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THE ANALYST.

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

An Ordinary Meeting of the Society was held on Wednesday, May 4, 1921, in the Chemical Society's Rooms, Burlington House. The President, Mr. Alfred Smetham, was in the Chair.

Certificates were read for the first time in favour of Messrs. W. N. Stokoe, B.Sc., A.I.C., and J. F. F. Rowland. Certificates were read for the second time in favour of Messrs. William Ellard Woolcott and Thomas Henry Pope, B.Sc., F.I.C.

Messrs. Percy Nicholas Mould and Walter Joseph Wright, F.I.C., were elected Members of the Society.

The following papers were read: "Detection and Estimation of Illipé Nut Fat used as a Substitute for Cacao Butter," by Francis G. H. Tate, F.I.C., and John W. Pooley, B.Sc., A.I.C.; "Notes and Demonstration on Apparatus for determining Hydrogen Ion Concentration," by G. W. Monier-Williams, O.B.E., M.C., M.A., F.I.C.; "A Note on the 'Oil' of Oats," by Ernest Paul, B.Sc., A.I.C.; and "Estimation of Potassium in presence of Sodium, Magnesium, Sulphates, and Phosphates," by H. Atkinson, B.A., A.I.C.

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DETECTION AND ESTIMATION OF ILLIPÉ BUTTER USED AS SUBSTITUTE FOR CACAO BUTTER.

By FRANCIS G. H. TATE, F.I.C., and JOHN W. POOLEY, B.Sc., A.I.C.

(*Read at the Meeting, May* 4, 1921.)

The identification and approximate estimation of certain of the fats used in the cheaper forms of chocolate present little difficulty, and these fats are easily identified by generally accepted methods and data provided in the leading textbooks.

The fat known commercially as illipé butter, however, appears not to have received that consideration which its extensive use and close similarity to cacao butter demand. In the state in which it is usually offered for sale, this fat can be distinguished from cacao butter by the absence of the typical smell of the latter and by its green colour.

It has, however, generally been considered impossible to detect with certainty the presence of illipé butter when mixed, even in appreciable quantity, with cacao

butter, and it became of great importance to investigate the physical and chemical features of these two fats. For this purpose a number of samples were obtained from widely different sources.

The following properties were considered: Specific gravity at 60°/15·5° C., and at 99°/15·5° C., coefficient of expansion, viscosity, melting-point, iodine value (Wijs), refraction $[n]_D^{40}$ (Zeiss), saponification equivalent, mean molecular weight of fatty acids, melting-point of free fatty acids, and "titre" test on fatty acids.

The methods throughout were adopted having in view the fact that the data obtained would be used under the ordinary routine conditions of a laboratory, and that the refined methods sometimes adopted in research would not always be possible.

It was obvious from the figures obtained that the coefficients of expansion were so close as not to form a good basis for comparison. The viscosity was determined at 60° C. in Redwood's instrument. A few readings were taken at 95° C. to establish the "viscosity temperature slopes," but the latter proving to be practically identical for the two fats, this investigation was not pursued.

Melting-Point.—The following method was adopted: A porcelain crucible containing mercury was placed on a wire gauze over a very small flame, the thermometer being suspended therein with the bulb fully immersed. A shaving of fat was placed upon the surface of the mercury, and the temperature raised gradually, at the rate of not more than one degree per minute. The reading was taken when the first signs of transparency were observed on the shaving. Before being finally adopted this method was tested by the work of three different observers, two of whom had no knowledge of the object of the investigation. The results confirmed our opinion that the personal factor could be ignored.

The Saponification Equivalent was determined in the usual manner. It was found, however, that the value of cacao butter and illipé butter covered almost identical ranges, but under the ordinary working conditions of the laboratory consistent results could not be obtained. It was therefore decided to reserve this constant for further investigation if considered desirable.

Fatty Acids.—The fatty acids were prepared by heating 10 grms. of the fat in a porcelain basin with 50 c.c. of alcoholic potassium hydroxide solution (40 grms. per litre) until the resulting soap became pasty. It was then dissolved in hot water, and the solution boiled and acidified with dilute sulphuric acid. The fatty acids were washed by boiling with water until free from sulphuric acid, and then dried and submitted to the following tests:

Melting-Point.—The method adopted for the fats was used.

Mean Molecular Weight.—This was estimated by titration against $\frac{N}{10}$ alkali, phenolphthalëin being used as indicator. As might be anticipated, the objection inherent in the saponification test applied equally to this test, and it was accordingly rejected. A summary of the observations is appended:

Cacao Butter.—(12 observations): Minimum reading, 272·6; maximum reading, 296·0; average reading, 280·5; and mean reading, 284·3.

Illipé Butter.—(8 observations): Minimum reading, 269·4; maximum reading 295·7; average reading, 277·6; and mean reading, 282·5.

TABLE A.—CACAO BUTTER.

Specific Gravity at 60° C.	Specific Gravity at 99° C.	Viscosity at 60° C.	Melting-point, ° C.	Iodine Value.	Melting-point of Fatty Acids, ° C.	Refraction $[n]_D^{40}$.
A. 0·8829	0·8581	101·3	30·2	35·2	48·5	1·4568
B. 0·8824	0·8579	100·7	30·6	40·1	48·8	1·4569
C. 0·8823	0·8575	99·1	31·0	35·8	48·1	1·4569
D. 0·8826	0·8575	100·4	31·0	35·0	48·3	1·4569
E. 0·8825	0·8575	99·1	30·7	37·5	48·4	1·4569
F. 0·8826	0·8576	100·3	31·2	36·1	47·7	1·4569
G. 0·8824	0·8572	100·3	30·9	35·6	48·5	1·4570
H. 0·8824	0·8573	99·3	30·9	34·9	48·0	1·4568
I. 0·8824	0·8574	99·9	30·6	36·4	48·5	1·4570
J. 0·8823	0·8573	99·7	31·2	34·6	49·2	1·4570
K. 0·8824	0·8574	99·8	31·1	35·1	48·6	1·4570
L. 0·8825	0·8578	99·0	30·6	35·6	48·5	1·4570
M. 0·8824	0·8574	99·3	30·2	35·7	—	1·4568
N. 0·8823	0·8576	99·6	31·1	36·5	—	1·4570
Average values : 0·8825	0·8575	99·9	30·9	35·9	48·4	1·4569

TABLE B.—ILLIPÉ BUTTER.

Specific Gravity at 60° C.	Specific Gravity at 99° C.	Viscosity at 60° C.	Melting-point, ° C.	Iodine Value.	Melting-point of Fatty Acids, ° C.	Refraction $[n]_D^{40}$.
A. 0·8831	0·8588	102·6	33·8	32·2	52·4	1·4561
B. 0·8835	0·8578	100·7	35·8	32·5	53·4	1·4562
C. 0·8827	0·8569	102·8	30·7	32·4	52·8	1·4568
D. 0·8835	0·8570	102·2	33·8	31·1	52·3	1·4565
E. 0·8827	0·8576	103·0	34·5	29·6	52·5	1·4568
F. 0·8826	0·8573	103·3	34·3	31·3	52·1	1·4568
G. 0·8840	0·8589	—	30·3	30·8	—	1·4564
H. 0·8829	0·8579	105·2	33·0	27·4	—	1·4573
I. 0·8824	0·8576	103·9	34·0	31·0	52·6	1·4568
J. 0·8824	0·8573	104·9	31·7	32·0	52·3	1·4569
K. 0·8824	0·8577	104·7	32·7	30·5	53·0	1·4569
L. 0·8826	0·8578	105·7	34·4	31·6	54·1	1·4571
M. 0·8826	0·8578	104·2	31·0	32·6	52·0	1·4570
N. 0·8820	0·8570	—	33·1	33·2	—	1·4572
O. 0·8823	0·8576	—	33·8	33·4	—	1·4568
P. 0·8826	0·8577	104·6	34·7	31·6	52·5	1·4570
Average values : 0·8826	0·8577	103·7	33·2	31·5	52·8	1·4568

TABLE C.—SUMMARY OF OBSERVATIONS.

	Cacao Butter.			Illipé Butter.		
	Maximum.	Minimum.	Average.	Maximum.	Minimum.	Average.
Specific gravity at 60° C.	0·8830	0·8823	0·8825	0·8840	0·8822	0·8826
Specific gravity at 99° C.	0·8581	0·8572	0·8575	0·8579	0·8569	0·8577
Viscosity...	101·3	99·1	99·9	104·9	100·7	103·7
Melting-point	31·2	30·2	30·5	34·7	31·0	33·2
Iodine value	40·1	34·6	39·8	33·4	27·4	31·5
Fatty acids, melting-point	49·2	47·7	49·4	54·1	52·0	52·8
Refraction, $[n]_D^{40}$ ...	1·4570	1·4568	1·4569	1.4573	1·4561	1·4568

"*Titre*" *Test.*—The following modification was adopted: Five c.c. were melted in a test-tube in which a thermometer was suspended without touching the sides of the tube. During the process of cooling, observations of temperature were taken at half-minute intervals, and a time-temperature curve constructed. The "titre"-point was in all cases found to be about 0·5° C. higher than the melting-point. This relation applied equally to the cacao butter and to the illipé butter, and there appeared to be no advantage in adopting this test, which was accordingly rejected.

The observations of the above-mentioned constants were as given in Tables A and B on p. 231, and Table C above.

Consideration of these values indicates that if the average constant could be taken as applicable in all cases, identification and estimation of either fat might be possible, but the variations observed extend the range of each constant, so that for the two fats they often overlap or approximate very closely to each other. It was noticed, however, that if the observed values were compounded so that they could be focussed in one factor this difficulty would be overcome. Further consideration of the values with this object in view demonstrated that all the constants given above would be operative except that for refraction, which is practically identical for both fats. As a general principle it was decided to multiply the various constants together. It was observed that unless all the constants of cacao butter were higher or lower than those for illipé butter, such multiplication would result in one constant nullifying another. To obviate this it was decided that in all cases in which the illipé constant was the lower, the reciprocal should be taken.

The "factors" calculated on this basis were, therefore, the products of the specific gravity at 60° C., the specific gravity at 99° C., the viscosity, the melting-point, the melting-point of the free fatty acids, and the reciprocal of the iodine value.

For twelve individual samples, each of cacao butter and illipé butter, the "factors" thus obtained were as follows:

TABLE D.

Cacao Butter: 3347, 3266, 3252, 3251, 3194, 3191, 3169, 3149, 3130, 3123, 2972, 2839.

Illipé Butter: 4771, 4714, 4652, 4535, 4503, 4487, 4463, 4397, 4279, 4112, 3901 3890.

It will be seen that by this method there are obtained two ranges which are distinct in a measure not to be observed with any individual constant. The respective average "factors" calculated from the average constants of those samples of which complete examination was made are:

Cacao Butter ... ... 3150 | *Illipé Butter* 4403

These figures appeared to afford a reasonable basis for the calculation of proportions in the case of mixtures of cacoa butter and illipé butter. It is, of course, evident that such a method, if applied generally and without modification, would be based upon a mathematical fallacy. For example, in the case of the two fats in question, there is an error varying with the relative percentages present and having its maximum in a mixture composed of equal proportions of the two fats.

Table E showing this error was compiled as follows: The constants for the cacao butter and the illipé butter are the accepted averages upon which the above composite factors were based. The values for the mixtures were obtained by numerical interpolation for each constant, and represent the values which should be obtained on average fats if it were possible to eliminate experimental error. The factors given in Column A were obtained by calculation from the interpolated values of the constants by the method described for the two fats under consideration. Column B shows the percentages which would be obtained by referring these "factors" to the two standard "factors."

TABLE E.

	Specific Gravity at 60° C.	Specific Gravity at 99° C.	Viscosity.	Melting-point.	Iodine Value.	Melting-point of Fatty Acids.	A. "Factor."	B. Illipé Butter calculated.
				° C.		° C.		Per Cent.
Illipé butter	0·8828	0·8576	103·6	33·5	31·5	52·8	4,403	100·0
90 per cent. Illipé butter	0·8828	0·8576	103·2	33·2	31·9	52·4	4,261	88·7
80 ,, ,, ,,	0·8827	0·8576	102·9	33·0	32·4	51·9	4,118	77·3
70 ,, ,, ,,	0·8827	0·8576	102·5	32·7	32·8	51·5	3,984	66·6
60 ,, ,, ,,	0·8827	0·8576	102·1	32·5	33·3	51·0	3,847	55·8
50 ,, ,, ,,	0·88265	0·85755	101·75	32·3	33·7	50·6	3,724	45·8
40 ,, ,, ,,	0·8826	0·8575	101·4	31·9	34·1	50·2	3,604	36·2
30 ,, ,, ,,	0·8826	0·8575	101·0	31·7	34·6	49·7	3,481	26·4
20 ,, ,, ,,	0·8826	0·8575	100·6	31·4	35·0	49·3	3,387	17·3
10 ,, ,, ,,	0·8825	0·8575	100·3	31·2	35·5	48·8	3,256	8·5
Cacao butter	0·8825	0·8575	99·9	30·9	35·9	48·4	3,150	0·0

It will be seen that the greatest error is 4·2. This error can, however, be eliminated by applying a correction either to the "factor" obtained or to the percentage calculated from it. The latter procedure is the one we would suggest, and the following table shows the correction to be applied at every even 10 per cent. observed value.

TABLE F.

Observed Percentage of Illipé Butter:	0,	10,	20,	30,	40,	50,	60,	70,	80,	90,	100.
Correction—Add:	0·0,	1·7,	3·0,	3·7,	4·0,	4·2,	3·9,	3·2,	2·2,	1·1,	0·0.

Having regard to the limit of experimental error which must be allowed, it is possible that a result approximating sufficiently closely to the actual value might be obtained by an average addition of 2·1—*i.e.*, half the maximum error—to all observed values.

We prefer, however, to apply the absolute corrections shown in Table F, those applicable to any value other than even intervals of 10 per cent. being easily interpolated.

In order to test the method two series of mixtures were prepared, and each mixture submitted to a full examination. It should be observed that no repetition of work was made, the object being to ascertain how far the method was applicable under ordinary routine conditions. Further, the fats used were not "average" fats obtained by mixing several samples, but were purposely taken at random, and, in the case of the first series, were not previously tested.

The results obtained were as follows:

TABLE G.

Specific Gravity at 60° C.	Specific Gravity at 99° C.	Viscosity at 60° C.	Melting-point.	Iodine Value.	Melting-point of Fatty Acids.	"Factor."	Illipé Butter Present.	Illipé Butter Found.
			First Series.					
			°C.		°C.		Per Cent.	Per Cent.
0·8826	0·8575	99·4	31·0	35·0	48·3	3320	10	15·8
0·8827	0·8580	99·8	31·2	33·2	48·8	3466	20	28·6
0·8828	0·8577	100·0	31·5	33·3	49·5	3546	30	35·3
0·8829	0·8577	100·7	30·0	33·3	51·0	3503	40	31·8
0·8830	0·8577	102·0	32·0	32·6	50·8	3853	50	60·1
0·8830	0·8578	102·0	31·4	32·3	50·5	3793	60	55·4
0·8830	0·8579	102·2	32·1	32·2	50·6	3905	70	64·2
0·8830	0·8580	102·8	32·0	31·7	51·0	4010	80	72·0
0·8830	0·8580	103·0	32·3	31·4	52·8	4227	90	87·5
			Second Series.					
0·8825	0·8577	98·3	32·1	37·3	48·8	3125	0	0·0
0·8825	0·8576	99·3	32·0	36·3	49·0	3245	10	8·9
0·8825	0·8576	99·1	32·5	35·8	50·0	3405	20	23·4
0·8826	0·8576	99·4	32·5	34·7	51·0	3595	30	31·0
0·8827	0·8578	100·2	32·8	34·6	51·2	3682	40	46·4
0·8827	0·8576	101·2	33·0	34·1	51·5	3780	50	54·5
0·8828	0·8578	101·8	32·0	33·5	52·0	3828	60	58·1
0·8827	0·8577	102·0	32·2	33·1	53·0	3983	70	70·0
0·8827	0·8578	102·9	32·8	32·6	53·5	4194	80	85·1
0·8828	0·8577	103·0	33·0	31·9	54·0	4357	90	96·7
0·8829	0·8579	103·4	34·7	31·2	55·0	4790	100	100·0

As a further check, two mixtures were made up containing cacao butter mixed with two illipé fats of different origin, the latter being in about equal proportions, and the following results were obtained:

0·8827	0·8574	100·4	30·9	34·2	49·0	3366	25	19·8
0·8830	0·8576	100·9	32·0	33·2	50·0	3681	50	46·5

It will be seen that the mean deviations were as follows:

TABLE H.

Percentage of Illipé Butter: 10, 20, 25, 30, 40, 50, 60, 70, 80, 90.
Mean Deviation: ... ... 3·4, 6·0, 5·2, 3·1, 7·3, 6·0, 3·2, 2·9, 6·5, 4·6.

The question which next engaged our attention was the fact that very often the amount available for examination does not exceed 5 grms., whilst complete examination would require at least 130 grms. Certain of the constants which involved the use of only a small quantity of fat were therefore chosen to give a "short" factor calculated in the manner already described. The constants so chosen were: Melting-point, melting-point of the free fatty acids, and the reciprocal of the iodine value. The "factors" so obtained were as follows:

TABLE J.

Cacao Butter: 4437, 4305, 4278, 4258, 4250, 4210, 4192, 4169, 4165, 4123, 3962, 3724.
Illipé Butter: 6119, 5889, 5882, 5875, 5769, 5712, 5685, 5682, 5499, 5182, 5002, 4945.

It will be seen that here, again, the two ranges of values are distinct, the respective factors calculated from the average constants of those samples on which complete examination was made being:

Cacao Butter ... ... 4166 | *Illipé Butter* ... ... 5615.

Values calculated on this basis, and corresponding with Table E, show approximately the following correction for every 10 per cent. of illipé butter observed.

TABLE K.

Observed Percentage of Illipé Butter: 10, 20, 30, 40, 50, 60, 70, 80, 90.
Correction—Add: ... ... 1·5, 2·6, 3·4, 3·9, 3·9, 3·6, 3·3, 2·2, 1·1.

Applying these "factors" and corrections to the values observed for the test mixtures, the following average results are obtained (*cf.* Tables G and H):

Although this "short" factor may be useful where only small quantities of fat are available, we advocate the application of the "long" factor wherever possible. The latter, depending on a large number of observations, is necessarily the more reliable, the possible variation for any constant not affecting so appreciably the final result.

In conclusion, we would point out that we do not regard this investigation as complete. The method is put forward tentatively mainly as a basis for discussion. Further work is proceeding and more data are being accumulated.

TABLE L.

Illipé Butter Present.	Illipé Butter Found.		
	First Series.	Second Series.	Mean Deviation.
Per Cent.	Per Cent.	Per Cent.	
10	8·8	12·2	1·7
20	32·3	28·7	10·5
25	20·7	—	4·3
30	39·3	46·1	12·7
40	33·0	51·4	9·2
50	60·3 49·0	60·2	7·1
60	55·1	59·0	2·9
70	64·2	71·7	3·7
80	71·0	85·7	7·3
90	87·8	98·2	5·2

We have to thank the Government Chemist for permission to publish these results, and for facilities for carrying out the work.

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DISCUSSION.

Mr. KNAPP pointed out that the general principle of the authors' method might be applicable to other fats, although, of course, there were many pitfalls. He hoped that the authors would give the origin of the cacao butters used, including such details as the type of beans, and he drew attention to the fact that the name "illipé nut" was meaningless, and used in connection with many oleaginous nuts. He supposed that the fat which they had examined was derived from Borneo tallow, but he wondered what would be the interference of other fats, such as shea nut, which might be present. He further drew attention to the fact that equal quantities of given fats would not necessarily give a figure half-way between those of the original components. For example, this was well known to be the case in regard to melting-points. In this connection he asked how long the fats had been kept before the melting-points were taken, as this also affected the melting-point. As this method would be used to detect foreign fats in chocolates, he thought that the fats should first be made into chocolate and then extracted in order to carry out the analysis, as simple mixtures not made into chocolate might not give the same results.

Mr. L. K. BOSELEY observed that it was common practice to grind chocolate in a conche machine continuously for three days and three nights at a temperature of 150° C., and he thought that such treatment might produce some alteration in the analysis of the fats as compared with a simple mixture made in the laboratory.

Mr. E. M. HAWKINS asked upon what principle the authors' system of multiplication and division was based, and raised the question whether the multiplication of certain "factors" might not really magnify the error of experiment.

Mr. E. R. BOLTON thought the method of calculation proposed by the author was distinctly novel, and that the principle might well be applied to other calculations in connection with oil and fat analysis. The method of taking the melting-point appealed to him as being more free from personal error than the standard method. He criticised, however, the procedure of saponifying the fat in a dish, and suggested that a more complete saponification could be achieved in a flask under a condenser, and thus eventually produce purer fatty acids. He asked if all the fats examined were one season's fats, as he had observed a distinct difference from season to season, and he wondered if so far the authors had had any trouble with the fat produced from a species of *dicopsis*, which was a very awkward fat to distinguish when mixed with illipé butter.

Mr. TATE, in replying, said that one particular advantage in their melting-point method lay in the fact that shavings could be taken off the fat and tested without previous melting. It took three days to obtain a constant melting-point after a fat had been melted.

With regard to the question raised by Mr. Knapp and Mr. Boseley, they had extracted the fat direct from test samples of chocolates received from different sources, and the method invariably indicated percentages of illipé butter approximating very closely to the actual. In no case had illipé butter been indicated where not present or missed when used in manufacture.

In connection with the point raised by Mr. Hawkins, Mr. Tate did not agree that the method suffered by any magnification of reasonable experimental error. He explained in detail the process of deduction by which the method was devised. He considered that examination of the values obtained for the mixtures would be found to justify the calculation of the theoretical table.

The authors had examined the fat from two seasons, and the results had been reasonably concordant. Mr. Tate agreed with Mr. Knapp's remarks about the composition of illipé butter. Their work had been done on the fat known commercially by that name, which, in its individual physical and chemical constants, could not be differentiated with certainty from cacoa butter. Should there be any fundamental alteration in the character of illipé butter placed on the market, they would immediately detect it, as they were constantly examining fresh samples, and their composite factor would be modified accordingly. At present there was no indication that this would be necessary. If an extraneous fat were present it would be indicated by variation of one or more of the constants.

A NOTE ON THE "OIL" OF OATS.

By ERNEST PAUL, B.Sc., A.I.C.

(*Read at the Meeting, May* 4, 1921.)

Having had occasion to extract some pounds of freshly ground whole oats with petroleum spirit and, with the exception of a paper by Stellwaag,[1] being unable to find in the chemical literature on oils anything very definite as to the characteristics of the oil, advantage was taken of this opportunity for recording them.

The variety used was "Black Tartary," grown on a Fen farm near Peterborough. It yielded 4·32 per cent. of petroleum spirit extract, calculated on the dry sample.

The whole oats were dried to a moisture content of about 4 per cent. at 36° C. in a fruit-drying closet, ground, and extracted by percolation under pressure with hot petroleum spirit of boiling-point 40°–70° C., as long as any appreciable extract was obtained. A bright filtrate was obtained from the spirit extract with the aid of a mixture of kaolin and kieselguhr. On distilling off the solvent and partially drying the residue on a water-bath, it was found that this residue, previously liquid, had become semi-solid owing to the separation of a light brown granular mass, which proved to consist largely, if not entirely, of lecithins. In order to get rid of this, the whole mass was dissolved in dried ether and poured into excess of acetone, the lecithide being thus reprecipitated as a dirty white granular precipitate, which was filtered off by suction and proved to contain nitrogen and phosphorus; when dry and fat-free it only amounted to about 1 per cent. of the total extract.

The oil obtained by evaporation of the filtrate on a water-bath until constant in weight was a clear yellow-green fluid at ordinary temperatures, becoming thick and granular at 10° C., and solidifying completely in twenty-four hours at 5° C.; it had a marked acrid and irritating taste.

The constants of this oil are tabulated below, together with those obtained by Stellwaag on a filtered ether extract not otherwise treated for the separation of lecithins.

König[2] states that the ether extract of oats consists chiefly of free fatty acids While this work was in progress a comprehensive article by R. A. Berry[3] on oats, including some particulars about the oil, appeared. The main points of interest in this connection are that, according to Berry, the free fatty acids in the oil from freshly ground oatmeal are negligible, but that hydrolysis of the oil occurs to an extent depending upon the time elapsing between the grinding and the extraction, and that the effect of drying is to decrease the rate of hydrolysis, but not to prevent it.

Although no special precautions were taken to guard against hydrolysis or oxidation during extraction, it seems unlikely that free acid to the extent of over one-third of the total extract could have been produced in this way; it seems more probable that, even in the fresh oat, the true fatty matter is associated with a considerable amount of free fatty acid.

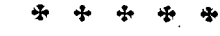
[1] Stellwaag, A.: *Landw. Versuchs. Stat.*, 1890, **37**, pp. 135-155.
[2] König, J.: *Landw. Versuchs. Stat.*, 1874, **17**, p. 1.
[3] Berry, R. A.: *J. Agric. Science*, **10**, Part IV., October, 1920.

The analytical figures resemble those of corn (maize) oil. The drying properties of the oil were tested by making films on a ground-glass plate, and it was found that at ordinary temperatures gumming had only commenced in two months; at 99° C. a hard varnish was produced in from two to three days.

On keeping the oil for three months in a corked vessel the acid value was found to be practically unchanged, but the odour had become decidedly rancid.

	Paul.	Stellwaag.
Melting-point	Approx. 8° C.	20° C.
Specific gravity at 15°/15° C.	0·925	—
Acid value	68·90	70·26
Free fatty acid (as oleic acid)	34·70	35·38
Neutral fat, per cent.	64	59·21
Saponification value	189·8	192·4
Iodine value (Wijs), per cent.	114·2	—
Unsaponifiable matter, per cent.	1·30	2·65
Refractive index $[n]_D^{40}$	1·4701	—
Total insoluble fatty acids, per cent.	93·6	92·76
Acid value of fatty acids	196·6	200·8
Mean molecular weight of fatty acids	284·8	279·0
Iodine value of fatty acids (Wijs)	127·1	—
Melting-point of fatty acids	27·5° C.	—
Refractive index $[n]_D^{40}$	1·4635	—

The work described above was carried out at the Research Station of the Olympia Agricultural Co., Ltd., The Bury, Offchurch, Leamington, and I have to thank Dr. Crowther, Director of Research, for permission to publish this note.



NOTES.

The Editor desires 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 DETECTION OF AGAR-AGAR.

THE detection of agar-agar in foodstuffs, etc., by chemical means offers some difficulty owing to the lack of well-defined reactions and the low chemical activity of this substance.

A method recommended by Macara and others is destruction of the organic matter by oxidation or ignition and examination of the sediment or ash microscopically for the presence of diatoms, which agars usually contain ("Aids to the Analysis of Food and Drugs," Moore and Partridge, Fourth Edition, p. 162. "Food Inspection and Analysis," Leach, Fourth Edition, p. 1002. "Microscopy of Vegetable Foods,"

Winton, Second Edition, p. 320. "Materia Medica," Greenish, Third Edition, p. 217. "Allen's Commercial Organic Analysis," Fourth Edition, Vol. I., p. 438).

This requires extreme care, much skill and experience, and is not always successful. Some samples of agar contain very few diatoms, and the material is only used in small amounts; moreover, when found, diatoms are not always conclusive evidence of the presence of agar. Kieselguhr or diatomaceous earth is sometimes employed as a filtering agent, especially for fruit juices, and the resultant product will often contain diatoms, so that, unless certain diatoms can be definitely identified as being characteristic of agar, the test is unreliable.

Another method recommended is to test the gelatinising power of the solution after removal of the more readily soluble constituents ("Allen's Commercial Organic Analysis," Bolton and Revis, Fourth Edition, Vol. VIII., p. 193).

A modification of this method, when carefully carried out, has been found to give the most reliable evidence of the presence or absence of agar.

For routine testing of fruit products the following procedure is recommended: Fifty grms. of raw-fruit pulp and 100 c.c. of warm water, or, in the case of jams, 50 grms. of jam and 500 c.c. of water at about 50° C., are well mixed and left to stand in a warm place, with occasional stirring until disintegrated, then allowed to settle and filtered through a folded filter. A little alumina cream may be added to assist filtration if sluggish. The insoluble matter is washed with warm water at about the same temperature. This removes sugars and other soluble substances which might interfere with the subsequent test. As a temperature of at least 90° C. is necessary to melt an agar gel, the majority of the latter will be left on the filter. The filter and contents are then transferred to a porcelain or metal vessel and boiled for a few minutes with about 50 c.c. of water and immediately thrown on to a folded filter. The filtrate, on cooling, will set into a gel if it contain any appreciable quantity of agar. If only a small amount, such as 0·1 per cent., be present, it may be necessary to evaporate the filtrate to small bulk before it will gel successfully. The method rarely fails in the presence of agar.

The residue on the filter may be ignited, the ash taken up and gently boiled in a small quantity of dilute hydrochloric acid, the insoluble matter allowed to settle, and the sediment examined microscopically for the presence of diatoms, which, if found in conjunction with a positive gel test, may be considered as additional evidence.

The process can be modified to give a rough approximation of the amount of any agar present by well washing the first filter with warm water and the second filter with boiling water, evaporating the whole of the second filtrate to dryness, and weighing the residue. A pure jam under the same conditions shows only a trace of solid residue.

The amount of agar found should be checked against a solution of agar of approximately the same strength. Agars themselves contain constituents which are soluble in water.

ALBERT E. PARKES.

LEAD IN PEATY WATERS.

The brown colour of certain peaty waters is sometimes so strong as to interfere with the recognition of small traces of lead by treatment with an alkaline sulphide. In such a case the lead may be conveniently recognised and estimated by the following process: Permanganate is added to the water until it is distinctly pink. The water is then rendered alkaline with ammonia and kept for about forty-eight hours, when, unless too much permanganate has been added, the water will be colourless and a precipitate will have formed. This precipitate contains the whole of the lead. It can be collected on a filter, dissolved in a few drops of concentrated hydrochloric

acid, and the resulting solution, after being diluted to a suitable measure, can be tested with alkaline sulphide in the usual way.

This procedure not only produces a colourless solution, but will serve to concentrate the lead without important loss when the proportion of lead in the water is too small for direct examination. The composition of the precipitate has not been studied, but it would appear that peroxide of lead is formed and is carried down either mechanically by manganese dioxide, or, possibly, as a compound with one of the oxides of manganese. THOMAS TICKLE.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Comparison of the Acidity of Goat's, Cow's, and Human Milk. E. W. Schultz and L. R. Chandler. (*J. Biol. Chem.*, 1921, **46**, 129-131.)—The acidities were determined by means of standard buffer salt solutions, brom-thymol blue, methyl red, and brom-phenol blue being used as indicators. Fresh goat's milk gave P_H values between 6·7 and 6·4, fresh cow's milk from 6·5 to 7·2, and fresh human milk from 6·86 to 7·46. Completely soured goat's milk showed a range of from 4·4 to 3·7, and was considerably more acid than soured cow's milk. T. J. W.

The Size of Fat Globules in Goat's Milk. E. W. Schultz and L. R. Chandler. (*J. Biol. Chem.*, 1921, **46**, 133-134.)—Goat's milk, although possessing a similar fat content to cow's milk, rarely forms a cream layer, owing probably to the small size of the fat globules. Measurements made by the authors by means of dark-ground illumination gave the following results: Below 2μ, 57; 2–4μ, 34; 4–6μ, 7; and 6–8μ, 2 per cent. The fat is thus more finely divided than in the case of cow's and human milk. T. J. W.

Estimation of Moisture in Milk-Powder. N. Schoorl and S. C. L. Gerritzen. (*Pharm. Weekblad*, 1921, **58**, 370-378.)—If milk-powder be dried at either 95° or 103° C. an apparently constant weight is reached in three to four hours, but the results, compared with the actual moisture found by exposure *in vacuo* over phosphorus pentoxide for twenty-four hours at 95° C., are 1·6 to 1·9 per cent. too low; admixture of sand is without influence on the test. Further heating at 95° C. causes gradual loss, but even after twenty-nine hours the correct moisture is not indicated. On prolonged heating at 103° C. the loss in weight fails to reach a limit, and a brown coloration is produced due to decomposition of milk-sugar in presence of phosphates. If milk-powder be heated at 100° C. in air which has been dried by sulphuric acid, the moisture-results after three to four hours are about 0·5 per cent. too low; the correct moisture is indicated only after thirty-six hours' drying. Heating at 110° C. in undried air gives results which are correct within 0·1 to 0·3 per cent. if the time of drying does not exceed two hours, and risk of

decomposition may be avoided by placing the weighing bottles on asbestos instead of directly on the metal shelf of the oven. W. J. W.

Fatty Acids of Egg-Yolk Lecithin. P. A. Levene and I. P. Roaf. (*J. Biol. Chem.*, 1921, **46**, 193-207.)—The fatty acids were prepared by acid hydrolysis of the salt precipitated by the addition of cadmium chloride to a purified alcoholic solution of the lecithin. These acids were found to consist of a mixture of oleic, palmitic, and stearic acids in molecular proportions. The oleic acid was identified, after separation, by its iodine value and analysis of its hydrogenation product, whilst the two solid fatty acids were identified by their elementary analysis, their melting-points, and their molecular weights. These results tend to indicate the existence of more than one lecithin in egg yolk. T. J. W.

Electrical Conductivity of Water Extracts of Wheat Flour. C. H. Bailey and F. A. Collatz. (*J. Ind. Eng. Chem.*, 1921, **13**, 319-321.)—Details are given of experiments made to determine the influence of the time and temperature of extraction upon the conductivity of the resulting extracts. This property is slightly increased by extending the time from 15 to 960 minutes, and is more marked in the case of clear flours than in that of patent flours. An increase in temperature also leads to a slight increase in conductivity, the highest values being given at a temperature slightly lower than 60° C. For the comparison of different flours a uniform procedure must be followed, and the following has been adopted: one part of flour is extracted with 10 parts of conductivity water at 25° C. for exactly thirty minutes, with intermittent shaking, whirled in a centrifuge for five minutes, and the clear solution filtered. The conductivity is determined in a modified Freas cell at a temperature of 30° C. in the usual manner. The conductivity of the extracts is apparently due to inorganic salts of phosphoric acid formed by hydrolysis of phytin by the action of phytase, and is proportional to the ash content, thus serving as an index of the grade of the flour. T. J. W.

Available Carbohydrate in Thrice-Boiled Vegetables. L. O'Reilly and E. H. McCabe. (*J. Biol. Chem.*, 1921, **46**, 83-89.)—The materials investigated were boiled in cheese-cloth bags for three periods of thirty minutes each. The insoluble residues were boiled with water, treated with taka-diastase, hydrolysed by boiling with dilute hydrochloric acid, and the resulting solution titrated with Benedict's solution. The results obtained indicate that very few vegetables lose the whole of the available carbohydrate present by boiling three times in water. The use of a larger volume of water for boiling ensures a more complete extraction, and the addition of 0·05 to 0·1 per cent. of sodium bicarbonate to the water greatly accelerates the removal of the available carbohydrate. On extracting 100 grms. of vegetable by boiling with three portions of water (2 litres each time), vegetable marrow, lettuce, and celery were completely freed from available carbohydrate, whilst in the case of spinach, asparagus, turnips, beet, and onions, the removal was practically complete. After the same treatment string beans, cauliflower, pumpkin, cabbage, and carrots retain approximately 0·5 per cent. of the carbohydrate. T. J. W.

Analysis of South Tunisian Palm Wine. A. M. d'Aymeric. (*J. Pharm. Chim.*, 1921, **23**, 272-273.)—The "wine" is obtained by puncturing the palm tree, about 7 litres being obtained from a tree per day for a period of more than one month. It is usually consumed in a fresh condition, but readily undergoes fermentation. Analysis of the liquid kept for eight days yielded the following results: Sp. gr., 1·0295; alcohol, 4·5; total acidity (as H_2SO_4), 0·70; glycerol, 2·0; reducing sugars 0·20; gums, 3·0; total solids, 10·5; and ash, 0·20 per cent. W. P. S.

Critical Temperature of Solution of Fats in Mixtures of Ethyl Alcohol and Amyl Alcohol. A. J. J. Vandevelde. (*Bull. Soc. Chim. Belg.*, 1921, **30**, 14-16; 58-62.)—Attempts were made to find a mixture of 94 per cent. ethyl alcohol and amyl alcohol which could be used in place of 99·1 per cent. ethyl alcohol in the Crismer method. It was found, however, that a mixture which gave the same value as ethyl alcohol when tested with the standard petroleum spirit yielded low results when used for fats. Further, a mixture which, with beef fat, gave the same value as ethyl alcohol yielded high results when tested against the standard petroleum spirit or coconut oil. The differences appear to be due to the relative solubility of the fats in amyl alcohol (*cf.* Fryer and Weston, ANALYST, 1918, **43**, 3). W. P. S.

Preserved Edible Fungi. J. Gerum. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1921, **41**, 123-126.)—Methods for the canning or bottling of edible fungi (*Boletus edulis*) are described, and it is shown that when they are preserved by sterilising in brine, there is a loss of about 43 per cent. of the nitrogenous food substance if the fungi are subsequently washed and cooked in fresh salt solution. The protein matter so lost is the portion most easily digested. Two samples of the fungi removed from the liquid in which they were preserved had the following composition: water, 92·53 and 93·46; mineral matter, 1·41 and 0·88; organic substances, 6·06 and 5·66; sodium chloride, 1·16 and 0·51; and nitrogen 0·24 and 0·27 per cent.

H. E. C.

Detection of Lactic Acid. L. Hartwig and R. Saar. (*Chem. Zeit.*, 1921, **45**, 322.)—Lactic acid may be detected by means of Denigès' reaction, which consists in heating 0·2 c.c. of the solution with 2 c.c. of concentrated sulphuric acid for two minutes in a water-bath, and treating the cooled liquid with one or two drops of a 5 per cent. alcoholic guaiacol solution; in presence of lactic acid, a deep rose-red persistent coloration is obtained, provided that the percentage of the acid in the original solution is not appreciably greater than 0·2. The method is applicable, with the same restriction as regards concentration, to baking powders, which now often contain calcium hydrogen lactate as their acid constituent. The lactic acid may be extracted by stirring about 3 grms. of the baking powder in a mortar with just sufficient water to dissolve it until most of the carbon dioxide has been evolved, then adding a drop of methyl orange solution and running in phosphoric acid solution from a burette until a distinct red tint appears. The liquid is then ground with sand and burnt gypsum to a doughy consistency and left for some hours, the hardened mass being powdered and extracted with ether in a Soxhlet apparatus.

The extract, filtered if necessary, is evaporated in a weighed flask and dried in a steam oven. If lactic acid be present, the residue consists of a viscous, brown syrup, which has an acid taste and fumes in the oven. This is dissolved in water to give a solution of not more than 0·2 per cent. concentration, which is filtered through a hardened filter and tested as above for lactic acid. The reaction is not shown by 0·2 per cent. solutions of formic, acetic, malic, citric, tartaric, benzoic, salicylic, and tannic acids, but is quite distinct with a solution containing 0·022 per cent. of each of these acids and of lactic acid. T. H. P.

Active Constituents of Capsella Bursa Pastoris. H. W. van Urk. (*Pharm. Weekblad*, 1921, **58**, 553-556.)—The author failed to detect the presence of alkaloids in a chloroform extract of *Capsella;* very weak reactions obtained with iodine-potassium iodide solution, Mayer's reagent, and tannin were probably due to traces of cholin extracted by the chloroform. A test for glucosides by Bourquelot's method also gave negative results, and Hager's contention (*Pharm. Praxis*, **1**, 604) therefore remains unconfirmed. W. J. W.

Alkaloids of Valerian. A. Goris and C. Vischniac. (*Comptes rend.*, 1921, **172**, 1059-1061.)—The authors confirm the existence, in valerian root, of the two alkaloids, chatinine and valerine, discovered by Waliszewski (*Union Pharm.*, 1893, **34**, 251); the former is soluble in ether, and the latter insoluble in ether but soluble in chloroform. These alkaloids have but slight physiological activity, and together form only about 0·01 per cent. of the fresh root. T. H. P.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Variation in the Zinc Content of Vertebrate Animals. G. Bertrand and R. Vladesco. (*Comptes rend.*, 1921, **172**, 768-770.)—Examination of a number of animals and fish of various ages shows that the proportion of zinc in the body reaches a maximum at an early stage in the development of the animal, afterwards diminishes to a minimum, and begins to increase in old age. The following amounts of zinc were found in the fresh material: Mice, 2·5 to 4·4; rabbits, 3·1 to 4·9; guinea pigs, 2·3 to 5·6; hens, 3·3 to 31·4; tench, 8·02 (7 months) to 706 (7 years); and herrings 69 to 122 mgrms. per 100 grms. T. H. P.

Nutritive Value of Lard. J. C. Drummond, J. Golding, S. S. Zilva, and K. H. Coward. (*Biochem. J.*, 1920, **14**, 742-753.)—The authors have investigated the question of the known deficiency of lard in the fat-soluble accessory factor (vitamin A). Pigs were fed on diets respectively deficient and rich in vitamins; the pigs were then killed, and the vitamin content of the fat produced under the different conditions of feeding was ascertained by means of feeding experiments with rats. Experiments were also made with pig fat of average character, as used for the manufacture of lard, and with commercial lard obtained from a large firm of bacon-curers. The results of experiments indicate that—(1) When the pig is fed on a diet (*e.g.*, grass) rich in vitamin A, supplies of that factor are stored in the body-

fat. (2) When the diet is deficient in vitamin A (*e.g.*, a diet consisting almost entirely of toppings and whey), the body-fat is deficient in that factor. (3) The process of manufacture of lard on a large scale in this country causes a marked destruction of the vitamin present, probably owing to exposure of the fat to oxygen at a high temperature. It appears, therefore, that the low nutritive value of lard is due to the deficiency in vitamin A of the usual diet of fattening pigs in this country and to destruction of the vitamin during the manufacture of lard.

R. G. P.

Factors Influencing the Nutritive Value of Lard and Lard Substitutes. J. C. Drummond. (*J. Soc. Chem. Ind.*, 1921, **40**, 81-83T.)—The nutritive value of lard cannot be estimated solely in terms of digestibility and coefficients of utilisation, since it depends largely on the amount of the so-called vitamin A present; the diet of the pig is the chief factor controlling the quantity of vitamin in the fat. In the manufacture of lard, loss of vitamin may be prevented by avoiding aeration or close contact with air or oxidising substances at high temperatures. Lard substitutes do not contain appreciable quantities of the vitamin, since this is not usually present in the constituents (cottonseed oil, earthnut oil, oleostearin, etc.). The vitamin content of beef fat is also dependent on the diet of the animal. Hydrogenated fats do not contain the vitamin. W. P. S.

Anhydrides of Higher Aliphatic Fatty Acids as Food. D. Holde and I. Tacke. (*Ber.*, 1920, 53B, 1898-1907; *Chem. Abstracts*, 1921, **15**, 862.)—The anhydrides of higher fatty acids are readily absorbed by the intestine, and oleic anhydride purified by distillation under reduced pressure has been used as a salad oil, although when used as a frying oil it undergoes some decomposition. The anhydrides of linseed and rape oils and of olein were prepared by heating the glycerides beneath a reflux condenser with a smaller excess of acetic anhydride than in Albitzky's method, and were purified by distillation and treatment with sodium hydroxide. The resulting products were semi-solid at 18° C., rape oil anhydride being partly crystalline, and they melted at about 23° C. They resembled the original glycerides in having a viscous consistency and slight volatility, whilst their refractive indices were about the same as those of the glycerides and higher than those of the free fatty acids.

Use of Edestin in Determining the Proteolytic Activity of Pepsin. J. F. Brewster. (*J. Biol. Chem.*, 1921, **46**, 119-127.)—Edestin was prepared by extracting the fat from hempseed, then extracting the air-dried meal with 5 per cent. sodium chloride solution, and recrystallising the edestin which separated. After filtration the residue was washed with 50 per cent., 95 per cent., and absolute alcohol, and finally with ether, afterwards being air-dried at the ordinary temperature. The proteolytic activity of pepsin was estimated by adding to a series of test-tubes definite increasing amounts of 1 per cent. edestin solution, followed by $\frac{N}{10}$ hydrochloric acid, 10 per cent. sodium chloride solution (which precipitates the edestin), and finally a 1 per cent. dilute hydrochloric acid solution of the pepsin under examination. The tubes were suspended in a constant temperature bath at 37·5° C., and the time noted

which elapsed between the addition of the pepsin solution and the point at which the precipitated edestin was completely liquefied, as shown by the sudden clearing of the contents of the tube. A straight line is obtained by plotting the required time of digestion in minutes against the concentration of substrate, and the relative activities of different pepsins are proportional to $\frac{\text{time of digestion}}{\text{volume of substrate}}$. Initially the activity of one sample of pepsin is determined by the U.S.P. method, and the remainder compared with it by the method described above. The use of edestin as described is based upon the work of Osborne (*J. Amer. Chem. Soc.*, 1894, **16**, 663, 703, 757).

T. J. W.

Effect of Cooking on the Digestibility of Phaseolin. H. C. Waterman and C. O. Johns. (*J. Biol. Chem.*, 1921, **46**, 9-17.)—Feeding experiments made upon rats have shown that the raw proteins of the bean *Phaseolus vulgaris*, added to an otherwise adequate diet, are insufficient for normal growth, but this is achieved if the protein or bean meal be boiled for a short time with distilled water. To investigate this behaviour samples of the raw and boiled phaseolin were digested in a solution containing 0·1 per cent. of pepsin and $\frac{N}{20}$ acid for one and a half hours at 37° C. Sodium carbonate containing trypsin was then added to show, after neutralisation, an excess of both substances of 0·5 per cent., and the digestion continued for two and a half hours. After digestion the enzymic activity was destroyed by heating the liquid to 80° C. for five minutes, the solutions filtered, and duplicate estimations of amino-nitrogen were made upon 10 c.c. portions of the filtrates. The results obtained show that by cooking the phaseolin for five minutes there was a distinct increase in digestibility, the maximum effect being obtained by boiling the phaseolin for three quarters of an hour, when an approximate increase of 30 per cent. amino-nitrogen was produced calculated on that yielded by the uncooked material.

T. J. W.

Vitamins and Yeast Growth. R. J. Williams. (*J. Biol. Chem.*, 1921, **46**, 113-118.)—The vitamin content of various vegetables and yeast was determined by grinding 1 grm. of the dry material with carborundum powder, suspending it in 100 c.c. of water at 60° C. overnight, and filtering the liquid. The amount of filtrate employed in each experiment was such as to give an increase of from 5 to 20 mgrms. of yeast. The results obtained are in good agreement with those obtained by Osborne and Mendel (*J. Biol. Chem.*, 1920, **41**, 451) in rat-feeding experiments. By the yeast-growth method baker's yeast shows a higher vitamin content than does brewer's yeast, but this is reversed in feeding experiments on animals. The extract of each yeast has a greater effect upon the growth of the corresponding yeast than upon that of the other. It is suggested that the antiscorbutic vitamin C may act as a secondary factor in the stimulation of yeast growth. T. J. W.

Differentiation of Yellow Plant Pigments from the Fat-soluble Vitamin. M. Stephenson. (*Biochem. J.*, 1920, **14**, 715-720.)—Experiments with young rats fed on a diet free from fat-soluble vitamin to which was added (*a*) crude alcohol-

petroleum spirit extract of dried carrot, (*b*) pure carotin; and comparative experiments with butter-fat before and after removal of colouring matter by means of birch charcoal, led to the following conclusions: (1) The addition of the crude alcohol-petroleum spirit extract of dried carrot to a fat not containing vitamin (palm-kernel oil was used) confers on it the properties of promoting growth and of protecting the animal from, or curing it of, kerato-malacia. (2) That carotin is not responsible for the above action. (3) That the vitamin content of butter is not affected by the removal of colouring matter by filtration through charcoal.

R. G. P.

Action of Methylene Blue and other Dyes on Living and Dead Yeast. C. G. Fraser. (*J. Phys. Chem.*, 1920, **24**, 741-748; *Chem. Abstracts*, 1921, **15**, 872.)—On treating suspensions of living and dead yeast with an aqueous solution (0·5 grm. per 100 c.c.) of methylene blue or erythrosin living cells are not stained after several minutes, whereas dead cells are stained within one minute. Congo red, fuchsin, safranin, gentian violet, and methyl green are less suitable dyes for distinguishing between living and dead yeast cells.

Influence of Minute Quantities of Metallic Salts on the Bacteriological Content of Water. E. L. Atkinson and R. C. Frederick. (*J. Royal Naval Medical Service*, April, 1921. *Reprint.*)—Standard solutions of lead (acetate), copper (sulphate), iron (ferric chloride), and zinc (sulphate) were added to four types of water (distilled water and waters low, high, and very high in total solids), so that each sample contained 0·25 or 0·5 part per 100,000 of the respective metals—*i.e.*, amounts within the possible limits of contamination from metal pipes or tanks. One c.c. of an eighteen-hour broth culture of *B. typhosus* was injected into each sample, and also into samples of the waters free from the metals, and these were subcultured at daily intervals for three days into ordinary nutrient broth tubes, which were then kept under extremely favourable conditions, and finally subcultured by the usual routine method. The results, given in tabular form, are contrasted with those obtained in the absence of metallic contamination, and show the very pronounced effect which traces of copper, and sometimes of zinc compounds, may have upon a bacteriological examination of water. It is shown that a water which is unfit for drinking purposes owing to serious contamination with excreta might, if containing minute quantities of metallic compounds, be passed as bacteriologically pure. The results also emphasise the importance of the chemical analysis of any sample of water. The products of contamination of water are readily recognisable by chemical methods, whereas it is admittedly difficult to isolate pathogenic organisms, especially from water contaminated with sewage.

Vitality and Viability of Hæmolytic Streptococci in Water. G. S. Livingston. (*Amer. J. Hygiene*, 1921, **1**, 239-251.)—Streptococci have been found in water contaminated by excretions, the organism of most common occurrence in water contaminated with human fæcal matter being *S. fecalis*, a non-hæmolytic variety. Hæmolytic streptococci appear to be rarely present in excretions, and the probability

of their reaching water in this way is slight, although cases of such contamination are on record (Wilson, *War Medicine*, **2**, 556). Hæmolytic streptococci are capable of retaining their vitality for periods up to forty days in sterile water, but under more natural conditions the interference of other organisms is an important factor. For example, all streptococci died in less than seven days in pond, ditch, and river water, whilst virulent strains died more rapidly than the least viable of the non-virulent strains, most of them not surviving for twelve hours. The evidence of the experiments described is that virulent hæmolytic streptococci recently isolated from lesions in the human body are poorly adapted to survive storage in water, but that old strains long accustomed to artificial media survive much longer.

Hæmolytic Streptococci from Milk and from Human Lesions. R. C. Salter. (*Amer. J. Hygiene*, 1921, **1**, 154-181.)—Hæmolytic streptococci differing in many respects from human pathogenic streptococci occur normally in unboiled and in pasteurised milk, and apparently belong to a homogeneous group. The morphological differences between milk and human hæmolytic streptococci are, however, too slight for purposes of differentiation, but the reaction of milk streptococci in litmus milk is distinctive. Most of the milk species produce acid and cause a reduction of the litmus within twenty-four hours, whereas human pathogenic strains usually produce only a slight acidity, although there are numerous exceptions—*e.g.*, *S. viridans*. Pathogenic hæmolytic streptococci ferment carbohydrates more readily, a constant difference between the two groups being the failure of the milk species to ferment saccharose. They may also be differentiated by their thermal death-rates in milk, the pathogenic streptococci dying rapidly, whilst the milk species die slowly at 60° C. The identification of hæmolytic streptococci should always be confirmed by a test for hæmolysis in a broth culture. The true Beta type produces hæmolysin in broth, whilst other streptococci of very similar type do not. Hæmolytic streptococci resembling the pathogenic type in every respect have been isolated from "certified" milk. They had only a low resistance to heat, and could be destroyed by proper pasteurisation.

A Streptococcus which produces Ropiness in Milk. H. Violle. (*Ann. Inst. Pasteur*, 1921, **35**, 218-229.)—A description is given of the streptococcus which produces ropiness in milk; the organism is widely distributed in milk, butter, cheese, etc., and grows rapidly in media containing lactose, maltose, or sucrose; lactic acid being produced. It does not attack lævulose, dextrose, xylose, starch, dextrin, or casein, but it utilises acid amines. The organism developes rapidly at 30° C.; its growth is inhibited at 46° C., and it is destroyed by heating for thirty minutes at 60° C. The streptococcus does not appear to have any injurious effect on human beings. W. P. S.

Relation between Lactic Acid Production and Bacterial Growth in the Souring of Milk. J. C. Baker, J. D. Drew, and H. J. Conn. (*New York Agric. Exper. Stat., Bul.* No. 74, 1919.)—Different cultures of lactic acid bacteria grow or multiply at different rates; one culture examined by the authors multiplied at a rate

almost in accordance with geometrical progression, whilst with another culture the rate was arithmetical. The rate of acid production per individual cell per hour also varies with the culture, being lower in the case of the organism multiplying at an arithmetical rate than in that which multiplies normally (geometrically). For a vigorous culture, the rate of production of lactic acid lies between 5×10^{-10} and 10×10^{-10} mgrms. per hour. The ratio of total acid produced to the number of organisms present is fairly constant, and is directly proportional to the amount produced per generation by each individual cell. There is a tendency for the acid to increase at a geometrical rate up to the coagulating point; at this point, multiplication of the bacteria appears to cease, and there is a considerable decrease in the rate of acid production. For counting the number of bacteria, the authors consider the direct count method to be preferable to the plate culture method.

W. P. S.

WATER ANALYSIS.

Estimation of Active Carbon Dioxide in Water. I. M. Kolthoff. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1921, **41**, 97-112.)—The solubility product of calcium carbonate, and the equilibrium between carbonate, bicarbonate, and carbon dioxide in water are discussed. It is shown that the value of the solubility product obtained by Tillmans and Heublein (*Gesundheitsin.*, 1913, **35**, 669) is too high; and the amounts of free carbon dioxide calculated from the equilibrium between calcium carbonate and bicarbonate on the basis of their figure is only correct when the combined carbon dioxide is less than 100 mgrms. per litre. Similarly Tillmans and Heublein's table, from which the active carbon dioxide (*i.e.*, the carbon dioxide which will act on other carbonates) can be calculated, gives erroneous results when the calcium and bicarbonate concentrations are not equivalent. A table is given from which the amount of the active carbon dioxide in a water can be accurately determined when the free carbon dioxide and the calcium content have been estimated. For the estimation of the free carbon dioxide the author adds 10 drops of 1 per cent. phenolphthalein to 100 c.c. of the water in a stoppered flask, and titrates with $\frac{N}{10}$ sodium hydroxide until the faint pink colour persists for five minutes.

H. E. C.

Estimation and Significance of the Hydrogen Ion Concentration in the Examination of Drinking Water. I. M. Kolthoff. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1921, **41**, 112-122.)—The use of the hydrogen electrode is not convenient for the estimation of the hydrogen ion concentration in drinking waters, but the colorimetric method gives good results: neutral-red should be used as indicator. The P_H values are found by comparing the colour with that of standard buffer solutions in suitably calibrated glass wedges. A simple method obviating the use of buffer solutions consists in comparing the colour of 10 c.c. of the water, containing 0·1 c.c. of 0·1 per cent. neutral-red solution, with 10 c.c. of standard mixtures of cobalt nitrate (72·8 grms. of crystallised cobalt nitrate in 1 litre of 1 per cent.

hydrochloric acid) and ferric chloride (45·05 grms. $FeCl_3.6H_2O$ in 1 litre of 1 per cent. hydrochloric acid). The P_H values are as follows:

Mixture.	P_H.	Mixture.	P_H.
9·0 Co, 1·0 Fe ...	7·0	5·0 Co, 5·0 Fe ...	7·6
7·0 ,, 3·0 ,, ...	7·2	4·0 ,, 6·0 ,, ...	7·8
5·5 ,, 4·5 ,, ...	7·4	3·0 ,, 7·0 ,, ...	8·0

The carbon dioxide content of the water may easily be calculated from the P_H concentration and the bicarbonate content. The P_H value of drinking water is almost always between 7·0 and 8·0, is affected only slightly by the hardness, and is of but little value in assessing the quality of the water. H. E. C.

ORGANIC ANALYSIS.

Guanidine Carbonate as a Standard Alkali. A. H. Dodd. (*J. Soc. Chem. Ind.*, 1921, **40**, 89-90T.)—Guanidine carbonate prepared from dicyanodiamide is readily obtained in a pure state by precipitating it from its aqueous solution with alcohol, repeating the precipitation, washing the product with 80 per cent. alcohol, and drying it at 110° C. It is not hygroscopic, and should form a useful substance for standardising acid solutions. W. P. S.

Indicator Properties of Two New Phthaleïns. W. Csanyi. (*Zeitsch. Elektrochem.*, 1921, **27**, 64-68; *J. Chem. Soc.*, 1921, **120**, II., 270.)— The phthaleïn prepared from phthalic anhydride and 1 : 2 : 3-xylenol affords a useful indicator, the change of which ranges from colourless to blue, takes place within the hydrogen ion concentrations, P_H 8·9 to 10·2, and is unaffected by excess of alkali or of alcohol. Ortho-α-naphtholphthaleïn is a less useful indicator than the above, because its colour changes are less sharp, although they take place over the same range of hydrogen ion concentration. Also it is only slightly soluble in water and in alcohol. H. E. C.

Analysis of Liquid and Gaseous Mixtures of Ether, Alcohol, and Water. I. Masson and T. L. McEwan. (*J. Soc. Chem. Ind.*, 1921, **40**, 29-32T.)—The method is based on the fact that paraffin, water, ether, and alcohol together form two layers, the upper containing all the paraffin, most of the ether, and a part of the alcohol. By working under standard conditions the increase in weight or volume of the upper layer serves to indicate the amount of ether present, and from the specific gravity of the original liquid and the amount of ether found the amount of alcohol present is estimated by referring to the table of gravities of known mixtures of alcohol, ether, and water determined by the authors. The following is the method of estimating ether in the case of liquids: A separating funnel, containing 200 c.c. of petroleum spirit (sp. gr., 20/4°, 0·68; b.-pt., 40-75° C.), is weighed (to 0·1 grm.); 100 c.c. of the sample are then added and the funnel and contents again weighed. Two hundred c.c. of water, containing 2 per cent. of sulphuric acid to prevent emulsification, are then poured in, and the funnel is shaken vigorously and set aside for a few minutes, with occasional rotation to assist in separation of the two layers. After running off the aqueous layer the gain in weight represents ether, etc.,

absorbed by the petroleum spirit, the gain in weight being about 90 per cent. of the ether present, and from the result the true value is found by reference to curves drawn from the tabulated results of examination of known mixtures. The results are accurate within 1 per cent., except where much water is present. Where only a small quantity of liquid (*e.g.*, 20-30 c.c.) is available, the process is carried out volumetrically in a 100 c.c. burette, the conditions of experiment differing slightly from those recorded above, and the actual content of ether is found by reference to special tables of results obtained by this method with known mixtures. In the case of gaseous mixtures, the gas is first dried by passing it through a drying-tube packed with ignited alumina; the ether and alcohol are then absorbed by two acid-bubblers, containing 10 c.c. and 5 c.c. respectively of pure sulphuric acid (sp. gr., 1·84). The gas taken for analysis should not contain more than 5 grms. of ether; any alcohol absorbed by the alumina should be displaced by a stream of dried air. It is advisable to stand the acid-bubblers in cold water. The gain in weight of the alumina tube gives the amount of water present, and that of the acid-bubblers the alcohol and ether. The contents of the first acid-bubbler are placed in a Mohr-shaped burette, filled to the lowest mark with mercury, and having a 50 c.c. graduated stem and a bulb of not less than 80 c.c. The first bubbler is then washed out with the contents of the second, and the acid layer made up to 20 c.c. with sulphuric acid (sp. gr., 1·84); mercury is then run out and 40 c.c. of paraffin added (sp. gr. at 20/4° C., 0·75; b.-pt., 120-140° C.). More mercury is then run out and 50 c.c. of water are carefully introduced into the burette, held in a tilted position, after which the burette is corked and the contents mixed gently while the burette is cooled under the tap. After thorough mixing of the contents by inverting the burette twelve to fifteen times, the liquids are allowed to separate and the gain in volume of the paraffin is read. All readings of the burette are made at the same temperature as indicated by the thermometer attached to the stem of the burette. The gain in volume of the paraffin under these conditions is from 70 to 84 per cent. of the volume of ether absorbed, and the actual amount of ether is obtained by reference to tabulated results obtained by this method with known mixtures. R. G. P.

Non-Identity of Elæostearic Acid Tetrabromide from Tung Oil with Ordinary Linolic Acid Tetrabromide. B. H. Nicolet. (*J. Amer. Chem. Soc.*, 1921, **43**, 938-940.)—α-Elæostearic acid (m.-pt., 48° C.) from tung oil yielded, on bromination in glacial acetic acid solution, only 5 per cent. of a solid tetrabromide, m.-pt., 115° C.; the corresponding tetrabromide from linolic acid obtained from cottonseed oil melted at 114° C., but when mixed with the α-elæostearic tetrabromide gave a mixture melting at 103° C. Further, ethyl tetrabromo-α-elæostearate (m.-pt., 50° C.) was prepared, and its melting-point was found to be lowered by the addition of ethyl tetrabromolinolate. The two tetrabromides are, therefore, not identical, but isomeric, and there is thus no evidence that the two acids are space isomers.

W. P. S.

Determination of the Acetyl Value of Fatty Substances. E. André. (*Comptes rend.*, 1921, **172**, 984-986.)—From the saponification values, S and S', of a

fat before and after acetylation, it is possible to calculate exactly the corresponding acetyl value. If the acetyl value be denoted by A, it is easily shown that $A=(S'-S)\left(1+\frac{\lambda S}{1-\lambda S}\right)$, where λ has the value 0·75. Calculation of the acetyl value from this expression is simpler and more rapid than Lewkowitsch's method for determining it. T. H. P.

Composition of French Oil of Turpentine. M. Vèzes. (*Comptes rend.*, 1921, **172**, 977-980.)—The polarimetric method described by Darmois (Thesis, Paris, 1911) has been applied to the estimation of the proportions of pinene and nopinene in a number of samples of oil of turpentine. Two hundred c.c. of the oil are distilled to five equal fractions of 40 c.c., the specific gravity (d) of each of these being measured at 15° C., and also the rotation (α) of a column 1 cm. in length for the three colours, yellow ($\lambda=578\ \mu\mu$), green (546), and indigo (436), furnished by a mercury vapour lamp and isolated by filtration through suitable glasses. The corresponding values of $[\alpha]=\frac{\alpha}{d}$ are calculated and are plotted as ordinates, with the values of λ as abscissæ. Two points representing, for two colours, each of the five fractions are joined by straight lines, the five lines meeting in a point, C; straight lines are then drawn between C and the analogous points P and N, corresponding with mixtures of the optical antipodes of pinene and nopinene respectively. By the ordinates of the points where it cuts the vertical lines representing the two colours considered, the line CP defines the rotatory powers for these colours of the pinene in the sample, and similarly CN indicates the rotatory powers of the nopinene present. From the known values of the rotatory powers of pinene and nopinene for one of the colours chosen, it is possible to calculate the proportions of the two hydrocarbons by the law of mixtures. The points P and N lie on the λ-axis, their abscissæ being: for P, 818 and 730·85 for the yellow-green and yellow-indigo pair respectively; and for N, 1042 and 1480·58 respectively. The points C, found experimentally for different samples of genuine oil of turpentine, differ but slightly from one another and also from the point of intersection, C_1, of the two lines representing, for each pair of colours, the rotatory dispersion of the pinene and nopinene contained in French oil of turpentine. The co-ordinates of C_1 are $\lambda=740$ and $[\alpha]=15{\cdot}10°$ C. for the yellow-green pair and $\lambda=661{\cdot}66$ and $[\alpha]=21{\cdot}05°$ C. for the yellow-indigo. The divergences of C from C_1 may be due to experimental error or to the existence of varieties of pinene and nopinene differing in rotatory power. T. H. P.

Estimation of Chlorides in Petroleum. R. R. Matthews. (*J. Ind. Eng. Chem.*, 1921, **13**, 325-326.)—The sample is thoroughly mixed, and 500 c.c. are transferred to a graduated 2,000 c.c. stoppered cylinder into which 125 c.c. of acetone are introduced, and the liquids mixed for approximately three minutes. The total volume is then made up to 2,000 c.c. by the addition of water, and the whole mixed for five minutes. The cylinder is allowed to stand until approximately 500 c.c. of the acetone-water mixture have settled out, and about 400 c.c. are

removed by means of a glass siphon, filtered to remove suspended oil, and concentrated, and an aliquot part titrated with $\frac{N}{20}$ silver nitrate solution, potassium chromate being used as indicator. Good agreement is shown between estimations made upon different portions of the same sample of oil. T. J. W.

Estimation of Benzene Hydrocarbons in Coal-Gas. E. Berl, K. Andress, and W. Müller. (*Zeitsch. angew. Chem.*, 1921, **34**, 125-127.)—The adsorptive properties of charcoal are utilized in a method for estimating benzene hydrocarbons in coal-gas. The inherent disadvantages of the dinitrobenzene and paraffin-oil methods are avoided, and the fact that even small amounts can be estimated accurately renders the method superior to that of Deville, in which the minimum amount which can be determined is 26·8 c.c. per cb. m. A layer of charcoal is put into a U-tube, the lower portion of which is widened, and into the open ends of which are placed stop-cocks in communication with inlet and outlet tubes. A current of coal-gas is passed over the charcoal at a velocity of approximately 250 litres per hour. When adsorption is complete, the limbs of the tube are connected with a steam-supply and a condenser and measuring-burette respectively, and the tube is heated in a bath to 110° to 120° C., while a current of steam is passed through it; the hydrocarbon is thus distilled and is measured in the burette. By this method the amount of benzene hydrocarbon in a sample of illuminating gas was found to be 20·2 to 23·9 c.c. per cb. m., whilst the results obtained by the dinitrobenzene and paraffin-oil methods were 16·0 to 17·2, and 9·74 to 10·1 cm. per cb. m. respectively. W. J. W.

Characteristic Reaction of Mercury Fulminate. A Langhans. (*Zeitsch. anal. Chem.*, 1921, **60**, 93-94.)—The test is made as follows: Twenty parts of fulminate are moistened with 5 parts of alcohol, 50 parts of water are added, and the mixture is shaken with 100 parts of 20 per cent. sodium sulphantimoniate solution. A yellow precipitate forms, the colour changing after a time to green and then black. The filtrate obtained from the precipitate is coloured red by nitric acid, and this red colouring matter is soluble in ether. W. P. S.

Reagent for Wood and Vanillin. J. Grüs. (*Ber. Deut. Bot. Ges.*, 1921, **38**, 361-368; *J. Chem. Soc.*, 1921, **120**, II., 284.)—When a wood shaving is dipped in a solution of vanadium pentoxide in dilute phosphoric acid the cell walls gradually assume a yellowish-brown colour. The addition of vanillin to this reagent produces a reddish-brown precipitate, or if the test be applied on a microscope slide reddish-brown crystals may be observed. H. E. C.

Origin, Development, and Value of the Thalleioquin Reaction. W. B. Hart. (*J. Soc. Chem. Ind.*, 1921, **40**, 72-73T)—The green coloration produced when a quinine solution is treated with chlorine and ammonia was first mentioned by H. A. Meeson (*Phil. Mag.*, 1835, 158), and the test has since then been developed in numerous ways by the substitution of bromine for chlorine, by different methods of liberating the halogen, etc. The author, as the result of an investigation of the reaction, finds that excess of bromine and its prolonged action are detrimental to the production of

the coloration, 6 atoms of bromine per molecule of quinine yielding the greatest depth of colour, and with this proportion the reaction may be obtained with a quinine solution of 1 : 200,000. The limit of sensitiveness is 1 : 250,000 in a depth of 2·25 inches of solution. The reaction is governed by so many variables that it is of little, if any, use for quantitative work. W. P. S.

INORGANIC ANALYSIS.

Identification of Mercury as Cuprous Mercuric Iodide. P. Artmann. (*Zeitsch. anal. Chem.*, 1921, **60**, 81-88.)—A bright red coloration is obtained when a drop of a mercury salt solution is added to precipitated cuprous iodide. The reaction is best carried out by coating a piece of filter-paper with freshly-precipitated cuprous iodide, drying the paper at a low temperature, and then placing a drop of the mercury solution on the treated surface. A solution containing 2 mgrms. of mercury per litre will give a coloration with the test. Bismuth salts, easily reducible substances, and excess of acid (greater concentration than $\frac{N}{10}$) must not be present. W. P. S.

Nitroso R-Salt, a Reagent for the Detection of Cobalt. H. S. van Klooster. (*J. Amer. Chem. Soc.*, 1921, **43**, 746-749.)—When R-salt (sodium 2, 3, 6-β-naphtholdisulphonate) is treated with nitrous acid, a nitroso compound is obtained which yields a bright red dye with cobalt salts and renders possible the detection of cobalt in the presence of large quantities of nickel. To prepare the nitroso salt, 35 grms. of R-salt are dissolved in 400 c.c. of water, 10 c.c. of concentrated hydrochloric acid are added, the solution is cooled to 8° C., and a solution of 7·2 grms. of sodium nitrite in 20 c.c. of water is added drop by drop during thirty minutes. The orange-coloured precipitate is collected, washed with a small quantity of cold water and then with alcohol. It is but slightly soluble in cold water, still less in alcohol, and fairly soluble in hot water. To detect cobalt in the presence of nickel and other metals, 2 c.c. of the solution to be tested are treated with 1 grm. of powdered sodium acetate and 2 c.c. of the reagent (0·5 grm. of nitroso R-salt dissolved in 100 c.c. of water); the mixture is boiled, 1 c.c. of concentrated nitric acid is added gradually, and the boiling continued for one minute. A red coloration indicates the presence of cobalt. The colours produced by iron, nickel, copper, etc., are destroyed by the nitric acid. W. P. S.

Estimation of Iron by the Cupferron Method. G. E. F. Lundell. (*J. Amer. Chem. Soc.*, 1921, **43**, 847-851.)—Investigation of the cupferron method for the estimation of iron and the separation of this metal from manganese, showed that iron is precipitated completely by the reagent even from solutions containing as much as 20 per cent. by volume of hydrochloric acid or sulphuric acid; the precipitate is also insoluble in cold dilute hydrochloric acid, but is slightly soluble in dilute ammonia. W. P. S.

Thallous Ferricyanide. Estimation of Thallium. V. Cuttica and G. Canneri. (*Gazz. Chim. Ital.*, 1921, **51**, I., 169-174.)—The preparation and properties of

thallous ferricyanide are described. The following procedure is recommended in the estimation of thallium as chromate. The liquid containing the precipitated chromate is filtered under pressure through a filter supported by a platinum cone, the precipitate being washed with 80 per cent. alcohol, in which it is practically insoluble, until a drop of the alcohol fails to react for chromic acid. The thallous chromate is then dissolved in dilute sulphuric acid and estimated by addition of potassium iodide and titration with thiosulphate: 3I corresponds with Tl_2O. The liquid to be titrated should contain the amount of thallium from about 0·2 grm. of the chromate, and 100 c.c. of 2 *N*-sulphuric acid, diluted to about 400 c.c., should be employed. Two grms. of potassium iodide are added, the end-point of the titration in presence of starch paste being shown by the change from dark green to golden yellow. The method gives rather lower results than the gravimetric method, but is more rapid.

T. H. P.

Preparation of Zirconia from Brazilian Ore and a New Method of Estimation. E. C. Rossiter and P. H. Sanders. (*J. Soc. Chem. Ind.*, 1921, **40**, 70-72T.)—The finely ground ore is fused with sodium hydroxide, leached with water, the residue evaporated with hydrochloric acid to dryness and extracted with hot water, and the extract filtered. The dilute solution of the chlorides is treated with sulphur dioxide in slight excess of the amount required to reduce the ferric chloride and heated to boiling, after which sufficient *N*-sulphuric acid is gradually added to replace the chlorine in the zirconium oxychloride; after a short time the whole of the zirconia is deposited as a bulky precipitate of basic sulphate, $5ZrO_2.2SO_3$. The precipitation, which can be made quantitative, affords a means of separation from iron and alumina, but not from titania. The solution, containing 0·2 grm. of zirconia in 150 c.c., is boiled with 25 c.c. of sulphur dioxide solution until the iron is reduced. It is then neutralised with ammonia, 10 c.c. of sulphur dioxide solution and 2 c.c. of *N*-sulphuric acid added, and the liquid again boiled. The precipitate is filtered off, washed, and dissolved in hydrochloric acid. The solution is evaporated just to dryness, the residue dissolved in water, the precipitation with sulphurous and sulphuric acids repeated, and the precipitate washed and ignited to oxide. The method is not suitable for the separation of large quantities of iron, as the basic zirconium sulphate is slightly soluble in the ammonium salt derived from the acid formed by the reduction of the ferric salt.

W. R. S.

Estimation of Iodic Acid and Silver by Electrometric Titration. W. S. Hendrixson. (*J. Amer. Chem. Soc.*, 1921, **43**, 858-866.)—The electrometric titration method is trustworthy for the estimation of iodate; the latter is reduced by the addition of an excess of standardised iodide solution in dilute sulphuric acid, and the excess is titrated with permanganate solution. Iodide may be titrated directly with iodate in dilute sulphuric acid solution by the electrometric method. Hydrochloric acid cannot be used in place of the sulphuric acid. The titrations may, however, be made in the presence of chloride in concentration not exceeding $\frac{N}{10}$, and in the presence of nitric acid of higher concentration if it contains only traces of nitrous acid. Silver may be accurately estimated by electrometric titration, iodide and permanganate solutions being used for the purpose.

W. P. S.

Volumetric Estimation of Sulphuric Acid. C. Pezzi. (*Giorn. Chim. Ind. Appl.*, 1921, **3**, 10-11.)—Raschig's method of estimating sulphuric acid volumetrically by precipitating it as benzidine sulphate and titrating the latter, suspended in water, with $\frac{N}{10}$ sodium hydroxide solution at 50° C., is useless in presence of ferric salts, which tenaciously retain occluded benzidine sulphate. The author finds that titration of the precipitate with standard sodium nitrite solution gives results low by only about 0·2-0·3 per cent., even when ferric salts are present. Two grms. of benzidine are dissolved in 750 c.c. of water containing 3 c.c. of hydrochloric acid, the clear solution being made up to a litre; 150 c.c. are sufficient for 0·1 grm. of H_2SO_4. This solution is added in the cold, with continual stirring, to the sulphate solution containing hydrochloric acid, the precipitate being allowed to settle. The mother-liquor is filtered in small quantities through two filter-papers on a Büchner funnel, the precipitate being afterwards introduced and washed first with the mother-liquor and finally several times with hot water, 15 c.c. in all being used. The precipitate is washed into a beaker, and should be finely divided in the liquid, whilst the papers are destroyed in a small beaker by means of 15 c.c. of concentrated hydrochloric acid, and this solution added to the suspension of the precipitate. The whole liquid is then titrated with $\frac{N}{20}$ sodium nitrite solution at 10-12° C. until, even 15 minutes after the titration is finished, a drop of the liquid colours potassium iodide-starch paper. One c.c. of $\frac{N}{20}$ sodium nitrite corresponds with 0·002452 grm. H_2SO_4 or 0·0024015 grm. SO_4. T. H. P.

Detection of Volatile Alkamines in the Presence of Ammonia and of Tertiary Alkamines in the Presence of Primary and Secondary Alkamines. H. E. Woodward and C. L. Alsberg. (*J. Biol. Chem.*, 1921, **46**, 1-7.)—The foodstuff is distilled with water, and the distillate received in a slight excess of acid. The acid solution is evaporated to a small volume, made alkaline by the addition of sodium hydroxide, and distilled into about 1 c.c. of 40 per cent. formaldehyde in a test-tube. One c.c. of a solution containing 12 per cent. of potassium bromide and 18 per cent. of mercuric bromide is added, and the test-tube gently warmed. In the presence of over 0·5 mgrm. of amino nitrogen there is produced a white precipitate of mercurous bromide which is insoluble on the addition of more formaldehyde. This reaction is approximately quantitative when employing about 10 c.c. of solution containing 1 c.c. of formalin and between $\frac{N}{100}$ and $\frac{N}{200}$ of the amine. Under these conditions the precipitate obtained is nearly twenty times the weight of the amino nitrogen. To detect tertiary alkamines in the presence of others, the volatile substances are distilled off and collected in a slight excess of dilute acid. The acid solution is evaporated to a small bulk, filtered if necessary, and precipitated by the addition, from a graduated pipette or burette, of Mayer's reagent containing 45 grms. of mercuric iodide and 33 grms. of potassium iodide per 100 c.c., until no more precipitation is observed. Each c.c. of the reagent precipitates 59 mgrms. of trimethylamine. By filtration and distillation of the precipitate with a solution containing sodium hydroxide and sodium sulphide, the trimethylamine may be recovered free from traces of mono- and dimethylamine. T. J. W.

Decomposition of Nitrous Acid. E. Oliveri-Mandalà. (*Gazz. Chim. Ital.*, 1921, **51**, I., 138-140.)—The methods usually employed for the detection of nitric acid in presence of nitrous acid give uncertain results owing to the formation of appreciable traces of nitric acid by partial decomposition of the nitrous acid. The author finds that the action of hydrazoic acid on nitrous acid proceeds quantitatively in accordance with the equation $HNO_2 + HN_3 = H_2O + N_2O + N_2$, no trace of nitric acid being formed. To test for nitric acid in a solution containing also nitrous acid, the liquid, acidified with acetic acid when the nitrous acid is present as salt, is treated with either a few c.c. of dilute hydrazoic acid or a little of its sodium salt. Since excess of hydrazoic acid interferes with all the colour reactions employed to identify nitrates and nitrites, the liquid is boiled to expel such excess, and a portion of it tested with an acetic acid solution of naphthylamine and sulphanilic acid to ascertain if all the nitrous acid has been destroyed; this being the case, the remainder of the solution is examined for nitric acid. T. H. P.

Comparison of Scales' Method and the Use of Devarda's Alloy for the Reduction of Nitric Nitrogen. A. P. Harrison. (*J. Biol. Chem.*, 1921, **46**, 53-56.)—Two hundred c.c. portions of a sodium nitrate solution containing approximately 0·1 mgrm. of nitric nitrogen per c.c. were measured into Kjeldahl flasks, to one set of which 3 grms. of Devarda's alloy (aluminium 50, copper 45, and zinc 5 per cent.) and 2 c.c. of saturated sodium hydroxide solution were added. A second set was prepared by adding 80 grms. of zinc-copper couple, 5 grms. of sodium chloride, and 1 grm. of magnesium oxide. Both sets were distilled into 50 c.c. of 4 per cent. boric acid solution to a total volume of 200 c.c. One drop of brom-phenol blue was added to each and the liquids titrated by artificial light with $\frac{N}{14}$ sulphuric acid. The results obtained indicate that both methods are equally accurate, but that the Scales' method is more convenient and occupies less time, the same zinc being used repeatedly and recoppered before each estimation. T. J. W.

PHYSICAL METHODS, APPARATUS, ETC.

Adsorption of Alkalis by Cellulose. I. M. Kolthoff. (*Pharm. Weekblad*, 1921, **58**, 46-56.)—Although cellulose does not adsorb sodium and potassium hydroxides in the physical sense, they are taken up by it from solutions, and with concentrations up to $4N$ the amounts are proportional to the concentration. Thus, from sodium hydroxide solutions of $4N$, $2N$, N, $\frac{N}{2}$, $\frac{N}{10}$, $\frac{N}{20}$, and $\frac{N}{100}$ strength, undried cellulose removed 3·2, 1·4, 0·65, 0·35, 0·085, 0·04, and 0·11 mol.-equivalents per grm. respectively, and in the equation $\frac{x}{m} = ac$ these results therefore show $a = 0{\cdot}8$. With dried cellulose the following figures were obtained: from $4N$, 2·1; $2N$, 1·1; N, 0·58; $\frac{N}{2}$, 0·3; $\frac{N}{10}$, 0·055; and $\frac{N}{20}$, 0·03, giving $a = 0{\cdot}55$. Between concentrations of $4N$ and $6N$, the amounts taken up are constant and independent of concentration: 3·3, 3·2, and 3·1 for $4N$, $5N$, and $6N$ respectively, and chemical combination takes place. Above $6N$ a sharp increase occurs and amounts again become constant: 5·2 and 5·5 for $7N$ and $8N$; a different combination is formed. Alkali carbonates are not taken up by cellulose.

From barium hydroxide more is taken up than from alkali hydroxides; thus, filter paper took up 0·53, 0·52, 0·28, 0·30, 0·14, and 0·03 mol.-equivalent per grm. from barium hydroxide solutions of 0·29*N*, 0·27*N*, 0·095*N*, 0·087*N*, 0·028*N*, and 0·0084*N* concentration respectively, and with cotton wool the amounts were 0·62, 0·39, 0·22, and 0·14, with 0·3, 0·15, 0·06, and 0·03*N* solutions. The amounts taken up are proportional to the square root of the concentration. Ammonia is taken up only in presence of other alkalis. W. J. W.

Adsorption of Lead and Copper by Cellulose. I. M. Kolthoff. (*Pharm. Weekblad*, 1921, **58**, 152-159.)—Neutral solutions of lead salts are not completely adsorbed by cellulose. If an alkali hydroxide containing carbonate be added to the solution, all the lead is removed, but this is due to precipitation of most of the lead, after which the remainder is adsorbed. With very dilute lead solutions, the amount adsorbed by cellulose is dependent on the concentration; in stronger solutions the amounts taken up are proportional to the alkalinity of the ash of the cellulose. Thus, filter paper having an ash-alkalinity equivalent to 1·17 c.c. $\frac{N}{10}$ per grm., adsorbed 1 to 1·4 c.c. $\frac{N}{10}$ per grm. from $\frac{N}{5}$, $\frac{N}{10}$, $\frac{N}{20}$, and $\frac{N}{50}$ solutions of lead acetate, whilst only 0·22 to 0·30 c.c. were adsorbed by filter paper with an ash-alkalinity of 0·22 c.c. $\frac{N}{10}$. In the case of copper salts, adsorption or fixation from neutral solutions is very slight and is not influenced by the alkalinity of the cellulose ash; about 0·1 c.c. $\frac{N}{10}$ per grm. was fixed from solutions of concentrations between $\frac{N}{4}$ and $\frac{N}{40}$. Fixation is greater from ammoniacal copper solutions, and is in inverse ratio to their concentrations. Thus, from solutions of 0·14*N* $CuSO_4$ in 1·2*N* NH_3, and of 0·07*N*, 0·028*N*, and 0·011*N* $CuSO_4$ in 0·6*N* NH_3, the amounts fixed were 0·35, 0·37, 0·67, and 0·84, respectively per grm. of paper. A considerable increase in the fixation takes place by addition of sodium hydroxide to the ammoniacal solution: the amounts with 0·14*N* $CuSO_4$ in 1·2*N* NH_3 and 0·1*N* NaOH, and with 0·07*N* and 0·014*N* $CuSO_4$ in 0·6*N* NH_3 and 0·1*N* NaOH were 3·12, 5·55, and 5·62 cc. 0·1*N* Cu. per grm. of filter paper. W. J. W.

Adsorption by Asbestos. I. M. Kolthoff. (*Pharm. Weekblad*, 1921, **58**, 401-407.)—Asbestos possesses adsorptive properties only when in an impure condition; material which has been purified by treatment with hydrochloric acid is no longer adsorbent. Adsorption varies with different qualities: with five samples, 3·25, 0·65, 0·2, 2·6, and 1·0 c.c. of $\frac{N}{10}$ hydrochloric acid were adsorbed per grm. of asbestos, whilst with another sample, 0·5 c.c. of $\frac{N}{10}$ silver nitrate and 1·37 c.c. of $\frac{N}{10}$ sodium hydroxide were adsorbed. Metals are adsorbed from their neutral solutions. When 1 grm. of asbestos was treated with 50 c.c. of a solution containing 20 mgrms. per litre, and the metal remaining in the solution was estimated colorimetrically after treatment, the following figures were obtained: nickel, 18; copper, 8; iron, 8; silver, 7; and lead 0 mgrms. per litre. The high adsorption of lead may be utilised in its removal from drinking water. Adsorption is found to take place in accordance with the adsorption isotherm: $x/m = \alpha c^{1/n}$. The following values of $1/n$ and α were obtained with various solutions: hydrochloric acid, 0·65 and 4·9; silver nitrate, 0·66 and 0·28; copper sulphate, 0·29 and 0·1; lead nitrate, 0·35 and 0·125; sodium

hydroxide, 0·33 and 0·30; and morphine chloride, 0·53 and 0·2. With higher end concentrations silver is more completely adsorbed than copper or lead, but with lower end concentrations the reverse occurs. Thus, with $\frac{N}{10}$ and $\frac{N}{100}$ silver nitrate, $x/m = 0{\cdot}075$ and 0·016; with 0·1 mol. and 0·01 mol. copper sulphate and 0·05 mol. and 0·005 mol. lead nitrate, $x/m = 0{\cdot}05$ and 0·028, and 0·0436 and 0·023. In addition to being adsorbent, impure asbestos is hygroscopic. For quantitative estimations it must be boiled with hydrochloric acid, and the purified material should not adsorb more than 0·1 c.c. $\frac{N}{10}$ acid per grm. W. J. W.

Adsorbent Properties of Glass-Wool. I. M. Kolthoff. (*Pharm. Weekblad*, 1921, **58**, 463-471.)—Glass-wool is not a true adsorbent, as in contact with water it liberates alkali, which affects the composition of solutions filtered through the material; further, its adsorptive capacity varies with the temperature, and a state of equilibrium is not reached. The alkalinity, calculated as equivalent of $\frac{N}{10}$ hydrochloric in 50 c.c. of water which had been shaken with Jena glass-wool, was 0·05 after five minutes', and 0·5 after ten days', shaking; after boiling for half an hour the alkalinity was 2·6. Previous treatment of the glass-wool with hydrochloric acid slightly increased its action with water, owing to a larger surface being exposed. From very dilute solutions of metallic salts relatively large amounts of the metals are taken up. Estimation of the metal in the residual solution after shaking 60 c.c. (10 mgrms. per litre) for two minutes with 1 grm. of glass-wool at normal temperature and 50° C., respectively, gave the following results: Lead, 1·5 mgrms. per litre and nil; copper, 2 to 3 mgrms. per litre and nil; zinc, 8 and 1 mgrms. per litre. In each case the adsorbed metal was completely recovered by treating the glass-wool with acetic acid. Investigation of the applicability of the adsorption formula $x/m = ac^{1/n}$ shows that after shaking with hydrochloric acid for one hour, there is no relationship between the end-concentration and the amount absorbed; for values of c of 0·096, 0·0485, 0·02428, and 0·0093, the values for x/m were 0·10, 0·096, 0·072, and 0·070. If the glass-wool be shaken with the acid for five hours to one week, the adsorption equation holds good, and the rate of adsorption is directly proportional to the concentration if there be no interference from diffusion. With end-concentrations of 0·0835–0·0082, the values for a and $1/n$ are 0·89 and 0·35 after five hours' shaking, and 10·0 and 0·89 after one week. Beyond this period, no relationship exists between x/m and c, but the amount which is adsorbed steadily increases. From sodium hydroxide solutions much smaller amounts are adsorbed than from hydrochloric acid; after four hours' shaking, 0·15–0·28 c.c. $\frac{N}{10}$ NaOH were taken up from solutions of $\frac{N}{10}$ to $\frac{N}{100}$ concentration.

From solutions of metallic salts and of alkaloids only small amounts are adsorbed. In the case of silver nitrate solutions, adsorption after four days was 0·25–0·30 c.c. $\frac{N}{10}$; lead solutions undergo a higher adsorption. From quinine hydrochloride only 1·5–2·0 per cent. was taken up. After boiling for fifteen minutes higher results were obtained, the adsorption with $\frac{N}{10}$ silver nitrate, copper sulphate, and quinine hydrochloride, being 2·0, 7·75, and 9·0 c.c. $\frac{N}{10}$ respectively. W. J. W.

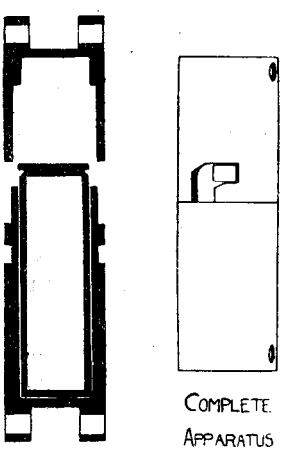
Analysis by means of Reducing Flames; Detection of Traces of Manganese in Presence of Iron or other Substances. J. Meunier. (*Comptes*

rend., 1921, **172**, 678-681.)—This method (*Bull. Soc. Chim.*, 1919, **25**, 57), which permits of the detection of traces of compounds of lead, manganese, calcium, strontium, potassium, lithium, etc., when other methods fail, is based on the principle that, when an oxide or a salt undergoes reduction on introduction into a flame, the latter exhibits a line spectrum of the corresponding metal. The hydrogen flame gives better results than coal gas, and an apparatus is described by means of which the powdered material is projected into the tube leading the hydrogen to the orifice where it is burnt. Behind the flame is the slit of a spectrograph. The results obtained with oxalate or oxide of iron are given, the number of lines in the spectrum increasing as the exposure of the photographic plate is increased. The presence of a trace of manganese in the iron was readily detected.

T. H. P.

Arsenic Reduction Tube with Electrical Heating. K. Zwicknagl. *Chem. Zeit.*, 1921, **45**, 418.)—In order to obviate the possible danger of hydrogen arsenide passing through the usual form of tube without decomposition on account of insufficient heating of the gas at the centre of the tube, a tube with an internal diameter of 10 mm. is flattened out into a roughly rectangular cross-section by heating and pressing between sheet-asbestos until the walls are about 2 mm. apart. When in use, the flattened part of the tube (15 to 20 cm. in length) is held in a vertical position, and is heated by means of resistance-wire about 0·5 mm. in diameter wound round the tube and separated by and insulated with asbestos-paper —a current of 3 amps. at 120 volts is sufficient to give the necessary temperature; the capillary tube is of the usual dimensions—viz., 1 to 1·5 mm. in diameter.

R. G. P.



Apparatus for Sodium Peroxide Fusions. H. J. Hodsman. (*J. Soc. Chem. Ind.*, 1921, **40**, 74T.)—The reaction vessel consists of a thin-walled steel tube closed at the bottom and provided with a well-fitting cap which has so small a surface of contact that a joint cemented by solidified melt can be broken readily or freed by solution in water. The mixture of substance and sodium peroxide is placed in this tube, which is then inserted in an outer casing having a lid held in place by a bayonet catch; the latter, when drawn up, holds the cap of the inner tube in position. The apparatus is supported in an inclined position, and the base is heated by a burner until the charge has fired.

W. P. S.

REVIEWS.

OFFICIAL YEAR-BOOK OF THE SCIENTIFIC AND LEARNED SOCIETIES OF GREAT BRITAIN AND IRELAND. Pp. 351. London: Charles Griffin and Co., Ltd. 1920. Price 15s.

The thirty-seventh annual issue of this Year-Book, which has recently been published, shows a great advance on the previous year in the number of papers contributed on scientific subjects during the session of 1919 to 1920, and is thus an indication of the recovery of the learned societies from one of the effects of the war.

The work is so well known and so permanently established that it is hardly necessary at the present day to emphasise its value to all workers in any branch of science, literature, or art. The complete lists of papers read before the different societies, which are conveniently classified into fourteen groups, enables anyone to learn what has recently been done in the subjects in which he is particularly interested, and in many cases this would not be possible in any other way.

As usual, the book is well printed and on good paper, and has an index which is sufficient for the societies as a whole, but it would be a great advantage if the publishers could see their way to issue every few years a classified subject index.

EDITOR.

COCOA AND CHOCOLATE, THEIR CHEMISTRY AND MANUFACTURE. Second Edition by R. WHYMPER. Pp. 568. London: J. and A. Churchill. 1921. Price 42s. net.

Nine years have elapsed since the appearance of the first edition of this book, which is the standard work in English on the chemistry and manufacture of cocoa and chocolate. Whilst the first edition probably owed more than a little to the standard work in German (Zipperer's "Manufacture of Chocolate"), the 241 pages of new matter in this edition owe nothing to that source of information. The book may conveniently be considered under the three main sections into which it is divided.

The first section, entitled the History, Botany, and Agriculture of Cacao, contains as much information on these subjects as the analyst of cocoa and chocolate could desire. This section has received ample additions in the historical portion, and a separate chapter is now devoted to Statistics (to 1918). One cannot read the interesting but somewhat discursive eighteen pages on the fermentation of cacao without realising how many problems the subject contains for the chemist. The author's whole-hearted approval of the washing of the cacao bean, as practised in Ceylon and Java, is not echoed by other authorities, who have observed that washing away the pulp greatly reduces the toughness of the shell, and renders it brittle and liable to break on handling. The chapter on the characteristics of the principal kinds of commercial cacao bean is excellent, and contains new data of interest to the chemist.

It is the second section, which deals with the Manufacture of Chocolate and Cocoa Powders, which has received the most important and valuable additions, the amount of information being twice that in the former edition. The new material is largely derived from the author's personal experience; it is up to date throughout, and contains much that is of value to the analyst. The few inaccuracies in the first

edition have been corrected, save one referring to theobromine. The author still quotes some results of his which show that large quantities of theobromine are lost on roasting the beans—thus, fifteen minutes at 120° C. show a loss of 0·34 per cent., and eight minutes at 230° C. a loss of 0·86 per cent. The author has evidently been the victim of an inferior method of estimation of theobromine. Were his figures true, the flues leading from roasting machines, in which tons of beans are roasted every day, would soon be clogged with theobromine, and a very valuable by-product thus produced. The new chapter on the Nutritive Value of Cocoa Preparations is unsatisfying, not because of any fault on the author's part, but because the modern work published on this subject is meagre.

The third and concluding section, occupying 177 pages, is of especial interest to the analytical chemist. This deals with the chemistry of cacao and its products, and gives a complete survey of the component parts and the methods of analysis. The author has collected in his book almost all the analytical figures of cacao products that have ever been published, the only notable exception being those of Winton, Silverman, and Bailey. He also gives the range for the various constituents. In the reviewer's opinion the fat in roasted nib is rarely as low as 45 per cent., and the theobromine in cocoa powder never as low as 0·7 per cent. The various analytical methods are set out clearly and fully, and very few processes have escaped mention. A more critical spirit would have been an advantage here; for example, as many as seven methods of estimating the fat are described. The methods of Kreuz and Hughes might with advantage have been replaced by the expeditious process due to S. B. Phillips (ANALYST, 1916, xli., 122). Those who, having no knowledge of the subject, consult the book for specific guidance, will find the wealth of material, ancient and modern, confusing, and many of the chapters would profit by a succinct summary of conclusions. Milk chocolate deservedly receives considerable attention, and a chapter has been added, unfortunately not conclusive, on the detection of husk in cocoa, the author's opinion being that "with modern machinery there can be no excuse for the presence of any quantity of husk in a cocoa or chocolate."

On p. 396 theobromine is stated to sublime at 134° C. The correct temperature is about 220° C. Maupy's process does not estimate the mixed alkaloids, as stated on p. 501, but gives theobromine alone.

The text is illustrated by sixteen plates showing the production of the raw product, twenty-nine figures of machinery, and nine photo-micrographs of cocoa and sugar. The photo-micrograph showing crushed cacao nibs is feeble and unrepresentative.

In carefully reading the book only one printer's error was noticed (on p. 515 Method I. should read Method II.). The arrangement of the book is logical throughout, and the printer's work leaves nothing to be desired.

ARTHUR W. KNAPP.

THE EXTRA PHARMACOPŒIA, Vol. II., Seventeenth Edition. By W. H. MARTINDALE, Ph.D., F.C.S., and W. W. WESTCOTT, M.B. Pp. xxxii + 688. London: H. K. Lewis and Co., Ltd. 1921. Price 17s. 6d. net.

The first volume of the seventeenth edition of this work was issued in June, 1920, and has already been reviewed in this journal (ANALYST, 1920, Vol. XLV., p. 400).

The subjects included in the present volume are arranged in the same sequence as in Vol. I. They consist mainly of analytical addenda to the Materia Medica given in that portion of the work, followed by chapters on Animal Organo-therapy, Bacteriological Notes, and a posological table arranged in alphabetical order.

As in previous editions, we find in this volume a very useful section on the examination of blood, urine, water, and milk, in addition to other physiological and pathological substances. In most instances the methods of analysis described are sound and the deductions made are correct, but a few points call for comment.

In describing the estimation of fat in milk only one method, the Werner-Schmidt process, is given, and the details of procedure in this case are not those usually adopted at the present time.

On p. 450 appears the statement that skimmed and separated milk should contain at least 9 per cent. of milk solids, whereas the Sale of Milk Regulations of 1901, to which the authors refer, have been superseded by the Regulations of 1912, fixing a minimum standard of 8·7 non-fatty solids.

Under water analysis the phenol-sulphonic process is the only one described for the estimation of nitrates, and no reference is made to the other more usual methods.

The present edition retains the chart for the recognition of organic chemical bodies used in therapeutics that has proved its utility in previous issues, and other very useful sections are those on mineral waters, antiseptics, and proprietary medicines. Ninety pages are devoted to Bacteriological and Clinical Notes with reference to special diseases, and the information here recorded is concisely and clearly expressed.

A number of legal enactments and reports on various diseases are referred to, and frequently brief abstracts are given. The methods of bacteriological examination and tests for the detection of specific organisms are described at some length, and the many references to original articles on these subjects show the great trouble taken by the authors to make the book as complete as possible.

The present edition in every respect reaches the high level of excellence attained by its predecessors and should not only find its way to the bookshelves of those specially interested in pharmacology and therapeutics, but should also prove of great value to the large number of analytical chemists who are frequently consulted on matters referred to in its pages. P. A. Ellis Richards.

Volumetric Analysis. By Charles. H. Hampshire, B.Sc., F.I.C. Third Edition. Pp. 120. London: J. and A. Churchill. 1921. Price 7s. 6d. net.

The larger part of this book is devoted to the processes of volumetric analysis and gives a clear presentation of the elementary theory underlying them; there are in addition sections in smaller type dealing with that side of the subject which concerns particularly the pharmaceutical student, for whom the book is primarily intended.

The first six chapters give in detail a variety of estimations based on the use of standard acids and alkalis; the theory of indicators is explained without reference to ionisation. There follow four chapters on reactions involving oxidation and reduction. Two chapters are devoted to precipitation reactions, and finally there is a useful

collection of miscellaneous exercises. Throughout the volume the calculation of results is well illustrated by a series of fully worked numerical examples. The book contains also a short index and a table of the atomic weights of the commoner elements.

As presenting an introductory course in volumetric analysis the small volume can on the whole be recommended; at the same time it is open to criticism on certain points of detail. Need the beginner be burdened with the chemical names and structural formulæ of the dyes used as indicators? If the elementary student is to rely upon his textbook for instruction in manipulation, that instruction should be thoroughly comprehensive. In this book a knowledge of the accurate use of the balance is taken for granted, and no details are given as to the best methods of getting a weighed quantity of solid into solution without loss; on the other hand, the manipulation of the various measuring vessels is described at length. If limitations of space prevent a complete treatment of sources of error, it would surely be better to leave such treatment entirely to the teacher. While the student receives warning on such points as the cleanliness of apparatus and the temperature at which his flasks, etc., hold exactly the right volume, no mention is made of errors of parallax in reading a burette, or of the procedure to be followed if the presence of water in freshly washed apparatus should introduce errors into his results. Omissions such as these are just as fruitful of error as many of those points which are fully dealt with.

A. F. KITCHING.

THE FUNDAMENTAL PROCESSES OF DYE CHEMISTRY. By H. E. FIERZ-DAVID, translated by F. A. Mason. Pp. 235 (45 illustrations, including 19 plates). London: J. and A. Churchill. Price 21s. net.

The title of this book scarcely indicates its technical character, and the word "Manufacture" might well replace "Chemistry." The translator is to be congratulated for introducing to the English-speaking chemist a volume novel in both its manner of presentation and contents. The translation is a very literal one, which is to be welcomed provided the result is a readable volume. This is generally the case, although one notes sentences such as: "For the rest, the index will afford any further information in cases of doubt" (p. 4), and "The development of the dye industry has brought it about . . ." (p. 210). The plates are well produced, but it is unfortunate that the figures are not better adjusted to relate to the adjacent text. Fig. 43 ("Distillation of a liquid of high boiling-point") appears very elementary and quite unnecessary. The publishers are well advised to refrain from the recent practice of producing books on cheap paper and adopting other economies in preference to making a small addition to the cost.

The novel method of inserting as a marginal note the quantities used in a preparation is retained in the translation. Preparations of many important intermediates are given in detail. Objection must be taken to the author's habit of interrupting the description of a preparation by inserting a discussion of the methods which may be used; thus the description of the preparation of naphthalene nitrotrisulphonic acid 1. 3. 6. 8 ceases at the top of p. 14, and is recommenced in the middle of a paragraph on p. 15. The method of stating the amount of an amino-

compound by giving the amount of sodium nitrite in grms. required for its diazotisation is particularly useful, but it should not be introduced in a book of this character without an explanation, as is done on p. 19 ("28 nitrite naphthylamine-trisulphonic acid").

The sections on "Intermediates" and "Dyes" are accompanied by valuable "Notes on Works Technique and Practice" for each preparation. The section on "Technical details" deals with works operations, plant, management, and costing, and a valuable "Analytical section" completes the book.

The author has made an extremely valuable addition to chemical literature. In view of its wide scope, the volume should undoubtedly be in the hands of all interested not only in dyestuffs but also other organic chemicals, either as students, chemists in works, or persons responsible for plant in chemical works.

F. W. Atack.

Recent Practice in the Use of Self-contained Breathing Apparatus. By Lieutenant Rex C. Smart, M.C., R.E. Pp. xiii + 243. London: Charles Griffin and Co., Ltd. 1921. Price 15s.

The subject-matter of this book deals with rescue work in connection with military mining operations, but, as Professor Cadman remarks in a Foreword: "Rescue work in collieries would be conducted more efficiently and with less danger by the application of some, at least, of the rules and regulations, and by the adoption of the standardised system of training which were employed by the military authorities in connection with the rescue operations conducted by the travelling companies of the Royal Engineers during the war." It may be added that, not only in collieries but in many industrial operations where cases of "gassing" are liable to arise, the use of self-contained breathing apparatus, and proper instruction in its use, would prove to be of material benefit.

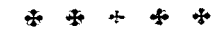
The author deals in ten sections with (1) the organisation of mine rescue schools, (2) training of personnel, (3) self-contained breathing apparatus, (4) testing and repairing of apparatus, (5) formation of characteristic mine gases in military mining, (6) resuscitation from mine-gas poisoning, (7) organisation of mining companies in mine rescue work, (8) rescue and recovery work in the trenches, (9) use of breathing apparatus in mine warfare, and (10) care of mice and canaries.

The "Proto" type of apparatus, which is described in detail, was the one chiefly used; it has been proved to be in advance of other types, and the extended use to which it was subjected enabled its defects to be detected, and the results of wear and tear to be determined. Doubtless some of its faults were due to the conditions obtaining in military mining operations, and would not arise in civil use where greater care in control and inspection of the apparatus might be anticipated.

The "Salvus" breathing apparatus was used only for inspectional purposes; its wider applicability is disputed in the light of tests carried out with it. A brief description of the "Novita" apparatus for the administration of oxygen in gas-poisoning cases is given.

It is to be regretted that the information in this book has not been presented in a clearer manner; the too frequent use of heavy type, even for minor paragraphs,

confuses the reader and tends to obscure more important items. The paragraph title "Abnormalities and Impurities in Mine Air" on p. 142 is repeated on p. 145. On this page also the incomplete sentence, "Gelignite on detonation 7 per cent. CO, burning 30 per cent. CO," has apparently become detached from its context; whilst on p. 154, a section headed "(*b*) German Carbon Monoxide Testing Papers" is given undue prominence and should have been inserted at the top of the page after "4 (*a*) Test paper . . . British method." Notwithstanding these defects, the author is to be congratulated on having collected and assembled a large amount of detail in regard to the use of breathing apparatus, which is a valuable addition to the rather limited information published. W. J. WRIGHT.



THE INSTITUTE OF METALS.

FOR the first time since its formation, in 1908, the Institute of Metals will be paying in the autumn a return visit to a provincial city. This meeting will be held in Birmingham, on September 21-23, the first autumn meeting of the Institute having also been held in that city in November, 1908. The arrangements for the meeting are in the hands of the Committee of the Birmingham section of the Institute, of which Dr. H. W. Brownsdon, M.Sc., is Chairman. The programme will include a reception by the Lord Mayor of Birmingham, visits to the University and works in the neighbourhood, as well as excursions in the locality.

INTERNATIONAL UNION OF PURE AND APPLIED CHEMISTRY.

BELGIAN BUREAU OF CHEMICAL STANDARDS.

A REPORT has been made to the Belgian Chemical Society (*Bull. Soc. Chim. Belge*, 1921, **30**, 41) by M. J. Timmermans upon the Bureau of Chemical Standards, the establishment of which was entrusted to Belgium at the International Chemical Conference (*cf.* ANALYST, 1921, p. 120).

Attention is first directed to the various technical and scientific uses for standard chemical products, and to the difficulty of procuring such products. In Germany, "normal metals" of guaranteed purity can be obtained from the Physikalisch-Technisches Reichsanstalt at Charlottenburg, which has also investigated the methods of purification suitable for technical products and the methods for their analytical control; whilst in the United States an analogous task has been undertaken by the Bureau of Standards at Washington.

The work outlined for the Belgian Bureau falls under three headings: (1) The preparation of pure organic liquids, which at the present time are particularly difficult to obtain. (2) Formation of a depot for pure and standard products prepared in the United States and other countries within the International Chemical Union. (3) The establishment of a centre for the distribution of samples of rare substances, and of information on everything concerning pure products, literature upon the subject, etc.

The work of the Bureau may be assisted by pecuniary help, by supplies of crude material, and by presents of pure chemicals or rare products capable of being used as chemical standards. It is also requested that chemists should send copies of any papers published by them upon subjects cognate to the work of the Bureau.

THE INSTITUTE OF CHEMISTRY.

FINE CHEMICALS, LABORATORY GLASS, AND PORCELAIN.

THE Council of the Institute of Chemistry, while recognising that the political considerations underlying some of the issues arising from the production, in this country, of laboratory requirements, such as reagents, research chemicals, glass, and porcelain, are outside the scope of the activities of the Institute, nevertheless feel that they are called upon to put before all users of laboratory materials certain definite facts and certain conclusions which may be drawn from those facts.

They have therefore published a memorandum in which it is pointed out that many instances have proved that British manufacturers are capable of producing chemicals in a state of purity fully comparable with that of pre-war supplies from abroad, and that in this matter they feel that it is their duty to emphasise the importance of encouraging home production. They are emphatically of opinion that users of chemicals should make themselves acquainted with what is available as the result of the very substantial progress made by British manufacturers, and, with a view to helping to spread this knowledge, have appointed a special Committee which will deal with questions relating both to reagents and chemicals, and will be prepared to assist chemists to obtain any materials which they may need. All users of such chemicals are strongly urged to make themselves fully acquainted with the circumstances, and to consider the ultimate effect of failing now to aid in building up a stable chemical industry.

Referring to glass apparatus, the Council remark that certain manufacturers have shown ability and readiness to produce the articles required, but that these manufacturers are now under the impression that the support promised to them during the war has not been extended to them in a measure sufficient to make them hopeful of the stability of this part of their industry.

So far as the Council have been able to obtain evidence, complaints regarding glass of recent manufacture, marked with the names of known makers, have been few in number. With the approval of the Board of Trade, the Board of Education, and the Department of Scientific and Industrial Research, the Institute has recently issued a letter to a large number of users urging them to purchase only laboratory glassware which bears the manufacturers' distinctive marks, since without those marks it is impossible to trace the source of any articles which may be the subject of complaints, and to take steps to remedy the faults disclosed.

British-made scientific glassware, equal at least in quality to any hitherto obtainable elsewhere, is forthcoming and at a price which is not unreasonable in the present circumstances having regard to the high cost of materials and production. There is a prospect, moreover, that when once the confidence of the manufacturers is restored and the industry established firmly, prices will compare favourably with those of articles now imported.

The same general considerations apply to the desirability of affording manufacturers of British laboratory porcelain such support as will enable them to complete the final stages of development necessary in order to supply porcelain of at least as high quality as that obtained from abroad. The Council feel that many of the complaints which have been made relate to apparatus of doubtful origin, and for that reason have appointed a Committee which is prepared in the interests alike of users and manufacturers to investigate any complaints which may be brought to their notice.

Summarising the whole position, the Council of the Institute state that they earnestly desire to do all in their power to ensure that chemists shall be able to obtain their professional requirements from home sources, and to aid manufacturers in meeting successfully such requirements.

THE INTERNATIONAL NORMAL WEIGHT FOR THE SACCHARIMETER.

At the present time the normal weight that is in almost universal use for the saccharimeter (or polarimeter, reading the percentage of sugar directly) is 26 grms., this standard having been adopted by the International Commission for Uniform Methods of Sugar Analysis in 1900. On dissolving 26 grms. of pure sucrose in water, and making the liquid up to 100 metric c.c. at 20° C., a solution is obtained which, in a 200 mm. tube, reads exactly 100 scale divisions, corresponding with $34{\cdot}657 \pm 0{\cdot}023$ angular degrees with spectrally purified sodium light. In France a normal weight of 16·29 grms. is largely used.

The proposal, was, however, recently made by Dr. C. A. Browne and other American chemists to adopt a sugar scale having a normal weight of 20 grms., the principal advantages claimed being: (1) It is a compromise between the 26 and 16·29 grm. scales; (2) the results obtained are easily converted into percentages by multiplying by 5; (3) aliquot portions of 50, 25, 20, and 5 c.c. of the 20 per cent. solution represent even gramme quantities; and (4) the specific rotation of sucrose at a concentration of 20 grms. in 100 c.c. (18·62 per cent. by weight) is about the maximum. It was further argued that the factor for convertion into circular degrees, namely $100 = 34{\cdot}657$, is inaccurate in view of the work of Bates and Jackson published in 1916, and that the present is an opportune time for adopting a new factor and a new standard. French chemists decided to support their American colleagues in adopting the 20 grm. normal weight.

In order to elicit the opinion of British chemists in the matter, a committee was formed consisting of the following: Professor Arthur R. Ling, F.I.C. (who acted as Chairman); Professor Thomas Gray, D.Sc., Ph.D., F.I.C.; L. J. de Whalley, B.Sc., F.I.C.; Hugh Main, B.Sc., and J. P. Ogilvie, A.I.C. (Secretary). This committee drew up a statement of the arguments *pro et contra*, nearly 2,000 copies of which were sent to chemists engaged in the sugar and allied industries, both in this country and in the British colonies.

An analysis of the replies received shows about 72 per cent. of the correspondents to be opposed to the adoption of the new standard. Most of these replies stated as the reason for the decision that the advantages claimed for the proposed new standard were too slight to compensate for the considerable inconvenience, expense, and confusion that would (it was considered) be involved by its adoption. A very frequent additional reason was that, owing to the smaller amount of sample taken for the assay, the accuracy of observation would be diminished. In an addendum to his reply, A. F. Blake, Chief Chemist, Atlantic Sugar Refineries, Ltd., St. John, N.B., Canada, said that he thought the 26-grm. value should be retained regardless of whether Herzfeld's or Bates and Jackson's factor is the correct one, or whether future investigations provide still another, "new instruments being made according to the best factor available at the time, and old ones being controlled and corrected by quartz plates standardised according to the latest factor."

It would therefore appear that British chemists are largely in favour of retaining the present international standard, which in fact is now in almost universal use (excepting in France and in Mauritius).