in respect of composition and character.
- (6) Cheese sold without name or classification shall be deemed to be full or whole-milk cheese, and shall comply with the standard prescribed therefor.
- (7) (a) *Processed cheese, cheese paste or cheese spread*, not otherwise named to indicate the variety of cheese, shall contain not less than 45 per cent. of milk-fat in its water-free substance,

not more than 45 per cent. of water, and not more than 3 per cent. of harmless emulsifying agents.

- (b) Every packet of processed cheese shall bear a label in which shall be written in bold-faced sans-serif capital letters of not less than 12 points face measurement in such colours as to form a distinct colour contrast to the ground, the word "Processed."
- (8) *Luncheon cheese, cheese mixture or club cheese* is a cheese compound prepared from cheese with or without wholesome foodstuffs and condiments, and shall contain not less than 48 per cent. of milk-fat in its water-free substance, and not more than 35 per cent. of water.
- (9) The addition to luncheon cheese, cheese mixture or club cheese of sulphur dioxide (or sulphites calculated as sulphur dioxide) in proportion not exceeding 2 grains to the pound is hereby permitted.

d-SORBITOL IN DIABETIC CHOCOLATE.—An application was received for permission to use *d*-sorbitol in place of sucrose in the manufacture of diabetic chocolate. In view of the fact that its use is allowed by the existing standard for invalids' foods, permission was given. The addition of 3 grains of saccharin per lb. was also permitted.

ALKALINE CREAM.—A large number of samples of cream brought into New South Wales from an adjoining State were found to contain an alkaline preservative, probably sodium peroxide. After successful prosecutions samples of cream no longer gave reactions for peroxide, but still contained a foreign alkaline substance either used as a neutralising agent or being the residue from a preserving process. This addition was detected by determining the *pH* value, and legal proceedings were taken whenever the *pH* exceeded 7. The recorded values for the *pH* of cows' milk show a considerable range (6.4 to 7.2). The normal range given by Van Slyke and Barker (*J. Biol. Chem.*, 1919, 345) is 6.5 to 6.75. It is unlikely therefore that the *pH* of any commercial cream would exceed 6.85 unless alkaline neutralisers had been used. Fifteen samples of cream found to contain alkaline peroxide had *pH* values ranging from 7.20 to 7.50, whilst 8 samples, reported to contain an alkaline neutraliser but no peroxide, had *pH* 7.18 to 7.45. Later samples showed values less than 7.0, and in some of these it is probable that the alkalinity from the peroxide process had been balanced by an addition of acid. A low freezing-point (Hortvet) (−0.57 to −0.58) supported this conclusion.

NITROUS OXIDE FOR WHIPPED CREAM.—An application was received for permission to use nitrous oxide in whipped cream. The application was not recommended because the introduction was for the purpose of increasing the bulk, and also in view of the absence of definite information as to the harmlessness or otherwise of nitrous oxide.

DIETHYLENE GLYCOL IN ESSENCE OF LEMON.—Two samples of essence of lemon contained diethylene glycol as the vehicle. As this substance cannot be considered harmless, its use as a solvent in essences is a contravention of the standard under the Act.

STANDARD FOR MALTED MILK.—The following standard, based on the results of the examination of all the brands of malted milk sold in New South Wales, has been adopted as reasonable for both the manufacturer and consumer.

- (1) Malted milk powder is the product made either
- (a) by combining whole milk with the liquid separated from a mash of ground malt and wheat or other cereal flour in such a manner as to secure full enzymic action of the malt extract and reducing the product to dryness by desiccation under reduced pressure; or
 - (b) by mixing dried whole milk with dried malt extract.
- (2) Malted milk powder shall contain not less than 7.5 per cent. of milk-fat, not more than 3.5 per cent. of water, and may contain salt (sodium chloride) not exceeding 1 per cent. It shall be free from rancidity.
- (3) Malted milk for retail sale shall be packed in airtight receptacles.

STANDARD FOR TRIPE.—With a view to ensuring that a uniform article shall be supplied, the following revised standard has been adopted:

Tripe as sold for human consumption shall not be prepared so as to impair its nutritive properties, nor contain any added foreign matter except salts, and its reaction value (determined on the liquid obtained by exudation and pressure) shall not be less than *pH* 6.5 nor greater than *pH* 7.5.

ZINC IN CANNED VEGETABLES.—The deep green colour of certain vegetables was found to be due to the presence of organically combined zinc. Successful prosecutions caused the manufacturers to abandon the use of zinc.

Other samples of canned vegetables were found to be coloured with an aniline dye; this practice was also stopped by legal proceedings. Subsequently a request for permission to use an aniline dye for this purpose was received, but after consideration permission was refused.

Patiala (Punjab) India

REPORT OF THE CHIEF CHEMIST FOR THE YEAR ENDED APRIL 12TH, 1940

THE Central Analytical Laboratory of the State of Patiala was established in March, 1939, at the Mahendra College, under the direction of Mr. K. L. Budhiraja, M.Sc., A.I.C. It was set up primarily to exercise control over the Ghee Grading Station at Namaul, and to analyse soils and minerals from the Development Department, but in practice it has been doing work for all the State Departments. The 307 samples submitted consisted mainly of ghee, but there were also 69 samples of soils, 11 samples of minerals, and 16 samples of water. When the Pure Food Act comes into force the work of the Laboratory is certain to expand.

GHEE.—Of the 211 samples submitted, 83 were received from the Ghee Grading Station, 73 from executive authorities, and the remainder from Medical Officers of Health, etc. In the absence of any standards for purity, the Patiala State adopted those laid down by the Agricultural Marketing Adviser to the Government of India. Fifty-five of the samples (other than those from the Grading Station) were genuine, 69 were adulterated, and the others were of doubtful purity. The degree of adulteration ranged from 5 to 70 per cent., and 15 samples consisted of pure hydrogenated vegetable fat popularly called vanaspati or vegetable ghee; this is the most common adulterant. Prosecutions were undertaken and fines were imposed as the result of analyses at the Laboratory. The Department was also helpful in checking the smuggling of vegetable ghee products in evasion of the local (octroi) duties.

Ceylon

ANNUAL REPORT OF THE GOVERNMENT ANALYST FOR THE YEAR 1939

The Government Analyst (Mr. J. V. Collins) reports that the war has not affected the analytical work of the Department, and that there has been a small increase in the number of articles examined (4255 as compared with 4095 in 1938). Much of the work is concerned with criminal investigation; 1091 cases were investigated as against 1041 in 1938.

STAIN CASES.—There were 128 cases in which identification of blood was required, and 150 cases (with 323 exhibits) in which identification of the species of blood was asked for. Of these exhibits, 195 gave positive reactions for human blood and 20 for blood other than human.

An interesting case was one in which a heavy blood stain on the vest of an accused person was found, very unexpectedly, to be a mixture of ox blood and pig blood. On inquiry it was found that the accused had been arrested by a public-spirited butcher, who had knocked him down with the first weapon that came to hand—a joint of beef. This joint was on the butcher's stall, and above it was suspended a joint of pork.

POISONING CASES.—Exhibits from the Courts were examined in 84 cases, and poison was detected in 46. The poisons identified included copper salts (7), powdered glass (5), arsenic (4), arsenic and mercury (2), nitric acid and nitrates (3), prussic acid (3), disinfectants (3), croton oil (3), quinine (2), nux vomica (2), dyestuffs (2), mydriatic alkaloids (2), acetic acid (2), bleaching powder, a saponin, an unidentified alkaloid, etc.

Copper salts are becoming increasingly popular, owing apparently to a misguided belief in their efficacy in abortion cases. The dyestuffs are probably used as "charms" or are added mischievously, as the usual history is that some one introduced an unknown substance into a pot of cooking rice, which subsequently became brightly coloured.

Quinine Poisoning.—In the cases of quinine poisoning two hospital patients received an overdose of quinine hydrochloride amounting to nearly 100 grains. Both patients died shortly after, and fits were a symptom in each case. The following quantities of anhydrous quinine (in grains) were recovered:—Stomach and contents, 10, 11; kidneys, 1/3, 5/13; liver, 1 1/3, 1; brain, trace in each case.

DECIPHERING NUMBERS ON BICYCLES.—Sixty bicycles, as compared with 3 in 1938, were examined to discover the original number. There was no evidence of alteration of the number on 16 of these; on 10 it was not possible to restore the original number; some of the original figures could be made out on 9, and on 25 the whole of the original number could be deciphered.

MILK.—Of the 659 samples examined, 391 were adulterated. It is remarkable that for many years past a higher percentage of genuine samples has been received from Local Authorities than from the Medical Department—46 per cent. as against 32 per cent. Cane sugar was found in 20 samples, mostly of milk supplied on contract to Government hospitals. Turpentine, an unusual constituent, was found in one sample.

ARRACK.—In connection with the control of Government arrack, 124 samples from distilleries and warehouses were examined for the Excise Department. As a result of cleaner collecting and better fermentation of the toddy, the quality of the spirit is improving yearly and the acidity figure is falling to a more satisfactory level.

Standardisation of Testing Methods for Textiles

DEFINITIONS OF ATMOSPHERIC HUMIDITY AND THE MOISTURE CONTENT AND REGAIN OF TEXTILE MATERIALS*

THE following definitions are given for reference in connection with Standard Specifications. Standardised methods for determining the physical constants here defined are to be published later.

ABSOLUTE HUMIDITY AND RELATIVE HUMIDITY.—The absolute humidity of the atmosphere is defined as the mass of water present per unit volume. The relative humidity is the ratio of the actual amount of water per unit volume to the amount necessary to saturate a unit volume at the same temperature and pressure. The relative humidity is usually expressed as a percentage.

DEW POINT.—If moist air is cooled, condensation occurs as the state of saturation is approached. The temperature at which condensation begins is termed the dew point.

MOISTURE-CONTENT.—The percentage moisture-content means the amount of moisture in a material when expressed as a percentage of the total weight.

REGAIN OF TEXTILE MATERIALS.—When the moisture in a textile material is calculated as a percentage on the oven-dry weight, the percentage is termed the regain. Thus, with a moisture-content of 20 per cent., the regain is $100 \times 20 \div 80$, *i.e.* 25 per cent.

Standard Regains.—These are arbitrary values chosen as the most useful for commercial purposes. The correct conditioned or "correct invoice weight" of a consignment shall be calculated from its oven-dry weight and the recognised standard regain.

More or less arbitrary percentage moisture-contents and regains have been attributed to the different textile fibres and the yarns and cloth made from them, so that for trade purposes they may be declared "in correct condition" when they contain the recognised percentages of moisture or the equivalent "regain." The following "standards of regain" are commonly, but not universally, accepted:

	Standard moisture-content Per Cent.	Standard regain Per Cent.
Cotton	7.83	8.5
Silk	9.91	11
Flax and hemp	10.71	12
Jute	12.09	13.75
Wool and waste	13.79	16
Wool (tops combed with oil)	15.97	19
Wool (tops combed without oil)	15.43	18.25
Worsted yarns	15.43	18.25
Carded woollen yarns	14.53	17
Wool noils (ordinary)	12.28	14
Wool noils (scoured and carbonised)	13.79	16
Woollen and worsted cloths	13.79	16
*Viscose and cuprammonium rayons	9.91	11
*Cellulose acetate rayon	5.66	6

* Other standards are applicable in certain circumstances.

When these standards were first made, it was assumed that when 100 parts of the fibre in the oven-dry condition were exposed to the "usual condition of the atmosphere" they would "regain" moisture to the extent shown in the foregoing table. When the moisture-contents and the regains of the material agree with the standards in the table, they are regarded as "100 per cent. in correct condition."

Equilibrium Regain.—After exposure for a sufficient time to an atmosphere whose relative humidity and temperature are kept constant, the regain of the material reaches a steady value termed its "equilibrium regain."

STANDARD ATMOSPHERE.—Before mechanical or physical tests are applied, textile materials must be "conditioned" by exposure under standard conditions of relative humidity and temperature. In this country and in the United States the conditions denoted by 65 ± 1 per cent. relative humidity at $70^\circ \pm 2^\circ$ F. are being widely observed. On the Continent the corresponding standards are $65, \pm 1$ per cent. and $20^\circ \pm \frac{5}{2}^\circ$ C., the greater tolerance for temperature being allowed to avoid, as far as possible, refrigeration in the summer.

DETERMINATION OF RELATIVE HUMIDITY*

Under normal conditions the ventilated wet and dry bulb hygrometer and the sling hygrometer give results of sufficient accuracy for commercial purposes. Other methods, capable of greater accuracy, depend on the direct determination of the dew point or the weighing of the moisture in a measured quantity of air.

Various empirical relations connecting the wet bulb and dry bulb temperatures with the dew point have been derived and tables have been computed to avoid the calculation of the relative humidity. Glaisher's *Hygrometric Tables* are no longer used in scientific work. For the ventilated hygrometer and the sling hygrometer the psychrometric tables issued by the Russian Meteorological Institute have been amplified and are widely used at the National Physical Laboratory, Teddington. The tables in the Appendix to the Standards on Textile Materials prepared by Committee D13 of the American Society for Testing Textile Materials are based on the Smithsonian Meteorological Tables. Marvin's *Psychrometric Tables*, published by the U.S.A. Dept. of Agriculture, are used fairly generally; they can be obtained from Casella & Co., Ltd., Fitzroy Square, London, W.1.

In other instruments there is a sensitive material, such as human hair or goldbeaters' skin, and the changes in this resulting from the absorption or loss of water are recorded. These instruments should not be used in cases of dispute and should be calibrated regularly by comparison with a standard hygrometer.

British Standards Institution

BRITISH STANDARD No. 598—1940. METHODS FOR THE SAMPLING AND EXAMINATION OF BITUMINOUS ROAD MIXTURES†

THIS Specification, first issued in 1936, has recently been revised, and several modifications have been introduced to keep it up-to-date with present practice. In addition to the complete revision of certain sections, a modified test for the rapid determination of bitumen-content and two alternative methods for the recovery of bitumen have been introduced.

The rapid method for the determination of soluble bitumen is that described in the *J. Inst. Petroleum*, 1939, 25, 185, 168–177, but in cases of dispute the standard method, which is essentially that described in *J. Soc. Chem. Ind.*, 1931, 50 (*Chem. and Ind.*), 599, must be used.

The two alternative methods for the recovery of soluble bitumen are to be regarded as tentative until experience in their use has been gained.

DEFINITIONS.—The following definitions, which (with the exception of that for asphalt) have been adopted by the Permanent International Association of Road Congresses, are applicable throughout.

Bitumen.—Mixtures of hydrocarbons of natural or pyrogenous origin or combinations of both (frequently accompanied by their non-metallic derivatives) which can be gaseous, liquid, semi-liquid or solid, and which are completely soluble in carbon disulphide.

Asphaltic Bitumen.—Natural or naturally occurring bitumen, or bitumen prepared from natural hydrocarbons by distillation or oxidation or cracking; solid or viscous, containing a low proportion of volatile products; possessing characteristic agglomerating properties, and substantially soluble in carbon disulphide.

Asphalt.—A natural or mechanical mixture in which asphaltic bitumen is associated with inert mineral matter. An indication of the origin or nature of "asphalt" should always be given.

Tar.—A bituminous product, viscous or liquid, resulting from the destructive distillation of organic material. The word "tar" should be preceded by the name of the substance from which it was derived, and its mode of production should also be indicated.

CONTENTS OF THE SPECIFICATION.—Part I, Definitions. Part II, Sampling: (1) Asphalt, (2) Tarmacadam, (3) Information to be supplied with sample. Part III, Methods of Examination: Pot stain test—Apparent specific gravity—Voids—Soluble bitumen-content: (1) Hot and cold asphalt, (2) Tentative method for recovery of bitumen, (3) Tarmacadam.—Water-content: (1) Asphalt, (2) Tarmacadam.—Mineral aggregate: (1) Tests on mineral aggregate, (2) Preparation of sample, (3) Petrographical identification—Certificate of analysis—Appendix: Method for the rapid determination of soluble bitumen.

* *J. Text. Inst.*, 1940, 31, 55.

† Copies of the Specification can be obtained from the British Standards Institution, 28, Victoria Street, London, S.W.1. Price 2s.; post free 2s. 3d.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS

Food and Drugs

Simplified Alkali-libability Determination for Starch Products. T. J. Schoch and C. C. Jensen. (*Ind. Eng. Chem., Anal. Ed.*, 1940, 12, 531-532.)—Raw starch decomposes in hot aqueous alkali to give simple acidic substances, principally formic, acetic and lactic acids as well as pyruvic aldehyde, and this suggests that the reaction is initiated by enolisation of free terminal aldehyde groups as formulated by Evans (*J. Amer. Chem. Soc.*, 1926, 48, 2665; 1930, 52, 294) for the aldose sugars. With acid-modified starches alkaline decomposition proceeds more rapidly, indicating increased aldehyde-content. The reaction provides a simple and precise measurement of alkali-libability, the rate of decomposition of the starch (the alkali number) being the number of ml. of 0.1 N sodium hydroxide solution consumed by each g. of starch during digestion with alkali at 100° C. for one hour. The starch should be pulverised to pass through a 60-mesh sieve, and its moisture-content determined separately by drying *in vacuo* at 105° C. for 4 hours, the alkali number being calculated for the dry substance. When a starch product contains sufficient added acid or alkali to affect the alkali number, 1 g. is gelatinised in hot water and neutralised to thymol blue with standard acid or alkali, and the alkali number is corrected for this titre. The digestion with alkali may conveniently be made in a feeding bottle provided with its rubber cap which is pierced by means of a hot needle to allow an exit for steam. The starch (500 mg.) is gently agitated with 10 ml. of water to form a uniform suspension, 25 ml. of 0.4 N sodium hydroxide solution are added with continued agitation and finally 65 ml. of hot water, and the bottle is capped and placed in a vigorously boiling water-bath. If the starch product gelatinises in cold water, it is first treated in a dry bottle with 1 or 2 ml. of benzene before addition of the alkali and water. The bottle is heated for

exactly an hour, after which it is placed in cold water, and 50 to 75 ml. of water are added to stop the decomposition process. The excess alkali is titrated to the yellow end-point of thymol blue with standard acid and, as a blank determination, 25 ml. of the alkali solution are titrated with the standard acid. Strict adherence to the conditions described is recommended. The method has been applied to a number of theoretical and practical problems. Commercial maize and wheat starches have higher alkali numbers than the tuber starches, and this is attributed to different molecular configuration rather than to hydrolytic decomposition during manufacture. A particular starch is not necessarily composed of identical molecules. By leaching maize starch at a temperature just below gelatinising point by the method of Baldwin (*J. Amer. Chem. Soc.*, 1930, 52, 2907) a small amount of soluble carbohydrate with a high alkali number and probably consisting of small molecules was obtained, and a typical acid-modified thin-boiling starch exhibited a similar heterogeneity. When raw maize starch is digested with hot alkali and the neutralised solution is treated with alcohol the precipitated carbohydrate has an alkali number lower than that of the original starch, but even when the pre-digestion with alkali is continued until nearly all the starch is destroyed the alkali number of the residual carbohydrate does not fall below 4.0. With increasing degree of acid conversion, the alkali number rises progressively and reaches values from 56 to 66 for the white dextrans and Lintner's soluble starch. When white dextrin is dried at 105° C. there is a progressive fall in alkali-libability, and this may be due to the formation of anhydrides less susceptible to attack by alkali. Alkali-libability must not be regarded as a quantitative evaluation of aldehyde-content but merely as an empirical index of the degree of hydrolysis. A. O. J.

Component Acids and Glycerides of some Indian Ox Depot Fats. T. P. Hilditch and K. S. Murti. (*Biochem. J.*, 1940, 34, 1301-1311.)—The analytical charac-

teristics of four different Indian ox depot fats (taken mainly from the perinephric tissues) were as follows:

Fat from	Sap. val.	Iodine val.	Free fatty acids (as oleic) Per Cent.	Unsap. matter Per Cent.	Fat, m.p. (open tube) ° C.	Mixed acids, setting-point ° C.
Bombay cow, original ..	280.1	26.6	1.4	—	—	—
" " neutralised ..	282.2	26.5	0.1	0.35	50.5	47.6
" bullock, original ..	282.0	26.1	2.4	—	—	—
" " neutralised ..	283.2	25.8	0.1	0.3	50.0	48.5
Calcutta cow and bullock, original	283.2	31.0	1.3	0.2	49.0	48.4
Calicut cow, original ..	283.5	31.1	0.2	0.4	50.5	48.7

From each fat, neutralised, if necessary, the mixed fatty acids were liberated and separated by the usual lead salt and alcohol method; the methyl esters of the respective acids were

then fractionally distilled. The mixed acids were found to possess the following composition (in mols. per cent.):

	Bombay cow	Bombay bullock	Calcutta ox and bullock	Calicut cow
Saturated acids:				
Lauric	0.2	0.3	0.3	0.5
Myristic	5.2	4.4	2.8	3.7
Palmitic	43.3	39.1	38.8	34.7
Stearic	22.9	27.9	25.5	27.9
Arachidic	0.4	1.0	0.4	—
Unsaturated acids:				
Tetradecenoic	0.5	0.5	0.4	0.4
Hexadecenoic	1.4	1.0	2.2	1.6
Oleic	25.1	24.8	28.0	29.5
Octadecadienoic	0.9	0.9	0.9	1.3
C ₂₀₋₂₂ unsaturated	0.1	0.1	0.7	0.4

Compared with the composition of English, North and South American and Australian ox depot fats previously examined, the unsaturated acid content (27 to 33 per cent.) is low, the acids from other ox depot fats never containing less than 40 per cent. and frequently as much as 50 per cent. The deficiency of oleic acid in the Indian fats is made up by an increased proportion not of stearic acid, but of palmitic acid, and the proportion of this acid is indeed higher than has hitherto been observed in any animal depot fat. For a wide range of land animals (including birds) the palmitic acid content has been found to be 25 to 30 per cent. As a consequence of the high proportion of palmitic acid the

"melting-points" of the fats and the setting-points of their mixed fatty acids are little higher than those of the usual ox depot fats; a high proportion of stearic acid, on the other hand, would have resulted in a considerably higher melting-point. The Bombay cow and Calicut cow depot fats were further examined to determine the nature of their component glycerides. The fats were resolved by systematic crystallisation from acetone into three fractions of different consistence and iodine value, and these were separately examined as previously described (Hilditch and Paul, *Biochem. J.*, 1938, **32**, 1775). The probable component glycerides (mols. per cent.) are as follows:

	Bombay cow	Calicut cow
Fully saturated:		
Tripalmitin	3.0	—
Dipalmitostearin	22.6	16.5
Palmitodistearin	10.3	11.8
Mono-"oleo"-disaturated:		
Hexadecenodistearin	—	0.1
Hexadecenopalmitostearin	2.4	—
"Oleo"-dipalmitin	18.2	11.0
"Oleo"-palmitostearin	33.0 to 32.2	41.5 to 38.4
"Oleo"-distearin	—	1.8
Di-"oleo"-monosaturated:		
Palmitodi-"oleic"	9.7 to 10.5	14.2 to 17.3
Stearodi-"oleic"	0 to 0.8	0 to 3.1
Tri-"oleic"	0.8 to 0	3.1 to 0

The glycerides of the Indian ox depot fat are thus of the mixed type characteristic of all other animal depot fats, but the Calicut fat shows a much closer resemblance to an English fat than does the Bombay fat; the total of fully saturated components in the Calicut fat (28 per cent.) is, however, higher

than in English fat (17 per cent.). The Bombay cow depot fat is remarkable in possessing the highest proportion of fully saturated glycerides (36 per cent.) yet observed in an animal depot fat; the proportions of dipalmitostearin and of "oleo"-dipalmitin are abnormally high.

F. A. R.

Mixed Unsaturated Glycerides of Liquid Seed Fats. II. Low Temperature Crystallisation of Cottonseed Oil. T. P. Hilditch and L. Maddison. (*J. Soc. Chem. Ind.*, 1940, **59**, 162-168; cf. *ANALYST*, 1940, **65**, 364.)—The method of crystallisation of solid fats from acetone at 0° C. or -10° C., has been extended to cottonseed oil as representing the more liquid and unsaturated fats, but much

lower temperatures have been used. The component acids (per cent. by weight) of the cottonseed oil were: myristic, 1.4; palmitic, 23.4; stearic, 1.1; arachidic, 1.3; tetradecenoic, 0.1; hexadecenoic, 2.0; oleic, 22.9; linolic, 47.8. The oil was separated into 6 fractions by systematic crystallisation from acetone at temperatures down to -35° C., and the acids in each fraction were determined. The

technique was simplified by adding solid carbon dioxide directly to the acetone solution in the following manner:—The acetone solution of the fat was placed in a 3-litre round-bottomed flask contained in an insulated rectangular cardboard box, which had a movable fibre partition and was fitted with a similar lid, circular holes being cut in each to take the neck of the flask. At the bottom of the box there was a layer of three inches of cotton wool. The fraction of the cotton-seed oil to be crystallised was dissolved in dry acetone (3 to 7 ml. per g. of fat) in the flask, and small pieces of solid carbon dioxide (not larger than $\frac{1}{4}$ -inch cubes, wiped free from adherent frost) were added to the solution, with vigorous stirring, further portions being added when the evolution of carbon dioxide gas ceased, until the temperature had fallen to the desired point. The space between the movable partition (resting on the shoulder of the flask) and the lid of the box was then loosely packed with larger lumps (about 1-in. cubes) of solid carbon dioxide. In this way the solution could be held for any desired period (*e.g.* up to 7 hours) at a temperature varying by not more than $\pm 2^\circ\text{C}$., but thorough stirring at intervals was necessary to prevent local over-cooling. The separated fraction was removed by filtration with the aid of a pump, the low temperature being maintained by insulating the Buchner funnel in a box packed with crushed solid carbon dioxide. The stem of the funnel passed through a hole in the base of the box and was connected with a large filter-flask which was not artificially cooled. The solid fat fraction collected in the funnel was washed with 100–200 ml. of acetone previously cooled with solid carbon dioxide to -45°C ., and was then heated at 100°C . *in vacuo* to remove all acetone, whilst the acetone filtrates were distilled and the residual dissolved fat was also heated at 100°C . *in vacuo*.

The possible component glycerides in each fraction thus obtained were then considered in the light of the component acids found, but without the help which with the more saturated fats was to be had from a study of the fully saturated components present, or of the products of hydrogenation of the fractions. Sufficient separation was effected to show that each fraction was to a very large degree made up of only 2 of the 4 possible categories of mixed saturated unsaturated glycerides—fully saturated, mono-unsaturated-disaturated, di-unsaturated-mono-saturated and tri-unsaturated glycerides. Large proportions of mixed oleolinoleo-glycerides and extremely small proportions of the simple forms, trilinolein or triolein, appeared to be present in each fraction. Conclusive quantitative data for the component glycerides in each fraction could not be obtained from the component acid analyses alone, but the following conclusions were reached:—The chief glycerides in the cotton-seed oil examined were about 58 per cent. of saturated (mainly palmito-) di-unsaturated glycerides, accompanied by about 28 per cent.

of tri-unsaturated glycerides; smaller proportions (13 per cent.) of mono-unsaturated-disaturated glycerides, and very small traces of palmitin. Of the main components, 35–40 per cent., were probably palmito-oleolinoleins, with 20 per cent., or somewhat more, of palmito-dilinoleins and possibly small amounts of palmito-diolein; oleodilinoleins formed probably almost the whole of the tri-unsaturated glycerides, although here again the presence of small amounts of triolein was not excluded. The minor quantities of mono-unsaturated glycerides were made up of somewhat more linoleo- than oleo-compounds, and the saturated acyl radicals present quite possibly included one palmitic and one minor component saturated acid (myristic, stearic or arachidic) in most of the triglyceride molecules of this group. One fraction (32.5 per cent. of the whole fat) consisted almost wholly of mono-saturated di-unsaturated glycerides, and palmitic acid formed over 80 per cent. of the saturated acids of this fraction. Hydrogenation at as low a temperature as possible gave a saturated product which on crystallisation yielded β -palmitodistearin (m.p. 67.7°C .; synthetic β -palmitodistearin has m.p. 68°C .). It is thus established that, in the monopalmito glycerides of cotton-seed oil, the palmitic group is exclusively attached to the β - or central hydroxyl group of the glyceryl radical.

D. G. H.

Determination of Iron and Copper in Butter. G. M. Moir and E. D. Andrews. (*New Zealand J. Sci.*, 1940, **21**, 249–265.)—

Iron.—Ten-g. samples of the butter are melted in 15-ml. centrifuge tubes, 1 ml. of 5 per cent. sodium hydrosulphite ($\text{Na}_2\text{S}_2\text{O}_4$) solution is added, and the tubes are shaken and left overnight at $35^\circ\text{--}40^\circ\text{C}$. One ml. of 20 per cent. trichloroacetic acid is introduced, the tubes are left for 30 minutes at $40^\circ\text{--}50^\circ\text{C}$., and the melted fat is removed by suction. The aqueous layer is shaken with 5 drops of 10 per cent. sodium tungstate solution and filtered cold through papers washed, immediately before, with 5 per cent. nitric acid. The filtrate is shaken with 0.5 ml. of saturated potassium persulphate solution, and then with 2 drops each of nitric acid and hydrogen peroxide. One ml. of 30 per cent. ammonium thiocyanate is added, and the contents of all the tubes, including the standards, are made up to the same volume, and shaken with 2 ml. of amyl alcohol to extract the colour. To clear the amyl alcohol layer, the tubes are chilled in ice-water overnight, and the colours of the samples and standards are matched in a colorimeter. Very accurate results were obtained with the Klett bio-colorimeter. In 6 samples of butters the amounts of iron ranged from 0.3 to 0.9 p.p.m.

Copper.—Twenty-five-g. samples of the butter are melted at $40^\circ\text{--}50^\circ\text{C}$. in 50-ml. centrifuge tubes, and then each is treated with 5 drops of 3 per cent. hydrogen peroxide, 12 drops of conc. hydrochloric acid and 2.5 ml. of 20 per cent. trichloroacetic acid. The tubes

are well shaken and kept at 40°–50° C. for 30 minutes, after which the separated fat is removed. After cooling, 5 drops of 10 per cent. sodium tungstate are added to the aqueous layers in each of the tubes, and the tubes are again shaken and left for 30 minutes. Each sample is filtered, and the precipitate is washed with 3 ml. of 4 per cent. nitric acid and then with 5 per cent. trichloroacetic acid. The filtrate is treated with 2 ml. of 20 per cent. sodium citrate solution (to prevent precipitation of phosphate) and with a few drops of phenolphthalein solution. Sufficient ammonia (the same amount for each tube) to make the liquid alkaline is added, and each sample is made up to 40 ml. and treated with "a few grains" of solid sodium diethyl dithiocarbamate and 5 ml. of amyl alcohol. The tubes are shaken, allowed to stand for 3 or 4 hours, and again shaken, to complete the extraction of the coloured compound. The colours of the amyl alcohol extracts are then compared in a colorimeter with standards prepared from copper sulphate. In test experiments, in which known quantities of copper (0.1 to 0.5 p.p.m.) were added to a butter (copper content, 0.09 p.p.m.), the results were in close agreement with the calculated amounts. The copper-contents of 370 samples of creamery butter ranged from less than 0.1 to over 1.0 p.p.m. (1 sample); in 64 per cent. of the samples it did not exceed 0.2 p.p.m.

If the principal reagents are adequately purified, it is possible to obtain blank results so low that no appreciable error is caused by adding the reagents to the standards. For this purpose the trichloroacetic acid must be distilled *in vacuo*. The sodium tungstate and sodium citrate solutions are treated with a few mg. of sodium diethyl dithiocarbamate, followed by 2 or 3 extractions with amyl alcohol in a separating funnel. The aqueous solution is then ready for use.

Wet Oxidation Method.—More concordant results were obtained by the following modification of the wet oxidation method of W. Williams (*J. Dairy Res.*, 1931, 3, 93) than by the dry ashing procedure:—Twenty-five-gram samples of the butter are weighed into 50-ml. centrifuge tubes, 8 ml. of nitric acid (A.R. quality) are added to each, and the tubes are placed for about an hour in a boiling water-bath, their contents being stirred at intervals to ensure thorough mixing. The samples are cooled until bubbles of gas (nitrous fumes) cease to be evolved from the aqueous layer and are then centrifuged. Most of the fat is removed by means of a pipette attached to the water-pump, after which 5 ml. of petroleum spirit (b.p. 120° C.) are introduced, and this is removed in the same manner. The contents of each tube are transferred to a 200-ml. Kjeldahl flask, which is heated over a small flame until only about 1 ml. of liquid remains. Two ml. of sulphuric acid are introduced into each flask, and the heating is continued until nitrous fumes are no longer evolved; if necessary, the oxidation may be completed by adding a few drops of 30 per cent. hydrogen

peroxide. The crystalline residues are dissolved in water that has been redistilled from glass, and the solutions are transferred to Pyrex test-tubes, neutralised and treated with the reagents (citrate, etc.), and the analyses are completed in the colorimeter as before. Blank estimations are made simultaneously and a correction applied. Results thus obtained with 23 samples of butter (copper, 0.04 to 0.79 p.p.m.) agreed closely with those given by the filtration method.

Composition of the Fatty Acids in Chinese Coconut Oil. H. Nobori. (*J. Soc. Chem. Ind. Japan*, 1940, 43, 199–200.)—The dried kernels of coconuts growing in Hainan Island, South China, contained 65.69 per cent. of oil with the following characteristics:—sp.gr. at 30/4° C., 0.9203; n_D^{30} , 1.4536; m.p., 24.2° C.; solidif. p., 22° C.; saponification value, 260.6; iodine value, Wijs, 8.7; Polenske value, 12.5; Reichert–Meissl value, 6.6; Hehner value, 90.7; thiocyanogen value, 6.7; acid value, 1.1; unsaponifiable matter, 0.38 per cent. The mixed fatty acids had sp.gr. at 30/4° C., 0.8999; m.p. 24.5° C.; solidif. p., 23° C.; n_D^{30} , 1.4410; saponification value, 268.4; iodine value, 9.9; neutralisation value, 263.5. The mixed fatty acids (500 g.) were esterified with an equal volume of methyl alcohol to which 2 per cent. sulphuric acid had been added and 492 g. of pale yellow liquid esters were recovered having sp.gr. at 20/4° C., 0.8776; n_D^{30} , 1.4352; saponification value, 254.1; iodine value, 9.5; acid value, 8.1. Twelve fractions were obtained by distillation of 470 g. of esters, and the residue was fractionated into a further three fractions. These fractions were refractionated in the usual manner, and the constants of the liberated fatty acids were determined. From the iodine and thiocyanogen values the fatty acids of the original coconut oil were calculated to contain: saturated acids, 92.2; oleic acid, 5.5; linolic acid, 2.3 per cent. Redistillation of the fatty acids from the higher-boiling fractions (14 and 15) indicated the presence of arachidic acid; each fatty acid fraction from 8 to 13 was separated into solid and liquid parts by the lead salt and alcohol method, but no hexadecenoic acid, $C_{18}H_{30}O_2$, found by Longenecker (*J. Biol. Chem.*, 1939, 130, 167), could be detected. The composition of the saturated fatty acids was calculated to be caprylic, 8.7; capric, 8.1; lauric, 51.3; myristic, 13.1; palmitic, 7.5; stearic, 2.0, with a small quantity of arachidic acid.

D. G. H.

Factors Affecting the Efficiency of Materials used in Packaging Frozen Foods. W. Rabak. (*Paper Trade J.*, 1940, 111, T.A.P.P.I. Sect., 110–112.)—The resistance of carton boards to penetration by moisture may be measured from the increases in weight of anhydrous calcium chloride, which is placed in an air-tight container the lid of which is the carton material, in 6 days at

50 per cent. relative humidity and at 0° and 70° F. The calcium chloride (50 g.) was weighed into shallow weighing-bottles, which were placed in heavy-gauge aluminium containers (diameter 13.4 cm., depth 6.5 cm.), having a flat shoulder (width 1.8 cm.) on which were placed a pair of heavy rubber rings. The sample was then clamped tightly between the rings so as to seal the container. Results obtained at 0° and 70° F., respectively, were:—Coated boards (6 samples), 0.15, 0.78; impregnated boards (6 samples, types not specified), 0.24, 2.3 g. per 1000 sq.cm. per day. When a moist material was placed in direct contact with the carton (thickness, 0.020 in.) the figures obtained at 70° F. were:—Coated paperboard, 6.2; impregnated paperboard, 38.2; untreated cardboard, 110.2 g. of water per 1000 sq.cm. per day. Wax-treated papers were found to allow little penetration of moisture vapour, especially at low temperatures, whilst with vegetable parchment waxed on both sides the transmission was nil or negligible in 6 days. Creasing, however, produced marked increases in transmission, especially with waxed papers and at the higher temperatures. Moisture-proof viscose and laminated rubber materials were very efficient at all temperatures, even when creased. The losses in weight of loose frozen peas in various types of packages were determined by weighing at intervals for 9 months during storage at 0° and 15° F. under commercial conditions. The efficiency of the package in preventing losses in weight was found to depend mainly on the wrapping material used, but also on the efficiency of the seal. Satisfactory results were obtained with heavily-waxed paper, vapour-proof viscose and laminated rubber sheets, but ordinary viscose was less satisfactory. In general, a package which allows the loss of not more than 0.1 per cent. of the weight of the contents at 0° F. and 1.0 per cent. at 15° F. in 9 months, is regarded as satisfactory. J. G.

Biochemical

Human Foot Perspiration and Upper Boot Leather. A. Colin-Russ. (*J. Hyg. Camb.*, 1940, 40, 447; *J. Int. Soc. Leather Trades Chem.*, 1940, 24, 359.)—The effect of human perspiration on full chrome upper leathers tanned with basic chrome sulphate liquors is important in connection with the suitability of upper material for boots. The collagen complex of the leather is disrupted by the acidic and saline constituents of the perspiration, and solubilised chromium and ionisable sulphate are progressively released, the process being more pronounced at higher temperatures and when the tannage is more basic. The process may be regarded as double decomposition resulting in the formation of sodium sulphate, which may sometimes render the leather fibre brittle.

Estimation of Serum Iron and Pseudo-haemoglobin Iron with o-Phenanthroline. G. Barkan and B. S. Walker. (*J. Biol.*

Chem., 1940, 135, 37–42.)—Two ml. of blood or serum are measured into a short rimless test-tube, 1 ml. of 1.2 per cent. hydrochloric acid is added, and the tube is stoppered and kept at 37° C. for at least 1 hour. After cooling, 1 ml. of 20 per cent. trichloroacetic acid is added, and the mixture is allowed to stand for 1 hour at room temperature. The resulting suspension is centrifuged, and 2 ml. of the clear supernatant liquid are transferred to the cell of an Evelyn colorimeter. To the solution are added 0.5 ml. of saturated sodium acetate solution, 0.5 ml. of 1 per cent. hydrazine sulphate solution (to reduce the iron to the ferrous condition) in 2 M-acetate buffer solution (pH 4.5), and 0.5 ml. of 0.1 per cent. o-phenanthroline monohydrate solution. A blank solution, prepared by mixing 0.5 ml. of each of the above reagents with 0.5 ml. of the hydrochloric acid, 0.5 ml. of the trichloroacetic acid and 1 ml. of water, is introduced into another cell. The cells are stoppered and left for at least 1 hour at room temperature, after which 2.5 ml. of water are put into each cell, and the colours are measured in an Evelyn photoelectric colorimeter with filter 490. Ferric iron added to horse serum was estimated, with a maximum error of 4 per cent.

To determine pseudo-haemoglobin iron ("easily split off" blood iron), defibrinated blood, filtered through glass wool, is diluted with 4 volumes of water, and 10 ml. of this diluted blood are oxygenated by shaking in air, heated with 5 ml. of 1.2 per cent. hydrochloric acid, incubated at 37° C. for 16 to 24 hours and cooled, after which 5 ml. of 20 per cent. trichloroacetic acid are added, and the mixture is left for 1 hour at room temperature. It is then filtered and the filtrate is treated as described above to produce the red ferrous phenanthroline complex. F. A. R.

Improved Method for the Estimation of Non-haemin Iron. G. Brückmann and S. G. Zondek. (*J. Biol. Chem.*, 1940, 135, 23–30.)—One g. of the tissue pulp is ground in a mortar with powdered glass, 5 ml. of saturated sodium pyrophosphate solution and 10 ml. of 10 per cent. trichloroacetic acid. The mixture is transferred to a wide centrifuge tube, heated in a boiling water-bath for exactly 7 minutes and centrifuged. The residue is washed twice with 4 ml. of a mixture of the two reagents in equal parts. The combined centrifugate and washings are made up to volume, and aliquot portions are used for colorimetric estimations of iron either with thioglycollic acid or with o-phenanthroline. When great accuracy is required, the residue may be re-digested with sodium pyrophosphate solution and trichloroacetic acid, but the additional iron recovered in this way is seldom more than 5 per cent. of the total, and may usually be ignored. An extinction photometer (Leitz) with filter 495m μ is used for measuring the colours of the final solution. The results obtained by the method were compared with those obtained by digestion with pyrophosphate and trichloroacetic acid at room temperature for 3 days, and by the

hydrochloric acid extraction method of Starkenstein and Weden (*Arch. exp. Path. Pharmacol.*, 1928, **134**, 274). In addition, the total iron-contents of the same tissues were measured after ashing with sulphuric and perchloric acids, and the haemin iron was estimated by the method of Yabusoe (*Biochem. Z.*, 1925, **157**, 388): One g. of the tissue pulp is ground with powdered glass, 1 ml. of *N* hydrochloric acid and a few ml. of ice-cold absolute methyl alcohol. The mixture is centrifuged, and the residue is washed with methyl alcohol. The combined centrifugate and washings are shaken with 1 g. of powdered magnesium sulphate (iron-free), left for 30 minutes and centrifuged. The colour of the solution is compared with that of a haemin standard solution (filter 620 μ), or, better, its iron-content is estimated after ashing. The value so obtained may be subtracted from the total iron-content, giving yet another estimate of non-haemin iron content. The agreement between this value and that found by the new method was very close, the maximum divergence being 4 per cent. The hydrochloric acid method gave inconsistent results.

F. A. R.

Polariscopic Determination of Proteolytic Activity. Q. Landis. (*Cereal Chem.*, 1940, **17**, 468-472; *J. Inst. Brewing*, 1940, **46**, 380.)—The method is based on the fact that the approximately linear decrease in the sp. rotation of gelatin as the temperature rises from 15° to 30° C., is affected by the action of proteolytic enzymes on the gelatin to an extent which depends on the enzymic activity. A fresh solution of gelatin is prepared by soaking 2 g. in 98 ml. of cold water and then warming the mixture to 40° C., an acetate buffer (to produce pH 4.8), and toluene (as preservative) being also added. A mixture of 25 ml. each of this solution and of an infusion of the enzyme in 2.5 per cent. sodium chloride solution (both at 40° C.) is digested in a 50-ml. stoppered Erlenmeyer flask for 5 hours at 40° C. If the solution is then turbid, it is cleared by immersion in boiling water until the soluble protein flocculates. The flask is then cooled rapidly, immersed in ice-water for 1 hour and then warmed to 20° C. and the value of $[\alpha]$ is determined; solutions which are turbid at this stage should be filtered. Blank solutions containing (a) no enzyme and (b) no gelatin should be made concurrently, but the former one should be poured into the polarimeter tube within 1 minute after cooling or it will gel. The following relationship between the concentration (c) of the enzyme (in milli-units per 50 ml. of reaction-mixture containing 25 ml. of the enzyme infusion) and the fall in rotation (p°) was deduced from experiments with a preparation of known proteolytic activity:

$$\log c = (0.728p + 0.378).$$

This holds for values of *c* ranging from 10 to 25. The original paper also contains a modified calibration method for use at high summer

temperatures, and tabulated values obtained with papain and with brewers' and distillers' malts.

J. G.

Determination of Tungsten in Biological Materials. J. C. Aull and F. W. Kinard. (*J. Biol. Chem.*, 1940, **135**, 119-121.)—The sample (e.g. 2 ml. of blood, 10 ml. of urine or 2 g. of organ) is heated gradually in a 500-ml. Kjeldahl flask with 4 ml. of conc. sulphuric acid, 10 ml. of a 1:1 mixture of conc. nitric acid and 70 to 72 per cent. perchloric acid, and a few glass beads. If charring occurs, more of the nitric and perchloric acid mixture is added until the solution clears. The heat is increased until white fumes are no longer evolved. After cooling, the solution is diluted with water and made distinctly alkaline to litmus with 40 per cent. sodium hydroxide solution. The solution is boiled vigorously for several minutes, cooled, transferred to a graduated flask of suitable size, and made up to volume. After filtering, a 5.0-ml. aliquot portion (containing not more than 0.1 mg. of tungsten) is measured into the tube of an Evelyn photoelectric colorimeter and the following reagents are added with shaking: 1.0 ml. of 15 per cent. potassium thiocyanate solution, 4.0 ml. of conc. hydrochloric acid and 1.0 ml. of titanium chloride reagent prepared by boiling 1.0 ml. of 10 per cent. titanous chloride solution with 2.0 ml. of conc. hydrochloric acid to expel traces of hydrogen sulphide and then diluting to 80 ml. with conc. hydrochloric acid. After 10 minutes the tubes are placed in the colorimeter and read against a blank tube containing reagents only, using filter 420. The tungsten-content is calculated by reference to a standard curve prepared from solutions containing 0 to 0.1 mg. of tungsten. The range of recoveries of added tungsten was from 95 to 105.8 per cent., with an average of 99.9 per cent.

F. A. R.

[Preparation of] Stable 2,6-Dichlorophenol-indophenol Solutions. I. Stone. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 415.)—Solutions of sodium 2,6-dichlorophenol-indophenol used for the determination of vitamin C (*cf.* Mottern, Nelson and Walker, *ANALYST*, 1933, **58**, 48) are relatively unstable, and must frequently be replenished and stored in the cold. Thus, after 8 days' storage of a solution at 2° and 30° C., the results of an assay fell, respectively, from 153 to 143 and from 153 to 90 mg. of reagent per 100 ml. of solution. Pre-extraction of the dry reagent with ether (*cf.* Knight, Dutcher and Guarrant, *Science*, 1939, **89**, 184) did not effect much improvement. The following procedure is suggested:—Slightly more than the requisite weight of reagent (usually approximately 0.2 g. per 100 ml., for 0.005 *M* solutions) is stirred well with distilled dioxane (see below) containing 1 ml. of glacial acetic acid per 100 ml. After 15 minutes no more lumps should remain, and the solution is then filtered through a dry No. 42 Whatman paper. The clear red filtrate is standardised against pure ascorbic

acid in the usual way, the volume of dioxane added being less than 10 per cent. of the total volume in the titration-vessel, as larger proportions obscure the end-point. Since peroxides are produced in the commercial product if it is allowed to stand, 500 ml. of the dioxane should be distilled, the first and last 50 ml. being rejected. Traces of peroxides, however, may still remain, but they do not interfere with the vitamin C assay, although they affect the iodine and sulphuric acid method of standardisation (Menaker and Guerrant, *ANALYST*, 1938, **63**, 201); hence the use of pure ascorbic acid for this purpose. The rate of evaporation of dioxane is not appreciably greater than that of water, and assays made at intervals during $3\frac{1}{2}$ months with 0.005 *M* solutions (initial assay, 146 mg. per 100 ml.) showed decreases in strength of only about 2 mg. per month.

J. G.

Estimation of Aneurin by a Modification of the Melnick-Field Method. A. D. Emmett, G. Peacock and R. A. Brown.

(*J. Biol. Chem.*, 1940, **135**, 131-138.)—The method of estimating aneurin devised by Melnick and Field (*cf. ANALYST*, 1939, **64**, 367) was applied to complex mixtures containing some of the other vitamins. The only modifications introduced were a simplified technique for adsorption and elution, and the substitution of the Lovibond tintometer for the colorimeter. Using pure solutions of aneurin, it was found that the concentration of the vitamin was directly proportional to the colour measured in red units and a standard curve was constructed from the data so obtained. The estimation of aneurin in tablets and ampoules gave results ranging from 95 to 103 per cent. of the theoretical value. The presence of other synthetic vitamins (riboflavin, vitamin B₆, nicotinic acid, pantothenic acid and ascorbic acid) had no effect on the accuracy of the aneurin assay, except when relatively much ascorbic acid was present, *e.g.* 2 mg. (aneurin 30γ). This interference by ascorbic acid could be overcome by cautious addition of either 0.01 *N* iodine solution or 1 per cent. potassium ferricyanide solution, but there is a risk that excess of the reagent will be added and this will attack the vitamin B₁. A better method of overcoming the interference is to adsorb the aneurin on "Superfiltrol" (see below). The following pharmaceutical preparations were assayed by the direct method: wheat germ extract, emulsion of vitamins A, B and D, fortified liver extract, stomach extract with vitamin B₁. With the exception of the liver extract, the results obtained were in close agreement with the values obtained by biological assay. In some instances the vitamin may be present either in a complex soluble mixture or in a combined form, as in yeast, and it is necessary to digest the material to split off the vitamin; interfering factors must then be removed and this is best done by adsorption of the aneurin. A stock solution of the test material is prepared, of such a concentration that it contains between 5 and 20γ

of aneurin per ml. Of this solution, 2-, 3- and 4-ml. portions are transferred to three 15-ml. centrifuge tubes and diluted to 5 ml. The pH is adjusted to 4-5 with dilute hydrochloric acid, 0.1 to 0.15 g. of "Superfiltrol" is added, and the tubes are shaken intermittently for an hour; they are then centrifuged and the supernatant liquid is discarded. To each of the adsorbates are added 3 ml. of water and 3 ml. of 95 per cent. alcohol containing 5 mg. of phenol per ml. The pH is then adjusted to 7-8 (thymol blue) with dilute sodium hydroxide solution, and 6 ml. of the diazo reagent are added. The mixture is left for at least 2 hours at room temperature and then filtered on a small Hirsch filter. The adsorbate is washed with 5 ml. of water and transferred, with the filter-paper, to a dry centrifuge tube, and 2 ml. of 95 per cent. alcohol are added. The tube is stoppered, shaken to elute the pigment, and then centrifuged. The supernatant liquid is transferred to a tintometer cell, and the colour is measured, the results being calculated from the standard curve. Whereas low results were obtained with a dried yeast, a wheat germ extract and an elixir by the direct assay method, results given by the adsorption method agreed closely with the biological values for these products.

F. A. R.

Chemical Estimation of Nicotinic Acid: Inhibitory Effect of Cyanogen Bromide upon the Aniline Side Reactions. D. Melnick and H. Field.

(*J. Biol. Chem.*, 1940, **135**, 53-58.)—In the method previously described (*cf. ANALYST*, 1939, **64**, 367) for the estimation of nicotinic acid in biological material by reaction with cyanogen bromide and aniline, a blank was prepared by simply diluting the test solution with an alcoholic buffer solution. Harris and Raymond (*Biochem. J.*, 1939, **33**, 2037), however, advocated the addition of the base (in their method *p*-aminoacetophenone) to the blank solution, as it was found that the base reacted with substances in the hydrolysates to produce colours indistinguishable from that obtained with nicotinic acid. It has now been found that aniline behaves in a similar manner, but that in presence of cyanogen bromide the interfering side-reaction does not occur. Thus the addition of aniline to the blank solution is not recommended.

F. A. R.

Assay Method for Pantothenic Acid. D. Pennington, E. E. Snell and R. J. Williams.

(*J. Biol. Chem.*, 1940, **135**, 213-222.)—The pantothenic acid content of tissue extracts can be estimated by measuring the growth-response of a culture of *Lactobacillus casei* ε. A basal medium containing alkali-treated peptone, glucose, sodium acetate, alkali-treated yeast extract, acid-hydrolysed casein, cystine, riboflavin and inorganic salts is prepared and the pH is adjusted to 6.6 to 6.8; 5-ml. portions of the medium are introduced into each of 10 bacteriological test-tubes. To some of the tubes are added volumes up to

5 ml. of a standard pantothenic acid solution and to others the samples for analysis. The tubes are sterilised, cooled and inoculated with 1 drop of a 24-hour culture of the organism in normal saline. The tubes are then incubated at 37° C. for 72 hours. The response of the organisms to pantothenic acid can be measured either by titrating the lactic acid produced during growth or by measuring the turbidity in a thermoelectric turbidimeter or in an Evelyn photoelectric colorimeter. The response, by both methods of measurement, was found to be nearly directly proportional to the amount of pantothenic acid present when this was between 0.05 and 1.2 mg. units (1 mg. unit = 0.08% of calcium *d*(+)-pantothenate). A standard curve must be prepared with each set of assays. With pure solutions of pantothenic acid, recoveries of 105 to 109 per cent. were obtained by the titrimetric method and of 94 to 105 per cent. by the turbidimetric method. Recoveries of a similar order were obtained when pantothenic acid was added to yeast extract, liver extract or whey; such extracts were prepared by autoclaving, preferably after autolysis under benzene. Some substances, however, did not show such good recoveries and the assay figures did not agree when calculated from different doses. The most important of these substances was urine; others were oysters and mushrooms. The error could be rectified by adding to the basal medium a supplement prepared by autoclaving the sample in question with sulphuric acid, and then removing the sulphuric acid by treatment with baryta. F. A. R.

Antiscorbutic Values of Fruits and Vegetables. M. Olliver. (*Lancet*, 1940, II, 190-195.)—The following values were found for the amounts of ascorbic acid (mg. per 100 g.) in a number of fruits and vegetables as normally consumed. (a) Raw fruits and vegetables: watercress, 58 to 72; strawberry, 44 to 93; orange-juice, 33 to 77; lemon-juice, 29 to 60; grapefruit-juice, 28 to 64; raspberry, 21 to 37; tomato, 12 to 42; lettuce, 5 to 30; apple, 4 to 17; banana, 6 to 8; melon, 2 to 3; pear, 2 to 3. (b) Home-cooked or commercially canned fruit and vegetables: black currant, 80 to 152; brussels sprouts, 19 to 44; asparagus 13 to 44; strawberry, 19 to 35; spinach, 9 to 50; cauliflower, 16 to 42; gooseberry, 13 to 31; cabbage, 11 to 33; potato (new), 10 to 20; turnip, 11 to 17; pea, 6 to 20; raspberry, 4 to 17; potato (old), 7 to 10; carrot, 1 to 15; stringless bean, 2 to 6; greengage, apple, plum, 1 to 4; apricot (dried), 1 to 2; prune (dried), 1. With the exception of potatoes, the outer tissues of fruits and vegetables generally contain more ascorbic acid than the inner tissues. Ordinary methods of drying fruits readily destroy vitamin C, whereas cooking or canning cause only a partial loss. In household cooking this loss is brought about not so much by the effect of temperature as by extraction of the vitamin in the liquor; with fruit, the syrup or water is consumed, but with vegetables the liquor is thrown away. The more water

used in boiling the vegetables and the longer the time of heating, the greater is the loss of vitamin. The presence of alkali, however, does not increase this loss, contrary to earlier work which suggested that the addition of soda to boiling greens had an adverse effect on the ascorbic acid. In canning there is little loss of ascorbic acid during the cooking process, most of the loss observed occurring during the preliminary blanching or hot-water treatment used to destroy enzymes; the loss by extraction is less than in home-cooking because less water is used. The material used by the canner must be fresh, whereas that purchased by the housewife has usually been stored for some time, and it has now been shown that considerable losses of ascorbic acid occur during storage. Thus, canned fruits and vegetables tend to contain more of the vitamin than the corresponding materials purchased in shops and cooked at home. Vitamin C, contrary to certain beliefs, withstands the heat treatment of jam boiling, the apparent loss observed being due chiefly to dilution with added sugar and water. The costs of vitamin C from various sources are compared. F. A. R.

Methylene Blue Reducing System of Palestine Orange Peels investigated by the Thunberg System. L. Frankenthal. (*Enzymologia*, 1939, 6, 287-306.)—Peel juice, prepared with the aid of a hydraulic press, was decolorised with animal charcoal. The filtrate had a high reducing activity towards methylene blue in presence of phosphate; this could not be due to ascorbic acid, which is adsorbed by animal charcoal. In absence of phosphate, reduction was very slow. The reducing substance was heat-labile, unlike that present in orange-juice, but, notwithstanding its heat-lability, it passed readily through membranes, thus lacking the colloidal nature ascribed to enzymes. The properties of the substance were compared with those of a number of reducing systems, and were found to agree very closely with those of dehydroascorbic acid and its more stable isomer, 2:3-diketogulonic acid. The presence of dehydroascorbic acid in orange peel juice was confirmed (a) by incubating the juice with glutathione (this produced ascorbic acid, which was then titrated with dichlorophenolindophenol) and (b) by isolating the 2:4-dinitrophenylsazone of dehydroascorbic acid, m.p. 271 to 273° C. It was also shown that the dehydroascorbic acid was actually present as such in the peel and not produced subsequently either by catalytic oxidation by traces of metal or as a result of the charcoal treatment. Dehydroascorbic acid was found not to be stable in the juice for long, but changed into diketogulonic acid, this being responsible for the reduction of the methylene blue; old juice contained no dehydroascorbic acid. No dehydroascorbic acid was found in orange juice. The results suggest that orange peel contains an enzyme capable of oxidising ascorbic acid to dehydroascorbic acid. F. A. R.

Erratum. September issue, p. 519 (Estimation of Phosphorus), line 12 of the Abstract. For "about 0.4 mg. of phosphorus" read "not more than 0.4 mg. of phosphorus."

Organic

Determination of Hydroxyl Groups with the Grignard Reagent. W. Fuchs, N. H. Ishler and A. G. Sandhoff. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 507-509.)—The method of Zerewitinoff (*Ber.*, 1907, **40**, 2023; 1908, **41**, 2236) for the determination of active hydrogen based upon the evolution of methane in reactions with magnesium methyl iodide is not highly accurate, and the improved method of Kohler, Stone and Fuson (*J. Amer. Chem. Soc.*, 1927, **49**, 3181) is too intricate for occasional use. It was found necessary therefore to devise a simpler and more rapid method based upon that of Zerewitinoff. The apparatus consists of a reaction vessel connected with a manometer and a gas burette. The reaction vessel consists of a lower portion containing the reagent and an upper portion carrying a vertical capillary tube and two horizontal side tubes connected with manometer and gas burette in the usual manner. The upper portion fits over the lower portion by means of a ground-glass joint and is firmly held in position by brass springs. The apparatus differs from conventional design chiefly in that the sample is weighed into steel cups, which are suspended from protuberances on the vertical capillary tube in such a manner that they can be lowered into the reagent by means of an electromagnet. The air and moisture in the apparatus are replaced by pure dry nitrogen. No volume changes occur during manipulation, and a dibutyl phthalate manometer makes pressure adjustments rapid and accurate. The sample, which has been dried at 100° C. in a 2-mm. vacuum (or if volatile at a lower temperature and higher pressure), is weighed into two steel cups which are then hung on the capillary tube in the reaction vessel containing the Grignard reagent dissolved in 15 to 20 ml. of iso-amyl ether. The apparatus is quickly fitted together and flushed out with dry nitrogen which has been passed through iso-amyl ether. The reaction vessel is then surrounded with a water-bath at 70° C., the flushing with nitrogen is continued and the gas burette is filled with nitrogen. The water-bath at 70° C. is replaced by one at room temperature, and the system is adjusted repeatedly to atmospheric pressure until pressure conditions remain stable for 5 or 10 minutes. One of the cups is then lowered into the reagent, and the reaction, which is usually vigorous, is allowed to proceed. After reaction has ceased the usual procedure in gas analysis is followed, and the methane evolved is measured. The second portion of sample serves for a duplicate determination, and its cup can be lowered into the reagent as soon as the first determination is completed. Calculations were based on the density of methane at the

temperature at which the system was maintained during the determination. Grignard reagent prepared with *n*-butyl ether as solvent reacted more quickly than the reagent in iso-amyl ether, with equally precise results, but pyridine was found unsuitable as a solvent. Benzoic acid, anisic acid, α -naphthol and β -naphthol gave results closely in accord with theory. The results obtained with acetoacetic ester and malonic ester agreed with the theoretical values for the completely enolised compounds. The toluic acids gave high results, probably due to impurities. The three phthalic acids, trimesic acid, pyromellitic acid and benzene pentacarboxylic acid did not yield methane with the reagent, the apparent explanation being that, since these compounds are insoluble in the medium, the Grignard reagent does not react in heterogeneous systems. Picric acid did not react, probably owing to its insolubility, but tribromophenol reacted completely. Resorcinol, hydroquinone, toluhydroquinone and phloroglucinol either failed to react or reacted incompletely. Their solubility was slight and they are known to be capable of keto-enol tautomerism. Resorcinol in *n*-butyl ether solution apparently reacts as a monoketomonoenol compound. A. O. J.

Photometric Estimation of Furfural. R. A. Stillings and B. L. Browning. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 499-502.)—The colorimetric method for the estimation of furfural by means of the red colour produced with salts of aniline or xylidene has the advantage over the gravimetric and volumetric methods that the methylfurfural and hydroxymethylfurfural, always present in the distillates from plant tissues, are not included in the determination. For transmission measurements a General Electric recording spectrophotometer was used, and for photometric measurements an absorption meter and a Weston Photronic cell connected through a potentiometer with a microammeter. A Jena BG 18 glass filter was fitted to the opening of the photronic cell. Glass cells providing a thickness of liquid of 5 mm. were used. Owing to the difficulty of obtaining pure xylidene or its isomers (or constant mixtures of these) the reaction with aniline acetate was considered the more satisfactory for photometric work. A study of the effects of variations of temperature and of the concentrations of acetic acid, aniline, sodium chloride and furfural yielded the following results:—Increase of acetic acid concentration considerably increased the stability of the colour, and a concentration of 200 to 500 g. per litre proved satisfactory. Neutralisation of the distillate prepared by the usual methods (containing about 12 per cent. of hydrochloric acid) introduced up to 200 g. of sodium chloride per litre, and the final concentration after the addition of other reagents was 20 g. per litre. It is recommended that this concentration should be maintained. The statement of other investigators (Barta, *Biochem.*

Z., 1934, **274**, 212; Riffart and Keller, *Z. Unters. Lebensm.*, 1934, **68**, 113; Tolman and Trescott, *J. Amer. Chem. Soc.*, 1906, **28**, 1619; Abst., ANALYST, 1907, **32**, 49) that Beer's Law is valid for the furfural-aniline colour was confirmed for furfural concentrations of 0.5 to 4.5 mg. per litre in presence of the above-mentioned amount of sodium chloride, and under these conditions a concentration of aniline of 50 g. per litre provided high sensitivity, reasonable stability and relatively slight variations in colour with variations in aniline concentration. Rise of temperature decreased the stability of the colour and the time required to reach the minimum transmission. The transmission at its minimum value is not affected over the range 15° to 30° C., but for precise work the temperature should be controlled, preferably at 20° ± 0.5° C., at which temperature minimum transmission is reached in 55 minutes. The error introduced by presence of methylfurfural and hydroxymethylfurfural is less than 1 per cent. if the concentration of these substances is less than that of the furfural, as it is in the usual pentosan distillates. The method is as follows:—The solution containing 0.05 to 0.45 mg. of furfural is exactly neutralised to phenolphthalein with 10 per cent. sodium hydroxide solution and, after dilution of the liquid with water to about 40 ml., sufficient sodium chloride solution (200 g. per litre) is added to produce a final concentration of 20 g. per litre. Freshly distilled aniline (5 g.) is added by means of a specially graduated pipette to 50 ml. of glacial acetic acid and, after adjustment of the temperature to 20° C., this reagent is added to the liquid, which is then diluted with water to 100 ml. and stored in the dark at 20° ± 0.5° C. for 55 minutes. The transmission is then measured, and a control determination is made with the reagents by the same procedure; the result of this is taken to be equivalent to 100 per cent. of furfural. The quantity of furfural present in the sample is found by means of a calibration curve constructed for the instrument.

A. O. J.

Linolenic Acid in the Leaves of Land Plants. M. Tsujimoto. (*J. Soc. Chem. Ind. Japan*, 1940, **43**, 208–209.)—The leaves of certain land plants were dried and extracted with ether or petroleum spirit, the extract was saponified with alcoholic potassium hydroxide, the unsaponifiable matter was removed, and the fatty acids obtained by decomposition with hydrochloric acid were dissolved in ether. The solution was chilled and treated with a very large excess of bromine, and the precipitated bromides were collected, weighed and analysed. The bromide yields (as per cent. of the ethereal extracts) were as follows:—*Sasa paniculata* (Nemagaridake), 2.6; *Sasa albo-marginata* (Kumazasa), —; *Pinus Thunbergii* (black pine), 0; *Ginkgo biloba*, green leaves, 0.5; yellow leaves, 0.7; *Platanus orientalis*, 1.6; *Trifolium repens* (clover), 2.0. The linolenic bromide from the

leaf oil of *Sasa paniculata* melted at 177° to 178° C. and contained 63.28 per cent. of bromine. Except in the oil from *Pinus Thunbergii*, linolenic acid was present in all the leaf oils, but as far as the present experiments go, highly unsaturated acids, similar to those present in fish oils and in algae (*cf. Chem. Umschau*, 1925, **32**, 125), are not present.

D. G. H.

Determination of Sludge in Used or Oxidised Motor Oils. C. G. Ludeman. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 520–525.)—Motor oil sludge is composed of the alteration products of the oil, dust, dirt, metal wearings, emulsions, etc., but this investigation was confined to the alteration products formed by oxidation and polymerisation, and the oils used were artificially sludged by blowing air through them at 175° C. The components of the sludge may be grouped into asphaltenes (bitumens soluble in carbon disulphide but insoluble in paraffin naphtha), carbenes (bitumens soluble in carbon disulphide but insoluble in carbon tetrachloride) and carboids (insoluble in carbon disulphide). About 10 ml. of oil were weighed in an Erlenmeyer flask, and the sludge was precipitated by adding 200 ml. of solvent a portion of which was retained for washing down the sides of the flask. The tightly stoppered flask was set aside in the dark at room temperature (about 25° C.) and, after exactly 24 hours the contents were filtered through a 30-ml. Coors No. 4 Gooch crucible which contained a mat, 3 mm. thick, of Powminco asbestos and had been thoroughly dried at 110° C. The supernatant liquid was passed through the crucible first, the sludge being retained as far as possible and added all together at the end. When the contents of the flask had been completely transferred, the cake of sludge was sucked dry until it was broken by cracks. During the washing of the crucible with the solvent the suction was interrupted three times for two minutes while the crucible was half-full of solvent. Usually 150 to 200 ml. of solvent were required for washing. After the final washing the crucible was dried at 110° C. for an hour and weighed. A number of solvents were examined for their precipitating properties, and isopentane was found to precipitate the greatest amount of sludge. An attempt to relate the chemical and physical properties of solvents with their precipitating power was unsuccessful, and, since the possibility of the exact reproduction of solvents consisting of the various naphthas from crude sources is small, it is recommended that definite chemical compounds, such as isopentane, carbon tetrachloride and carbon disulphide, should be used as standard precipitants. A fourth reproducible solvent is propane, as recommended by Hall, Levin and McMillan (*Ind. Eng. Chem., Anal. Ed.*, 1939, **11**, 183). The following is a complete analytical procedure for used or oxidised motor oils:—(a) asphaltenes, carbenes and carboids with inorganic material by precipitation with isopentane; (b) carbenes and carboids with

inorganic matter by precipitation with carbon tetrachloride; (c) carboids with inorganic matter by precipitation with carbon disulphide; (d) soluble sludge by propane precipitation of the oil obtained by evaporation (preferably *in vacuo*) of the filtrate from (a); (e) dissolved sludge by propane precipitation of an oil sample filtered at 25° C., with or without pressure; (f) inorganic material by ignition of the precipitates (a), (b) or (c). The individual components are then found as follows:—Soluble sludge is given directly by (d); asphaltenes are given by the difference between (a) and (b); carbenes by the difference between (b) and (c); carboids by the difference between (c) and (f); inorganic material is given directly by (f); undissolved sludge is given by the difference between (a) and (e), and dissolved sludge directly by (e). The sludge was also determined by a volumetric method based upon its separation by precipitation and centrifuging by a standardised procedure, but there appeared to be no simple relation between the sludge found by this method and that found by the gravimetric method.

A. O. J.

Effect of Glycerin on the Distillation Method for Water. R. B. Trusler. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 509–510.)—

Xylene is frequently recommended as the distillation medium in the determination of water in soap by the method of Dean and Stark (*J. Ind. Eng. Chem.*, 1920, **12**, 486; *Abst., ANALYST*, 1920, **45**, 270), but it is necessary to determine whether soaps containing glycerin (e.g. liquid potassium soap, potassium-vegetable oil soap and the so-called cold-made soaps) behave in the same manner towards the distillation medium as do the majority of bar, flaked and powdered soaps containing no glycerin. In order to determine the effect of the presence of glycerin, water was added to weighed quantities of anhydrous glycerol and the distillation was carried out with the precautions described in a previous paper (*Oil and Soap*, 1939, **16**, 239). The data obtained with benzene were similar to those obtained with toluene, the maximum average error obtained when 4 to 10 ml. of water were added to 10 g. portions of glycerol being +0.095 ml. with benzene and +0.17 with toluene. With these media the error increases with increase of water. With xylene the absolute error is greatest in absence of water, and least when the weights of water and glycerol are approximately equal. Since the b.p. of xylene is much higher than that of benzene or toluene some glycerin is undoubtedly carried over with the xylene vapour. The difference between water determinations made with benzene and those made with toluene did not exceed 0.1 per cent. and, since dehydration is more rapid with toluene, analytical comparisons were made only between toluene and xylene as distillation media. A series of moisture determinations made upon a number of soaps showed that with xylene the error per cent. of sample ranged from 0.05 for an "all soda" powdered laundry soap to

1.10 for a cold made coco-olive bar soap. When xylene is used instead of toluene, therefore, the error in the amount of water found may exceed 1 per cent. A greater error will be observed in the analysis of soaps made with fatty oils of the coconut oil type than with soaps made from longer chain fatty acids since the former type yields more glycerol. Toluene and xylene gave identical results with soaps free from glycerin. In order to obtain universal agreement in the determination of anhydrous soap by the distillation method, toluene should be used as the distillation medium, and the commercial variety known as 10° toluene is recommended. A. O. J.

Detection and Determination of Ethylene Glycol in Lubricating Oil. H. Lamprey, E. E. Sommer and A. D. Kiffer. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 526–527.)—

The examination of crank case oil for anti-freezing materials is sometimes necessary to detect leakage of engine-cooling solution into the crank case and to determine the effect of such substances upon sludge formation in the crank case. The most common anti freezing materials are methanol, ethanol and ethylene glycol, although glycerin, diethylene glycol, propylene glycol, isopropanol, sugars and inorganic salts are sometimes used. Specific methods for the identification of methanol and ethanol are known (Levin, Uhrig and Stehr, *Ind. Eng. Chem., Anal. Ed.*, 1939, **11**, 134), but no satisfactory procedure for the detection or determination of ethylene glycol has been described. The method now suggested is based upon isolation of the glycol and its identification by its physical properties. To 100 ml. of the oil, 100 ml. of xylene are added, and the mixture is distilled for 2 hours by the method described in "Standards in Petroleum Products and Lubricants," p. 345 (American Society for Testing Materials, Philadelphia, 1937). If calcium chloride or oxalic acid has been detected, 1 g. of sodium carbonate should be added to the mixture before distillation. The lower aqueous layer of the distillate is fractionated in a small column to separate the ethylene glycol, a micro equipment being used if the concentration of the glycol is low (Morton, "Laboratory Technique in Organic Chemistry," 1st Ed., pp. 50, 73, 74; New York, McGraw Hill Book Co., 1938). In the rare event of the occurrence of more than one anti-freezing agent, further fractionation will be necessary. The ethylene glycol is finally identified by determination of the refractive index and sp. gr. of the fraction boiling within the range 195° to 200° C., and tables for this purpose are given in the original paper. If no other high boiling anti-freezing agent is present in the distillate, the method may be made quantitative by measuring the volume and n_D (or volume and sp. gr.) of the aqueous portion of the distillate. The following anti-freezing agents do not interfere with the method:—calcium chloride, oxalic acid, glycerin, sugar and diethylene glycol. Later experiments have shown that ethylene glycol

may be identified in presence of much larger quantities of propylene glycol, diethylene glycol and methyl and ethyl cellosolves. The sensitivity of the method may be increased by increasing the amount of sample originally taken. A. O. J.

Identification of Aromatic Halogenonitro Compounds as Piperidyl Derivatives. M. K. Seikel. (*J. Amer. Chem. Soc.*, 1940, **62**, 750-753.)—Piperidine reacts more readily than other bases, such as ammonia, aromatic amines or morpholine, with compounds containing reactive halogen derivatives, while the reaction products crystallise readily and, in general, contain few by-products. Two general procedures have been developed: (A) in which the compound is heated for an hour under reflux with piperidine, and (B) in which an alcoholic solution is heated with piperidine for 15 minutes on the steam-bath. Procedure A yielded identifiable compounds in 79 per cent. of the 37 halogen nitro compounds studied, and procedure B in 74 per cent. Definite piperidyl derivatives were obtained with a large number of the compounds. In procedure A, 0.5 g. of the halogenonitro compound is treated with 1.5 ml. (1.0 ml. for bromo compounds) of piperidine, and the mixture is heated under reflux in an oil-bath for 1 hour, cooled, treated with water and filtered. If the product is an oil, it may be made to crystallise by chilling with ice. The paper gives the melting-points and other characteristic properties of the possible piperidyl derivatives, together with observations which will be of assistance in identifying the aromatic halogenonitro compounds studied.

Detection of Aromatics in Air. G. R. Gilbert and R. E. Tannich. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 433-436.)—Existing methods (listed by Yant, Pearce and Schrenk, *U.S. Bur. Mines, Rept. Investigations*, 1936, 3323) are unsuitable for small proportions of aromatic hydrocarbons, or necessitate the use of an interferometer or spectrograph, and refractometric methods involve the use of low temperatures, with the result that ice from atmospheric moisture blocks the apparatus. In the method suggested, blends of known volumes of methyl alcohol vapour with known volumes (*a*) of the sample and (*b*) of air containing known volumes of the aromatic hydrocarbons, are condensed by cooling, and the *n* of each condensate is determined in an Abbé refractometer at identical temperatures. Methyl alcohol is used because its *n* (while less than that of the aromatic hydrocarbons) is similar to that of water, and any errors due to variations in the humidity of the sample of air are thereby minimised. Several samples of the air are obtained in 5-litre, short-necked Pyrex flasks fitted with a rubber stopper containing a thermometer and 2 tubes with stop-cocks (diameter, 4 mm.), which reach just below the neck. If the flasks are evacuated (to about 2 mm. of mercury, abs. pressure) the sample may be taken by inserting the outer end of one of the tubes in

the air under investigation and opening the stop-cock. The temperature is then recorded. Exactly 0.5 ml. of methyl alcohol is introduced into the flask through one of the tubes and evaporated by warming the closed flask to approximately 38° C.; the contents of the flask are then condensed by cooling to -56.7° C., and the *n* of the condensate (which is removed in a long fine capillary pipette) is determined. Solutions containing 0.1, 0.5, 1.25, and 2.5 ml. of the liquid contaminant in 100 ml. of methyl alcohol are next prepared, and 0.5 ml. of each is measured into an evacuated flask, the vacuum being then broken by admission of air (free from hydrocarbons). This procedure (heating and cooling) is repeated, and the values of *n* obtained are plotted against the corresponding volumes of contaminant originally present in the 0.5 ml. of solution, so as to produce a calibration curve. A portion of the liquid responsible for the contamination is distilled (Engler), and its aromatic content and density at 15.6° C. before and after extraction with 4 volumes of 99.5 per cent. sulphuric acid (*d* and *d'*, respectively) are determined. Then *d'*, the density (at 15.6° C.) of aromatic hydrocarbons present in the liquid contaminant = $[d - (1 - x)d']/x$, where *x* is the aromatic content of the liquid sample (expressed as a fraction, in terms of vol.). Also the aromatic content (in p.p.m. of vapour by vol.) is given by $9120cxd'(460 + t)760/pm$, where *c* is the volume of total hydrocarbons (expressed in ml. of liquid) present in the sample of air (as read from the calibration curve); *m* the mol. wt. of the aromatic hydrocarbons; *t* the temperature (in °F.) and *p* the abs. pressure (in mm. of mercury) of the contaminated air. The derivation of the above equations and worked examples are set out in the original paper. For concentrations of aromatic hydrocarbons of the orders of 100 and 400 p.p.m., the errors are approximately 15 and 80 p.p.m., respectively. J. G.

Separation and Determination of 2,4-Diaminodiphenylamine. I. S. Shupe. (*J. Assoc. Off. Agr. Chem.*, 1940, **23**, 719-721.)—This constituent of certain hair dyes is chemically similar to *p*- and *m*-phenylenediamines. The free base is said to be more stable in air than its salts, but aqueous solution of base and salts are rapidly oxidised to form brown to purple solutions. When purified by sublimation *in vacuo* it has m.p. 130° to 131° C. The method for its determination is based upon its solubility in carbon tetrachloride, in which most of the diamines are insoluble. The aqueous solution (25 ml.) is made alkaline with excess of sodium bicarbonate, 0.1 g. of sodium sulphite is added to inhibit oxidation, and the liquid is extracted immediately with five 20-ml. portions of carbon tetrachloride. The extracts are washed with two 20-ml. portions of water, and the washing water is extracted with another portion of the solvent. The combined carbon tetrachloride extracts are filtered through cotton-wool, evaporated on a steam-bath and dried in a desiccator.

The residue may be dried at 100° C. for 30 minutes without appreciable decomposition, but this yields a discoloured product. If aminophenols are present, the carbon tetrachloride extract is given a preliminary washing with 25 per cent. sodium hydroxide solution. It is important that extraction with carbon tetrachloride should be made immediately after rendering the solution alkaline, because the base rapidly becomes crystalline and difficult to extract. Although the diamino-diphenylamine is almost insoluble in petroleum spirit whereas 4-aminophenylamine is soluble, attempts to separate these compounds by means of this solvent were not completely successful, apparently because the diamino-diphenylamine crystallises out and carries with it some of the 4-aminodiphenylamine. The residual crystalline material, dissolved in hydrochloric acid and reprecipitated with sodium bicarbonate gave an additional yield of the 4-amino compound when extracted again with petroleum spirit. Carbon tetrachloride extracts both of these compounds from alkaline solutions.

A. O. J.

Determination of Thioglycollates in Depilatories. E. M. Hoshall. (*J. Assoc. Off. Agr. Chem.*, 1940, **23**, 727-734.)—Of the organic sulphur compounds (including calcium thioglycollate, calcium dithioglycollate, calcium thiolactate, the calcium salt of pseudo-thiohydantoin and the aliphatic mercaptans) that occur in modern depilatory preparations, calcium thioglycollate appears to be used most frequently. The following method serves for its determination:—If the sample is a non-oily paste, 3 to 5 g. are washed with warm water through a filter into an iodine flask. If the sample is an oil-in-water emulsion, a portion (4 to 10 g.) is shaken with 100 ml. of water until no lumps remain, and the mixture is diluted to 200 ml. and filtered, the first 20 ml. of the filtrate being rejected. The filtrate (100 ml.) is then transferred to an iodine flask. Depilatories rarely occur as water-in-oil emulsions, but probably the oil phase could be removed by extraction with chloroform and the residue treated by either of the above-mentioned methods. The clear liquid is slightly acidified to litmus with 0.1 *N* hydrochloric acid, the flask is quickly closed and shaken, and the stopper is sealed with 0.1 *N* iodine solution. The stopper is gently opened, the iodine is allowed to run in and titration is continued with 0.1 *N* iodine solution in presence of starch. Each ml. of iodine solution is equivalent to 0.018423 g. of calcium thioglycollate with 3 molecules of water of crystallisation. Satisfactory results were obtained by this method with depilatory pastes containing 4 to 6 per cent. of calcium thioglycollate made in imitation of commercial preparations. Calcium thioglycollate reacts quantitatively with one-half molecule of iodine in neutral solution. The reaction has not been investigated, but it appears to be similar to the oxidation of cysteine to cystine. In order to detect calcium thioglycollate in depilatories,

the following tests may be applied to the clear filtrate obtained from an aqueous suspension:—With 5 per cent. copper sulphate solution a violet-brown precipitate, soluble in dilute acids and in ammonia, is formed. With a few drops of 0.1 *N* ferric chloride solution, an evanescent indigo blue colour appears in neutral or slightly acid solutions, and on further addition of the reagent the colour changes to green, then to purple-brown and finally to yellow with formation of a precipitate. The test used by Hamence (*ANALYST*, 1940, **65**, 152) to distinguish molybdenum from tungsten with thioglycollic acid may be adapted to the detection of thioglycollates. The dilute solution of the thioglycollate is treated with 1 ml. of 3 per cent. ammonium molybdate solution and 0.5 ml. of hydrochloric acid, and the mixture is diluted to 50 ml. A greenish yellow to greenish yellow-brown colour forms and becomes green, with separation of a precipitate, on addition of ammonia. The usual components of thioglycollate depilatory preparations do not interfere with these tests.

A. O. J.

Separation of *p*-Aminophenol, *p*-Methylaminophenol and *o*-Aminophenol. I. S. Shupe. (*J. Assoc. Off. Agr. Chem.*, 1940, **23**, 721-727.)—These aminophenols are commonly used in certain classes of hair dyes. To determine *p*-methylaminophenol in presence of *p*-aminophenol and *o*-aminophenol a portion not exceeding 400 mg. of the mixture is treated with 5 ml. of 10 per cent. hydrochloric acid and sufficient water to make 15 ml. The mixture is cooled to 15° C., mixed with 5 ml. of 10 per cent. sodium nitrite solution and extracted with five 20-ml. portions of ether free from alcohol and peroxides. The first four extracts are washed with 15 ml. of water, then with 10 ml. of saturated sodium bicarbonate solution and finally with 15 ml. of water, and the three washing solutions are shaken individually with the fifth ethereal extract. The combined ethereal extracts are filtered through cotton-wool, the greater part of the solvent is removed on the water-bath, and the last portion is allowed to evaporate spontaneously. The residual *p*-nitrosomethylaminophenol is dried in a desiccator and weighed, and the weight is multiplied by 0.809 to obtain the corresponding weight of *p*-methylaminophenol. To determine *p*-aminophenol in presence of *o*-aminophenol and *p*-methylaminophenol, the mixture (not exceeding 200 mg.) is treated in a stoppered vessel with 5 ml. of 10 per cent. sodium acetate solution, 50 mg. of powdered sodium bisulphite, and finally with 0.5 ml. of benzaldehyde for each 50 mg. of sample. The mixture is heated for 5 minutes on the steam-bath and, when cold, is shaken thoroughly with 25 ml. of petroleum spirit and allowed to stand at about 4° C. for 3 hours. The aqueous and petroleum spirit layers are filtered through a Gooch crucible, and the flask and crucible are washed with petroleum spirit. The remaining precipitate is transferred and washed with

cold water, and the crucible is dried at 100° C. The weight of the benzaldehyde derivative, multiplied by 0.553, gives the corresponding amount of *p*-aminophenol. When recrystallised from alcohol and water, the benzylidene-*p*-aminophenol melts at 185° to 186° C. To separate *o*-aminophenol from *p*-aminophenol and *p*-methylaminophenol, the dry mixture (not exceeding 200 mg.) is dissolved in 2 ml. of glacial acetic acid, 8 ml. of chloroform are added and the mixture is stirred to induce crystallisation and left for 30 minutes. The crystalline precipitate is collected in a Gooch crucible, washed with chloroform, and dried in a desiccator. The weight of *o*-aminophenol acetate, multiplied by 0.645, gives the corresponding amount of *o*-aminophenol. Under the conditions stated, about 2.5 mg. of *o*-aminophenol remain in solution, and this amount may be added as a correction to the quantity found. Experiments with mixtures of known composition showed that the methods yielded results within 5 per cent. of theory. A. O. J.

Colorimetric Estimation of Indigo.

J. Lotichius. (*J. Soc. Dyers and Colourists*, 1940, **56**, 433-434.)—By a modification of Lotichius' method for estimating indigo in indigo vats (*cf. J. Soc. Dyers and Colourists*, 1939, **55**, 87), the indigo-content of commercial indigo may be estimated quantitatively as follows:—To a known weight (approx. 0.25 g.) of indigo in a 250-ml. flask, are added 100 ml. of water, 10 ml. of sodium hydroxide solution (sp.gr. 1.425), 7 ml. of sodium protoalbinat solution (*cf. Lotichius, loc. cit.*), and about 2 g. of sodium hydrosulphite powder. After standing stoppered, at a temperature not exceeding 30° C. for some hours (preferably overnight) until reduction is complete, a few ml. of 30 per cent. hydrogen peroxide solution are added. The blue colloidal indigo solution produced is made up to 250 ml.. Five ml. are diluted to 1 litre and the colour is matched with that of a standard solution of Sirius Blue B and Sirius Green 2B (*cf. Lotichius, loc. cit.*). Saponifiable organic constituents, which cause turbidity, must first be removed. By this method indigo may also be estimated in indigo printing paste in about 30 minutes. A sample of printing paste was found to contain 4.1 per cent. of indigo, the calculated proportion being 4.2 per cent. E. B. D.

Colorimetric Estimation of Sodium Hydrosulphite and the Hydrosulphite-content of Indigo Vats. J. Lotichius.

(*J. Soc. Dyers and Colourists*, 1940, **56**, 434-435.)—The method is based on that for the colorimetric estimation of indigo (see preceding abstract). To approximately 1 g. of indigo (if in the form of a powder it should be rubbed into a paste with alkaline water) are added a known weight (about 0.5 g.) of sodium hydrosulphite ($\text{Na}_2\text{S}_2\text{O}_4$) and air-free water containing 3 ml. of sodium hydroxide solution (sp.gr. 1.425) to make up 100 ml. After displacement of the remaining air by coal gas the flask is stoppered and shaken for 30 minutes

at room temperature (over-reduction occurs at higher temperatures), and the excess of indigo is allowed to settle for a further 30 minutes. Ten ml. of the supernatant liquid are then pipetted into 10 ml. of sodium protoalbinat solution (*cf. Lotichius, loc. cit.*) in another graduated flask, the tip of the pipette being kept below the surface of the protoalbinat solution, and the mixture is oxidised with 1 ml. of 30 per cent. hydrogen peroxide solution, and diluted a hundred-fold, and colorimetrically tested. The factor for calculating indigo into sodium hydrosulphite is 0.66. For the estimation of the hydrosulphite content of indigo-alkali-hydrosulphite vats, the indigo, I_1 (g. per litre) is estimated colorimetrically. The sodium hydrosulphite is then oxidised with excess of indigo, and the reduced indigo I_2 (g. per litre) is estimated colorimetrically as described above, with some modifications in detail. From ($I_2 - I_1$) the amount of sodium hydrosulphite may be calculated. E. B. D.

Estimation of Indigo on the Fibre. J.

Lotichius. (*J. Soc. Dyers and Colourists*, 1940, **56**, 435.)—A piece of cotton, 5 cm. square, dyed in indigo, is treated with 20 ml. of cold conc. sulphuric acid (sp.gr. 1.84) in a porcelain dish for 30 to 45 minutes. In one movement the resulting dark green solution of indigo sulphate is tipped into about 750 ml. of water, and the dish is rinsed in this. The indigo in the colloidal solution, which can be kept for some time, may be estimated after dilution to 1 litre. The standard solution of Sirius dyes (see abstract above) may be used for comparison (although this is not theoretically permissible because absorption coefficients may be different) because experimental results agree with those compared with standard indigo sulphate solution. The latter solution, which, unlike the dye solution, must be freshly prepared for each series of estimations, contains 10 mg. of chemically pure indigo, dissolved (as stated above) in 10 ml. of sulphuric acid and diluted to 1 litre. E. B. D.

Inorganic

Qualitative Reactions of Salicylaldoxime Derivatives with Inorganic Ions. J. F. Flagg and N. H. Furman.

(*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 529-531.)—A comparative study was made of salicylaldoxime and its 5-chloro, 3,5-dibromo, and 5-nitro derivatives in their reactions with 72 inorganic ions in neutral, slightly acid, and ammoniacal solution; the sensitiveness of the tests for copper, nickel, bismuth and lead was determined. It was found that the chloro and nitro compounds do not differ to a marked extent in behaviour from salicylaldoxime; the dibromo compound is unsuitable as a reagent, owing to its sparing solubility. Salicylaldoxime and its nitro derivative are equally sensitive in tests for copper, lead and nickel; the chloro compound is a less sensitive

reagent. The unsubstituted oxime answers best for the detection of bismuth. No definite reactions were obtained with the alkalis, alkaline earth metals and most of the metals of the third, fourth, fifth, sixth, and eighth groups of the Periodic System. W. R. S.

Determination of Ruthenium with Thio-nalide. W. J. Rogers, F. E. Beamish and D. S. Russell. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 561-563.)—Ruthenium is quantitatively precipitated from 0.2 to 0.5 N hydrochloric acid solution by thionalide (thioglycollic β -aminonaphthalide). The ruthenium solution is heated almost to boiling, treated with excess of an alcoholic solution of the reagent, and boiled until the precipitate coagulates (one atom of ruthenium requires two molecules of thionalide). The precipitate is collected, washed with hot water, charred in a tared porcelain crucible, ignited, reduced in hydrogen in the usual manner, and weighed as metal; no decrepitation or deflagration takes place. Sodium chloride does not interfere, but low results are obtained in solutions containing nitric acid. In the distillation method for the separation of ruthenium from other metals, the ruthenium tetroxide may be conveniently absorbed in 3 per cent. hydrogen peroxide in two ice-cooled receivers placed in series. The distillate is transferred to a beaker, treated with the requisite quantity of hydrochloric acid, boiled until oxygen is no longer evolved, and precipitated with thionalide. The hydrogen peroxide used should be pure and free from acetanilide, otherwise the ruthenium precipitate does not coagulate well and low results are obtained. W. R. S.

Determination of Rhenium in Molybdenite. C. F. Hiskey and V. W. Meloche. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 503-506.)—The finely powdered mineral (4 g.) is attacked with 20 ml. of strong nitric acid in a 250-ml. conical flask, 5 ml. of fuming nitric acid being added after violent action is over. When the attack has subsided, the contents of the flask are heated just below boiling-point and, after red fumes cease to be evolved, 50 ml. of strong hydrochloric acid are cautiously added. The liquid is heated until chlorine is no longer evolved, fresh hydrochloric acid being added as required (125 to 150 ml. in all), and eventually concentrated to not less than 25 ml. The solution is cautiously treated with strong sulphuric acid (75 ml.), and the liquid is transferred to a distilling-flask, and distilled for 2½ hours at 260° to 270° C. in a current of steam (2 parts) and carbon dioxide or air (1 part) until 250 ml. of distillate have been collected in the ice-cooled receiver. The distillate is treated with a slight excess of a potassium bromide solution of bromine to oxidise any sulphur dioxide and then with 100 ml. of strong hydrochloric acid and thiocyanate and stannous chloride solutions (*ANALYST*, 1939, **64**, 62). Standards containing 10, 50 and 100 γ of rhenium are also treated in the same manner. The solutions

are set aside for about 30 minutes, by which time the colour due to a little molybdenum carried over in the distillation will have faded, and the colours are then matched in 100-ml. Nessler tubes. The comparison is repeated after about 20 minutes, as a check on the complete decolorisation of the molybdenum complex. Selenium interferes in the process, selenium dioxide being volatile and reduced by the stannous chloride. It may be removed by precipitation with 1 g. of sodium sulphite from the chloride solution of the mineral prior to the addition of the strong sulphuric acid; when the selenium has settled, the solution is filtered, and the filtrate is concentrated to 25 ml. and treated with 75 ml. of strong sulphuric acid, etc., as described above. The rhenium found by this method in a number of molybdenite samples (chiefly American) ranged from *nil* to 40 p.p.m. W. R. S.

Determination of Sulphide Sulphur in Presence of other Sulphur Compounds.

J. B. Lewis. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 535.)—The determination of sulphide sulphur in solutions containing other sulphur compounds by precipitation as lead sulphide in presence of ammonium acetate (*cf.* Balde-schwieler, *Ind. Eng. Chem., Anal. Ed.*, 1934, **6**, 402) gives correct results if the solution is treated with sufficient sodium hydroxide to provide a minimum alkalinity of 2 per cent. This alkalinity is necessary to prevent precipitation of lead sulphite, which is not prevented by the presence of ammonium acetate, even in acetic acid solution. W. R. S.

Amperometric Titration of Fluoride with Thorium Nitrate. A. Langer. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 511-514.)—

Minute amounts of fluorine in the form of sodium fluoride can be titrated with 0.01 N thorium nitrate solution by the use of the dropping mercury cathode for the graphical determination of the end-point. The solution (50 ml.) should contain up to 0.2 mg. of fluorine and potassium chloride or nitrate (0.1 M) as the conducting salt. In smaller volumes of solution, quantities of 0.005 mg. of fluorine may be titrated with 0.001 N thorium solution. The error is of the order of one per cent. Lanthanum nitrate may be used instead of the thorium salt, but titration with calcium salt solution is unsatisfactory. W. R. S.

Detection and Elimination of Phosphate in Qualitative Analysis by means of Zirconium Salts. F. K. Pittman. (*Ind. Eng. Chem., Anal. Ed.*, 1940, **12**, 514-515.)—

A revised process has been based on Curtman's zirconyl chloride method (*Chem. News*, 1924, **129**, 299), which depends upon the fact that zirconium (with hafnium) forms a phosphate which is insoluble in strongly acid solution. The filtrate from the hydrogen sulphide precipitation (copper and tin group) is boiled until all hydrogen sulphide has been expelled, and then made up to about 100 ml., cooled to room temperature, neutralised with ammonium hydroxide, and made acid with 5 ml.

of 6 *N* nitric acid. To this acid solution 35 ml. of 0.015 *M* zirconyl chloride solution are added, a few drops at a time, with vigorous stirring throughout the addition. The precipitate is left to settle for 1 minute, decanted on to a very porous filter paper, and washed several times without the aid of suction, with small amounts of a 5 per cent. solution of ammonium nitrate, the washings being added to the filtrate. The precipitate is rejected. The solution will then contain less than 1 mg. of phosphate, and the last traces can be removed by adding 10 ml. of 0.05 *M* zirconyl chloride solution, heating the mixture just below its boiling point for 2 minutes, leaving it for 5 minutes and filtering. The filtrate is then treated as if phosphate had not been present. The small excess of zirconium will follow the iron and will not interfere with the usual ferrocyanide or thiocyanate tests for that metal.

In testing for phosphate a small portion of the filtrate from the copper and tin group is treated as directed for the removal of phosphate, except that the zirconyl chloride solution should be 0.005 *M* rather than 0.015 *M*. If very small amounts of phosphate are to be tested for, the main portion of the solution should be evaporated to about 15 ml. before the test portion is taken. In this way less than 1 mg. of phosphate can readily be detected. Iron, chromium, manganese, cobalt and nickel can readily be detected in the filtrate from the phosphate, if present to the amount of 1 mg. in the original solution; for aluminium and zinc, 2 and 5 mg., respectively, are required. There is no serious loss of alkaline earth metals. Except with arsenate, oxalate and ferrocyanide, no precipitate is formed when zirconyl chloride is added to a solution of any of the other common anions. Arsenate gives a white precipitate and ferrocyanide a yellow precipitate. Oxalate gives a transient white precipitate in weakly acid solutions.

Adsorption of Malachite Green by Clays and Allied Materials. V. L. Bosazza. (*Nature*, 1940, 146, 334.)—Certain minerals may be identified by the colours of the adsorption complexes produced by staining with a 0.05 per cent. solution of malachite green for 10 minutes and drying. Examples are:—kaolinite, deep blue-green; anauxite, light to deep blue-green; montmorillonite and beidelite, marine blue; pyrophyllite and talc, blue-green; margarite, no pronounced colour; quartz (which is commonly associated with these minerals), no colour. Pleochroism is pronounced in kaolinite, less marked but variable with anauxite, and does not occur in the other minerals mentioned. Washing with water, acid or alkali fails to remove the colour from kaolinite, although acid changes it to bright red. The extent of the adsorption varies according to the mineral concerned, and in some instances (*e.g.* anauxite) with its composition. Thus, under the above conditions, white kaolin (rich in kaolinite) adsorbs 85 per cent. of the dye in 1 minute and 93 per cent.

in periods of 60 minutes and over, whilst bentonitic clays and fuller's earth adsorb the dye from much stronger solutions. J. G.

Physical Methods, Apparatus, etc.

Cross Sections of Pulp Fibres. J. H. Graff, M. A. Schlosser and E. K. Nihlen. (*Paper Trade J.*, 1940, 111, T.A.P.P.I. Sect., 105–110.)—The possibility of identifying fibres used in paper manufacture from certain characteristics of their cross-sections was investigated. A small bundle of the fibres in question is desiccated and mounted in paraffin wax; and the cross-sections (diameter 5 microns) are cut and magnified (100 diameters), and drawings of them are made. The average "roundness factor" is then given (as a percentage) by $100 \times$ gross area of each fibre/area of a circle whose diameter is the width of the fibre; the larger this factor the "rounder" is the fibre. The gross areas of the fibres and the areas of the lumens were determined by means of a planimeter. Other data which may be calculated are the average number of fibres per unit area, area of the lumen and total area of air-space between the fibres when these are assumed to be "stacked" in a pile in the same direction so that their cross-sections are all in the same plane. Results for 10 different types of pulp fibres are tabulated, and statistical treatment of them shows that the values obtained are typical of the fibre concerned, and may be used as an aid to identification. Good agreement was obtained in duplicate determinations. Since the fibre-width is correlated with at least 6 of the other factors, it may prove possible to use it to calculate some of these, but more measurements are required before the conversion factors can be ascertained. The differences between the above values may also provide useful information about the treatment undergone by the fibre during manufacture into paper. J. G.

Accelerated Spray Test for Determining the Relative Atmospheric Corrodibility of Ferrous Materials. T. Swinden and W. W. Stevenson. (*Advance Copy Paper*, No. 2, 1940, of the Corrosion Committee, Iron and Steel Inst., 1940.)—A method of automatic accelerated spray testing is described, in which the specimens, on an endless belt, complete a cycle of treatment, consisting in spraying, followed by drying in air and in a warm chamber. The cycle is completed in 7 minutes, and the specimens are subjected to 60 cycles per day. A standardised douching with water is given at the beginning of the second and subsequent days, and the daily spraying and drying treatment is continued for 20 days. Comparative results of long-period field tests and spray tests are given for 14 irons and steels of the low-alloy constructional type. Good agreement is shown in the orders of merit obtained in one-year field tests and in the 20-day spray tests, using a spray liquid consisting of *N*/100

sulphuric acid and *N*/100 sodium chloride. Less satisfactory agreement is shown in field tests and spray tests, using *N*/100 and *N*/20 sulphuric acid without sodium chloride. It is suggested that the results obtained with the

sulphuric acid and sodium chloride solution are of sufficient promise to encourage other workers to re-examine the spray method as the basis of a satisfactory short-time laboratory corrosion test.

Reviews

CONVERSION OF PETROLEUM. By A. N. SACHANEN, D.Sc. Pp. 413. New York: The Reinhold Publishing Corpn.; London: Chapman & Hall, Ltd. Price 36s. net.

The subject of this book, as the name suggests and as set out in the sub-title, is "Production of motor fuels by thermal and catalytic processes," that is, by cracking and recently developed methods such as hydrogenation and isomerisation with the aid of catalysts.

Dr. Sachanen is a member of the research and development division of the Socony Vacuum Oil Company, and is, as the book bears out, well qualified to write fully and authoritatively on petroleum conversion. The subject being so large, the author has wisely confined himself to the essentials, and has omitted reference to products, processes and plant and equipment generally used in other sections of the petroleum industry.

The book is divided into seven chapters, of which the first three, taking up more than half the book, discuss the fundamental factors and the thermal and catalytic reactions on which the design of plant and its operation depend. The subsequent chapters deal with the plant and equipment used and with the properties and chemical treatment of the new types of motor fuels produced, followed by a concise chapter on cracked products other than gasoline, which, although relatively of little use to-day, will in the future undoubtedly prove to be suitable for special purposes.

The book opens with an account of the paraffins, and the chapter gives an outline of the reactions of dehydrogenation, splitting decompositions, catalytic cracking, the so-called alkylation, isomerisation, and aromatic cyclisation. Data obtained by the many research workers in these fields are supplemented by those from actual operating plants. The olefines are treated in a similar way, polymerisation with and without catalysts, hydrogenation, isomerisation, and cyclisation being discussed. A description of the commercial methods in these processes is also given. The naphthenes, defined as "all saturated cyclic hydrocarbons, monocyclic and polycyclic, which have not the aromatic character," are discussed in relation to dealkylation, alkylation, dehydrogenation, ring rupture and isomerisation. Aromatic hydrocarbons are similarly treated, the reactions of a number of hydrocarbons particularly characteristic of coal-tar being included. Short summaries are given of each of these sections, which are very helpful. Only brief reference is made to the oxygen, sulphur and nitrogen compounds, these being at present of relatively little importance and interest; there is a very full bibliography at the end of this important chapter. The references are mainly to work carried out during the last ten years.

In the second chapter the author discusses the various factors that influence the progress of the reactions, the effects of temperature and pressure on the main and secondary reactions, the formation of coke and the effects of recycling. Residue- and non-residue-cracking, viscosity breaking, and reforming are also dealt with, the data quoted in support being critically examined.

Hydrogenation forms the subject of a separate chapter which covers the whole subject completely, including applications of the process, such as the conversion of low-grade lubricating stocks and Diesel oils into high-grade products,

as well as the conversion of heavy asphaltic crudes and refinery residues into gasoline and gas oils. Much information is given on the subject of the catalysts used, but little concerning the actual plant, few details being as yet available for publication.

The equipment used for the cracking operations is well described and illustrated by reference to well-known types of plant in successful operation. Processes which have been of importance in the early development of cracking are not described, being mainly of historical value.

The development of cracking has given rise to problems of chemical refining much more complex than those which obtained in the days of only straight-run products; they take up two full chapters. The determination of the relative proportions of the various types of hydrocarbons in a complex mixture presents a problem that is at present far from being solved; data given exemplify this. It is only to specially fractionated cuts of very narrow boiling-point range that certain methods can be successfully applied. The complex questions of gum formation, inhibitors, octane numbers and susceptibility to lead tetra-ethyl are adequately discussed.

The book contains a mass of detail well selected, set out, summarised and often criticised. It is valuable, therefore, not only as a work of reference, but as one that can be read with pleasure. It should prove of great use, not only to the average petroleum chemist and works operator, but also to those who have specialised in one or more of the many aspects of this great modern industry.

J. KEWLEY.

TEMPLES OF ARMANT. By SIR ROBERT MOND, LL.D., D.C.L., F.R.S., and OLIVER H. MYERS. 2 vols. Pp. xii + 223; 107 plates. London: The Egypt Exploration Society. 1940. Price £3 3s.

Two reports on Armant by the same authors have already been published, namely, "*The Bucheum*" and "*Cemeteries of Armant I*" (cf. ANALYST, 1935, 60, 65; 1938, 63, 690), Sir Robert Mond financing the work and Mr. Myers, with the help of various assistants, undertaking it.

Armant is a town in Upper Egypt, about twelve miles south of Luxor. Anciently it was of considerable importance, though now it is only a small place, noted chiefly for its sugar factory. During the Christian period in Egypt Armant was the seat of a bishopric, and the church (of which only a few ruined columns remain) was one of the largest and finest in Egypt.

The book consists of a preface and twelve chapters, in the writing of which the chief author has had the help of five collaborators. Mr. Myers regrets that there are fewer analyses and expert reports than in his previous books, though these are by no means lacking, and the last chapter consists of technical reports by sixteen different experts, who respectively deal with pottery; glass; glass-making materials; metals; metal-smelting products; geology; mineralogy; zoology; wood; osteology. There is also a report, by Mr. Myers himself, on weights.

As the reviewer is a chemist and the review is for THE ANALYST, the chemical and allied aspects of the report will be especially considered. A description is given of the methods employed for cleaning and preserving carved and painted limestone (both *in situ* and in the laboratory), and also of the manner in which a bronze lamp, too fragile for ordinary methods, was dealt with, and Mr. Harden recounts how he treated decayed glass mosaics.

Mr. Myers finds a difficulty in explaining the fact that certain pottery, although overfired and cracked in the kiln, was still black in the centre, like ordinary underfired ware. I would suggest that probably there was a large proportion of alkali present, which became fused round the charred organic matter and so prevented its complete combustion.

A green corrosion product on a silver-copper alloy is called by one expert "verdigris," which he defines as a "mixture of basic copper carbonate and acetate," but that there was any copper acetate in this particular instance was not proved and seems unlikely, the expert apparently having been misled by the frequent, but wrong, use of the word "verdigris" to describe the similar-looking basic copper carbonate, or sometimes even copper oxychloride.

No book is, or can be, perfect, and a few minor points may be criticised. Thus, the German word *Fundplatz* occurs more than fifty times, and one of the experts uses its English equivalent "find-place," although the words "place"; "position"; "situation" and "locality" are available. The use of the French word *sondage* in place of "sounding" or "boring" is equally unnecessary and objectionable. Several Arabic words, for example, *gisr*, *maktub* and *saggia*, are used without any explanation of their meaning. Also, why employ the word *insulae*? On p. 23 the text reads "to let the plaster," a word or words apparently having inadvertently been dropped. Mr. Myers has invented a new verb, namely, "to corpus" with "corpusing," which is both unnecessary and ugly. Also he says that certain metal objects were "dissolved" by the treatment used, whereas "disintegrated" is meant.

These criticisms, however, are of small matters, and the chief author is to be congratulated on an excellent report that compels admiration for the detail with which the work was done and the manner in which it is recorded, for the orderly arrangement of the vast amount of material and for the way in which, by submitting what often must have seemed most unpromising specimens to experts, he has caused to be extracted from them useful and profitable results. It is manifest that an immense amount of hard work, which would have daunted many archaeologists, has been done both in the field and in the preparation of the report. The historical summary is interesting as well as valuable.

The book is well printed and bound, and three of the plates are in colour. The inclusion of a plate showing sixteen blocks of carved limestone from Armant, now in the Grenoble Museum, is a happy inspiration.

A. LUCAS.

A TEXT BOOK OF BACTERIOLOGY. By R. W. FAIRBROTHER, D.Sc., M.D. 3rd Edition. Pp. 451 + x, 6 plates. London: William Heineman (Medical Books), Ltd. 1940. Price 17s. 6d.

This book is in the main an outline of the medical aspects of bacteriology. In writing it the author's aim has been to restrict himself to branches of bacteriology of medical importance and to the application of bacteriological methods to the prevention, diagnosis and treatment of disease, while avoiding excessive technical detail, which, although required in the laboratory, is not necessary for the average medical man. There is, however, a concise account of general bacteriological technique and of general bacteriology, amply sufficient in detail for all students of bacteriology. The book is divided into three parts under the following headings:—I General Bacteriology, II Systematic Bacteriology, and III General Technique.

Part I contains twelve chapters, beginning with a historical survey and then discussing the biology, morphology, cultivation, multiplication and destruction of bacteria, and their classification. Other chapters deal with antigens, hypersensitiveness, idiosyncrasy, and allergy, and the relation of bacteriology to medicine. In Part II (22 chapters) there is a systematic description of the different pathogenic organisms, concluding with chapters on filterable viruses, the bacteriophage and the bacteria of water, milk and shell fish. Part III comprises three chapters, dealing respectively with the microscope and staining methods, the preparation of culture media, and serological technique.

The chapters in Part II are subdivided into sections and paragraphs dealing with the classification of the organism, its habitat, morphology and

staining reactions, cultural reactions, resistance, biochemical activity, serology, toxin production, pathogenicity, diagnosis, specific therapy and chemotherapy. This is often followed by a concise account of the disease or diseases which the organism causes, with bacteriological diagnosis, prophylaxis and therapy. In this way, under one or other of the headings, all the important points concerning a micro-organism and its related disease are dealt with, if only briefly.

The book is well up-to-date; for example, one finds reference to the relationship between virulence of strains of *Staphylococci* and production of coagulase, and to the modern typing of meningococci and Flexner dysentery bacilli. There is also a good description of Wilson and Blair's medium for the isolation of *B. typhosum*; obviously a book of this size cannot contain everything, but a description of the valuable method of E. R. Jones, using tetrathionate broth and brilliant-green-eosin agar, would be well worth including.

The book is very well written and arranged, the subject matter is well balanced and clearly expressed, the printing is in good bold type, and the paper and binding are excellent. The volume can be strongly recommended; it has been justly described by other reviewers as the best of its kind.

D. R. WOOD

CALCULATIONS OF QUANTITATIVE ANALYSIS. By CARL ENGELDER, Ph.D. New York: John Wiley & Sons, Inc.; London: Chapman & Hall, Ltd. 1939. Pp. viii + 174. Price 12s.

This book opens with a discussion of general matters such as errors, precision and accuracy. It deals very fully with the use of logarithms, numerous worked examples being given. Reagents and methods of expressing the strength of a solution also receive attention. Considerable stress is laid on the laws of mass action and combining weights. Part 2, under the headings of Precipitation, Neutralisation and Oxidation, is devoted to the calculations involved in volumetric analysis. In Part 3 the theory of gravimetric analysis is discussed, and the last section is devoted to the calculations required in systematic quantitative analysis. Numerous useful tables, including logarithms, solubilities, densities, and ionisation constants, are given. A satisfactory index is also given.

Each section contains a very clear treatment of the appropriate underlying theory. Thus in the section on neutralisation we find a very full discussion of ionisation, pH value and titration curves. Oxidation-reduction processes are treated from the point of view of electron transfer, and in gravimetric analysis the concept of solubility-product is used. Attached to each section is a set of excellent numerical examples, making in all a total of three hundred.

Since this book is intended as a theoretical adjunct to systematic laboratory work, no practical directions are given. Used as suggested by the author, it will greatly enhance the value of practical work and convert analytical procedure, which is too often regarded as a mystic ritual, into an intelligent occupation. The treatment is modern and has therefore a definite physical bias. It is safe to predict that one who has faithfully worked through the suggested course will have acquired a satisfactory foundation for advanced work.

HAROLD TOMS

THE ANALYST

THE JOURNAL OF THE

Society of Public Analysts and other Analytical Chemists

A MONTHLY JOURNAL DEVOTED TO THE ADVANCEMENT
OF ANALYTICAL CHEMISTRY

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ERRATA:

VOL. 65, 1940:

- p. 27. Delete last line of "Official Appointments," *i.e.* Lincs. County, etc.
- p. 411. In list of Official Appointments for "Westmorland County Council, Kendal Borough, W. H. Roberts" read "W. H. Roberts as Public Analyst for that part of the County of Westmoreland within the boundaries of the Borough of Kendal."
- p. 519. Line 12 of Abstract on "Estimation of Phosphorus." For "about 0.4 mg. of phosphorus" read "not more than 0.4 mg. of phosphorus."
- p. 530. 2nd column: 7th line from bottom. For "*J. Chem. Soc.*" read "*J. Amer. Chem. Soc.*"
- p. 596. Last line but two of the Note: for " $\pm 0^\circ \text{C.}$ " read " $\pm 0.1^\circ \text{C.}$ "

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