

THE ANALYST.

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

THE COLORIMETRIC METHOD OF DETERMINING HYDROGEN ION CON- CENTRATION: SOME APPLICATIONS IN THE ANALYTICAL LABORATORY.

By NORMAN EVERS, B.Sc., F.I.C.

(Read at the Meeting, June 1, 1921.)

HITHERTO in the literature of analytical chemistry the terms "acidity" and "alkalinity" have in general been very loosely employed. "Acidity" or "alkalinity" have usually been determined by titration, and expressed in one of two ways: (a) As the number of c.c. of standard acid or alkali required by a given weight of the substance in order to render it neutral to some indicator; (b) as the percentage of some acid or alkali to which the acidity or alkalinity is ascribed, in some cases quite arbitrarily. If we require to find the percentage of a single acid or alkali which we know to be present, there is no objection to the titration method, but in many cases the important point is not the total percentage of acid or alkali present, but the number of hydrogen or hydroxyl ions present.

Hydrogen ion concentration rather than the amount of acid present is the important factor in the curdling of milk, the precipitation of proteins, the hydrolysis of sugars, and processes of digestion or fermentation, to take only a few instances. The majority of bacteria, yeasts, etc., have a range of hydrogen ion concentration at which their growth is at a maximum, and for this reason this factor is of the greatest importance in the case of bacteriological media and in biological processes generally. The complete precipitation of metals as their sulphides in inorganic analysis is dependent on the hydrogen ion concentration of the solution.

If "acidity" or "alkalinity" were expressed in terms of the hydrogen ion concentration of a solution of known strength, this would merely involve the use of a number without reference to any particular indicator, acid or alkali.

Owing to the work carried out in recent years the accurate determination of hydrogen ion concentration is now a comparatively simple matter. If only reasonable accuracy is required determinations can be carried out as a routine test in any laboratory not involving any special apparatus or skill.

METHOD OF EXPRESSING HYDROGEN ION CONCENTRATION.—Dr. Monier Williams ANALYST, 1921, 315) has dealt with the theoretical considerations involved. The

symbol P_H , by which hydrogen ion concentration is expressed, indicates the logarithm of the reciprocal of the concentration of hydrogen ions in terms of normal—*e.g.*, when $P_H = 2$, the hydrogen ion concentration is $N \times 10^{-2}$, or one hundredth normal; when $P_H = 3$, one thousandth normal, and so on. At the point of absolute neutrality,—*i.e.*, for pure water— $P_H = 7$. When P_H is less than 7 the liquid is acid, and when P_H is greater than 7 the liquid is alkaline.

METHODS OF DETERMINATION—(a) *The Hydrogen Electrode or Electrometric Method*.—This is the most accurate method of determining hydrogen ion concentration. It has been described by Dr. Monier Williams (*loc. cit.*). Its disadvantages are the somewhat complicated apparatus required, and the fact that its use involves difficulties where a series of determinations is to be made.

(b) *The Colorimetric or Indicator Method*.—This method, though sufficiently accurate for ordinary purposes, is not capable of as great accuracy as the electrometric method. It requires, however, no special apparatus, and a large number of determinations can be carried out at the same time. It is inapplicable to very dark solutions, though it may be used for coloured solutions if the colour is not too deep. It may be used with success with turbid liquids such as milk.

The colorimetric method depends on the fact that with every indicator the colour change extends over a characteristic zone of hydrogen ion concentration; if, therefore, the hydrogen ion concentration of an unknown liquid lies within the range of a certain indicator, we can determine the factor with accuracy if we can find a solution of known hydrogen ion concentration which gives the same shade of colour with the indicator. The two essentials for this method are, therefore: (a) A complete series of indicators with well-marked colour changes which will cover a wide range of hydrogen ion concentration. (b) Solutions of known hydrogen ion concentration which are easily prepared and stable. By the use of some of the recently synthesised indicators of the phthalein series, together with methyl red, we can fulfil the first requirement. As the accompanying chart shows, these indicators show brilliant and permanent colour changes over a range of $P_H = 1$ to $P_H = 11$. If we have a solution of unknown hydrogen ion concentration, and we test it with various indicators until one is found to give a shade of colour intermediate between its extreme shades, we can then find a solution of known hydrogen ion concentration which gives the same shade of colour with the same amount of indicator.

SOLUTIONS OF KNOWN HYDROGEN ION CONCENTRATION.—It is not possible for this purpose to use solutions of acids or alkali of known strength on account of the susceptibility of such solutions to changes of hydrogen ion concentration from accidental causes, such as the alkalinity of the glass, etc., but this difficulty may be overcome by introducing certain salts, such as phosphates, borates, etc., which have a "buffer" action—*i.e.*, which have the property, when in solution, of causing the hydrogen ion concentration to be only slightly affected by the addition of small amounts of acid or alkali. For ordinary purposes the following four solutions, together with $\frac{N}{10}$ hydrochloric acid and $\frac{N}{10}$ sodium hydroxide, are all that are required. By taking definite volumes of any of these solutions, with different volumes of $\frac{N}{10}$ hydrochloric acid or $\frac{N}{10}$ sodium hydroxide, solutions of practically any P_H required may be obtained.

1. *Standard $\frac{N}{10}$ Sodium Citrate Solution.*—This is prepared by dissolving 21.008 grms. of pure citric acid in 200 c.c. of *N*-sodium hydroxide solution and diluting to 1,000 c.c. with water.

0.1 <i>N</i> -Citrate Solution.	0.1 <i>N</i> -HCl.	P_{H_2}	0.1 <i>N</i> -Citrate Solution.	0.1 <i>N</i> -NaOH.	P_{H_2}
C.c.	C.c.		C.c.	C.c.	
1.0	9.0	1.17	9.5	0.5	5.02
2.0	8.0	1.42	9.0	1.0	5.11
3.0	7.0	1.93	8.0	2.0	5.31
3.33	6.66	2.27	7.0	3.0	5.57
4.0	6.0	2.97	6.0	4.0	5.97
4.5	5.5	3.36			
4.75	5.25	3.53			
5.0	5.0	3.69			
5.5	4.5	3.95			
6.0	4.0	4.16			
7.0	3.0	4.45			
8.0	2.0	4.65			
9.0	1.0	4.83			
9.5	0.5	4.89			
10.0	0.0	4.96			

2. *Standard $\frac{N}{5}$ Sodium Borate Solution.*—This is prepared by dissolving 12.404 grms. of pure boric acid in 100 c.c. *N*-sodium hydroxide solution and diluting to 1,000 c.c. with water.

0.2 <i>N</i> -Borate Solution.	0.1 <i>N</i> -HCl.	P_{H_2}	0.2 <i>N</i> -Borate Solution.	0.1 <i>N</i> -NaOH.	P_{H_2}
C.c.	C.c.		C.c.	C.c.	
5.5	4.5	7.94	9.0	1.0	9.36
5.75	4.25	8.14	8.0	2.0	9.50
6.0	4.0	8.29	7.0	3.0	9.68
6.5	3.5	8.51	6.0	4.0	9.97
7.0	3.0	8.68	5.0	5.0	11.08
7.5	2.5	8.80			
8.0	2.0	8.91			
8.5	1.5	9.01			
9.0	1.0	9.09			
9.5	0.5	9.17			
10.0	0.0	9.24			

3. *Standard (M/15) Potassium Dihydrogen Phosphate Solution* is prepared by dissolving 9.078 grms. pure potassium dihydrogen phosphate (KH_2PO_4) in 1,000 c.c. water.

4. *Standard (M/15) Sodium Phosphate Solution* is prepared by dissolving 23.87 grms. pure sodium phosphate ($Na_2HPO_4 \cdot 12H_2O$) in 1,000 c.c. water.

The two last solutions are used in combination.

PHOSPHATE STANDARDS.

M/15 Na ₂ HPO ₄ Solution.	M/15 KH ₂ PO ₄ Solution.	P _H .	M/15 Na ₂ HPO ₄ Solution.	M/15 KH ₂ PO ₄ Solution.	P _H .
C.c.	C.c.		C.c.	C.c.	
0·0	10·0	4·49	6·0	4·0	6·98
0·1	9·9	4·94	7·0	3·0	7·17
0·25	9·75	5·29	8·0	2·0	7·38
0·5	9·5	5·59	9·0	1·0	7·73
1·0	9·0	5·91	9·5	0·5	8·04
2·0	8·0	6·24	9·75	0·25	8·34
3·0	7·0	6·47	9·9	0·1	8·68
4·0	6·0	6·64	10·0	0·0	9·18
5·0	5·0	6·81			

The following solutions are also required :

$\frac{N}{10}$ Sodium Hydroxide Solution (*Free from Carbonate*).—One hundred grms. of pure sodium hydroxide are dissolved in 100 c.c. water in a flask covered with tin-foil, and allowed to stand overnight for the carbonate to settle. The solution is then filtered quickly with the aid of the pump through a hardened filter. Ten c.c. are diluted to about $\frac{N}{5}$ strength with distilled water free from carbon dioxide, standardised against potassium hydrogen phthalate to phenolphthalein, and diluted to $\frac{N}{10}$ strength. This solution is stored in a bottle coated with paraffin wax and connected by a glass tube with a burette. The bottle and the burette have side-tubes joined to soda-lime tubes to prevent the entrance of carbon dioxide.

$\frac{N}{10}$ Hydrochloric Acid.—The ordinary laboratory solution may be used. The above solutions, with the exception of the $\frac{N}{10}$ sodium hydroxide, are kept in well-stoppered resistant glass reagent bottles. The only other apparatus required consists of burettes, graduated pipettes, suitable dropping bottles for the indicators, and racks for the test-tubes.

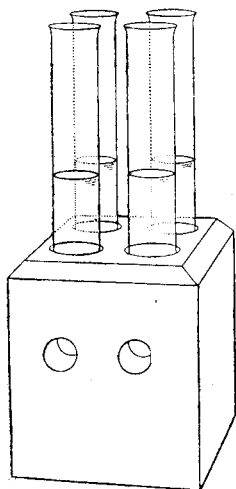
INDICATOR SOLUTIONS.—The following table shows a complete series of indicators which may be used for solutions from P_H = 1 to P_H = 11. These indicators are now readily obtainable on the market.

			Strength of Solution.	Range of P _H .
			Per Cent.	
Thymol blue	0·04 in water	} 1·2 to 2·8 8·0 to 9·6
Brom-phenol blue	0·04 "	
Methyl red	0·02 in 50 per cent. alcohol	4·4 to 6·0
Brom-cresol purple	0·04 in water	5·2 to 6·8
Brom-thymol blue	0·04 "	6·0 to 7·6
Phenol red	0·02 "	6·8 to 8·4
Thymolphthalein	0·04 in alcohol	10·0 to 11·0

METHOD OF DETERMINATION—1. *For Clear or Turbid Liquids Free from Colour.*

—The solution to be tested is tried with various indicators until one is found which gives a tint lying between its extremes of colour. Ten c.c. of the solution are then run into a clean test-tube, washed with neutral distilled water, and 5 or more drops of the indicator solution are added. With a little practice it is easy to judge from the shade of colour roughly what the P_H value is. Ten c.c. of a solution of this P_H value are then prepared in another test-tube from the standard solutions by consulting the tables—*e.g.*, if the solution gives a neutral colour with brom-phenol blue, the P_H may be judged to be about 3.5. From the tables we see that a mixture of 4.75 c.c. of $\frac{N}{10}$ citrate solution and 5.25 c.c. of $\frac{N}{10}$ hydrochloric acid has a P_H value of 3.53. This mixture is therefore prepared by running in the solutions from burettes or graduated pipettes. Five drops of the indicator are added and the colours compared. If the shades of colour do not match, another tube of a different P_H value is prepared until an exact match is obtained.

2. *For Coloured Liquids.*—For this purpose the piece of apparatus known as a comparator is used (see diagram). It consists of a cubical block of wood of $3\frac{1}{2}$ inches



side, with four holes bored vertically to hold four test-tubes. Two holes are also bored horizontally completely through the block, so that it is possible to look through two pairs of test-tubes simultaneously. In hole No. 1 is placed the tube containing 10 c.c. of the solution to be tested and 5 drops of indicator. Hole No. 2, behind this, holds a tube containing 10 c.c. of distilled water. In No. 3 we have the tube containing 10 c.c. of the standard solution with 5 drops of indicator, and, behind this, in No. 4 is a tube containing 10 c.c. of the coloured solution. In this way the colour is compensated, and we can compare the shades of colour without interference from the colour of the solution. If the colour of the solution is very dark, it may not be possible to distinguish the colour of the indicator, and recourse must be had to the electro-metric method. Where a large number of determinations has to be made it is convenient to have racks holding test-tubes containing a series of standard solutions with the indi-

cator, so that the tubes can be easily picked out for comparison and replaced. In this way a large number of solutions can be dealt with in a very short time. With practice it is quite easy to get a rough idea of the P_H value of a solution by merely adding the indicator without the use of standard solutions, and in many cases this may be all that is required.

THE DETERMINATION OF ACIDITY OR ALKALINITY OF COMMERCIAL ARTICLES.

—The method is very useful where a large number of samples have to be compared for acidity, such as gelatin, flour, starches, milk, sulphur, and many others. For merely comparative purposes all that may be necessary is to add the indicator to a suspension or solution of known strength and compare the colours without determining the actual P_H value.

Fine Chemicals.—Every pure salt when in a solution of definite concentration

should give a solution of definite P_H value. The method may therefore be used as an indication of the purity of salts and of the presence of an excess of the free acid or base. Where a strong acid is combined with a strong base the P_H of the solution should be about 7. If the base is stronger than the acid the P_H of the solution will be greater than 7, and if *vice versa*, the P_H will be less than 7.

The following are a few of the more interesting results obtained with commercial chemicals.

Sodium Salicylate.—Pure sodium salicylate prepared by dissolving pure salicylic acid in the calculated amount of pure sodium hydroxide solution was found experimentally to have in 2 per cent. solution $P_H=9.1$. A large number of commercial samples, on the other hand, were found in 2 per cent. solution to lie between $P_H=4.4$ and $P_H=5.8$. The reason appears to be that manufacturers are careful to keep an excess of salicylic acid present in order to avoid the discoloration which occurs if the salt is allowed to become alkaline. The excess of salicylic acid is found on titration to average about 0.1 per cent.

Morphine Hydrochloride.—The P_H of a 1 per cent. solution of morphine hydrochloride was found by experiment to be 3.65. Several commercial samples varied from 4.5 to 4.8, evidently containing a very slight excess of morphine. One sample, however, had a P_H of 6.5. It was not so readily soluble as usual, and was shown to contain free morphine.

Calcium Chloride.—A sample of crystalline calcium chloride was found to have a P_H in 2 per cent. solution of over 11. Calcium chloride, if pure, should give a practically neutral solution. The result was found to be due to the presence of free lime in the sample.

Potassium Iodide.—The reaction of this salt is of considerable importance from the pharmacist's point of view, as there must be no possibility of iodine being set free in the various combinations in which it is likely to occur. For this reason a salt with a slight alkaline reaction is to be preferred. If the P_H of a 2 per cent. solution is not less than 7 nor more than 9, the salt will be satisfactory from this point of view.

As other instances in which the method may be useful the determination of the alkalinity of natural waters and of soap solutions may be mentioned. For the standardisation of the reaction of bacteriological media it has to a very large extent replaced the old method of titration to phenolphthalein.

In the author's experience, in a commercial laboratory dealing with a large variety of materials, this method is finding fresh uses almost daily. There are probably many other analytical problems, besides those mentioned above, to which this method might be applied. The aim of this paper, for which but little novelty is claimed, is mainly to illustrate to those analysts who have not yet had experience of it what is, in the author's opinion, a valuable addition to analytical methods.

My thanks are due to Messrs. Allen and Hanburys, Ltd., in whose laboratories this work was carried out.

DISCUSSION.

Mr. W. PARTRIDGE said that the National Health Insurance Committee had given attention to the question of hydrogen ion concentration in so far as it was

applicable to bacteriological media, and the results of their investigation could now be obtained in pamphlet form. Referring to the personal element, which was so important in colour matching, he pointed out that the matching apparatus was independent of the original colour of the solution.

Mr. C. L. L. CLAREMONT questioned the method of expressing the results on the ground that it was a purely conventional way of expressing something we did not understand at all. In his opinion such methods as these did not mark any advance in general laboratory work, and he did not consider the method really useful. Many years ago, when other indicators were used, the results were quite as accurate and good as now.

Mr. B. S. EVANS said that there was considerable conventionality in the use of indicators, and that different indicators did give different results with different acids, and he considered it all to the good that some attempt should be made to express acidity, independently of the indicator. He would be glad to know if Mr. Evers knew of any apparatus for balancing extraneous colours in ordinary colorimetric work, as, for instance, in the colorimetric estimation of manganese in copper, where the colour of the iron present interferes with the matching of the permanganate. He cited the case of acid phosphates as one in which ordinary titration with indicators was useless.

Mr. HAWKINS said the chief difficulty was the question of carbon dioxide. Small quantities of carbon dioxide were liable to creep in, and everybody knew that in the case of the Kjeldahl process such small quantities made a distinct difference. He would be glad to have a little information as to an indicator sufficiently insensitive to carbon dioxide and yet acting as a sharp indicator in artificial light.

Mr. BOLTON asked if Mr. Hawkins had tried cochineal for Kjeldahl estimations, as he had found it sensitive both by day and night, and he thought it would be difficult to find a better indicator for that purpose.

Mr. CHAPMAN, after thanking the author for having brought the matter before the meeting in such a clear manner, remarked that everyone knew how unsatisfactory itmus, methyl orange, etc., were, on occasions, as indicators, and alluded, as an example, to the adjustment of the hydrogen ion concentration of soluble starch used in Lintner's method of determining diastatic activity. With these new indicators one could, at any rate, get definite results.

Mr. EVERS, in reply, stated that he had had considerable experience with this method and the methods in general use, and had obtained far more uniform results in this way than by the ordinary method of standardisation. Hydrogen ion concentration could be determined rapidly—rapidity was its great point, and the trouble of titration was eliminated. It was a means of getting something absolutely definite; you did understand what you were determining. So far as he knew, there was no previously described apparatus suitable for Mr. Evans's purpose, but probably the apparatus he had demonstrated would be suitable. He had always used brom-phenol blue for ammonia titrations, as he had found that it was not interfered with by carbon dioxide. He did not consider that cochineal had any advantages over these indicators, and it was not so effective for determining hydrogen ion concentration.

GEMSBOK BEANS.

(Bauhinia esculenta Burch.)

By G. T. BRAY, A.I.C.

"GEMSBOK" beans, derived from *Bauhinia esculenta* Burch. (N.O. *Leguminosae*), a plant indigenous to South Africa, are stated to be eaten by both the natives and animals of the South-West Protectorate of the Union of South Africa. No analysis of the beans appears to be on record, and the following results of the examination of a sample recently received at the Imperial Institute from South Africa are therefore of interest.

The beans were dark reddish-brown, roughly spherical in shape, and about $\frac{1}{2}$ to $\frac{3}{4}$ in. in diameter. They had a woody shell, the inner surface of which was covered with a thin layer of opaque, tough, white, horny material. The kernel was fairly hard, cream-coloured, and oily, and had a pleasant nutty flavour with a faint bitter taste. The beans were composed of 49 per cent. of shell and 51 per cent. of kernel. The average weight of a bean was 2 grms. and that of a kernel 1 gm.

The results of the examination of the beans are given below :

	Moisture.	Crude Proteins.	Fat (extd. with sodium-dried ether).	Carbo-hydrates, etc. (by difference).	Crude Fibre.	Ash.	Nutrient Ratio.	Food Units.
Husks alone (per cent.)	8.5	2.5	0.2	67.2	19.8	1.8	1 : 27.1	74
Kernels alone	4.0	32.8	41.6	17.2	1.3	3.1	1 : 3.4	203
Whole beans	6.2	18.0	21.3	41.6	10.4	2.5	1 : 5.0	140

The beans are free from alkaloids and cyanogenetic glucosides.

A quantity of oil was extracted from the kernels with petroleum spirit, and was a golden yellow, limpid liquid with a nutty odour and an agreeable taste. On analysis the following figures were obtained :

Specific gravity at 15/15° C.	0.9211
Acid value	0.6
Saponification value	190.0
Iodine value (Hübl, 17 hours)	95.6
Volatile acids (soluble*)	0.3
Volatile acids (insoluble*)	0.1
Unsaponifiable matter, per cent.	0.8
Refractive index, (n) _D ²⁰	1.464
"Titre" of fatty acids °C.	30.6

The residual cake left after the expression of the oil would have the following composition (when calculated on the basis of a 7 per cent. oil content): Moisture, 6.4; crude proteins, 52.2; fat, 7.0; carbohydrates, etc. (by difference), 27.4; crude fibre, 2.1; and ash, 4.9 per cent. Nutrient ratio, 1 : 0.83; food units, 175.

The above examination shows the kernels to be rich in crude proteins and oil. The oil is of good appearance, and has constants very similar to those of cottonseed

* Number of c.c. $\frac{N}{10}$ potassium hydroxide solution required to neutralise the acids from 5 grms. of oil.

oil, and should be of value as an edible oil. The cake left after the expression of the oil contains a very high percentage of protein and a low amount of crude fibre. Its value would probably be well above that of decorticated cottonseed cake, but it would be advisable to carry out feeding trials with it before making a definite recommendation.

In India the seeds of *Bauhinia esculenta*, and also those of other species of *Bauhinia*, are employed as food, whilst those of *B. Vahlia* are stated to have medicinal properties.

The author desires to acknowledge his indebtedness to Mr. P. F. C. Sowter, B.Sc., A.I.C., for his assistance in carrying out some of the work.

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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.

A ROUTINE TEST FOR THE PRESENCE OF SULPHITES.

THE following method has been found to be a useful routine one for the detection of sulphites added as a preservative or bleaching agent to foodstuffs, confectionery, and other goods.

It is a modification of the combined methods of Schmidt (*Arbeiten aus dem Kaiserlichen Gesundheitsamte*, 21, 226) and of Winton and Bailey (*J. Amer. Chem. Soc.*, 1907, 29, 1499), and in practice has been found to be speedy, sensitive, and efficient, without the disadvantages of the better-known method of reduction by means of zinc to hydrogen sulphide (*U.S. Dept. Agr. Bul.*, 107, A.O.A.C.).

Ten grms. of the material, such as dried fruit or minced meat or fish, are incorporated with 10 to 20 c.c. of water, by means of a pestle and mortar, and transferred to a small conical flask of about 50 c.c. capacity. In the case of fruit-pulp, glucose-syrup, or fruit juice, 10 c.c. may be diluted, when necessary, with 10 to 20 c.c. of water in the flask.

Ten c.c. of dilute sulphuric acid of about 2N-strength and two or three small fragments of marble chips are now introduced into the flask, and the mouth immediately covered with a piece of starch paper (impregnated with a 1 per cent. starch solution), which should be screwed round the neck of the flask, and held in place with a rubber ring. The reason for the addition of the marble is to set up a gentle current of carbon dioxide to sweep out the oxygen and the liberated sulphur dioxide. The top of the paper is moistened with 1 drop of a 1 per cent. solution of iodine.

In the presence of any appreciable quantity of sulphites the blue stain on the starch paper will be immediately discharged by the sulphur dioxide. If traces only be present, it may take a few minutes. The action takes place in the cold; it may be hastened by leaving the flask in a warm place.

If the drop of iodine solution used be of the magnitude of 0.1 c.c. it is obvious that the limit of sensitiveness of the test is the quantity of sulphur dioxide necessary

to reduce the iodine and discharge the blue colour—namely, 0.00025 grm.; and this is the limit usually found when using known amounts of sulphites, showing that practically the whole of the liberated gas is driven out of the flask. This amount, if 10 grms. of the material be taken, would represent 0.0025 per cent. of sulphur dioxide, 0.175 grain per lb., or 1.75 grains per gallon respectively.

By using a weaker solution of iodine the test could be made more sensitive, but for a routine qualitative test the strength suggested makes it sufficiently delicate for the amounts usually met with.

Traces of hydrogen sulphide do not seriously interfere with this method, but in practice 1 c.c. of a 5 per cent. solution of copper sulphate is added to the other ingredients when testing meat or fish, and this will retain as much hydrogen sulphide as is likely to be present.

ALBERT E. PARKES.



NOTES FROM REPORTS OF PUBLIC ANALYSTS.

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

BOROUGH ANALYST'S REPORT TO THE BOROUGH OF SALFORD FOR THE YEAR 1920.

VISITATION OF FARMS.—It is considered that the primary object of the department is to procure a pure milk supply, and that legal proceedings should only be taken in cases of apparent fraud, or where persuasion fails to effect an improvement. Hence, during the year 1920, the proportion of milk dealers personally interviewed has been much larger, whilst in cases where farm samples have been unsatisfactory the farms have been visited with the object of finding the cause, and, if possible, removing it.

"APPEALS TO THE COW."—In the case of twelve samples of milk deficient in fat by amounts varying from 6 to 15 per cent. "appeal to cow" samples were taken by the inspector. These were found to be deficient in fat, although the solids-not-fat figures were up to the average. The farm was therefore visited by the Veterinary Surgeon and the Borough Analyst. There were thirty cows, all in fair condition, yielding about twenty-six gallons of milk. The feed consisted largely of brewers' grains and turnips, with a proportion of maize meal and oats. It was considered that the albuminoid ratio was too low, and suggestions for improvement were made to the farmer. As a result, the milk supply has been permanently improved. After five weeks, four "appeal to cow" samples were again taken, and were found to contain 3.6, 3.0, 3.4, and 3.4 per cent. of fat respectively.

DIRT IN MILK.—The following method of estimation is used: The milk (500 c.c.) is allowed to stand for some hours in a cylindrical vessel, the bottom of which is drawn out and fitted with a tube graduated in 0.01 c.c. The volume of sediment is read directly and multiplied by two to obtain the parts per 100,000 of volume of sediment in the milk. Of the 613 samples examined, 396 gave no measurable sediment, 98 gave a sediment of 0.5 per 100,000, 62 gave 0.1, 3 gave 1.5, 29 gave 2.0, 6 gave 3.0, 1 gave 4.0, 6 gave 5.0, 3 gave 6.0, 2 gave 7.0, 3 gave 9.0, 1 gave 10.0, 1 gave 12.0, 1 gave 15.0, and 1 gave 20.0. As a provisional standard, samples giving a sediment of more than 5 volumes per 100,000 are being regarded as adulterated, although obviously this is too lenient to be accepted as a permanent standard.

SAMPLES UNDER THE RAG FLOCK ACT, 1911.—Three samples were found to contain 70, 50, and 100 parts respectively of soluble chlorine per 100,000, and there-

fore did not conform with the requirements for the standard of cleanliness fixed by the Ministry of Health in the Rag Flock Regulations, 1912 (viz., a maximum of 30 parts per 100,000). In each case a circular letter was sent by the Medical Officer of Health to the vendor, intimating that in future proceedings would be taken where the flock fell below the legal standard.

G. D. ELSDON.



ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Discoloration of Sweet Potatoes in Tins. E. F. Kohman. (*J. Ind. Eng. Chem.*, 1921, 13, 634, 635.)—The black coloration sometimes observed in tinned sweet potatoes is due to combination of a tannin-like constituent with iron derived from the container; since the iron must be in the ferric state in order to give the coloration, the access of air is necessary for the formation of the latter, and the necessity of tight seams is emphasised.

W. P. S.

Eggs and Egg Products. (*Report of Connecticut Agric. Exper. Stat. Bull.*, 227, 1921, 223-224.)—*Composition of Egg-Shells*: Air-dried egg-shells (without the inner membranous lining) contained 0.21 to 0.64 per cent. of moisture (15 samples), 49.40 to 53.28 per cent. of lime as CaO (26 samples), and 1.04 to 2.48 of magnesium oxide (7 samples), and lost on ignition 45.41 to 46.39 per cent. (15 samples). *Egg-Powder*: A sample stated to consist of sprayed egg yolk, albumin, salt, powdered skimmed milk, starch, gelatin, and sodium bicarbonate, had the following composition: Moisture, 6.83; ash, 9.47; protein (N \times 6.25), 20.48; starch, 26.40; fat, 13.85; and lecithin phosphoric anhydride, 0.46 per cent. No artificial colour was present. *Egg "Noodles"*: Eight samples gave the following results on analysis: Ash, 0.68 to 1.66; protein, 12.94 to 16.56; and lecithin phosphoric anhydride, 0.028 to 0.066 per cent. They were free from artificial colour. The lecithin phosphoric acid (P_2O_5) content was taken as the index of egg material present. Accepting Juckenack's standard (*Conn. Exp. Stat. Record*, 1904, p. 138), 0.0225 per cent. of lecithin phosphoric acid may be found in "noodles" prepared without eggs, and this amount is more than doubled by the addition of egg or egg yolk in the proportion of one to 1 lb. of flour. Figures less than 0.035 or 0.040 per cent. do not indicate appreciable amounts of eggs. *Dehydrated Eggs*: Two samples gave the following results: Moisture, 6.25 and 6.62; ash, 3.42 and 3.50; proteins, 40.44 and 43.00; fat, 45.12 and 42.29; and lecithin P_2O_5 , 1.37 and 1.33 per cent.

Adulteration of Egg-Powder with Cereal. (*U.S. Dept. Agric. Bureau of Chemistry. Service and Regulatory Announcements*, June, 1921. Suppl. 113, 96-97.)—An article sold as "whole egg-powder made strictly from fresh eggs" was found to consist essentially of maize starch, egg albumin, and a small amount of calcium and

aluminium phosphates, and to be artificially coloured with tartrazine. It was condemned in the District Court of the United States as adulterated, and also as misbranded, notwithstanding the fact that the following statement was inconspicuously printed on the label: "Artificially coloured, The contents of this packet contains powdered yolk of fresh eggs, albumin, corn starch, and phosphate."

Ice Cream. (*Connecticut Agric. Exper. Stat. Bull.*, 227, 1921, 236-249).—According to the Public Acts of 1919, ice cream manufactured in the State of Connecticut must contain not less than 8 per cent. of milk fat in (plain) ice cream, and not less than 6 per cent. in fruit and nut ice cream. It must be free from boric acid, salicylic acid, formaldehyde, saccharin, salts of copper, iron oxide, ochres, and injurious colours or flavours. Harmless permitted colours and flavours must be declared, and the use of harmless vegetable gums and gelatin is allowed. Ice cream containing less fat than the standard amounts may be sold provided the true fat content is declared. Of 400 samples examined in 1920, forty-seven contained less than the standard amounts of fat, but the average proportion was 8.8 per cent.

Estimation of Caffeine in Tea and Coffee. R. E. Andrew and E. M. Bailey. (*Connecticut Agric. Exper. Stat. Bull.*, 227, 1921, 256-258).—The following simple method, based on the methods of Power and Chestnut (*J. Amer. Chem. Soc.*, 41, 1300), of Stahlschmidt (*J. Amer. Off. Agric. Chem.*, 2, 3, 332), and of Deker (*Chem. Zentrbl.*, 1903, 1, 1, 62), is submitted for collaborative study with a view to its adoption as an optional official process: Five grms. of the tea or coffee, previously ground to pass through a $\frac{1}{25}$ in. sieve, are boiled with 10 grms. of heavy magnesium oxide in a 500 c.c. flask for two hours over a low flame, a narrow glass tube 30 ins. long being used as condenser. The contents of the flask are then cooled, made up to 500 c.c. and filtered, and 300 c.c. of the filtrate mixed with 10 c.c. of 10 per cent. sulphuric acid, and evaporated to about 100 c.c. in an Erlenmeyer flask. The liquid is filtered into a separating funnel, the flask being rinsed out with small portions of 1 per cent. sulphuric acid, and the whole then shaken with six successive portions of chloroform (25, 20, 15, 10, and 10 c.c.). The united extracts are treated with 5 c.c. of a 1 per cent. solution of potassium hydroxide, and the chloroform layer is subsequently drawn off, whilst the alkaline layer is washed with two portions (10 c.c. each) of chloroform, and the washings added to the main extract. The chloroform is distilled in a weighed flask, and the residue dried at 100° C. until constant in weight. If desired, the nitrogen in the residue may be estimated by Kjeldahl's method, and the caffeine calculated from the nitrogen multiplied by the factor 3.464. The quoted results obtained with various kinds of tea and with coffee by either method agree closely with those obtained by the more tedious methods of Stahlschmidt and of Power and Chestnut.

New Method for the Volumetric Estimation of Reducing Sugars. A. Jonescu and V. Varcolici. (*Ann. Chim. anal.*, 1921, 3, 229-234).—The reagent employed contains 46 grms. of potassium ferricyanide and 46 grms. of potassium hydroxide per litre; it is standardised by titration with 0.5 per cent. dextrose

solution, 10 c.c. of the reagent being mixed with 20 c.c. of water and heated to boiling, and the sugar solution added, drop by drop, from a burette until the yellow colour of the ferricyanide has disappeared completely. Ten c.c. of the reagent should be reduced by 0.05 gm. of dextrose. The method may be applied to the estimation of lactose in milk, after this has been treated with acetic and picric acids, filtered, and the filtrate neutralised. When the concentration of the sugar solution is much less than 0.5 per cent., it is advisable to boil a portion of the sugar solution with the reagent, and to titrate the ferrocyanide with permanganate in acid solution. One litre of *N*-permanganate solution is equivalent to 368.34 grms. of potassium ferrocyanide, 329 grms. of potassium ferricyanide, or 30 grms. of dextrose.

W. P. S.

Estimation of Maltose and Lactose in the Presence of other Reducing Sugars by Barfoed's Reagent. L. Le Grand. (*Ann. Chim. anal.*, 1921, 3, 240-244.)—Barfoed's reagent is prepared by dissolving 20 grms. of neutral copper acetate in 300 c.c. of water, adding 7.5 c.c. of 38 per cent. acetic acid, and filtering the mixture. It is reduced by dextrose, lævulose, and galactose, but not by maltose and lactose. To estimate the two latter in the presence of other reducing sugars, a mixture of 5 c.c. of the sugar solution (containing not more than 0.1 gm. of total sugars) and 15 c.c. of the reagent is boiled for three minutes, filtered, the cuprous oxide is dissolved in ferric sulphate solution, and the quantity of ferrous sulphate formed is found by titration with standardised permanganate solution. Each c.c. of the latter solution should be equivalent to about 0.006 gm. of copper. Reference to a table gives the quantity of monoses corresponding with the amount of cuprous oxide formed. The total quantity of reducing sugars is estimated in another portion of the original sugar solution by means of Fehling's solution; the difference between the two estimations gives the amount of maltose and lactose present.

W. P. S.

Detection and Estimation of Lævulinic Acid in Foods. L. Grünhut. (*Zeitsch. Untersuch. Nahr. Genussm.*, 1921, 41, 261-280.)—A very sensitive reaction for lævulinic acid (β -acetylpropionic acid) depends on the red coloration obtained when a dilute solution of the acid is treated with sodium nitro-prusside; the coloration is stable in alkaline and in acetic acid solution. Lævulinic acid is generally accompanied in foods by formic acid, and sometimes by acetic acid and lactic acid. To estimate these acids, the sample is acidified with phosphoric acid and extracted with ether; the extraction-flask should contain a quantity of sodium hydroxide solution to prevent volatilisation of the formic acid with the ether vapour. The ethereal extract is then shaken with dilute sodium hydroxide solution, the aqueous portion separated, evaporated to dryness, and the residue dissolved in a definite volume of water. If formic and lævulinic acids alone are present, the formic acid is estimated in a portion of the solution by the mercuric chloride method; another portion (50 c.c.) of the solution is then heated under a reflux apparatus for two hours with 20 c.c. of dilute sulphuric acid and an excess of potassium dichromate solution; the formic acid is oxidised to carbon dioxide and water, whilst the lævulinic acid is oxidised to carbon dioxide, water, and acetic acid, according to the equation:

$C_5H_8O_3 + 7O = C_2H_4O_2 + 3CO_2 + 2H_2O$. The mixture is then distilled, and the acetic acid titrated in the distillate; the amount of dichromate reduced corresponds with the sum of the quantities of formic and acetic acids. When the sample itself contains acetic acid, the amount found in the distillate will be correspondingly more than is indicated by the amount of dichromate reduced. If all four acids (formic, acetic, lævulinic, and lactic) are present, it is advisable to submit the solution to a simple distillation. The distillate will contain almost the whole of the acetic acid and most of the formic acid, but practically no lactic acid, whilst the residual solution will contain all the lævulinic acid, the lactic acid, and a portion of the formic acid. The two solutions are then analysed separately as described, oxidation with dichromate giving the amount of acetic acid corresponding with the lactic acid, as shown by the equation: $C_3H_6O_3 + O_2 = C_2H_4O_2 + CO_2 + H_2O$. To ensure complete oxidation of the lactic acid, the heating must be continued for a further two hours after 30 c.c. of concentrated sulphuric acid have been added to the mixture already oxidised as described.

W. P. S.

Composition of Hollyhock Seed and Oil. R. S. Hiltner and L. Feldstein. (*J. Ind. Eng. Chem.*, 1921, **13**, 635.)—Mature seeds from the pods of different varieties of hollyhock had the following composition: Water, 4.4; ash, 6.9; oil (ether extract), 11.9; crude protein, 21.2; crude fibre, 25.6; and starch, 9.1 per cent. The oil had a greenish-yellow colour; its physical and chemical characters were: Specific gravity at 15.6° C., 0.9275; $n_{D^{25}}$, 1.4722; and iodine value, 119.0. With Halphen's and Bechi's tests the oil gave positive reactions, thus resembling cottonseed oil; the coloration with Halphen's reagent was not obtained when the oil had been heated previously for ten minutes at 250° C.

W. P. S.

Vanilla Paste. (*Connecticut Agric. Exper. Stat. Bull.*, **227**, 1921, 258.)—Vanilla extract of standard quality should contain in 100 c.c. the soluble matter from not less than 10 grms. of vanilla bean (*Standards of Purity for Food Products*, U.S.D.A. Circ. 136 [1919]). The amount of vanillin obtained from 10 grms. of vanilla bean will depend upon the quality of the bean, and may vary from 0.07 to 0.24 gm. (*Conn. Exp. Stat. Report*, 1901, p. 150). A sample of vanilla paste recently examined consisted of gum, glycerin, and sugar, with 0.047 gm. of vanillin. The contents of the tube were stated to be equal to 1 pint of liquid extract of vanilla, and therefore should have contained at least 0.33 gm. of vanillin in the total quantity of 36.7 grms.

Derivatives of Sulphur in Commercial Salvarsan. H. King. (*J. Chem. Soc.*, 1921, **119**, 1107-1120.)—The difficulty experienced by manufacturers in preparing salvarsan of uniform toxicity is due to the substance being amorphous or doubtfully crystalline, and so not readily purified, and secondly to the use of sodium hypsulphite in the reduction process. Solutions of commercial salvarsan in methyl alcohol yield an insoluble deposit, which Fargher and Pyman (*J. Chem. Soc.*, 1920, **117**, 373) regarded as the monohydrochloride of the monosulphamic acid of 3 : 3' - diamino - 4 : 4' - dihydroxyarsenobenzene. An abnormal commercial sample recently examined yielded, when dissolved in methyl alcohol, 24 per cent. of a deposit which

had a similar elementary composition to that of Fargher and Pyman's preparation, but examination of several samples of the insoluble constituent showed that, in general, it consisted of a mixture of 3 : 3' - diamino - 4 : 4' - dihydroxy - 5 - sulphino-arsenobenzene monohydrochloride and "salvarsan sulphate" and "hydrochloride," the two latter probably being present chiefly in the form of the mixed salts. There was no evidence of the presence of a monosulphamic acid. The reduction of the basic property of the salvarsan molecule by the introduction of a sulphinic or sulphonic group has a distherapeutic effect, the influence of the sulphonic group being somewhat greater than that of the sulphinic group. The greater relative efficacy of pure sulphur-free salvarsan, prepared by means of hypophosphorous acid, compared with that of monosulphino-arsenobenzene, is shown by the results (described in detail) obtained by intravenous injection of the preparations into mice.

Detection of Neosalvarsan in a Complex Mixture. M. A. Morel. (*Ann. Chim. anal.*, 1921, 3, 215-216.)—In the case of a mixture of iodides of mercury and potassium, sodium bicarbonate, arrhenal, and vegetable substance of brown colour, supposed to contain 0.6 per cent. of neosalvarsan, it was impossible to detect novo-arsenobenzene owing to the fact that insoluble brown substances masked the colour reactions in acid and alkaline solutions. The mixture was therefore examined (a) alone, (b) after adding a quantity of novo-arsenobenzene equivalent to the amount supposed to be present. Three grms. were acidified with dilute hydrochloric acid (1 to 10) and treated with dimethylaminobenzaldehyde in hydrochloric acid. After twenty-four hours the precipitate was centrifuged off, washed free from acid and arrhenal, and, after destruction of organic matter, tested for arsenic in Marsh's apparatus. Arsenic was thus detected in the mixture to which novo-arsenobenzene had been added (b), but not in the mixture itself (a). A test for formaldehyde was also made in the distillates obtained after treatment with dilute sulphuric acid; formaldehyde was detected in (b), but not in (a). (*Cf. ANALYST*, 1921, 144.)

R. G. P.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Substance yielding Hydrogen Sulphide contained in the Seeds of Certain Papilionaceæ. M. Mirande. (*Comptes rend.*, 1921, 173, 252-253.)—Further investigation supports the view that the source of the hydrogen sulphide yielded by the auto-fermentation of *Lathyrus* and other members of the *Papilionaceæ* family (*ANALYST*, 1921, 291) is a substance of protein character; only part of the total sulphur present is emitted as hydrogen sulphide. T. H. P.

Investigation of the Products of the Enzymic Hydrolysis of Inulin by the Biological Method for the Detection of Dextrose. E. Bourquelot and M. Bridel. (*J. Pharm. Chim.*, 1921, VII., 24, 81-89.)—It has been found previously that, when dextrose is dissolved, either alone or together with other sugars, in 70 per cent. aqueous methyl alcohol, 82.6 per cent. of it is transformed into β -methylglucoside by the action of emulsin (*ANALYST*, 1920, 45, 175, 413). Application of this

method of investigation to the product resulting from the hydrolysis of inulin by the inulase of *Aspergillus niger* shows that this product consists solely of lævulose.

T. H. P.

Occurrence of Manganese throughout the Vegetable Kingdom. G. Bertrand and M. Rosenblatt. (*Comptes rend.*, 1921, 173, 333-336.)—Maumené (*Comptes rend.*, 1884, 98, 1416) gave a list of plants which, contrary to the general rule, he stated to be free from manganese. These included oranges, lemons, onions, and certain cruciferous plants. The authors have investigated these supposed exceptions, and in all cases found manganese to be present in appreciable proportions. For example, in the case of a lemon the rind contained 0.17; the white pith, 0.07; the pulp (without pips), 0.03; and the pips, 0.24 mgrm. of manganese per 100 grms.

T: H. P.

Relative Digestibility of Various Preparations of Proteins from Beans. H. C. Waterman and D. B. Jones. (*J. Biol. Chem.*, 1921, 47, 285-295.)—Experiments *in vitro* and *in vivo* on the proteins of Chinese and Georgia velvet beans (*Stizolobium niveum* and *S. deeringianum*) show that the dialysed proteins are not sufficiently digestible for animal nutrition, but that when the dialysed protein is coagulated by boiling the increased digestibility is sufficient for animal growth. The raw and cooked beans contain dihydroxyphenylalanine, which also is a limiting factor. The digestibility of the proteins *in vitro* in terms of amino-nitrogen is 30 per cent., which is increased to 57 per cent. (determined by digestion with pepsin and trypsin) on coagulation. Parallel experiments *in vivo* lead to the conclusion that whilst there is no definite quantitative relationship between digestibility *in vitro* and *in vivo*, there is a general agreement and rough proportionality, and variations are in the same direction. To account for the indigestibility of certain proteins, it is thought that the amino-acid residues are united in a different order from that in others which are readily assimilated by the animal and which are acted upon by the proteoclastic enzymes.

H. E. C.

Viscous Lactic Streptococcus in Milk. H. Violle. (*Ann. Inst. Pasteur*, 1921, 35, 218-229.)—An objectionable form of lactic streptococcus is frequently present in milk and milk products. When grown on nutrient gelatin it forms large aggregates of crystalline appearance, whilst in saccharine media it produces lactic acid at the expense of most of the sugar, and yields a glairy substance. It is not pathogenic, and may be distinguished from the pathogenic streptococci of man and of the cow by its biological characteristics and the appearance of its cultures. It also differs in certain particulars from ordinary lactic streptococci, and ought, therefore, to be classified with the paralactic or pseudolactic bacteria.

Toxicity of Various Fractions and Combinations of Fractions of Coal Tar Creosote to Wood-destroying Fungi. H. Schmitz and S. M. Zeller. (*J. Ind. Eng. Chem.*, 1921, 13, 621-623.)—The results obtained in the investigation recorded show that there is no visible cessation of growth of either *Lenzites scapiaria* or *Poly-*

porus lucidus when the concentration of the coal tar creosote is below 1 per cent. In the majority of cases, the toxic point (the minimum percentage of creosote which will inhibit completely the growth of the organisms) lies between 2 and 4 per cent. The fact that these results are at variance with those recorded by other observers is due to the methods used and to the disturbing influence of many factors. It is evident that *Lenzites sapinaria* is more resistant to greater concentrations of creosote than is *Polyporus lucidus*. As would be expected, the toxic point varies with the nature of the wood. The most toxic fractions are those which distil between 235° and 270° C. and between 270° and 315° C.; there is no great difference in the toxicity of any of the combinations of the various fractions except when the residue above 355° C. is omitted. This residue is toxic only in very high concentration. The method employed for determining the toxic value consisted, briefly, in impregnating dry sawdust with an alcoholic solution of the creosote, and exposing the impregnated sawdust to the atmosphere for twenty-four hours to allow all the alcohol to evaporate; 3 grms. of the treated sawdust were then saturated with water in a flask, sterilised in an autoclave, cooled, and inoculated with the cultures. The computed percentages were calculated on the dry sawdust, without taking into account the water present during the growth of the organisms; in commercial practice, the amount of impregnation is calculated as lbs. of creosote per cb. ft. of wood. Impregnated timbers, such as railway sleepers, may be placed under conditions, as regards moisture, very favourable to the growth of fungi, and the tests were intended to imitate these natural conditions.

W. P. S.

Toxicity of Wood Preservatives. C. J. Humphrey, R. M. Flemming, and E. Bateman. (*J. Ind. Eng. Chem.*, 1921, 13, 618-621.)—The toxicity of a number of wood preservatives was tested by means of a method described previously (*ANALYST*, 1915, 40, 447), the fungus used, *Fomes annosus*, being incubated at 26° C. on agar medium containing varying quantities of the preservatives. With beechwood creosote, the toxicity of the fractions increased with the boiling-point, that of the fraction boiling at 225° C. having, at a concentration of 0.05 per cent., five times the killing power of the fraction boiling at 180° C. The toxicity of various commercial preservatives corresponded with their cresylic acid content. The most toxic substance examined was a preparation containing about 24 per cent. of dinitrophenol; this, in a concentration of less than 0.01 per cent., inhibited the growth of the organism.

W. P. S.

Yeast Test as a Measure of Vitamin-B. W. H. Eddy, H. L. Heft, H. C. Stevenson, and R. Johnson. (*J. Biol. Chem.*, 1921, 47, 249-275.)—A critical study of yeast stimulation as a method of testing for water-soluble vitamin-B has been made, the technique of Funk and Dubin (*J. Biol. Chem.*, 1920, 44, 487) being used with various extracts; it is found that, owing to lack of a basal medium providing an optimum of all factors except vitamin-B, the test is of little value as an estimation of vitamin content, as compared with rat-feeding experiments, though there is an approximate agreement if the extracts are dilute. When the solution is

very dilute, a destructive effect, due to the alkali, begins to appear, but the results are very contradictory. (Cf. ANALYST, 1921, 146, 246.) H. E. C.

Fat-Soluble Vitamin and Yellow Pigmentation in Animal Fats. H. Steenbock, M. T. Sell, and M. V. Buell. (*J. Biol. Chem.*, 1921, 47, 89-109.)—It was considered that fat-soluble vitamin might be identical with yellow pigments of the carotinoid type, but experiment shows this is not so (cf. ANALYST, 1921, 246). In cod-liver oil there is much fat-soluble vitamin, with very small amounts of yellow pigments. In butter there is a seasonal variation in fat-soluble vitamin content, which does not run parallel with its yellow pigment; but, in general, owing to the content of the cow-feeds, highly pigmented butters are rich in the vitamin. Pale butters are looked upon with suspicion, and may contain as little vitamin as oleo-margarine. In the case of beef-fats, the most pigmented samples are generally the richest in the vitamin. The fact that fat-soluble vitamin is resistant to saponification by severe methods shows that it is not a fat or an ester. H. E. C.

Fat-Soluble Vitamin Content of Peas in Relation to their Pigmentation. H. Steenbock, M. T. Sell, and P. W. Boutwell. (*J. Biol. Chem.*, 1921, 47, 303-308.)—Following the author's theory that fat-soluble vitamin is related to certain yellow-plant pigments, it is shown that in the case of ripe peas, out of six samples investigated the green peas which contained a quantity of yellow pigment were far richer in content of fat-soluble vitamin than yellow peas which contained much less yellow pigment. H. E. C.

Anti-scorbutic Principle of Potato Juice. Bezssonoff. (*Comptes rend.*, 1921, 175, 417-419.)—Raw uncrushed potato has a strong anti-scorbutic power, but the expressed juice has only a weak action, which is only slightly intensified by the addition of the residual *marc*. This instability of the anti-scorbutic principle appears to be due to enzymic action, and, in particular, to an oxydase (Bertrand's laccase). By expressing the juice from the potato in the presence of citric or tartaric acid the action of the laccase is inhibited, and a product which does not turn brown on exposure to air and has a strong anti-scorbutic power is obtained. The juice expressed from new potatoes has a much more pronounced anti-scorbutic acid than that derived from old potatoes.

Effect of Heat and Oxidation upon the Anti-scorbutic Vitamin. R. A. Dutcher, H. M. Harshaw, and J. S. Hall. (*J. Biol. Chem.*, 1921, 47, 483-488.)—Experiments previously made indicated that rhubarb juice and orange juice could be boiled for fifteen minutes without losing their anti-scorbutic power. Analogous results were obtained by Delf (*Biochem. J.*, 1920, 14, 211) with juices extracted from fresh cabbages, Swedish turnips, and oranges, and it was suggested that the stability of the anti-scorbutic vitamin at temperatures above 100° C. might be due to the absence of air. Further experiments have shown that the anti-scorbutic vitamin in fresh orange juice is not destroyed by pasteurisation for thirty minutes at 63° C. in closed vessels, or by boiling for thirty minutes beneath a reflux condenser.

Hydrogen peroxide has some destructive influence on the vitamin when added to the juice at the ordinary temperature, and this action is intensified by heating the mixture of orange juice and hydrogen peroxide at 63° C. and 100° C. Hence, the conclusion is drawn that the anti-scorbutic properties of orange juice are susceptible to oxidation, but, in the absence of oxidising agents, are stable at temperatures up to the boiling-point of the juice.

Estimation of the Diffusible Calcium of Blood Serum. L. v. Meysenbug, A. M. Pappenheimer, T. F. Zucker, and M. F. Murray. (*J. Biol. Chem.*, 1921, **47**, 529-537.)—From 30 to 40 per cent. of the calcium in ox serum is in a non-filterable colloidal form and apparently in combination with proteins (Cushny, *J. Physiol.*, 1919-20, **53**, 391). If blood serum be dialysed against a calcium-free Ringer's buffer solution (NaCl, 36.0; NaHCO₃, 10.08; KCl, 1.68 grms., and water to 2,000 c.c.), there appears to be progressive dissociation of the colloidal calcium compound, until, after seven days, about 90 per cent. of the total calcium will have passed into the dialysate. When, however, calcium is added to the dialysing fluid equilibrium is obtained within twenty-four hours. For this purpose an equal volume of standard calcium chloride solution containing approximately the same amount of calcium as the serum (10.5 to 11 mgrms. per 100 c.c.) was mixed with the dialysing fluid, which, like the serum, had previously been saturated with a mixture of carbon dioxide and air of known tension. The added calcium was deducted from the amount estimated in the dialysate, and the result deducted from the total amount in the original serum. By this method the diffusible calcium of the serum of normal men and dogs was found to vary from 60 to 70 per cent. of the total serum calcium.

Gasometric Estimation of Urea in Urine. R. L. Stehle. (*J. Biol. Chem.*, 1921, **47**, 13-17.)—The following is a rapid and accurate method for estimating urea in urine: Ammonium salts are removed by treatment of 25 c.c. of the diluted urine (1:10) with 4 grms. of permutit for four minutes; 1 c.c. of the filtered ammonia-free urine is transferred to Van Slyke's carbon dioxide apparatus, rinsed in with 1 c.c. of water, followed by 1 c.c. of sodium hypobromite solution. A correction is made for the gases contained in the urine, rinsing water, and hypobromite solution; the solubility of air in diluted urine is the same as in water, and the hypobromite solution contains 0.006 c.c. at 15° C. to 20° C. and 0.005 c.c. at 21° C. to 25° C. Nitrogen is liberated quantitatively from the urea in about half a minute, but not appreciably from other nitrogenous urinary constituents. H. E. C.

Zinc-Potassium Ferrocyanide as a Reagent in Urine Analysis. Thiéry. (*J. Pharm. Chim.*, 1921, **23**, 494-503.)—The xanthine bases and uric acid are estimated together in 200 c.c. of urine and expressed as uric acid. In another 200 c.c. of the urine the xanthine bases are precipitated with 20 c.c. of potassium ferrocyanide solution (150 grms. anhydrous salt per litre), followed by 20 c.c. of zinc acetate solution (112 grms. anhydrous salt per litre acidified with 5 to 10 c.c. of glacial acetic acid), the precipitate is separated, and 120 c.c. of filtrate (= 100 c.c. of urine) are used for the estimation of uric acid by Haycraft and Denigès' method

(*cf.* ANALYST, 1896, 21, 212). The difference between the amount of uric acid found and the amount of (apparent) uric acid found in the original untreated sample is calculated as xanthine. The precipitate given by potassium ferrocyanide and zinc acetate contains all the creatinine and xanthine bases, and most of the alloxan, alloxantin, and allantoin, whilst all the uric acid passes into the filtrate.

R. G. P.

Dinitrosalicylic Acid as a Reagent for the Estimation of Sugar in Urine.

J. B. Sumner. (*J. Biol. Chem.*, 1921, 47, 5-9.)—The method is based on the fact that 3, 5-dinitrosalicylic acid is reduced by glucose to a highly-coloured nitroamino compound, but it is not reduced by other urinary constituents, except when glucose is present. A correction may be applied for the reduction which even normal urine produces after the destruction of its sugar by boiling with caustic alkali. Dinitrosalicylate solution is prepared containing 2 per cent. of the acid and 2 per cent. of sodium carbonate, and also standard glucose solutions containing 1 and 0.5 mgrm. per c.c. preserved with toluene. For the estimation 1 c.c. of the dinitrosalicylate solution and 2 c.c. of 1.5 per cent. sodium hydroxide solution are added to 1 c.c. of the clear urine (which, if containing more than 0.4 per cent. of sugar, must be diluted), and the test tube is plugged with wool and heated in boiling water for five minutes. The cool mixture is diluted in volume to match the colour of a standard similarly prepared from 1 c.c. of the stronger (1 mgrm.) glucose solution. To correct for the reducing action of uric acid and polyphenols in normal urine, heat 1 c.c. of the urine in boiling water for fifteen minutes with 1 c.c. of 3 per cent. sodium hydroxide solution, cool, add 1 c.c. of glucose solution (containing either 1 mgrm. or 0.5 mgrm., according to whether the total reduction was equivalent to more or less than 1 mgrm. per c.c.) and 1 c.c. of dinitrosalicylate solution, and proceed as before. The amount of glucose indicated, less the amount added, gives the reducing value due to the urine as distinct from its sugar. Details are given for preparing the 3, 5-dinitrosalicylic acid.

H. E. C.

Estimation of Veronal. L. Van Itallie and A. J. Steenhauer.

(*Pharm. Weekblad*, 1921, 58, 1062-1068.)—For the separation of veronal from urine, ethyl acetate is preferable to ether, the solubility of veronal in this solvent being 1 : 8.9. A preliminary treatment of the urine with lead acetate or basic lead acetate effects precipitation of some of its constituents and prevents emulsification during the solvent extraction. The use of charcoal for purifying the extracted veronal leads to low yields on account of adsorption; treatment with potassium permanganate gives quantitative results. The method of estimation is as follows: 100 c.c. of urine are mixed with 10 c.c. of lead acetate, and filtered, and 100 c.c. of the filtrate are evaporated to 25 c.c. and acidified with acetic acid. The solution is shaken with twice its volume of ethyl acetate, and filtered, and the residue, after distillation of the solvent, is dissolved in 10 c.c. of boiling water, 5 c.c. of dilute sulphuric acid are added, and the heated mixture is treated with $\frac{N}{10}$ potassium permanganate until the supernatant liquid above the precipitate becomes colourless. The manganese oxide which separates is decomposed with a few drops of hydrogen peroxide, the solution is again

shaken with ethyl acetate, filtered, and evaporated, and the residue dried at 100° C. For estimating veronal in human organs, these are extracted with alcohol in presence of acetic acid. After filtration, and evaporation to about 25 c.c., absolute alcohol is added until no more precipitate forms, and the solution is again filtered and evaporated. The residue is boiled with water, filtered while hot, and shaken twice with ethyl acetate. The further treatment is carried out as described above. W. J. W.

AGRICULTURAL ANALYSIS.

Relation of Hardness to Protein Content of Wheat. H. F. Roberts. (*J. Agric. Research*, 1921, **21**, 507-522.)—There is some indirect evidence in support of the commonly accepted belief that the hardness of wheat is associated with and possibly dependent upon its protein content, and it is generally accepted that hard wheats are relatively high in protein. In experiments to obtain direct evidence on the point three methods of testing wheat for hardness have been tried: (1) The method devised by the author, in which hardness is expressed in terms of the crushing-point (*Kans. Agric. Exper. Stat. Bull.*, **167**, 1910, 371-390); (2) The method of Harper and Peters, in which the hardness is determined by means of a piston with a cutting edge, which is pressed down upon the kernel by weights added directly from above (*Kentucky Agric. Exper. Stat. Bull.*, **113**, 1904, 1-12); and (3) the method of Shaw and Gaumnitz, in which the apparatus consists of ordinary pliers, one arm of which is attached to a wooden block, whilst weights are suspended from the extremity of the upper arm until the wheat grain, placed between the jaws of the pliers, is cut (*Calif. Agric. Exper. Stat. Bull.*, **212**, 1911, 315-394). No correlation could be established between the crushing or breaking point of the kernel in grms. and the percentage of protein, nor could any relationship be found between protein content and either specific gravity or volume of the grain.

ORGANIC ANALYSIS.

Volumetric Estimation of the Methoxy Group. J. Troeger and E. Tiebe. (*Arch. Pharm.*, 1920, **258**, 277-287; *J. Chem. Soc.*, 1921, **119**, ii. 135.)—The substance (0.1 gm.), contained in a boat and placed in a tube fitted with a mantle and inspection window, is heated in a stream of dry hydrogen chloride until bubbles of gas are seen to escape from it. Air is displaced before heating and the methyl chloride produced is collected and measured over 30 to 35 per cent. sodium hydroxide solution (which only absorbs 0.3 c.c. of methyl chloride per hour, compared with 7.0 c.c. in the case of water). It is not necessary to correct the volume of gas for the vapour tension of water, but air derived from the acid used to generate the hydrogen chloride must be determined as unabsorbed gas when the gas is transferred to a eudiometer over water, and a correction must also be made for methyl chloride absorbed by the sodium hydroxide. Results obtained with a number of alkaloids were not absolutely correct, but indicated clearly the number of methoxy groups present. The method is inapplicable to substances which volatilise below the temperature of reaction. The Zeisl method is not applicable to substances containing sulphur; the present

method, however, afforded an approximate result with galipenesulphonic acid, but not in the case of another sulphonated compound.

R. G. P.

Detection of Nitrogen in Organic Compounds. C. D. Zenghelis. (*Comptes rend.*, 1921, 173, 308-310.)—The proportion of the nitrogen present in an organic compound which is converted into ammonia when the compound is heated with soda lime is increased by previous addition of one part of powdered electrolytic copper to the dry soda lime; any hydrogen sulphide or arsenide which may be formed when sulphur or arsenic is present is fixed by the copper. To detect nitrogen in an organic compound, a small quantity of the latter is mixed in a porcelain crucible with the soda lime and copper mixture, a little of the latter being superposed and the crucible covered with a watch-glass, on the lower surface of which hangs a drop of the author's formol-silver nitrate reagent (*ANALYST*, 1921, 381), whilst a little water on the upper surface acts as a condenser. The crucible is placed on a sand-bath or, better, a quartz plate previously heated strongly, and the heating maintained by means of a small flame; when moisture begins to condense round the reagent, the flame is withdrawn somewhat to one side. In presence of nitrogen in the compound, a silver ring soon forms, and later, possibly a mirror. This reaction is extremely sensitive, even when applied to compounds containing nitrogen united with oxygen. Thus, it is capable of detecting nitrogen in 0.00001 gm. of nitrophenol or 0.00005 gm. of acetoxime, nitrosonaphthol, or picric acid. When the substance to be examined distils or decomposes at a relatively low temperature, use is made of a glass tube 20 cm. in length, closed at one end. Into this are introduced successively a little sugar, soda lime-copper mixture, the latter mixed with the substance, and lastly more soda lime-copper mixture. The tube is closed by a stopper carrying a small tube which is bent at right angles and ends just beneath the watch-glass with the reagent. The closed tube is heated in a horizontal position, the heat being applied first to the soda lime-copper mixture, and last to the sugar, the gases from which expel the ammonia from the tube.

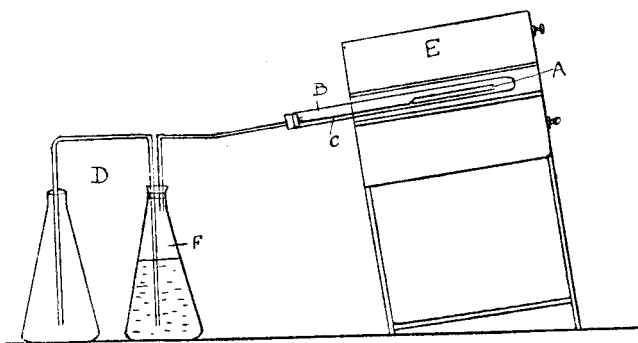
T. H. P.

Decomposition of Potassium Cyanide and Estimation of Formic Acid in the Presence of Hydrocyanic Acid. G. Harker. (*J. Soc. Chem. Ind.*, 1921, 40, 182-185r.)—The yield of gaseous hydrocyanic acid through the action of dilute sulphuric or other acid upon solid potassium cyanide approximates to 30 per cent. of the volume theoretically obtainable, the larger proportion remaining in the solution. The author has carried out experimental work to determine the conditions necessary to ensure the maximum yield of the gas. Warming the reaction mixture leads to decomposition and consequent loss of hydrocyanic acid, but by cooling the mixture and exhausting the apparatus to a pressure of from 16 to 30 mm. of mercury, the loss amounted to 2.9 per cent. only. Hydrolysis of hydrocyanic acid, by heating with either alkali or acid, proceeds slowly; in the former case ammonia and potassium formate are produced quantitatively, but when sulphuric acid is employed the formation of these substances is incomplete. For the estimation of a mixture of formic and hydrocyanic acids the solution is rendered alkaline with potassium hydroxide, and the cyanide estimated as usual by titration with standard

silver nitrate solution. To the solution a second portion of silver nitrate is added exactly equal to that employed for the titration, and the precipitated silver cyanide is removed by filtration. The filtrate is rendered strongly alkaline by the addition of sodium carbonate, gently warmed, and titrated with standard potassium permanganate solution for the estimation of the formic acid present. Experiments made with known quantities of hydrocyanic and formic acids yielded excellent results.

T. J. W.

New Method for Estimating the Volatile Matter yielded by Coals up to Various Temperatures. W. A. Bone and L. Silver. (*J. Chem. Soc.*, 1921, 119, 1145-1152.)—The "American," and the "crucible," and Lessing's methods for estimating volatile matter in coal are open to the objections that the temperature is not accurately regulated, and that air is not excluded. The proposed new method obviates these defects, and indicates precisely the moment when the expulsion of volatile matter ceases. Five grms. of dried coal, which has passed a No. 30 sieve, are placed in the silica tube *A* (12×1.5 cm.), which is supported in its position in the larger silica tube *B* (30×2.5 cm.) by a rod *c*. This "retort" part of the



apparatus is connected with the wash-bottle arrangement *D*, and slowly introduced into the electric furnace *E*, which is inclined at an angle, as indicated, and heated to a temperature of 900° C., controlled by a pyrometer. The heating is continued till no further gas is evolved, as shown by the water in *D* (usually forty minutes), when the retort system is slowly withdrawn from the furnace, but kept in connection with *D*. The water drawn back into *F* prevents the access of air. The loss in weight gives the volatile matter at 900° C. Coals with a high percentage of volatile matter are introduced into the furnace at 500° C. and gradually heated to 900° C. The results obtained are much more concordant than by other methods, and are in general agreement with the results by the American or crucible tests. H. E. C.

Estimation of Volatile Matter in Graphite. O. L. Shinn. (*J. Ind. Eng. Chem.*, 1921, 13, 633-634.)—The most trustworthy results are obtained by heating the graphite in a platinum boat in a current of pure nitrogen; this method is somewhat tedious, and necessitates the preparation and purification of nitrogen, so the following is proposed as a working method: 1 gm. of the sample is placed in a

platinum crucible provided with a well-fitting lid, the crucible is supported about 10 mm. above a good Meker burner and heated for exactly thirty seconds, then cooled, and re-weighed. The ordinary method of determining volatile matter in coals is unsatisfactory when applied to graphite, owing to oxidation of the latter; at the same time, the short-heating method described above cannot be used for coals and cokes.

W. P. S.

Calcium Chloride Method for the Estimation of Water in Gasoline and in Certain Other Substances. C. W. Clifford. (*J. Ind. Eng. Chem.*, 1921, 13, 628-631.)—Of the various methods recommended for the estimation of water in gasoline that depending on a calculation based on the difference in sp. gr. before and after dehydration is inaccurate, as is also the method in which the water is frozen out by chilling the sample to a low temperature. Dilution with oil dissolving much less water than gasoline is difficult, and the only satisfactory principle is selective absorption by a specific reagent. For this purpose calcium chloride has been found to be the most suitable absorbent. In this method a current of air, dried previously by calcium chloride, is passed through the sample at the rate of 5 to 15 litres per hour for one to two hours; if a part of the water has settled out in the sample it must be kept in suspension by frequent shaking or by the current of air itself. The moisture abstracted from the sample is absorbed by passing the air through two calcium chloride tubes. If the sample consists of a volatile liquid, such as gasoline, benzene, chloroform, or carbon disulphide, a current of dry air must be passed subsequently through the calcium chloride tubes for one hour in order to expel vapour from the tubes before these are weighed. The method is also applicable to sugar, certain pigments, flowers of sulphur, and rubber stock, but cannot be used in the case of acetone, pyridine, ethyl alcohol, and glycerol.

W. P. S.

Estimation of Water in Transformer Oils. E. Rengade and J. Clostre. (*Comptes rend.*, 1921, 173, 311-313.)—None of the methods previously described for this estimation giving satisfactory results, the authors recommend the following procedure: 200 c.c. of the oil are placed in a short-necked flask, closed by a three-holed stopper, carrying a thermometer and entry and exit tubes for passing air through the oil. The exit tube is bent downwards at an acute angle, and connected, by means of a small rubber stopper, with a condensing tube, consisting of an inclined branch, ending in a U-tube immersed in a d'Arsonval vessel; the orifice of the condenser may thus be brought within a very small distance of the stopper of the flask. The whole is maintained at 80° C. by means of an oil-bath, which surrounds the flask up to the neck and is continued above as an air-bath of asbestos card, through which pass the thermometer and the tubes. Thus no vapour condenses before reaching the condenser, which is cooled by either liquid air or solid carbon dioxide. Air, dried by means of sulphuric acid or phosphoric anhydride, is passed through the heated oil at the rate of 20 litres per hour. The increase in weight of the condenser during the first fifteen minutes represents solely water, and gives a minimal value for the water content; probably a more accurate value is obtained in thirty minutes,

the small amount of oil then condensing being counterbalanced by the water found to be liberated during the third period of fifteen minutes. T. H. P.

True Oxygen Absorption of Drying Oils. S. Coffey. (*J. Chem. Soc.*, 1921, 119, 1152-1161.)—The so-called "oxygen value" of drying oils calculated from the maximum increase in weight is really only an apparent value, since, owing to the simultaneous evolution of volatile decomposition products, the real oxygen absorption is considerably greater than the percentage increment in weight. For the estimation of the true value a modified form of Genthe's apparatus (*Zeitsch. angew. Chem.*, 1906, 19, 2087) was used. A filter-paper (11 cm. in diameter) was saturated with a "standard" solution of linseed-oil in petroleum spirit (b.pt. 40° to 60° C.), and suspended in the apparatus, which was then closed, completely filled with hydrogen, and placed in a thermostat at 100° C. When the temperature of the film of oil was constant, the apparatus was rapidly filled with oxygen, and manometer readings were taken at intervals. The volume of the apparatus was determined by measuring the volume of water required to fill it, corrections being applied for the volume of absorbents and manometer leads. Broken pumice, soaked in sulphuric acid, and a stick of sodium hydroxide wrapped in copper gauze were used as absorbents. The oxygen absorption of pure linseed oil (iodine value, 184.1) thus determined ranged from 28.6 to 29.2 per cent., and that of the fatty acids of the same oil from 30.05 to 30.20 per cent. In most cases the error in the estimation is within 1 per cent. The true oxygen absorption is much higher than the values hitherto recorded, and, unlike the apparent oxygen absorption, is a definite quantity for a given oil. The ratio of oxygen absorption to iodine absorption is 0.156. The oxidation is thus not a simple molecular autoxidation, for the oxygen absorption is about 25 per cent. higher than it would be on this assumption, which requires an oxygen absorption of 22.7 per cent. This leaves an *excess* oxygen absorption of 6.0 per cent., which may give rise to the volatile acid products, and it is possible that a relationship might be traced between the amount of oxygen in these products and the *excess* oxygen absorption.

Linolenic and Hexabromostearic Acids. S. Coffey. (*J. Chem. Soc.*, 1921, 119, 1306-1310.)—Hehner and Mitchell (*ANALYST*, 1898, 23, 310) prepared linolenic acid from purified linolenic acid hexabromide (m.pt. 180° to 181° C.) and found it to have sp. gr. 0.9288 at 15.5°/15.5° C. On re-bromination it yielded only 46 to 50 per cent. of the theoretical amount of hexabromide. Erdmann and Bedford (*Ber.*, 1909, 42, 1324) showed that this was due to the fact that linolenic acid obtained by reducing linolenic acid hexabromide with zinc consists of a mixture of α - and β -linolenic acids, only the α -modification being of natural occurrence. More recently Erdmann (*Zeitsch. physiol. Chem.*, 1911, 74, 179) claims to have prepared pure α -linolenic acid by fractional crystallisation from alcohol of the zinc salts of the liquid fatty acids of linseed oil. The zinc salt $(C_{18}H_{29}O_2)_2Zn, \frac{1}{2}ZnO$, melted at 72° to 73° C., and the yield was nearly theoretical, corresponding with 18 per cent. of linolenic acid in the liquid fatty acid of linseed oil. The author, however, has been unable to isolate pure zinc linolenate, the differences in the solubility of zinc linolenate and

zinc linolate being not nearly so pronounced as stated by Erdmann; in fact, the most soluble product was a mixture consisting mainly of zinc linolate. The statements of Erdmann and Bedford (*loc. cit.*) concerning the mixtures of α - and β -acids obtained from the hexabromide have been confirmed. Pure alkaloidal salts of hexabromostearic acid have been prepared. The strychnine salt crystallises in flat microscopic rhombs (m.-pt. 185° C.), the morphine salts in rhombs (m.-pt. 181° to 182° C.), and the narcotine salt melts at 184° C.

Characterisation of Amylase Solutions. H. von Euler and O. Svanberg. (*Zeitsch. physiol. Chem.*, 1921, **112**, 193-230; *J. Chem. Soc.*, 1921, **120**, ii. 528.)—After reviewing the literature on the subject, a new "absolute" measure for the activity of amylase preparations is advocated which is analogous to the author's method for invertase (*Zeitsch. physiol. Chem.*, 1919, **106**, 201). The reaction velocity is measured with soluble starch (Lintner), previously boiled, at concentration of 0.72 to 2.8 per cent. and temperature 37° C. The P_H value should be approximately 5, and the enzyme concentration such that the constant (*Sf.*) is between 0.004 and 0.08. The constant *Sf.* then equals ($K \times$ maltose/enzyme preparation); the concentrations are expressed in grms. Here *K* is the velocity constant of the unimolecular reaction by which the hydrolysis proceeds to the formation of a maximum quantity of maltose. Experiment shows that 1,000 Lintner units = 26 *Sf.*, and 1,000 of Sherman's units = 38.5 *Sf.*

H. E. C.

Significance of "Lignin" Colour Reactions. E. C. Crocker. (*J. Ind. Eng. Chem.*, 1921, **13**, 625-627.)—The colorations obtained with phloroglucinol, *p*-nitroaniline, etc., are due to the presence of traces of an aldehyde which usually, if not always, accompanies lignin; it appears that there is one single aldehyde in wood, or that one aldehyde predominates, and that this aldehyde is probably coniferyl aldehyde. Vanillin or furfural, if present, constitutes only a small fraction of the aldehyde content of wood. Several non-aldehydic substances which yield reactions similar to those of "lignin" are shown to contain traces of aldehydes which are responsible for the colour formation; in the case of clove oil and sassafras oil, the aldehyde seems to be identical with that of wood. The Maule reaction (red coloration when wood is treated successively with permanganate, dilute hydrochloric acid, and ammonia) is essentially a chlorination of some wood constituent, followed by alkaline treatment; the red coloration is obtained generally in the case of deciduous woods, an indefinite brown coloration being given by coniferous woods, and the reaction may be of value in differentiating these woods.

W. P. S.

Estimation of Cresol by the Phenol Reagent of Folin and Denis. R. M. Chapin. (*J. Biol. Chem.*, 1921, **47**, 309-314.)—Factors affecting the accuracy of Folin and Denis's method of estimating phenols (*J. Biol. Chem.*, 1915, **22**, 305) are discussed, and the use of the following procedure and factors is recommended: Three c.c. of the silico-tungstic acid reagent made up by Wu's method (*J. Biol. Chem.*, 1920, **43**, 189) are added to a quantity of liquid containing about 0.5 mgrm. of phenol to produce the maximum colour, and the mixture diluted; then 5 grms. of

sodium bicarbonate are added and the mixture allowed to stand for thirty minutes before the addition of 5 c.c. of 10 per cent. sodium sulphite solution; a further fifteen minutes should be allowed before colorimetric comparison with the standard to allow of full development of the colour. When phenol is employed as a standard in the estimation of cresols the following are the factors equivalent to 1 part of phenol: Ortho-cresol, 1.19; *m*-cresol, 1.09; *p*-cresol, 1.22; or, for commercial cresol (35 : 40 : 25), the factor is 1.16. For the determination of phenolic preservatives in sera, 1 c.c. of the sample is treated in a 300 c.c. flask with 125 c.c. of water, 4 c.c. of 1 : 3 sulphuric acid, 4 c.c. of 12 per cent. silico-tungstic acid, and fragments of hot pumice. The contents are distilled into a 200 c.c. flask; when 100 c.c. have passed over, a further 100 c.c. of water are added to the contents in the distillation flask, and the distillation continued till 200 c.c. have passed over. The phenol in the distillate is then determined in 25 or 50 c.c., depending on whether phenol or cresol is the preservative present.

H. E. C.

Volumetric Estimation of Aminonaphthol-mono- and di-sulphonic Acids. G. R. Levi. (*Giorn. Chim. Ind. Appl.*, 1921, 3, 297-302).—Aminonaphthol-sulphonic acids may be titrated with sodium nitrite in acid solution, and also with a diazo-compound, such as diazobenzene or diazotised *p*-nitrobenzene, and the author has determined the nitrite number and the diazo number for the following acids of importance in the azo-dye industry: *M*-acid ($\text{NH}_2 : \text{OH} : \text{SO}_3\text{H} = 1 : 5 : 7$), *S*-acid (1 : 8 : 4), γ -acid (2 : 8 : 6), *J*-acid (2 : 5 : 7), *R*-acid (2 : 3 : 6), 2-Sacid ($\text{NH}_2 : \text{OH} : \text{SO}_3\text{H} : \text{SO}_3\text{H} = 1 : 8 : 2 : 4$), *H*-acid (1 : 8 : 3 : 6), *K*-acid (1 : 8 : 4 : 6), and 2 *R*-acid (2 : 8 : 3 : 6). The nitrite number is determined by running $\frac{N}{2}$ -sodium nitrite solution slowly into a fresh solution or suspension of the acid containing mineral acid. Considerable excess of the latter does not increase the velocity of diazotisation appreciably, but this is influenced by rise of temperature, so that it is advantageous to carry out the first part of the diazotisation at 10° to 12° C., and then to raise the temperature to 25° or even 30° C. If the sulphonic acid be of good quality, the difference between the nitrite and diazo numbers should not exceed 0.5 or 0.2 per cent. for dry and pasty products respectively.

T. H. P.

Analysis of Wool Waste or Refuse. A. Demolon. (*Ann. Chim. anal.*, 1921, 3, 244-246).—Discrepancies in the estimation of total nitrogen in wool waste are probably due to difficulty of obtaining a representative sample for the estimation, and to insufficient heating during the digestion with sulphuric acid in the Kjeldahl method. It is recommended that about 10 grms. of the material be heated gently in a weighed porcelain basin with 15 c.c. of sulphuric acid until a fluid mass is formed; calcium sulphate is then added gradually, with stirring, so as to produce a dry powder and increase the total weight to 80 grms. Eight grms. of this powder (equal to 1 gm. of original material) are then taken for the estimation of the total nitrogen. The heating with sulphuric acid (Kjeldahl method) should be continued for two hours after a clear solution has been obtained.

W. P. S.

INORGANIC ANALYSIS.

Note on the Gasometric Determination of Nitrogen. R. L. Stehle. (*J. Biol. Chem.*, 1921, **47**, 11.)—In the process previously described (ANALYST, 1921, 110), the absorption of oxygen by pyrogallate is unnecessary if copper sulphate be omitted from the Kjeldahl digestion, as then only nitrogen is liberated on treatment with the sodium hypobromite. H. E. C.

Estimation of Metals as Sulphides. L. Moser and A. Schattner. (*Chem. Zeit.*, 1921, **45**, 758-759.)—The quantitative estimation of metals in their compounds is effected by precipitating them as sulphide, which is then obtained in a form suitable for weighing by heating in a current of hydrogen sulphide. The method gives excellent results with zinc, cadmium, manganese, and silver. In the case of iron, however, it is necessary to heat the sulphide in presence of both hydrogen sulphide and hydrogen in order to avoid the formation of higher sulphides. Zinc sulphide is obtained in a stable amorphous or partially crystalline form by heating it for thirty to forty-five minutes in the current of gas at 570° to 590° C., the temperature being controlled by means of a thermo-element placed near the crucible. Zinc oxide, either dried or ignited, may be quantitatively converted into zinc sulphide by heating it to redness in hydrogen sulphide. The same method is also applicable to cadmium oxide or carbonate, to manganese sulphate, or the oxides Mn_3O_4 and MnO_2 , and to silver chloride, nitrate or oxide, the respective sulphides being quantitatively obtained in each case. W. J. W.

Zinc Purpurate as a Reagent for Mercury Salts. G. Denigès. (*Bull. Soc. Pharm. Bordeaux*, 1921; *Ann. Chim. anal.*, 1921, **3**, 252-253.)—The reagent is prepared as follows: Two grms. of uric acid are heated with 2 grms. of nitric acid until solution is complete; 2 c.c. of water are then added, the mixture again heated and diluted with water to 100 c.c.; 10 c.c. of this solution are boiled for five minutes with the addition of 2 grms. of zinc. The solution becomes orange-yellow owing to the formation of zinc purpurate. A characteristic pink-coloured precipitate of mercury purpurate is obtained when a solution containing a trace of mercury (*e.g.*, 0.4 mgrm. per c.c.) is treated with a few drops of the reagent followed by a small quantity of sodium acetate solution. Silver salts, under the same conditions, yield a violet-coloured precipitate. W. P. S.

Compounds of Halogenated Derivatives of Mercury and Thallium.
[Separation of Mercury from Thallium.] J. Barlot and J. Pernot. (*Comptes rend.*, 1921, **173**, 232-234.)—The separation of mercury from thallium involved in the analysis of compounds of the type, $HgCl_2$, $TlCl$, may be effected by taking advantage of the fact that thallos sulphide, but not mercuric sulphide, dissolves easily in dilute mineral acid. The double salt is transformed into a mixture of nitrates by treatment with nitric acid, the liquid being approximately neutralised with ammonia and the two sulphides precipitated by excess of ammonium hydrosulphide. After filtration, the precipitate is treated with 10 per cent. nitric acid, which dissolves the thallos

sulphide; the liquid is boiled to expel hydrogen sulphide, neutralised exactly with ammonia, and treated with potassium iodide, the thallium being weighed as thallose iodide; the solubility of the latter is diminished by the presence of excess of potassium iodide.

T. H. P.

Quantitative Separation of Tin and Antimony. M. Mouret and M. J. Barlot. (*Bull. Soc. Chim.*, 1921, 29, 743-745.)—The following modification of Plato's method is recommended: The alloy is dissolved in *aqua regia*, the solution evaporated almost to dryness, and the residue dissolved in hot concentrated hydrochloric acid. An equal volume of water is added, then 50 c.c. of a solution containing not less than 4 grms. of crystalline orthophosphoric acid, and the solution is heated to 80° to 90° C., and a current of hydrogen sulphide is passed through it for thirty minutes. The antimony is completely precipitated as sulphide, and, after standing fifteen minutes, the precipitate may be filtered on a Gooch filter of double thickness, washed with hydrogen sulphide water, and dried in a current of carbon dioxide. Alternatively, the antimony sulphide may be electrolysed in a solution containing hydroxylamine hydrochloride, a current of 0.5 ampère at from 2 to 2.5 volts being used. The phosphoric acid solution containing the tin is heated to eliminate hydrogen sulphide, and a portion, containing not more than 0.04 gm. of tin, is treated with "cupferron," and continually stirred until the precipitate is flocculated. After filtration, the residue is washed with cold water, dried at a low temperature in a vacuum desiccator, ignited, and weighed. The method described is rapid, and the results obtained are in close agreement with the quantities of each metal present in an alloy.

T. J. W.

The Dimethylglyoxime Reactions of Iron and Cobalt. W. Vaubel. (*Zeitsch. öffentl. Chem.*, 1921, 27, 163-164.)—Ferrous salts yield an intense Bordeaux red coloration when treated with dimethylglyoxime solution and ammonia; the coloration is distinct from the red colour and precipitate given by nickel salts. Ferric salts also give the reaction, but only after reduction by a trace of ammonium sulphide, added after the dimethylglyoxime. If 20 c.c. of a cobalt salt solution, containing 2 mgrms. of cobalt, be mixed with 20 c.c. of dimethylglyoxime solution, and the mixture diluted to 50 c.c. and treated with 10 drops of ammonium sulphide solution and 10 drops of ammonia, a blue-violet to deep red coloration is obtained. This reaction may be used for the detection of cobalt in the filtrate from the precipitate obtained with nickel.

W. P. S.

Estimation of Vanadium in Steels and Iron Alloys. L. Rolla and M. Nuti. (*Giorn. Chim. Ind. Applic.*, 1921, 3, 287.)—Vanadium is quantitatively precipitated from a solution containing about 1 per cent. of free hydrochloric or sulphuric acid by cold 4 per cent. "cupferron" solution; the precipitate is filtered off, washed with 2 per cent. hydrochloric or sulphuric acid solution containing 2 per cent. of the 4 per cent. cupferron solution, dried in an oven at about 70° C., burned and calcined to constant weight, and weighed as V_2O_5 . The separation of iron and chromium from vanadium may be effected by means of boiling sodium hydroxide solution, the

first two elements being eliminated as hydroxides; in order to avoid the formation of a voluminous precipitate, the bulk of the iron may be removed previously by means of ether, as in Campagne's method. Tungsten may be eliminated as tungstic anhydride in the ordinary way. Since vanadium may be separated also from phosphorus in a solution containing hydrochloric or sulphuric acid, the method may be applied to ferrovandium: About 0.5 gm. is disaggregated in an iron crucible by treatment with six times its weight of sodium peroxide, the mass being taken up in water, acidified, and freed from silica; the filtrate, free from iron, is treated with cupferron as described above. In the case of steels sodium peroxide cannot be employed, since the large quantity of alkali salts formed would necessitate such dilution that the resulting solution would not contain a measurable amount of vanadium. Use is made, therefore, of the solution in hydrochloric acid, the silica and tungsten being eliminated by filtration, and the iron by means of ether. The liquid containing the chromium and vanadium is then boiled with sodium carbonate and filtered, the filtrate being treated either with lead acetate or with acetic acid and lead nitrate. The lead vanadate thus precipitated is collected and dissolved in dilute hydrochloric acid, the solution being evaporated with concentrated sulphuric acid, the residue dissolved in water, and the solution filtered and treated with "cupferron" to estimate the vanadium.

T. H. P.

Detection and Estimation of Potassium as Picrate. S. Minovici and A. Jonescu. (*Bull. Soc. Chim. România*, 1921, 3, 25-33; *J. Chem. Soc.*, 1921, 120, ii., 520.)—Potassium is precipitated quantitatively in the form of yellow crystals by a saturated solution of picric acid in 95 per cent. alcohol containing 5 per cent. of glycerol; the crystals are collected, washed with ether, dried under reduced pressure, and weighed. Alternatively, the precipitate may be dissolved and titrated with quinine hydrogen sulphate, which in dilute solution precipitates picric acid completely. The method is adaptable to the microchemical estimation of potassium, being sensitive to 0.01 mgrm. Sodium and ammonium salts do not yield crystals.

H. E. C.

Estimation of Bromine in Saline Waters. P. Lebeau and M. Picon. (*Bull. Soc. Chim.*, 1921, 29, 739-743.)—The following modification of L. Figuier's method (*Ann. Chim. Phys.*, 1851, 33, 303) is described: In order to avoid the addition of excess of chlorine in the estimation a preliminary test is made by adding increasing amounts of an aqueous solution of chlorine containing 0.5 gm. per litre to a series of tubes each containing 1 c.c. of the water under examination. The volume of chlorine solution required to cause a faint decrease in the coloration due to the liberated bromine will be approximately double the volume required to displace the whole of the bromine present. In the actual estimation a volume of water, containing approximately 100 mgrms. of bromine, is introduced into a separating funnel, and 5 c.c. of 10 per cent. hydrochloric acid are run in, followed by a quantity of 0.5 per cent. chlorine solution, calculated from the preliminary experiment. Fifteen c.c. of chloroform are then added, and the liquid gently agitated to avoid emulsification, and, after separation, run into a flask containing 10 c.c. of 10 per cent. potassium

iodide solution. The saline solution is washed with further portions of chloroform until these remain colourless, when from 0.3 to 0.5 c.c. of chlorine solution is added to the contents of the separating funnel, and the chloroform extraction repeated until bromine ceases to be liberated. The chloroform extracts are shaken with the potassium iodide solution, and the whole titrated with $\frac{N}{10}$ sodium thiosulphate solution. The quantity of saline solution used should not exceed 25 c.c., and, if containing only a small quantity of bromine, should be concentrated by evaporation. The results obtained are accurate within 1 per cent. of the total bromine present.

T. J. W.

Analysis of Fibro-Cement. B. J. Smart and P. C. Pecover. (*J. Soc. Chem. Ind.*, 1921, 40, 185-186T.)—Abnormal values being obtained in the determination of the lime value of the cement contained in an asbestos cement by extraction with 20 per cent. hydrochloric acid, investigation by the authors showed that the residual asbestos had adsorbed silica from the solution, and that this silica was capable of removal by extraction of the asbestos with alkali. Five grms. of the coarsely ground material were covered with 200 c.c. of water in a porcelain basin, and 40 c.c. of hydrochloric acid gradually added, with constant stirring, after which the mixture was allowed to stand for twenty-four hours. The whole was filtered, and the residue washed with 20 per cent. hydrochloric acid until the washings were colourless, after which it was dried, ignited, and weighed. The filtrate and washings were diluted to a known volume, and an aliquot part used for the estimation of calcium, silica, etc. From the known calcium-silica ratio of the cement used the proportion of silica adsorbed was estimated, this being confirmed by extraction of the residual asbestos with caustic alkali and estimation of the silica dissolved in the solution. By this method the asbestos found in three estimations in a cement prepared in the laboratory and containing 16.7 per cent. amounted to 16.6, 16.4, and 16.6 per cent.

T. J. W.

ERRATA.

Production and Testing of Zirconia (*ANALYST*, 1921, 344).—For "sulphite," line 19, read "sulphur trioxide," and for "arsenious acid," line 24, read "arsenic."

PHYSICAL METHODS, APPARATUS, ETC.

Apparatus for the Industrial Analysis of Gases. G. Ardoyer. (*Comptes rend.*, 1921, 173, 237-238.)—This apparatus consists of a vertical measuring tube, which is divided into tenths of a c.c., is surrounded by a water-jacket, and is connected at its lower extremity with a levelling tube containing salt water; the upper end is fused to a short, narrow, horizontal tube carrying a three-way cock, from which the graduation commences, and, beyond the cock, a small funnel, the third way being left free. The gas is introduced into the measuring vessel through this third way, which is connected in turn with the various Orsat pipettes, after the volume of the gas has been measured. An explosion chamber or a combustion tube charged with metallised asbestos and heated with a small flame may also be connected. The apparatus is simple, strong, and portable, and yields accurate results.

T. H. P.

THE INSTITUTE OF BREWING RESEARCH SCHEME.

THIS scheme, organised and financed by the Institute of Brewing for the solution of problems connected with the brewing and allied industries, has given an impetus to investigation in various branches of science which have been somewhat neglected during recent years. Systematic work in connection with hops is now in progress. The culture of new varieties, manuring experiments, and chemical analysis of hops are conducted under a grant from the Institute and the Kent County Council at the East Malling Fruit Research Station and at Wye Agricultural College, whilst four experimental drying kilns have been recently completed for the Institute at Paddock Wood.

The estimation of the resin content of hops was carried out as follows: Whole hops were soaked in petroleum spirit for twelve hours at the ordinary temperature, and digested on a water-bath for a further five hours. The mixture was filtered, the filtrate diluted to a definite volume, an aliquot portion dried, and the residue of soft resins was weighed. The total (hard and soft) resins were estimated in a similar manner by substituting ether for the petroleum spirit previously used. The total resins in twenty-two varieties over a period of four years ranged from 10.6 to 22.1 per cent., whilst the soft resins varied between 6.7 and 11.6 per cent. The percentage of hard resin present appears to be characteristic for a particular variety of hop.

Manuring experiments in the field will be conducted upon separate plots, with the following variations in treatment: (a) Unmanured, (b) complete mineral manure, (c) complete mineral manure with and without (i.) organic manure (dung), (ii.) nitrogen, (iii.) phosphorus, and (iv.) potassium.

Chemical investigations are being carried out by Dr. Pyman in the College of Technology, Manchester, with the object of isolating and estimating the constituent or constituents to which the hop owes its antiseptic or preservative qualities.

Research upon timber used in the construction of casks is in progress from the botanical, biological, and chemical standpoints. American white oak, owing to the presence of starch in the heartwood, is liable to lead to the development of foreign organisms and the deterioration of beer, whilst American red oak is unsuitable for use in barrels, owing to its permeable nature. The chemical investigation consists of: (a) The estimation of variations in extract yielded to various solvents; (b) the conditions under which such extracts are taken up by beer; and (c) the determination of whether such extracts are capable of communicating undesirable flavours to beer. Extracts of various woods obtained up to the present by the use of benzene, alcohol, and water, show large variations in quality.

T. J. W.



STATUTORY RULES AND ORDERS, 1921, No. 1305.

FOOD CONTROL.

THE SALE OF FOOD ORDER, 1921, DATED AUGUST 16, 1921, MADE BY THE BOARD OF TRADE UNDER THE MINISTRY OF FOOD (CONTINUANCE) ACT, 1920 (10 AND 11 GEO. V., c. 47, AND THE MINISTRY OF FOOD (CESSATION) ORDER, 1921.

In exercise of the powers conferred upon them by the Ministry of Food (Continuance) Act, 1920, and the Ministry of Food (Cessation) Order, 1921, and of all other powers enabling them in that behalf, the Board of Trade hereby order as follows:

PART I. : BREAD.—1. All bread (other than bread sold for consumption on the premises of the seller) shall be sold by weight and not otherwise. 2. No loaf of bread shall be sold unless its weight be 1 pound or an even number of pounds. 3. No roll of bread exceeding 2 ounces in weight shall be sold. 4. Bread which may not under the foregoing provisions of this Order be lawfully sold shall not be offered or exposed or carried for sale or delivered under a contract for sale. 5. Any person authorised by a local authority or any Inspector of Weights and Measures may require any person offering or exposing or carrying any bread for sale or delivering any bread under a contract for sale to weigh such bread in the presence of such person or Inspector or permit such person or Inspector to weigh such bread.

PART II. : TEA.—6. (a) All tea sold by retail, whether contained in a package or not, shall be sold by net weight, and in ounces or pounds or in multiple of ounces or pounds and not otherwise, provided that this shall not apply to tea sold in quantity of less than 2 ounces.

(b) A person shall not place on any package of tea intended for sale or on any wrapper, band, or label affixed thereto, any statement as to weight, or sell or offer or have in his possession for sale by retail any package bearing on the package or on any wrapper, band, or label affixed thereto any statement as to weight, unless in either case such statement is a true statement of the net weight of the tea contained in such package.

PART III. : LABELLING OF IMPORTED PRODUCE.—7. (a) A person shall not expose for sale by retail any imported meat or any imported bacon, ham, or lard unless the article bears at the time of exposure for sale a label with the word "imported" or with a word or words disclosing the country of origin of the article clearly printed thereon, so as to be easily readable by the customers:

Provided that where only imported meat or only imported bacon, ham, or lard is exposed for sale on any slab, rail, or counter it shall be sufficient compliance with the requirements of this clause if the slab, rail, or counter bears in a conspicuous position such a label:

Provided also that where all the meat for the time being on sale in any premises is imported meat, it shall be a sufficient compliance with the requirements of this clause if there is exhibited on the premises in a conspicuous position, and so as to be easily readable by the customers, a notice stating that imported meat only is on sale:

Provided also that where pieces of home-killed and imported meat, not exceeding in any case 1 pound in weight, are exposed for sale on a slab, tray, or counter, the foregoing provisions of this clause shall not apply to the imported meat so exposed, provided that the slab, tray, or counter bears in a conspicuous position a notice containing the words "mixed home-killed and imported meat" clearly printed thereon.

(b) On the occasion of a sale, other than a sale by retail, of any imported bacon, ham, or lard, the seller shall give to the buyer an invoice accurately stating: (i.) that the bacon, ham, or lard is imported, and (ii.) in the case of bacon or ham, the country of origin.

(c) For the purposes of this clause: "Meat" shall include beef, mutton, lamb, pork, and veal, but shall not include bacon or ham, or cooked, canned, or potted meat, sausages or offals. "Bacon" shall include shoulders and picnics, but shall not include pickled pork or cured pigs' heads. "Lard" shall not include neutral lard or compound lard. "Imported" shall mean with respect to any bacon, ham, or lard, cured or manufactured outside the United Kingdom, or cured or manufactured in the United Kingdom from pigs raised outside the United Kingdom. Lard which contains any imported lard shall be deemed to be imported lard.

8. A person shall not sell or offer or expose for sale, whether by wholesale or retail, as fresh eggs or new-laid eggs or under any description of which the words "fresh" or "new-laid" form part, any eggs which have been imported into the United Kingdom, unless the description also includes the word "imported" or a word or words disclosing the country of origin.

PART IV. : JAM.—9. A person shall not sell or offer or expose for sale any jam unless the same complies with the following provisions: (a) The water-soluble extract of jam shall not be less than 65 per cent. of the jam. (b) Not more than 10 per cent. of the jam measured by weight shall consist of added fruit juice. (c) Where more than one variety of fruit or vegetable is used in the making of a jam [other than in the form of added fruit juice not exceeding the quantity specified in sub-clause (b)] each such variety must be mentioned in the description, but so that where any fruit or fruits contained in such jam is less than 25 per cent. of the total fruit content, the name of such fruit or fruits shall be prefaced in the description by the words "Flavoured with" in such form as to be easily readable by the buyer. (d) The provisions of sub-clause (c) shall not apply to jam sold under the description of "Mixed Jam" or "Mixed Fruit Jam," (e) Notwithstanding the provisions of sub-clause (c), jam made from rhubarb and preserved ginger may be sold under the description "Rhubarb and Preserved Ginger," provided that the quantity of preserved ginger contained in such jam is not less than 20 per cent. of the total fruit content.

10. Without prejudice to the provisions of Clause 9, a person shall not sell or offer or expose for sale any article under the description of marmalade or under any description of which the word "marmalade" forms part, unless (a) only citrous fruits, citrous fruit juices, and sugar or other sweetening substances have been used in the making thereof; or (b) each variety of fruit or vegetable used in the making thereof is mentioned in the description.

11. (a) A person shall not sell or offer or expose for sale or deliver pursuant to any contract of sale, whether by wholesale or by retail, any jam in a container unless such container bears (i.) the name and address of the manufacturer of the jam, or in the case of imported jam, a word or words disclosing the country of origin; provided that this sub-clause shall not apply to a sale of jam where the jam has been manufactured for a retailer under a contract to manufacture made between the manufacturer and the retailer and is sold by the retailer as manufactured for himself, but in any such case, in any proceedings against the retailer in respect of a sale or offer or exposure for sale of the jam, the provisions of Clause 21 of this Order shall not be available as a defence; (ii.) except where the jam is packed in usual containers with a net content of 1 pound, 2 pounds, 3 pounds, or 7 pounds, the guaranteed net weight of the contents.

(b) Notwithstanding the preceding provisions of this clause, jam imported from any of the British Dominions beyond the seas may be sold in a container which does not bear the guaranteed net weight of the contents, provided that in the case of a wholesale sale the seller shall give to the buyer an accurate statement in writing showing the net weight of the contents of the container, and that in the case of a retail sale the seller shall keep posted in a conspicuous position, so as to be easily readable by all customers throughout the time during which the jam

is being sold or exposed for sale, a notice showing in plain words or figures the net weight of the contents of the container, in accordance with the statement given to him by the wholesaler.

(c) A person shall not make or knowingly connive at the making of any false statement as to the matters set out in this clause, or alter or deface any label or other writing regarding such matters.

12. Every person selling jam shall produce all books of account, records, and invoices to any person authorised by the local authority to inspect the same, so far as necessary for the purpose of showing whether or not he is complying with the provisions of this Part of this Order as respects content, weight, description, and labelling.

13. For the purposes of this Part of this Order the expression "Jam" shall include jelly, conserve, and marmalade.

PART V. : FATS.—14. A person shall not sell or offer or expose for sale as dripping any substance unless such substance shall have been manufactured by a process other than the acid process from raw-beef fat or raw-mutton fat or beef or mutton bones, and does not contain more than 2 per cent. of free fatty acids or more than 1 per cent. in all of water and substances other than fat.

15. A person shall not sell or offer or expose for sale any substance (except lard, neutral lard, oleo oil, beef and mutton stearine and Premier Jus) manufactured from raw-beef fat or raw-mutton fat or beef or mutton bones, or from any other animal fat, which does not comply with the requirements of the preceding clause, otherwise than under the name and by the description of "Technical Tallow."

16. A person shall not sell or offer or expose for sale as an edible fat any mixture or compound (other than butter, margarine, or dripping) manufactured wholly or partly from oils or fats, which contains more than 0·5 per cent. of free fatty acids or more than 0·5 per cent. in all of water and substances other than oil or fat.

17. A person shall not manufacture or sell or offer or expose for sale any margarine which does not contain at least 80 per cent. of oil and fat.

PART VI. : GENERAL.—18. (a) Every local authority is hereby authorised to execute and enforce the provisions of this Order within their area, and except in Scotland to institute proceedings for any offences against this Order. (b) For the purposes of Parts I. and II. of this Order the local authority shall be the local authority for the purposes of the Weights and Measures Acts, 1878 to 1919. (c) For the purposes of Parts III., IV., and V. of this Order the local authority shall be any local authority authorised to appoint an analyst for the purposes of the Sale of Food and Drugs Acts, 1875 to 1907.

19. A person authorised in that behalf by a local authority to procure for analysis samples of any article to which Part IV. or Part V. of this Order applies shall have all the powers of procuring samples conferred by the Sale of Food and Drugs Acts, 1875 to 1899, and a person selling or exposing for sale any such article shall, on tender of the price for the quantity which he shall reasonably require for the purpose of analysis, sell the same to such authorised person accordingly.

20. In any proceedings in respect of an infringement of this Order the production of the certificate of the principal chemist of the Government laboratories, or of an analyst appointed under the Sale of Food and Drugs Acts, 1875 to 1907, shall be sufficient evidence of the facts therein stated unless the defendant shall require that the person who made the analysis shall be called as a witness. The certificate shall, so far as circumstances permit, be in the form required by the Sale of Food and Drugs Act, 1875.

21. (i.) If in any proceedings for an infringement of any of the provisions of this Order (other than the provisions of Part I. or Part III.) the defendant proves (a) (1) in the case of proceedings under Part II., that he purchased the article in the package in which he sold the same or offered or exposed it for sale, and with a written warranty of the net weight of the article con-

tained in the package, or with a statement on the package of such net weight, (2) in the case of proceedings under Part IV., that he sold or offered or exposed for sale the article in the container in which he bought it, (3) in the case of proceedings under Part V., that he bought the article with a written warranty as to such of the matters referred to in Part V., in respect of which it is proved that an offence has been committed ; (b) in any case that he had no reason to believe that the article did not, as respects content, weight, description, or labelling (as the case may be), comply with the provisions of this Order ; (c) that he has given due notice to the prosecutor that he intends to rely upon the provisions of this clause. Such person shall be entitled to be discharged from the prosecution.

(ii.) The provisions of sub-sections (3) and (4) of Section 20 of the Sale of Food and Drugs Act, 1899, shall apply as nearly as may be to proceedings under this clause in the same way as they apply to proceedings under the Sale of Food and Drugs Acts.

22. For the purposes of this Order percentages shall be calculated by weight.

23. Infringements of this Order are summary offences under the Ministry of Food (Continuance) Act, 1920.

24. The Orders mentioned in the schedule to this Order are hereby revoked, but without prejudice to any proceedings in respect of any contravention thereof.

25. (a) This Order may be cited as the Sale of Food Order, 1921, and shall come into force on September 1, 1921. (b) This Order shall not apply to Ireland. (c) The Interpretation Act, 1889, applies to the interpretation of this Order as it applies to the interpretation of an Act of Parliament.

By Order of the Board of Trade,

FRANK H. COLLER,

Secretary to the Food Department.

August 16, 1921.

THE SCHEDULE.—S.R. and O., 1918, No. 547, the Bread Order, 1918 ; S.R. and O., 1917, No. 318, the Tea (Net Weight) Order, 1917 ; S.R. and O., 1921, No. 403, the Bacon, Ham, and Lard (Sales) Order, 1921 ; S.R. and O., 1919, No. 1733, and 1920, No. 858, the Imported Meat (Labelling) Order (No. 2), 1919, as amended ; S.R. and O., 1920, No. 2408, the Eggs (Description on Sale) Order, 1920 ; S.R. and O., 1920, No. 1512, and 1921, No. 215, the Jam (Sales) Order, 1920, as amended ; S.R. and O., 1919, No. 511, the Dripping (Standard of Quality) Order, 1919 ; S.R. and O., 1919, No. 658, the Edible Fats (Standard of Quality) Order, 1919.



REVIEWS.

A DICTIONARY OF APPLIED CHEMISTRY. Vol. II.: Calculi to Explosion. By SIR EDWARD THORPE, C.B., F.R.S., assisted by eminent contributors. Revised and enlarged edition, 1921. London: Longmans, Green and Co. Price 60s. net.

A review of the first volume of this most comprehensive treatise has already appeared in the ANALYST (1921, p. 225), and the comments there made upon the general character of this edition apply equally to the second volume.

There is a further increase in the bulk; the subject matter of 547 pages of the 1912 edition now occupies 717 pages, to which must be added the space afforded by the deletion of a number of small articles, so that it will be apparent how much new matter has been incorporated. In addition to the bringing up to date of all the articles there are notable increases in the articles on the different dyestuffs and colouring matters, which, it is hoped, is a reflection of the gradual development of this industry in Britain.

A number of excellent articles on different alkaloids and drugs appear in this volume which were formerly grouped together in the general article on vegeto-alkaloids. For example, there is a new article of twenty-six pages by Howard and Chick, giving a very complete account of cinchona and its alkaloids. The articles on drugs reflect the considerable increase of knowledge of these substances during recent years. The reviewers would here venture a suggestion that the usefulness of some of these articles would be further enhanced by the inclusion of a brief account of the microscopic characteristics of the drugs for purposes of identification. It has been noticed, too, that there is an increased number of references to English chemical literature and abstracts; this is highly desirable in view of the time which may often be saved by reference to the English abstract of a foreign paper. Turning now to some of the articles in detail, a few points may be noted.

Under Carbohydrates there is a paragraph on heptoses, and mention is made of new sugars, and work done within the last few years is all incorporated. In view of the importance of cellulose esters in aeroplane manufacture the account of these substances included under cellulose might be somewhat enlarged, and a more complete list of references would be useful. The section on Cement does not appear to have been much altered from the 1912 edition, though a fuller account of the mechanical testing of cement is given. As the intermittent kiln is now practically obsolete, some space might be saved by the excision of the early types of these kilns. Under the heads of Chemical Affinity and Industrial Catalysis are found most interesting accounts of some of the newer industrial processes, including Haber's and Sabatier's reactions, and applications of enzyme reactions. The old article on Chromophores and Chromogens has disappeared and is replaced by a comprehensive

new article on Colour and Constitution, by Watson; this article is distinctly utilitarian; chemical theories are considered at some length, but physico-chemical theories are not discussed, because they have not led to the discovery of new dyes or the development of the industrial side of the subject.

The important subject of corrosion of metals and their protection therefrom is dealt with very fully. In addition to the articles on corrosion of iron and the fouling of ships, by Brame, there is a lengthy article on corrosion of metals in general by Friend. Although there are thus three separate articles on different aspects of the same general subject, there is little or no duplication of matter. The article on Colloids is comparatively short; it is surprising that no reference is given to the useful reports on this subject published under the auspices of the British Association.

A new article appears on Decolorising Carbons, in which is described in some detail the manufacture and special properties of different kinds of animal charcoal, which subject was deleted from Vol. I. The absorption of gases by charcoal is considered under the heading of Carbon.

The ingenious and useful film evaporator designed by Kestner now finds description in the revised article on Evaporators, and the article on Gaseous Explosion, rewritten by Bone and Wheeler, is quite up to date, and includes a summary of the large amount of new information worked out by Wheeler and his collaborators.

The new edition of this volume has, as a whole, been excellently revised, and there are few printer's errors; on p. 568, however, for "Dutton" should be "Dufton," and on p. 226 there should be inserted a reference to Wade and Finnemore (*Trans. Chem. Soc.*, 1904, 85, 938). There is so much new matter included that the technical chemist can scarcely afford to do without this edition of a unique treatise.

G. R. THOMPSON.

H. E. COX.

THE CHEMIST'S YEAR-BOOK, 1921. Edited by F. W. ATACK, M.Sc., B.Sc. Sixth Edition. Two vols., pp. 1123 and index. Manchester: Sherratt and Hughes, 1921. Price 21s.

This publication, previous editions of which have been reviewed in the ANALYST (1917, 42, 406), is now in its sixth edition, in two volumes. The book is to be regarded from several aspects. In the first place it is a book of chemical and physical data, and, as that, contains a variety of useful and important information. There are to be found here extended tables of specific gravities of solutions and of liquids, of solubilities of gases in liquids and of organic and inorganic solids, alcohol tables, tables of vapour pressures, constant boiling-point mixtures: gas volume corrections, tables, and many other data too varied to be enumerated. There are tables giving formula, density, solubility, and melting- and boiling-points of upwards of 1,000 inorganic and nearly 3,000 organic substances; others giving the composition and main physical properties of over 1,200 minerals, and the constitution of from 700 to 800 dyes. Another table gives the formulæ and certain properties of about 140 alkaloids. There are two very useful lists of the pharmaceutical names of synthetic compounds and of the trade names of drugs. In many cases the authority for the

data is given: the alcohol tables are those of the American Bureau of Standards, and are not, in the writer's opinion, presented in a very useful form.

In all this the editor has brought together a mass of useful information, and the book is one of much value for reference.

These two volumes are, however, far from being only a collection of tables. Introducing many of the tables will be found succinct accounts of the theory relating thereto. For instance, there is a good exposition of the questions of density and specific gravity and the units used; the correction of thermometer readings and of boiling-points is dealt with. Introducing the minerals table there is a "chapter" on crystallography: preceding the list of dyes is a general review of coal tar, intermediates, and dyestuffs. Other sections are similarly treated.

These sections form most instructive introductions to the tables, and are a valuable feature of the book.

Lastly, this book is a veritable treatise of analytical chemistry. In Vol. I. are to be found sections on qualitative and volumetric analysis, gas analysis, and spectrum analysis. In Vol. II., water analysis, analysis of fuels, of clays, fire-brick, and silica materials, of Portland cement, oils and fats, essential oils, dairy products, beer and brewing materials, of cellulose, paper and textile fibres, the analysis of urine, and the recognition of blood-stains—to mention only some of the matters dealt with.

These analytical sections, many of them by authors who are admitted experts in their subjects, are unequal in merit. The section on water analysis is inadequate, that on the bacteriological examination of water so inadequate as to be useless; the section on spectrum analysis is very meagre. Such a subject as the "adulteration of fats"—including oils—can obviously not be dealt with in two pages, and it should be noted that in this section there is a grave confusion between cholesterol and phytosterol. A chemist desiring, or compelled, to undertake an ultimate organic analysis would hardly be helped by the picture of the combustion furnace and accessories and the letterpress attached to it.

Considerations of space perhaps account for the inadequacy of these sections, but can hardly be accepted as a valid excuse. Such subjects should be, at any rate, fairly fully treated or left alone. This criticism does not apply to several sections—*e.g.*, amongst others, Cellulose and Paper, by C. F. Cross, Brewing Materials, by F. Robinson, Agricultural Analysis, by E. J. Russell, and the Identification of Dyestuffs. The difficulty of dealing with so wide a range of analytical procedure in a book of this nature is immense; the result is, as has been said, an uneven product.

The book is well produced and printed, though at times the small size and the character of the type are somewhat trying to the eyes—*e.g.*, the alcohol and the logarithm tables. Moreover, are not "log tables" more convenient in a separate book than when incorporated in a book of this character? The writer thinks that, by leaving out certain tables which every analyst has ready at hand, more space might be given to the other matter. To give another example, surely tables for giving the percentages of added water and deficiency of fat in milk are not required. On the other hand, the factors for gravimetric analysis might be extended.

A perusal of this edition discloses a certain number of errors, misprints, etc.;

these have been privately communicated to the author in preference to being detailed here.

The editor is to be congratulated on this further edition of a most useful book.

E. HINKS.

DAIRY BACTERIOLOGY. By PROFESSOR ORLA-JENSEN. Translated by P. S. ARUP. Pp. xii + 180. London: J. and A. Churchill, 1921. Price 18s. net.

To write a book like this must have been a difficult task, for Dairy Bacteriology is now such a wide subject that to cover the ground adequately requires much space; a less eminent man than Professor Orla-Jensen would have been led into discussions of dairy problems in which bacteriology plays a part, as well of the science of bacteriology, and a volume of great size would have resulted. The author has, however, been wise enough to realise that dairying problems are best learned either practically or from works on that subject, while the science and technique of bacteriology are treated of in many excellent textbooks, and has confined himself to matter which is neither wholly dairying nor bacteriology, and the result is a work of moderate dimensions, full of the knowledge of which he is a master. It is not a book for the elementary student, but is written rather for the graduate and the expert, and cannot adequately be appreciated except by those who have some knowledge of dairying, and have had a bacteriological training.

The English is good and reflects great credit on the translator, and the illustrations are superb; the main fault to be found is that the author refers to many of his own researches as well as those of others in outline, and gives references for those who wish to consult them in detail, and these references are to the original Danish sources, which are generally inaccessible to the English reader; and it is to be regretted that the translator did not see his way to include an abstract at least of these where an English reference was not available. To particularise, it would have been very useful if a fuller summary of Professor Orla-Jensen's work on the lactic acid organisms had been included; a fuller description of the methods for the direct count of micro-organisms in milk might also have been included, as this is rarely given in bacteriological textbooks.

The work is divided into two parts, the first a bacteriological introduction which contains a happy selection of salient facts, and the second and larger portion devoted to practical dairying problems dependent on bacteriology; the whole ground of milk production—the preservation of milk, butter and cheese-making, fermentation products, and the grading of milk—is covered, and to all engaged in the scientific control of dairying this book cannot fail to be of the greatest interest and use.

H. DROOP RICHMOND.

CHEMISTRY OF PULP AND PAPER-MAKING. By E. SUTERMEISTER. Pp. vii + 479. New York: John Wiley and Sons. London: Chapman and Hall, Ltd., 1921. Price 36s. net.

In a book which covers such a large field as the "Chemistry of Pulp and Paper-Making" the individual chapters relating to each branch are necessarily limited. Nevertheless, the outlines of the subject are well set out, and serve as a guide to the

practical worker and chemist in the mill who is desirous of possessing a general knowledge of the subject and its application.

Chapter II. describes the various fibres used in paper-making, with their chemical and microscopical characteristics ; these are illustrated with an excellent set of photomicrographs prepared by the U.S. Paper Section of the Bureau of Standards, and should be found very useful to the student.

Chapters III. to VII. deal very fully with the preparation of the typical raw materials, the actual boiling trials, more especially in the treatment of wood by the soda, sulphate, and sulphite processes, being illustrated with various set examples of the composition of the liquors and methods of analysis. This detailed work is certainly a departure from the usual textbook method, and the author is to be congratulated on including analytical details.

Chapters VIII. to XI. deal with the incidental operations of bleaching, sizing, loading, and colouring of pulps.

One very important detail in a textbook of paper-making is lacking, and that is the operation of beating. It is usually claimed, and rightly so, that the finished paper is made in the beating room ; hence the absence of any details of effects of beating upon the different fibres in the ordinary hollander, the various types of modern beaters with auxiliary circulation, and the incidental details of bed-plate and weight of beater rolls to hydration of pulp detracts somewhat from the value of the book to the paper-making student.

The same remarks may be applied to the absence of any details of paper-making machines—that is, Fourdrinier, mould and board machines. The chapters on coating and paper-testing follow the lines usually laid down in a book of this kind.

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