

THE ANALYST

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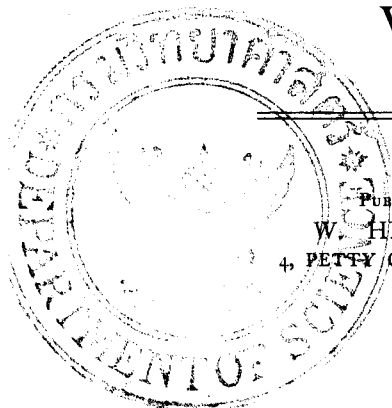
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THE ANALYST

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

AN Ordinary Meeting of the Society was held in the Chemical Society's Rooms, Burlington House, on Wednesday, 3rd December, 1924. The President, Mr. G. Rudd Thompson, F.I.C., was in the chair.

A certificate was read for the first time in favour of Mr. Thomas Mann.

Certificates were read for the second time in favour of:—Messrs. Arthur Chapman Barnes, B.Sc., F.I.C., John Jacob Fox, D.Sc. (Lond.), F.I.C., Thomas Congalton Hay, and Harold Rupert Jensen, M.Sc. (Liv.), A.I.C.

The following were elected members of the Society:—Messrs. Arthur Cecil Brooks, William Donovan, Albert George Flower, Clifford Hollingsworth, George Marshall Norman, Charles Edward Sage, Percy Frank Spendlove, Charles William Spiers, Leslie Herbert Trace, and James Rawson Walmsley.

The following papers were read:—"The Analytical Value of the Melting Point of the Insoluble Volatile Acids from Fats," by G. Van B. Gilmour, B.Sc., A.I.C., A.R.C.Sc.I.; "The Use of Mitchell's Ferrous Tartrate Reagent in Qualitative Analysis," by A. H. Ware; "The Influence of Hydrogen Ion Concentration on the Colorimetric Determination of Pyrogallol and Catechol Derivatives," by S. Glasstone, M.Sc., Ph.D., F.I.C.; and "The Futility of Sealing: A Suggestion for its Safeguarding," by B. S. Evans, M.B.E., M.C., B.Sc., F.I.C.

Obituary.

ROBERT HELLON.

ROBERT HELLON was born at Workington in Cumberland, on September 13, 1854, and died on November 9, 1924. Some few years after his birth his father was appointed manager of the Bank of Whitehaven, and the family moved to that neighbouring town. Here, at a private school his education was begun, and it was finished at a school in Wrexham. Returning to Whitehaven, he studied analytical chemistry for a while with Dr. Tosh, and then proceeded to the School of Mines, where he came under the influence of Frankland and Percy. After taking his Associateship in 1873, he went to Heidelberg and gained all that working with Bunsen meant to the chemical student of those spacious days. Then, after studying in Hoffmann's laboratory in Berlin (1877), and with Würtz in Paris, Hellon took his degree of Ph.D., with distinction, at Heidelberg, early in 1878.

Returning to France, he joined Dr. Tcherniac, who had been his fellow student, and thence onwards was to be his life-long friend, and, as the result of this collaboration, thiocyanacetone was first prepared. The account of this work was published in the *Comptes rendus* of the Paris Academy of Sciences, and elsewhere.

He left France in 1883, and, entering into partnership with his friend, Alfred Adair, began to practise as an analytical chemist on the north-east coast. South Africa tempted Adair, however, and Dr. Hellon decided to try his luck alone in Whitehaven, where his parents were still living.

When, in 1889, the position of Public Analyst for Cumberland, Westmorland, and the Borough of Kendal became vacant, he was the successful candidate. His later appointments were as Agricultural Analyst for Cumberland and Carlisle and Public Analyst for Carlisle.

He was a pioneer in the application of chemistry to the leather industry, and was one of the founders of the International Association of Leather Trades Chemists.

He twice served on the Council of the Society of Public Analysts (1904–1905 and 1919–1920). He became an Associate of the Institute of Chemistry in 1878, and a Fellow in 1889.

In innumerable ways he did much for the spread of scientific education in the county of Cumberland. He was appointed Justice of the Peace in 1913.

From his wide experience in the field of chemistry and physics and his natural aptitude for, and deftness in, experimental work, he was particularly well equipped for the occupation that filled his days. Unfortunately his constant ill-health prevented him from communicating to his colleagues, save by word of mouth, the many ingenious methods of analysis he devised and brought to perfection.

The saddest day of his life was April 6, 1915, when his devoted wife died. Without children, and sometimes, during the long stretch of a dreary northern winter, confined for weeks together to the house with that distressing complaint,

asthma, he was lonely indeed. At such times he lived largely in his private laboratory. Latterly, in the summer time, he took up botany, and became one of the leading authorities on the flora of his native county. When the failure of his eyesight and hearing became serious, wireless telephony came to him as a heaven-sent gift. As in everything he undertook, he threw his whole energy into his installation. He was ever grateful for this renewal of a contact with the outer world, which had been slipping away from him rapidly, and which he feared might cease almost completely.

The outstanding feature of his character was his gentleness. That must have been felt by everyone who came in contact with him. The writer has had the joy of his companionship for over thirty years, and he never heard him speak an unkind word to, or of, anyone. To the wayward he was ever generous. Children loved him, and it was his great delight to gather a turbulent band round his study fire and tell thrilling tales of adventure in the fairy fields of science.

He was what most of us wish to be, an able scholar and a very perfect gentleman.

THOMAS MANN.

On Certain New Methods for the Determination of Small Quantities of Arsenic, and Its Occurrence in Urine and in Fish.

BY H. E. COX, M.Sc., Ph.D. (LOND.), F.I.C.

(Read at the Meeting, November 5, 1924.)

So much has been written in the past on the determination of traces of arsenic that one would hesitate to offer any remarks on the subject were it not for the fact that some rather startling results have been reported by the late Prof. Bang to a Commission on Arsenical Poisoning in Sweden, and the conviction that the wet combustion process recommended by the Joint Committee of this Society and the Society of Chemical Industry in 1902 does not always give satisfactory results. Using new methods, the Swedish Commission reports the presence of quite considerable quantities of arsenic in the urine of normal persons, the amounts being such that in many cases chronic arsenical poisoning would be suspected. It has been generally assumed, without satisfactory reason, that urine is normally almost arsenic-free. In medical literature 0.03 mgrm. per litre is widely recognised as a maximum figure.

In the light of the results given by Bang (Arsenic Commission Bil. XI.) it appears that 60 per cent. of the population of Sweden could be suspected, on the

above basis, of suffering from chronic arsenical poisoning. Thus, the following figures are given for 100 persons on a known hospital diet:

	As ₄ O ₆ Mgrm. per litre.	Percentage of cases.
1.	0·00 – 0·01	21·1
2.	0·01 – 0·03	18·4
3.	0·04 – 0·06	23·7
4.	0·06 – 0·09	7·9
5.	0·10 – 0·20	21·1
6.	0·20 – 0·30	2·6
7.	0·30 – 0·70	5·2
		100·0

It will be appreciated how important such figures are in view of any criminal proceedings respecting arsenical poisoning. If 0·1 mgrm. or upwards of arsenious oxide were found in the urine, it would raise grave suspicion in the minds of chemists and physicians, yet it appears that seven times this amount may occasionally be present in the urine of a normal healthy person who is not taking arsenic in any form, except as a natural impurity in certain foods.

A search of the literature on this subject reveals that surprisingly few investigations have been made on normal urine, although there are plenty on real or supposed cases of chronic arsenical poisoning; also that in many instances the methods used have been, at best, only approximate. Thomson in 1905 (*Proc. Manc. Lit. Phil. Soc.*, 1905, 49, 1) did, in fact, find from $\frac{1}{1000}$ to $\frac{1}{100}$ grain of arsenious oxide per gallon in a few samples from various places, and attributed the fact to arsenic in the air. Other workers, e.g. Sanger and Black (*J. Soc. Chem. Ind.*, 1907, 1123) have elaborated methods for the determination of arsenic in urine, but assumed it to be completely absent in the normal condition. Only last year Billeter and Marfurt (*Helv. Chim. Acta*, 1923, 6, 780) stated that the average human body contains about 0·1 mgrm. arsenic as As₄O₆. This must be incorrect, as the urine alone may contain far more. It is considered by the Swedish Commission that the results obtained by earlier workers are low on account of the use of defective methods, and new processes are described. At the suggestion of some members of this Society I have carefully investigated these new processes and compared the results with those obtained by the older method.

Briefly put, my results confirm the main findings of the Swedish Commission, although their method of estimating the arsenic after the preliminary treatment is, in my opinion, open to criticism.

As the proposed methods, which really involve no new principle, are not available in English, French or German, they are abstracted here in some detail. For the original papers I am indebted to Prof. Karl Petrén, of Lund; these were in the Swedish language, and my thanks are due to Mr. N. T. Foley, who was good enough to translate them for me.

A. BANG'S ORIGINAL METHOD.

(Bang and Bjork--Arsenic Commission Supplement VII.)

This method consists in the destruction of all organic matter by nitric and sulphuric acids by a modification of the Kjeldahl digestion; the arsenic is subsequently determined by distillation with hydrochloric acid and titration with dilute iodine solution.

To about 20 grms. of the substance to be tested are added 20 to 22 c.c. of sulphuric acid in a 300 c.c. Kjeldahl digestion flask, the mixture is warmed over a

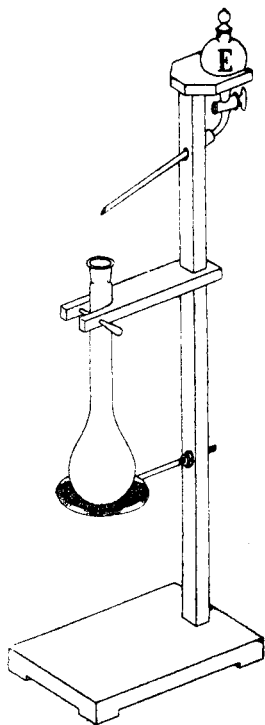


Fig. 1.

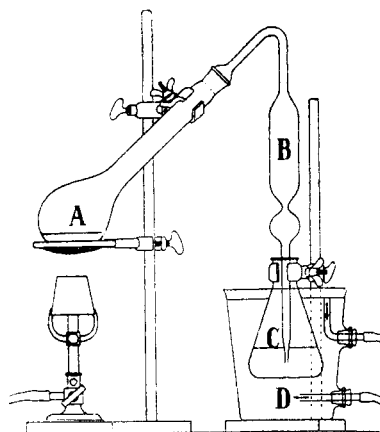


Fig. 2.

small flame, and concentrated nitric acid is added in drops, at the rate of about 8 per minute, by means of the apparatus shown (Fig. 1). Heating is continued until all organic matter is destroyed and a clear colourless liquid remains; the acid should fall directly on to the mixture, and the rate of heating should be so regulated as to avoid any large separation of carbon. When all the colour has disappeared strong heating is continued for 15 minutes, during which time all nitric acid is expelled. If any darkening appears more nitric acid must be added. After cooling, 50 c.c. of water are added, and the mixture is boiled, to remove nitrous

compounds, until white fumes again appear. A few crystals of potassium permanganate are now added, the colour of which should not be discharged. The volume of nitric acid added should be noted in order to apply the necessary corrections if it contains any perceptible amount of arsenic.

After completion of the combustion there are added 30 c.c. of water, 2 grms. of ferrous ammonium sulphate, 20 grms. of potassium chloride, and 2 grms. of potassium bromide. Then the flask is connected for distillation in the manner shown in the diagram (Fig. 2), except that the end of the tube does not touch the surface of the receiving liquor. The receiver contains 30 c.c. of 20 per cent. nitrite-free sodium hydroxide solution, 100 c.c. of water and 2 drops of phenolphthalein, and is cooled by running water. Distillation is continued until the colour of the alkaline solution becomes intensely red, then suddenly disappears; the acid distillate is neutralised by adding excess of sodium bicarbonate, and is then titrated with 0.005 *N* iodine solution.

It will be obvious that arsenic-free reagents must be used throughout; a blank test is made on the same quantities of acids and salts as are used in the determination, and the amount of iodine absorbed is deducted. It is not easy to obtain sufficiently pure reagents; the amounts employed are so large that serious error is easily introduced. Also, as is pointed out by Ramberg and Sjöström (Arsenic Comn. Supplement VIII.), there are errors inherent in the iodine titration due to absorption by substances other than arsenic. Boiling with water alone is not sufficient to remove all traces of nitric or nitrous acids, the complete elimination of which is a *sine qua non* of the process. Ramberg finds a preliminary treatment with dilute nitric acid always advantageous and sometimes essential; he removes nitrogen acids by ammonium oxalate and finally titrates with potassium bromate solution.

B. RAMBERG'S METHOD.

(This is the method finally adopted by the Commission in its Report).

A quantity of the substance which will contain not more than about 5 grms. of dry matter is introduced into a Kjeldahl flask and thoroughly wetted with about 15 c.c. of dilute nitric acid (sp. gr. 1.25). After a few minutes 22 to 23 c.c. of sulphuric acid are added, the mixture is heated, and concentrated nitric acid is dropped in, as in Bang's method. The atmosphere in the flask must at no time be free from red fumes. With substances difficult to burn the liquid is allowed to cool, 0.5 c.c. of nitric acid is added, and the flask re-heated; in this way the nitric acid is not vaporised too rapidly. When all organic matter is destroyed the addition of nitric acid is discontinued, and the heating continued until red fumes are no longer seen; then, after cooling, 25 c.c. of saturated ammonium oxalate solution are added and some fragments of glass, and the liquid is boiled until sulphuric acid fumes appear. After cooling, 20 c.c. of water are run in round the neck of the flask. Then there are added 50 c.c. of hydrochloric acid, 2 grms. of ferrous sulphate and 10 to 15 mgrms. of potassium bromide. If any yellow or

brown colour appears at this stage, nitrogen acids are present and the experiment must be rejected. The flask is connected for distillation (the apparatus must have glass joints only), 150 c.c. of water are put into the receiver, and the exit tube made to dip 1 cm. below the surface. A volume of 20–25 c.c. is distilled in 10 minutes; to the distillate is added one drop of methyl-orange solution (1:5000), then it is slowly titrated at a temperature of 35°–40° with potassium bromate solution (0.1485 gr./litre; 1 c.c.=0.20 mgrm. As_4O_6). The bromate solution is added a drop at a time towards the end of the titration, which is reached when the red colour of the methyl-orange is discharged; the end-point is quite sharp. A correction is applied for the blank on the reagents; this should not exceed 0.2 to 0.3 c.c.

Bang states that his method is accurate to 0.001 mgrm., that of Ramberg is put at 0.002 mgrm. For smaller quantities an electrolytic micro method is described, which is alleged to be accurate to 0.0001 mgrm.; in this case the acid solution containing the arsenic is introduced into an electrolytic Marsh apparatus of the type described by Trotman (*J. Soc. Chem. Ind.*, 1904, **23**, 177), and the mirror obtained is dissolved in 2 c.c. of 0.0005 *N* iodine solution in a small stoppered tube; then the excess of iodine is titrated with 0.0005 *N* sodium thiosulphate solution.

TESTS ON THE FOREGOING METHODS.

Neither the iodine nor bromate titration of arsenic is new, and both are known to be satisfactory for reasonable quantities of arsenic when the reagents are pure; but when large quantities of reagents are employed and the total arsenic to be estimated is of the order of 0.005 mgrm. of As_4O_6 the influence of impurities becomes important.

Using Bang's method, I have been quite unable to obtain concordant results with small quantities of arsenic, and even the blank is not constant.

The reasons for this appear to be:

- (1) Boiling with water does not remove all traces of nitrogen acids; the addition of ammonium oxalate is essential.
- (2) Alcoholic solution of phenolphthalein itself absorbs varying amounts of iodine.
- (3) The amount of reagents added is too large, so that impurities are introduced.
- (4) The sodium hydroxide and bicarbonate used to neutralise the distillate almost always contain some iodine-absorbing substances.

Ramberg's method is much superior to that of Bang. Two points in it are all important, *viz.* the complete oxidation of all traces of organic matter and the removal of all nitrous acid. When these are observed good results are obtained, *provided that the amount of arsenic to be estimated is not too small.* The following

table shows results obtained by me with this method; 4 grms. of starch were used in each test.

RAMBERG'S METHOD.

Experiment.	As ₄ O ₆ added. Mgrm.	Bromate solution taken. c.c.	As ₄ O ₆ found. Mgrm.
1.	Nil	0.20	—
2.	Nil	0.20	—
3.	Nil	0.23	—
4.	0.50	2.80	0.52
5.	0.30	1.75	0.31
6.	0.20	1.05	0.17
7.	0.10	0.70	0.10
8.	0.05	0.50	0.06
9.	0.02	0.35	0.030
10.	0.01	0.30	0.020
11.	0.005	0.25	0.010
12.	0.002	0.25	0.010

The results are quite satisfactory down to 0.01 mgrm.; for quantities less than this I find it best to use Ramberg's method of destruction and subsequently the Marsh-Berzelius process; better results are thus obtained than by the older method of the Joint Committee, and there is the advantage that the arsenic mirror is actually seen and may be proved to be arsenic by the ordinary recognised procedure.

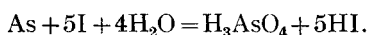
So far as the preliminary oxidation treatment is concerned there seems no doubt that the moist combustion recommended by the Swedish Commission is preferable to the older method. When there is a considerable mass of carbon to be extracted with acid, as is frequently the case in the Joint Committee's method, it is very doubtful whether all the arsenic is washed out. From experiments I have made it is almost certain that some is retained or adsorbed by the carbon, as is the common experience in the case of small amounts of copper or lead in foodstuffs. Also, if the heating is continued sufficiently long to ensure a clear and colourless liquid, there is risk of loss of arsenic by volatilisation, and if the liquor is coloured by organic matter, the results of the subsequent estimation are liable to serious error. Naturally these difficulties arise only when considerable quantities of organic materials are being treated.

ARSENIC IN URINE.

Many of the results of the examination of urine reported by the Commission were obtained by Bang; hence, in the writer's opinion, they may be slightly too high. The following results have been obtained by a slight modification of Ramberg's method:

To 100 c.c. of the urine were added 10 c.c. of nitric acid, and the liquid was evaporated in a Kjeldahl flask to about 20 c.c., after which 10 c.c. of sulphuric acid were added, the digestion continued, and the nitric acid removed, as in Ramberg's method, 15 c.c. of ammonium oxalate solution being used. The

arsenic was determined in the clear colourless nitrite-free liquid by the Marsh-Berzelius process, and the blank with these quantities of reagents was quite constant at 0.0015 mgrm. The mirrors were estimated in some cases by optical comparison with standards, and when they appeared greater than about 0.01 mgrm. by dissolving them in dilute iodine solution and titrating with thiosulphate; this gives quite good results when compared with the ordinary optical method.



There could be no doubt whatever that the mirrors were of arsenic, as in many instances they were heated in air, with the result that characteristic crystals of As_4O_6 were produced.

Experiment No.	Volume excreted in 24 hours. c.c.	As_4O_6 found. Mgrms. per litre.	As_4O_6 in Mgrms. per day.
13.	1500	0.04	0.06
14.	1200	0.05	0.06
15.	1350	0.03	0.04
16.	1700	0.02	0.03
17.	1600	0.00	0.00
F. 18.	1750	0.10	0.18
19.	1000	0.09	0.09
20.	1200	0.05	0.06
21.	900	0.01	0.01
22.	620	0.03	0.02
23.	1100	0.07	0.08
F. 24.	750	0.25	0.19
25.	950	0.01	0.01
26.	1100	0.02	0.02
27.	1200	0.00	0.00
28.	—	0.02	—
29.	—	0.07	—
30.	1700	0.01	0.02
F. 31.	1050	0.58	0.61
F. 32.	—	0.33	—

For some of these samples I am indebted to Dr. Blackie of the London Homœopathic Hospital.

Samples No. 18, 24, 31, and 32 are from persons having a fish diet; No. 31 had three meals of plaice 24 hours before the sample was taken.

These figures, which are almost as high as those of Bang, show clearly that normal urine may contain quite substantial proportions of arsenic, and confirm the observation that after a fish diet there is an appreciable increase in the output of this metal.

ARSENIC IN FISH.

According to the Swedish Commission the amount of arsenic in fish is sometimes greater than has hitherto been supposed. Bang reports 4 parts per million in one case. In this connection it may be remarked that in the recent Report

on Oysters by the Ministry of Agriculture and Fisheries as much as 3.7 parts of As_4O_6 per million was found in certain Whitstable oysters (*cf.* ANALYST, 1924, 49, 484).

Several kinds of fish have therefore been examined by the Ramberg method of destruction and the arsenic subsequently determined by the Marsh process. The results show that quite substantial amounts of arsenic may be present, although no figures quite as high as those of Bang have been obtained.

It seemed possible that fish feeding in Swedish waters might contain more of this impurity than those from English waters, as sold in the London markets, and to enable me to test this point Prof. Petrén kindly had a case of fish sent from the Swedish Sund. No difference appears between the two sets of figures. The results obtained by the old method of the Joint Committee are set out, side by side with those of the new process and those of Bang and Ramberg, and show that, at least in its application to fish, the old method gives low results. The weight taken for the determinations was 20 to 25 grms. of the edible portion of the fish, including skin but not bone:

ARSENIC AS As_4O_6 PER 100 GRMS. OF FISH.

Fish	Joint Committee's method Mgrm.	Swedish Commission's method (Ramberg) Mgrm.	Amount found by Bang. Mgrm.
<i>(British)</i>			
Whiting	0.01	0.04	0.01-0.33
Plaice	0.08	0.14	—
”	—	0.25	—
”	—	0.30	—
Sole	0.02	0.03	—
Hake	0.02	0.03	—
Herring	0.01	0.03	0.04-0.08
Cod	0.01	0.05	0.08-0.20
John Dory	0.01	—	—
Haddock	0.01	0.06	—
Brill	0.01	0.03	—
Mackerel	0.02	0.05	—
Halibut	0.00	0.03	—
Turbot	0.02	0.05	0.06-0.18
Perch	—	—	0.08
<i>(Swedish)</i>			
Mackerel	0.01	0.01	—
Herring	0.01	0.03	—
Haddock	0.01	0.02	—
Plaice	0.03	0.05	—
”	0.05	0.09	—
Sole	0.01	0.01	—
Cod	0.02	0.04	—

It is evident that fish may, and often do, contain quite appreciable quantities of arsenic, the amount varying in the different individuals; two fish from the same batch may contain widely different proportions. The high figures obtained from plaice are remarkable; three specimens out of four examined contained more than the Royal Commission's limit of 1/100 grain per lb. one giving twice that quantity. It might be expected that other flat fish, such as the sole, would contain like amounts, but this is not borne out by the results. The reason for this probably lies in the difference in the food of these fish, plaice, as I was informed by Dr. J. H. Orton, of the Marine Biological Association, feeding largely on bivalves, which have been found to contain notable quantities of arsenic; sole feed only to a small extent on bivalves, but largely on worms, starfish and crustacea.

There appears to be ample support for the suggestion that arsenic in human urine is largely due to fish; vegetables and meat have not been found to contain substantial amounts. There is also the proved fact that fish diet gives rise to a large increase in the excretion of arsenic. L. Brahme (*Arsen in Blut*, Stockholm, 1923) claims to have demonstrated arsenic in the blood within an hour of the eating of fish. The source of the arsenic in fish is doubtless due to the food; as has already been noted, plaice get it from bivalves; other kinds get it from other foods or from algæ. In this connection it may be noted that Gautier (*Bull. Soc. Chim.*, 1903, 29 [iii.], 780) and Tassilly and Leroide (*ibid.*, 1911, 9 [iv.], 63) show that algæ, and even sea-water itself, contain this element in recognisable proportions, varying from mere traces up to 0.7 mgrm. in algæ, and 0.08 mgrm. per kilo in sea water.

SUMMARY.—The methods and results of the Swedish Commission on Chronic Arsenical Poisoning have been carefully tested, and the conclusion has been reached that the nitric and sulphuric acid digestion process, described by Bang and Ramberg, gives higher and more accurate results than the older wet combustion method adopted by the Joint Committee of the Society of Public Analysts and the Society of Chemical Industry in 1902. The iodine titration method of Bang is not reliable, but the Ramberg bromate method gives satisfactory results if the amount of arsenious oxide to be estimated is not less than 0.01 mgrm.; below this amount the Marsh-Berzelius method, following the Ramberg method of destruction (details of which are given) is preferable.

It has been found that normal urine may contain quantities of arsenic which have been thought to be associated only with chronic arsenical poisoning; amounts present in the urine of persons on known diets varied from 0.0 up to 0.58 mgrm. per litre. Large excretion of arsenic is due to the eating of fish, and it is shown that fish, especially plaice, may contain arsenic up to 3 parts per million. The eating of such fish leads to the appearance of quantities of arsenic in the urine within twenty-four hours.

DISCUSSION.

Mr. JOHN WEBSTER said that in dealing with such very minute traces of arsenic the titration method—with its controls and blanks—seemed to him very unsatisfactory. He, personally, when employing the Marsh and other well-known

methods, had not found, under normal conditions, anything like the amount of arsenic mentioned by the author. In his opinion the number of reagents added in the course of the tests was unfortunate, as, naturally, the more reagents added, the more care would have to be exercised as to their absolute freedom from arsenic.

Mr. EDWARD HINKS referred to the opinion of the late Mr. Wilkie, expressed at the Nottingham meeting in 1923, that it was quite usual to find easily-detectable quantities of arsenic in normal urine. As arsenic was so very widely distributed, it was obvious that it must be frequently introduced into the system, and so, presumably, must be excreted in the urine.

Mr. N. T. FOLEY said that, according to his recollection of the Swedish original, Ramberg did check his experiments by the Marsh test.

Mr. J. MYERS said that it was rather a curious coincidence that the Swedish method described by Dr. Cox was, in its essentials, very similar to the one he was in the habit of employing for the determination of arsenic in pyrites, which method, moreover, came to this country from Sweden, by way of a private communication to Mr. Rudd Thompson. With the reservation that his work on pyrites comprised the determination of percentages of arsenic ranging from 0.004 to 0.5 per cent., and that iodine and starch were employed for titrations, the method was the same, but he would not care to trust it for the extraordinarily minute quantities dealt with by Dr. Cox, because, no matter how careful one might be in the choice of the reagents employed, the blank in his (the speaker's hands) invariably exceeded the quantities of arsenic dealt with in Dr. Cox's paper.

Mr. A. E. PARKES asked whether it had been proved that the glass of the apparatus was free from arsenic.

Mr. C. A. MITCHELL asked whether there was any evidence of the cumulative action of arsenic introduced into the system in fish. It would also be interesting to know in what manner it would be distributed in different organs of the body. It had been proved by Strassmann and Kirstein that, if arsenic were introduced *post mortem* into the stomach, it would accumulate in the left kidney, whilst the right kidney would only absorb traces, so that it would appear that certain organs had a selective action.

Mr. G. R. THOMPSON expressed his personal appreciation of Dr. Cox's work on these Swedish methods, which had been carried out with that scrupulous attention to accuracy with which the speaker had been familiar for so many years when he and Dr. Cox were associated. Apparently nothing was without arsenic content, but he would hesitate to accept the results obtained by a method in which the quantity of reagents added was so large as to introduce the possibility of serious errors.

Dr. Cox, replying, said there was no doubt whatever that the substance determined was arsenic, as it had been proved by the formation of arsenic mirrors. With regard to the microchemical method, he, personally, did not endorse what the Swedish Commission had said upon it, and had merely reported their opinion. He had been asked whether the urine of fish eaters might not give a direct result in the Marsh test; it did, and in certain cases the results were checked that way. It was easy to prove that a considerable quantity was present. The method he recommended was the Swedish method of destruction of organic matter by oxidation followed by the Marsh process, so that the effect of the possibility of losses of arsenic—due to the use of the "wet" method only—was eliminated; also it was well known that in tin and lead determinations it was impossible to extract all the tin from a mass of carbon, and in the wet combustion process arsenic was also retained or adsorbed by the carbon. He quite agreed with all that had been said on the quantities of reagents required, but nitric, hydrochloric, and sulphuric acids were all obtainable free from arsenic. Regarding the possibility of there being arsenic

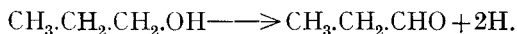
in the glass he had used for his experiments, it was possible nowadays to obtain, and he had obtained, glass free from arsenic. As to the cumulative effects of arsenic, the Swedish chemists had determined the amount of arsenic in the fish and the amount of arsenic eliminated by people having eaten the fish, and had found that the amounts agreed; further, they had proved that there was a cumulative effect until the fish diet was discontinued. Mr. Myers had said the methods were not new; that was so; they were only new as applied to foodstuffs, in which the amount to be determined was very much lower than in the case of pyrites.

An Apparatus for the Catalytic Dehydrogenation of Alcohols.

BY S. G. WILLIMOTT, PH.D., B.Sc.

(Read at the Meeting, November 5, 1924.)

IN the course of an investigation upon the synthesis of certain quinoline derivatives, it became necessary to prepare propyl aldehyde in quantity. The usual laboratory methods for the preparation of aliphatic aldehydes by oxidation of the alcohol are unsatisfactory, as the yields are erratic and low. In the present work with propyl aldehyde, for example, I have found that the yield of aldehyde-ammonia by the well-known Gattermann method was, at best, never more than 20 per cent. of the theoretical amount. Similar results have been obtained by Adams and Williams (*J. Amer. Chem. Soc.*, 1921, **43**, 2420), who studied the yields of acetaldehyde obtainable by the Gattermann method. These authors found the yield of aldehyde-ammonia to be only 23 per cent. of the theoretical quantity, and often to be lower than this. They cite Noyes (*Organic Chemistry for the Laboratory*, 1911, p. 89), who predicts a yield of only 15 per cent. for the method. My attention was therefore turned to the catalytic process of dehydrogenation for the preparation of aldehydes and ketones. Neave (*ANALYST*, 1909, **34**, 346) describes one form of apparatus for carrying out the dehydrogenation of alcohols, as suggested, in the first instance, by Sabatier and Senderens (*Bull. Soc. Chim.*, 1905 [iii.], **33**, 263). A primary alcohol such as propyl alcohol, which was used throughout this work is decomposed in the following way:



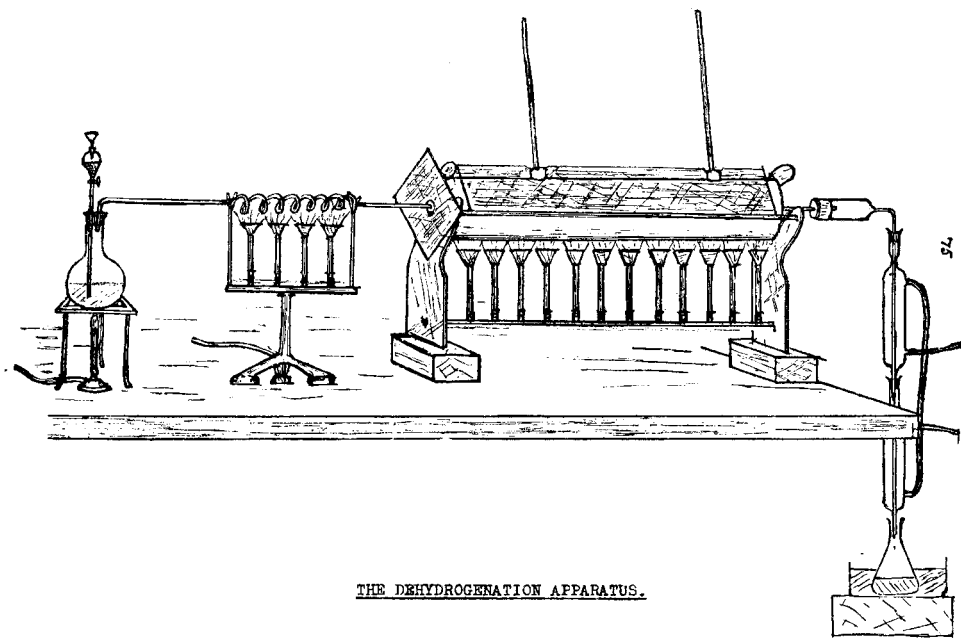
Neave's apparatus for carrying out a reaction of this type consists essentially of a 28 in. combustion tube of $\frac{1}{2}$ in. diameter, containing a 24 in. layer of finely divided copper prepared from powdered cupric oxide by reduction in a current of dry hydrogen at 300° C. To one end of the combustion tube is attached a bent capillary the vertical portion of which is joined on to a wider vessel. The other end of the combustion tube is connected with a receiver which is cooled by a mixture of ice and salt. An air bath consisting of an iron cylinder 26 in. long serves to

regulate the temperature. The bath, which carries two thermometers 12 inches apart, is heated by three Ramsay burners. When the temperature of the tube has reached 300° C., the alcohol (3 c.c.) is poured into the long receiver, from which it should drip steadily into the hot tube, become vaporised and, in its passage through the combustion tube, finally be catalytically decomposed by the copper.

In practice, however, this arrangement for the steady supply of alcohol into the tube was found to be only partly successful, and an improved method was devised. This consisted in driving the propyl alcohol vapour (b. pt. 97° C.) into a well heated copper spiral before its entry into the dehydrogenation tube. This apparatus consisted of a small round flask of 500 c.c. capacity, in which the alcohol was gently boiled, and had a cork with two holes, one for a small dropping funnel, the other for a delivery tube bent at right angles. This was connected by a short piece of rubber tubing with a copper tube of a diameter of $\frac{1}{8}$ in. At the end near the combustion tube and remote from the flask, a compact 6 in. spiral was connected directly with the combustion tube, and was heated by a Ramsay burner. An asbestos board protected the cork of the tube from carbonisation by heating of the spiral. The idea underlying this apparatus was simply to secure the alcohol passing over the catalyst as vapour at 300° C.; moreover, with this arrangement no liquid entered the combustion tube, and cracking, which was one of the disadvantages of the Neave apparatus, was avoided. A combustion tube of 40 in. length is preferable to the shorter one used by Neave, as then approximately 36 in. of the catalyst are available for acting upon the alcohol. A cylindrical air bath, 30 in. long and with a diameter of 4 in., was employed to regulate the temperature, and this had two thermometers, 12 in. apart, held in position by well-fitting corks protected by asbestos. A modification was introduced for the heating of the air bath. It was found satisfactory and convenient in practice, to use an ordinary combustion furnace in place of three Ramsay burners for the heating of the air bath. Usually it is only necessary to widen the jaws of the combustion furnace slightly to render it suitable for this purpose. The heating of the air bath and the regulation of its temperature is facilitated by placing a long piece of asbestos on either side of the air bath, as shown in the diagram. With this arrangement, once the required temperature has been reached, a small flame serves to maintain it constant. It is important to have an efficient condensing system for collecting the aldehyde or ketone produced, as otherwise the yield is seriously affected. Two Davis double-surface condensers were found to be efficient, and the receiver itself was allowed to stand in ice-water. The end of the combustion tube is connected with the condensers by means of an adapter, 1 inch in diameter and 8 inches in length, and at this point it narrows to a small tube bent at right angles to connect with the first condenser.

Pure, dry hydrogen, prepared by dropping 10 per cent. alkali on aluminium foil and passing the gas through permanganate solution and sulphuric acid, was used for the reduction of the cupric oxide, which was dry and powdery. The reduction was carried out at 300° C. and the tube kept full of hydrogen until required. The

supply to the hard glass tube of the alcohol to be dehydrogenated was easily controlled by simply regulating the boiling. The dehydrogenation was started at 290°C ., and the temperature allowed to reach 300°C . as the reaction proceeded. Occasionally it was also found convenient to employ "kupfer-bronz" (Kahlbaum's) when a ready catalyst was desirable. This is especially the case when only a small quantity of material is to be worked upon as, for example, in characterising primary, secondary and tertiary alcohols, as suggested by Neave. The "kupfer-bronz" was applied by intimately coating the walls of the hard glass tube with a uniform layer of the catalyst. The copper alloy, which was in a remarkably fine state of



THE DEHYDROGENATION APPARATUS.

division, readily adhered to the inner surface. The catalytic power of "kupfer-bronz" falls off sooner than when finely divided copper is used, but the yield is not greatly affected. The advantage of this modification is the saving of time, as no reduction is required, and immediately the efficiency of the catalyst becomes impaired it can be easily replaced.

The propyl aldehyde was obtained in a pure state by subjecting the liquid in the receiver to two fractionations using a Young column. On redistillation, the aldehyde boiled at 49.5°C . The alcohol remainder was neutralised with calcium hydroxide, as the presence of propionic acid rapidly destroys the activity of the catalyst. It was then redistilled and slowly passed over the catalyst, the process being continued until all the alcohol had been transformed. The average yield was 33 per cent. The complete apparatus is shown in the diagram.

SUMMARY.—1. The usual laboratory methods for the preparation of aliphatic aldehydes are shewn to be unsatisfactory.

2. An improved dehydrogenation apparatus is described, the chief advantages of which are:

- (a) The alcohol vapour, by passing through a heated copper spiral, enters the tube at 300° C., when it is readily dehydrogenated by the copper catalyst.
 - (b) The spiral pre-heater prevents liquid from entering the tube, with consequent cracking—a disadvantage of the Neave apparatus.
 - (c) The quantity of propyl alcohol vapour entering the dehydrogenation tube is controlled by simply regulating the boiling.
 - (d) The heating of the air-bath is facilitated by employing an ordinary combustion furnace with the introduction of asbestos sheeting which maintains the temperature uniform and reduces the gas consumption.
3. "Kupfer-bronz" can be used as catalyst in small operations.

Notes.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

THE COMPOSITION OF COCONUT OIL.

THE publication of remarks by Mr. G. D. Elsdon (ANALYST, 1924, 49, 423) on the determination of the composition of the mixed fatty acids of fats by the method of fractional distillation of the methyl or ethyl esters has brought to light an omission from the table contained in the Presidential address to the Society of Chemical Industry, recently delivered by the retiring President, Dr. E. F. Armstrong (*J. Soc. Chem. Ind.*, 1924, 43, 207T), which shows the percentage composition of fatty acids derived from certain fats.

Owing to the unfortunate omission of an identifying numeral the composition of coconut oil therein quoted, which is the work of E. Paulmyer (*J. Soc. Chem. Ind.*, 1907, 26, 881) has not been attributed to him. This is the more unfortunate, as Mr. Elsdon quotes these figures in his comments.

As was stated in the text of the address, the authors are in agreement, to a considerable extent, with Elsdon and with Channon, Drummond and Golding, that the method is capable of giving only an approximation to the true composition of the fat under examination, but they are convinced that many of the obviously erroneous results are to be attributed to the following causes:

- (1) The use of more or less imperfect fractionating apparatus, and an endeavour to arrive at fractional separation of the mixed esters which

have been prepared from fatty acids before any attempt has been made to separate the saturated and unsaturated constituents of the original fatty acid mixture.

- (2) Assumptions as to the composition of the fractions obtained without having identified the fatty acids which are assumed to be present in them.

It is proposed to publish in the near future the results of an extended investigation, now nearly completed, of the fatty acids obtained from coconut oil, in the carrying out of which special precautions have been taken to avoid, as far as possible, the sources of error which surround this particular method of analysis.

JOHN ALLAN.

DETERMINATION OF MILK SUGAR IN MILK.

THE following details for carrying out the iodimetric determination of milk sugar in milk (*cf.* Kolthoff, *ANALYST*, 1923, 48, 386) have been found to work well.

Ten c.c. of milk are weighed into a 100 c.c. flask and diluted with 50 c.c. of distilled water; 10 c.c. of Mayer's reagent and 2 c.c. of *N* sulphuric acid are added, the whole well shaken and the volume made up to 100 c.c. The solution is filtered, and 25 c.c. of the filtrate are neutralised to phenolphthalein (1 drop used); then 20 c.c. of 0.1 *N* iodine solution and 30 c.c. of 0.1 *N* sodium hydroxide solution are added. After 20 minutes, 4 c.c. of 0.1 *N* sulphuric acid are added, and the residual iodine titrated with 0.1 *N* thiosulphate solution.

The percentage of anhydrous milk sugar by weight is calculated thus:

$$\text{Per cent.} = \text{c.c. iodine used} \times 0.0682 \times \frac{100 - (0.3 + \text{Fat} \times 1.11)}{\text{Weight of milk}}$$

If desired, 10 c.c. of milk may be measured, and the weight of this taken as $10 \times \text{sp. gr.}$

For the determination of milk sugar in condensed milk 10 c.c. of a mixture of 1 part of condensed milk and 2 parts of water, both by weight, are used, or about 3.5 grms. are weighed direct into the flask.

H. DROOP RICHMOND.
L. R. ELLISON.

THE DETERMINATION OF FAT IN CONDENSED MILKS.

FOR the determination of fat in condensed milks the Röse-Gottlieb method is usually recommended. The Werner-Schmidt method gives low and variable results, especially in the case of sweetened condensed milk, with which there is a considerable amount of charring of the sucrose.

The following modification of the latter process was devised to overcome this difficulty, and has given very satisfactory results. It depends on the fact, embodied in Ritthausen's process, that the sugars can be filtered off after addition of copper sulphate solution to an emulsion of the milk, the fat remaining entangled in the precipitate. Copper sulphate was chosen as being the most convenient solution to use, but any other protein precipitant would probably serve equally well.

From 2 to 3 grms. of the condensed milk are weighed into a fat flask and completely emulsified by means of a glass rod with about 20 c.c. of cold water. Five c.c. of Fehling's copper sulphate solution are added, and the flask placed on

the water bath for a few minutes to complete the precipitation of the proteins. The contents are then filtered through a wet ribbed filter paper, the flask being washed out with warm water and the washings passed through the filter paper. Some of the precipitate remains adhering to the walls of the flask and need not be removed. The filter paper containing the precipitate is transferred to the flask, and 10 c.c. of hydrochloric acid are added. The filter paper is roughly broken up with the glass rod, and the rod, after washing, is then removed. The flask is placed on the water bath until the fat can be seen as clear globules on the surface of the liquid. Half an hour is usually sufficient. The filter paper by now is completely disintegrated and no charring takes place. The fat is obtained by three extractions with petroleum spirit, the extract being washed to remove traces of acid. The petroleum spirit is evaporated, and the fat weighed after the flask has been in the oven for about an hour.

The results obtained by this method have been checked by a second determination by means of the Röse-Gottlieb process, and in each case the two results have been in close agreement.

[R. W. SUTTON.

A CASE OF PRUSSIC ACID POISONING WITH LINSEED CAKE MEAL.

A SAMPLE of linseed cake meal which was alleged to have caused the death of a calf nine weeks old, and to have produced "stomach staggers" in the case of another calf, was recently sent in for examination. The food had been given in the form of a gruel made by soaking the meal for 12 hours in cold water.

The "free" prussic acid was determined by the method given in Auld and Ker's *Practical Agricultural Chemistry*, and 0.054 per cent. was found. This quantity is exceedingly high—in fact, the highest that has come to the writer's notice. There is no doubt that the method of preparation of the gruel, coupled with a cake of high glucoside content and enzymic activity, was responsible for the death of the animal.

H. T. CRANFIELD.

THE MIDLAND AGRICULTURAL AND DAIRY COLLEGE,
SUTTON BONINGTON, LOUGHBOROUGH.

DETERMINATION OF CADMIUM IN SPELTER AND ALLOYS.

IN view of the need of an expeditious method of determining cadmium in spelter, etc., it might be worth while to investigate the applicability of the precipitation of tetramminecadmium iodide (ANALYST, 1917, 42, 189) to the quantitative separation of cadmium from zinc. The scheme I have in mind consists in precipitating cadmium sulphide, mixed with a certain amount of zinc sulphide, from an ammoniacal solution by cautious addition of dilute sodium sulphide solution. The precipitate is dissolved in hydrochloric acid, the solution is evaporated, and the dry residue is taken up in a few c.c. of tartaric and dilute nitric acid solution. The cold solution is then treated with 30 c.c. of strong ammonia and 4 grms. of solid potassium iodide (ANALYST, 1919, 44, 275). The white crystalline precipitate is filtered off and washed free from zinc with a 4 per cent. solution of potassium iodide in ammonia (4:1 water); it is dissolved in a little sulphuric acid, and the cadmium determined as phosphate (ANALYST, 1916, 41, 124). The proposed method follows the lines of the separation and estimation of cobalt, described in the papers referred to above.

W. R. SCHOELLER.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

LABELLING OF MARGARINE.

ON November 7 the Glasgow Stipendiary Magistrate heard the arguments of counsel in a case brought before him under the Butter and Margarine Act, 1907. The facts were not in dispute, and the sample was proved to be margarine, but the point to be decided was whether the words on the wrapper, "Containing a small quantity of butter, Dutch made," formed part of the descriptive name of the article. These words were in small type beneath the words "Charmo Margarine" in large type, the name "Charmo" being a fancy name approved by the Board of Agriculture and Fisheries, and permitted to be combined with the word "Margarine" under Sec. 8 of the Butter and Margarine Act, 1907.

In giving his decision, the Stipendiary said that he had come to the conclusion that the words in question were part of the descriptive name of the accused's margarine, and that they were a contravention of Section 8 of the Butter and Margarine Act, 1907, as such name had not been, and admittedly could not be, approved by the Board of Agriculture and Fisheries. Counsel for the accused had submitted that there was a distinction between this case and the case of *Maypole Dairy Company v. Paterson* (1923, J.C. 85), and that this case was on all fours with the case of *Hawes v. Stephens* (K.B.D., May, 1924). He (the Stipendiary) could not see any difference between this case and that of the Maypole Dairy Company. The words in that case were "Mixed with Maypole butter," and he could see no difference between those words and the words in this case, *viz.* "Containing a small quantity of butter, Dutch made," which, in his opinion, were so associated with the words "Charmo Margarine" as together to constitute a descriptive name of the accused's margarine in the sense of the section libelled.

Accordingly he imposed a fine of £5, but agreed to state a case for appeal.

CUSTARD WITHOUT EGG.

ON November 5th a grocer was summoned at Durham County Petty Sessions for the sale of custard that was not of the nature, substance and quality demanded.

The Deputy Clerk of the Peace stated that six penny packets of "custard" had been purchased from the defendant, and that these, on analysis, had been found not to contain a mixture of eggs and milk. Although no standard had been laid down for custard, as in the case of milk, reference to the dictionaries showed that "custard" was "a mixture of eggs and milk."

The County Analyst (Mr. C. J. H. Stock) gave evidence to the effect that the samples analysed by him consisted entirely of maize starch, coloured and flavoured.

Dr. Hill, Medical Officer to the County Council, said that, in his opinion, the basis of all custard was egg, and that, in the making of custard, milk was a necessity.

The solicitor for the defence pointed out that not one person in a thousand went into a shop and asked for a box of custard. In this case the word "powder"

had been accidentally omitted from some of the smaller labels. There were on the market any number of powders which did not contain egg or milk. These ingredients were added afterwards.

The Court convicted the defendant, and imposed a fine of £3.

MILK "AS IT CAME FROM THE COW."

DAVIS *v.* BLACKMAN.

ON November 11th an appeal was heard in the Divisional Court, before the Lord Chief Justice and Justices Shearman and Salter, from a decision of the Southampton County Justices. The defence had been set up that the sample of milk, which had been found to be deficient in milk fat, was sold "as it came from the cow," and the question raised was whether this plea was sufficient to rebut the prosecution.

Mr. W. Blake Odgers, counsel for the appellant, an inspector under the Sale of Foods and Drugs Act, stated that the guidance of the Court was desired upon the question whether the justices should have allowed analytical evidence to be given to show the composition of other samples of milk produced under the same conditions in the same part of the country. The justices had excluded such evidence on the ground that it was irrelevant.

In giving his judgment the Lord Chief Justice said that in recent years the formula "as it came from the cow" had come to be regarded as almost of sacramental efficiency in cases relating to the adulteration of milk. In this case the appellant had sought to give evidence for the purpose of showing that the justices ought to be exceedingly careful before accepting evidence intended to show that milk was being sold in the condition in which it came from the cow. The reason given by the justices for not admitting this further evidence was that the appellant was confined, by the form of the information, to the regulations of the Board of Agriculture. In his opinion this view was erroneous.

The other Justices concurred, and the case was remitted to the Southampton justices for further consideration.

Queensland.

REPORT OF THE GOVERNMENT ANALYST FOR THE YEAR ENDED JUNE 30, 1924.

THE number of samples examined during the year was 6817, of which 1438 were for the Customs, 1533 for the Department of Health, 1606 for the Postmaster, and 991 for the Mines Department. The drop of 771 in the number of mines samples was mainly due to fewer samples being sent from the State arsenic mine.

Of the 615 legal samples taken by inspectors under the provision of the Health Acts, 367 were passed, 165 failed to reach the standard, and 62 were unfit for analysis. There were 38 prosecutions (30 for milk, 5 for spiritous liquors and 3 for poisons) and convictions were obtained in every case. The large proportion of "failures" to reach the standard arose through these samples having been found by the inspectors to be close to or below standard in their preliminary examination.

Of the 525 legal samples of milk, 65.5 per cent. passed the standard, 12.8 per cent. were adulterated with water, 3.6 per cent. were deficient in fat, 11.8 per cent. were unfit for analysis, and 6.3 per cent. were genuine, but slightly below standard.

INFANTS' FOODS.—The following analyses of infants' foods on the local market were made in order to ascertain their position with regard to the standard for infants' food suggested by the 1922 Food Conference.

Food.	Water. Per Cent.	Protein. Per Cent.	Fat. Per Cent.	Carbohydrate. Per Cent.	Mineral Substance. Per Cent.	Cold Water Extract. Per Cent.	Crude Fibre. Per Cent.	Remarks.
Glaxo	3.2	24.1	26.6	40.5	5.6	—	—	Desiccated milk with added lactose.
Lactogen	2.7	25.0	26.2	40.4	5.7	—	—	Desiccated milk with added lactose.
Horlick's Malted milk	5.1	12.5	7.9	70.9	3.6	74.5	Nil	Desiccated milk and desiccated malt extract.
BacchusMarsh malted milk	3.7	11.2	10.8	71.2	3.1	—	—	Desiccated milk and desiccated malt extract.
Mellin's Food	5.6	10.2	0.9	80.6	2.7	82.8	—	Desiccated malt extract.
Benger's Food	5.4	10.9	0.8	81.9	1.0	16.8	Nil	Wheat flour and pancreatic extract; very little unchanged starch when made as directed.
Savory and Moore's	6.7	9.8	1.2	81.5	0.8	—	—	Wheat flour and malt extract; most of starch changed when prepared as directed.
Allenbury's Malted Milk	4.9	9.7	0.8	83.5	1.1	—	—	Wheat flour and malt extract; most of starch changed when prepared as directed.
Nestles' Milk Food	3.3	12.8	4.1	78.1	1.5	66.0	0.2	Desiccated milk, bakedwheat flour, and cane sugar.
Neave's Food	3.2	13.4	1.2	81.1	0.7	6.6	0.4	Baked wheat flour.
Australian Groats	3.3	9.8	7.2	78.4	0.8	—	0.5	Ground oats.
Robinson's Groats	7.6	12.3	6.8	70.4	1.5	—	1.4	Ground oats.

COCOA POWDER.—Cocoa powder for use in a tropical climate should not contain more than 5 per cent. of water, as a proportion in excess of this limit tends to make the powder lumpy and liable to attack by fungoid growths. Several of the samples examined were lumpy and deteriorated owing to the water content being above the safety limit. The samples were free from foreign fat and added starch. Several of them contained fibre considerably in excess of the maximum proportion permitted to be present.

LIQUID PARAFFIN.—Only one sample of liquid paraffin failed to conform to the B.P. standard. This standard, however, is not comprehensive enough, in that it does not take cognisance of what is probably the most important feature of liquid paraffin for internal use, *i.e.* viscosity; a thin oil traverses the system rapidly and it is possible that, by reason of its solvent nature, it would be injurious. The samples examined ranged in viscosity from 62 to 212. The *Lancet* reported figures on commercial samples ranging from 67 to 440 and recommended a viscosity of at least 105 and a gravity not less than 0.88 (B.P. 0.86 to 0.89).

J. B. HENDERSON.

Government of Palestine.

ANNUAL REPORT OF THE DEPARTMENT OF HEALTH FOR THE YEAR 1923.

THE sections in this Report include Vital Statistics and Epidemic Disease, Medical Services, Sanitary Administration, Relief, Quarantine, and Laboratory Work comprising (a) Bacteriological, (b) Chemical, and (c) Work at the Antirabic Institute.

CHEMICAL DIVISION.—A summary of the work done during the year is given by Mr. G. W. Baker, the Government Analyst. Most of the Government Departments made use of the laboratory, and, in all, 4176 samples were examined. Of these, 3497 samples were milk, of which 846 were taken in the Jerusalem district. There were 236 prosecutions for the sale of watered or skimmed milk.

BUTTER.—The articles sold as butter in Palestine may be divided into three classes:

1. Australian butter imported chiefly for those merchants who cater for non-Palestinian customers. The three samples examined conformed to the English standard for butter.

2. Semni (or cooking butter) manufactured locally. This should consist entirely of butter fat from the milk of the cow, goat or sheep, and should be practically free from casein and water. Genuine semni commands a good price, and therefore invites adulteration with cheap fats; and a large proportion now offered for sale is mixed with, or consists entirely of, vegetable fats or oils.

3. Artificial butter. There are numerous butter factories producing a blend of milk with a vegetable oil or fat. These products can be purchased in printed wrappers marked "Artificial butter," "Almond butter," etc., but there is no doubt that they are also sold loose under the name of "butter" alone. Out of ten samples purchased as butter in the Jaffa district, nine proved to be composed of fat other than butter fat and contained an excessive amount of water. Under a new ordinance it will be possible to fix standards and take steps to prevent the sale of spurious articles.

CANNED ARTICLES.—Samples of jam of local manufacture have been found to contain zinc. Unused cans from the factory have been examined and zinc chloride found in the seams inside the can. Zinc chloride had evidently been used as a soldering flux. The manufacturer stated that he would shortly be in a position to use properly made cans. In the meantime he has been instructed to use rosin in the place of zinc chloride.

The importation of pickles from Egypt contained in old petrol tins has been investigated with the assistance of the Public Health Authorities in Cairo. Steps have been taken to prohibit the importation of pickles in metal containers.

ALCOHOLIC LIQUORS.—Further investigations into the trade in spurious liquors have been carried out. It has been established that potable spirits are manufactured in large quantities from "industrial" or refined spirit imported in iron drums at a strength of 96 per cent. alcohol. This alcohol is watered down to about 40 per cent. and suitable flavouring materials are added. It is sometimes distilled, but more often the "cold process" is employed, which means simple dilution without distillation. This diluted alcohol is then bottled and labelled; this is done both at the distillery and at retail establishments. Cognac, arak, rum, and whiskey are all made by this process.

A large variety of spurious labels has been found in the bottling establishments. These are mostly printed in France. Bottles filled from one cask of spirit may be labelled differently and sold at different prices. The statements on the labels are calculated to deceive the purchaser into the belief that he is buying a genuine imported article.

Measures of control have been considered, and, pending their application, prosecutions have been instituted in certain cases under a section of the penal code. These are looked upon as test cases. They are still in the Courts.

DEAD SEA WATER.—Experiments on the evaporation and crystallisation of water from the Dead Sea have been carried out, and a sample of carnalite was prepared from the water to be sent to the British Empire Exhibition.

LEGAL AND CRIMINAL INVESTIGATION.—The following poisons have been detected in cases of human poisoning:—Arsenic (1), mercury (1), alcohol (1), mydriatic alkaloid (seed of *Datura Metel*) (3), hydrastine (1), opium (1), and chloral hydrate (1). Arsenic was found in 13 cases of cattle poisoning.

The seeds of *Datura*, when used as a poison, are generally crushed and inserted into the middle of dried figs; robbery is usually the motive in these cases. Dried figs are also a favourite vehicle for arsenic.

A counterfeit coin contained 26 per cent. of silver, the remainder being chiefly copper and antimony.

The Problem of Atmospheric Pollution.*

In the year 1923 there were 3262 deaths in Salford, more than a third of which were due to respiratory disease, including lung consumption. Comparison of the borough statistics with those of England and Wales, as a whole, shows that the percentage of deaths from non-tubercular respiratory diseases in Salford is unduly high. The corresponding death rates from this cause per million persons living were:—Salford, 3092; England and Wales, 1867. The charts for years free from influenza epidemics (1916 and 1917) show a close relationship between fog periods and deaths from respiratory diseases, and confirm the results of clinical experience of the effects of smoke fog on patients whose respiratory organs are already damaged.

MEASUREMENT OF ATMOSPHERIC POLLUTION.—This is now systematically carried out by means of the "Standard Gauge," followed by analysis. The results for each month at four stations, as determined by Elsdon, are given in tabular form. The total and average monthly impurities for the 6 months from October, 1923, to March, 1924, were as follows, the figures representing metric tons per sq. kilometre:

	Tar.	Other insoluble carbonaceous matter.	Soluble matter. Loss on ignition.	Ash (soluble and insoluble).	Total solids.
Total, 6 months	1·45	17·07	13·75	47·13	79·40
Average, 1 month	0·24	2·84	2·29	7·86	13·23

Assuming the amount of atmospheric deposit at one of the Salford stations to represent an average for the Borough, the amount of "dirt" deposited was at the rate of about 3000 tons per annum.

* By H. Osborne, M.D., Medical Officer of Health, Salford.

The estimated consumption of coal in factories and in houses in the Borough is as follows:—Factories, 5877 tons per week all the year round; houses, 7077 tons per week (summer) and 11,305 tons per week (winter). In addition to this, the efficiency of combustion of household fires is only 65 per cent., as compared with over 90 per cent. in factories. Hence it would appear that the domestic fire is a greater cause of pollution than the factory fire.

CHARACTER OF POLLUTION FROM DOMESTIC FIRES.—In recent investigation on the problem of air pollution by domestic fires, carried out for the Manchester Air Pollution Board, the following comparative analyses were made by E. F. Greig:

ANALYSIS OF FLUE GASES from (1) A Coal Fire and (2) A sitting-room Gas Fire, per 1000 B.Th.U. available in fuel, the two fires being at maximum efficiency.

Unburned Gases, &c.	Coal Fire.	Gas Fire.
Carbon dioxide	1.38 cubic feet	1.03 cubic feet
Carbon monoxide	0.04 "	0.10 "
Methane	0.02 "	0.05 "
Unsaturated hydrocarbons	0.03 "	0.08 "
Soot	43.2 grains	2.6 grains

These results bring into prominence the fact that an ordinary gas fire produces much more carbon monoxide than a coal fire. They do not include the sulphur products (which are capable of setting up respiratory diseases) in the flue gases from either fire.

THE REMEDY.—It is not probable that the gas fire or radiator will replace the domestic fire, and from many points of view (*e.g.* health, value of visible rays) it is not advisable that they should do so. The solution of the problem appears to lie in the adoption of special carbonised fuel to be burned in open grates. The effect of carbonisation on the proportion of sulphur is shown in the following results:

1. Sulphur contained in raw Lancashire coal 2.93 per cent.
2. " " Smokeless fuel made from (1) without use of steam 2.14 "
3. " " Smokeless fuel made from (1) with use of steam 1.26 "

The steam motor wagon is a frequent cause of pollution, and since the prosecution of an owner, on June 2, 1920, it has been almost impossible to get a conviction. In that case, it was successfully contended that no penalty could be imposed, as the emission of smoke was due to "temporary or accidental causes," as stated in Locomotive on Highways Act, 1896. To be successful in a prosecution it is now necessary for an inspector to follow the steam wagon, and to prove that smoke was repeatedly emitted during its passage through the Borough.

Ministry of Health.

CIRCULAR 553. SALE OF FOOD AND DRUGS ACTS, &c.

THE following circular has been sent to the Clerks of Authorities administering the Food and Drugs Acts:—

SIR,

1. I am directed by the Minister of Health to request that a copy of the Report of the Public Analyst for the last quarter of the present year and a copy of the Medical Officer of Health's Report upon the administration of the Public Health (Milk and Cream) Regulations during the year, may be forwarded during the month of January.

2. An instance has been brought to the notice of the Department in which a part of a sample of dried milk taken under Article 5 of the Public Health (Dried Milk) Regulations, 1923, was placed in a paper envelope. The consequence was that, by reason of the absorption of water from the atmosphere into the dried milk and of fat from the dried milk into the envelope, the result of the analysis would not represent the actual composition of the article at the time of sale. It is desirable that samples of articles of this character should be placed in securely sealed bottles or other non-absorbent and air-tight containers, so that there may be no material change in the composition of the sample between the time of purchase and the time of analysis.—I am, etc.,

R. B. CROSS, *Assistant Secretary.*

December 29, 1924.

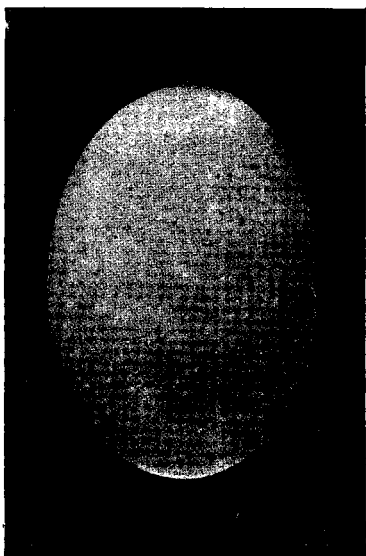
ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

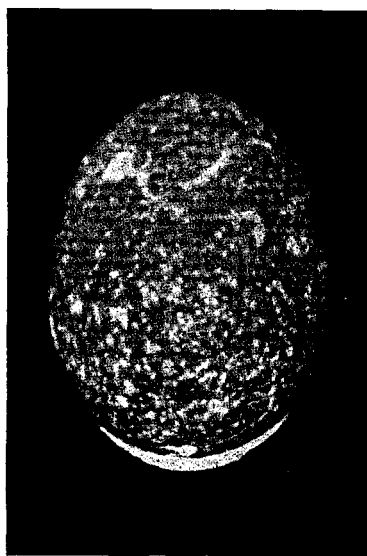
Modifications in the Transparency of the Shells of Eggs on Keeping.

A. M. Leroy. (*Ann. Falsificat.*, 1924, 17, 407-408.)—When a newly-laid hen's egg is examined by strong transmitted light (e.g. from a 50 c.p. metal filament lamp), the shell shows a uniform appearance, with darker points here and there due to calcareous granulations, the number and distribution of which are fairly constant for all the eggs from the same hen; about 10 to 12 hours after laying, light patches begin to appear on the dark background of the shell, and by the following day these show well-defined margins, and these are generally more concentrated towards the poles of the egg than in the intermediate zones. They are still more pronounced on the next day, but from that time onwards keep the same appearance until obvious alterations appear. By this means it is possible to distinguish between eggs laid the same day and those laid two or three days previously. There are pronounced variations in the number, relative size, and distribution of the

light patches in the shells of the eggs of different hens, but these characteristics are remarkably similar in the case of eggs from the same hen. The patches are independent of the protein membranes in the egg, and experiments have indicated



Newly laid Egg.



Egg, 12 hours after laying.

that their formation is due to certain parts of the shell being hygroscopic, whilst other parts rapidly lose their water of constitution by evaporation and become darker in colour. Their appearance is promoted by heat and retarded by humidity. Eggs kept in an atmosphere saturated with moisture will keep their transparency indefinitely.

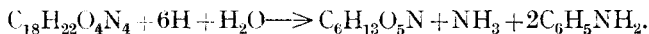
Colorimetric Determination of Sucrose in the presence of other Sugars.

H. Riffart and C. Pyriki. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **48**, 197-207.)

—After a detailed discussion on the influence of temperature, time, and concentration on the colour produced by various sugars with sulphuric acid, it is shown that sucrose can be determined with an accuracy of about ± 0.5 per cent. in the presence of other sugars by means of the colour produced by 70 per cent. sulphuric acid at 55° C. The colour is really due to fructose, and so is given by those sugars that produce fructose under the conditions of experiment; the method is therefore available in the presence of glucose, lactose, maltose, or galactose. Albumin or fat must first be removed, and when this is done good results are obtained in the determination of sucrose in condensed milk, biscuits, cake, chocolate, or beer. From 5 to 20 grms. of the material are made into a cream with water, treated with a clarifying agent diluted to 100 c.c. and filtered; 5 to 10 c.c. of the clear colourless filtrate are mixed with twice their volume of 70 per cent. sulphuric acid

and heated on a water bath at $55^{\circ}\text{C.} \pm 1^{\circ}$ for exactly 10 minutes, the thermometer being placed in the liquid as soon as that temperature is reached; the mixture is then cooled and the colour compared in Nessler glasses with that of a standard sucrose solution similarly treated. The more strongly coloured solution is diluted as required, but if its volume exceeds 50 c.c. it is advisable to start again with a fresh and smaller quantity. In the case of milks or chocolate the casein is removed with an acid mercuric chloride solution in the usual way. H. E. C.

Direct Method for the Determination of Glucose and other Carbohydrates. E. Knecht and E. Hibbert. (*J. Chem. Soc.*, 1924, 125, 2009–2012.)—By increasing the molecular proportion of phenylhydrazine to sugar to 12:1 the osazone is quantitatively formed and may be titrated with accuracy by means of titanous chloride without actually being isolated. To 10 c.c. of a 0.1 to 0.2 per cent. solution of the sugar are added 1 c.c. of saturated sodium tartrate solution and 0.25 grm. of phenylhydrazine dissolved in acetic acid; the mixture is heated for ten minutes in a briskly boiling water bath, then a large excess of standard titanous chloride solution is added, and the solution boiled over a free flame for two minutes, a current of carbon dioxide being passed into the flask meanwhile. The excess of titanous chloride is then titrated hot with a solution of crystal violet until a permanent red colour is produced. The crystal violet solution is standardised against the titanous chloride sugar, the equivalent of which can be calculated from the equation:—



Lavulose and invert or other sugars forming osazones may be similarly determined. H. E. C.

Acidity of the Oil in Olives. R. Marcille. (*Ann. Falsificat.*, 1924, 17, 398–400.)—The pulp of about 20 olives is crushed in a mortar, with the addition, from time to time, of small quantities of neutral anhydrous sodium sulphate until the mass is no longer sticky. Then, after standing for 2 or 3 minutes, the paste is covered with anhydrous redistilled carbon disulphide, and the grinding repeated. The extract is filtered, the residue washed two or three times with the solvent, so as to obtain in all about 50 c.c. of filtrate and washings. These are collected in a weighed glass basin, allowed to evaporate spontaneously; the residue is weighed and redissolved in neutral ether, and the solution titrated with standard alcoholic sodium hydroxide solution (phenolphthalein as indicator); or the residue may be dissolved in a neutral mixture of benzene and 95 per cent. alcohol (2:1), and Poirrier blue C4B used as indicator. Carbon disulphide is the best solvent, because it is not easy to remove petroleum spirit completely from the residue, and benzene and ether extract constituents other than oil from the epidermis of the fruit. For example, the following amounts of acid (in terms of oleic acid) were determined in the material extracted with these various solvents from the same batch of olives: Carbon disulphide (spontaneous evaporation), 0.39; carbon disulphide (final

evaporation in oven), 0.36; petroleum spirit, 0.36; benzene, 1.56; and ether, 2.18 per cent. The acidity of the oil in fresh olives of other varieties cultivated in Tunis ranged from 0.29 to 0.50 per cent. Olives delivered to the works for expression contained oil with an acidity of 0.62 per cent., rising to 1.3 per cent. after 6 days, and 2.18 per cent. when the fruit was bruised. The increase in the acidity was due to the lipolytic action of the mould, *Penicillium glaucum*.

Pectin in Fruits and Fruit Products. H. J. Wichmann. (*J. Ass. Off. Agric. Chem.*, 1924, 8, 123-130.)—The material is pulped by passing it through a meat grinder, care being taken neither to crush seeds nor to lose liquid. After thorough mixing, 300 grms. are boiled with 800 c.c. of water for 1 hour in a 1.3 to 2 litre beaker, the evaporated water being replaced occasionally. The mass is then transferred to a 2000 c.c. measuring flask, cooled, made up to volume, and filtered through a folded paper. To 200 c.c. of the filtrate are added 2 to 4 lumps of cube sugar if the solution does not contain sugar; 25 c.c. are evaporated and treated with 200 c.c. of 95 per cent. alcohol, allowed to settle and filtered, and the precipitate washed with 95 per cent. alcohol and transferred to the original beaker with hot water. The pectin solution is evaporated to 40 c.c., cooled to 25° C. or below, and, if water-insoluble substances separate during evaporation, it is stirred vigorously and, if necessary, treated with a few drops of 10 per cent. hydrochloric acid and warmed. It is cooled again, and from 2 to 5 c.c. of 10 per cent. sodium hydroxide solution, mixed with sufficient water to make a total volume of 50 c.c. are added, the bulk of the precipitate indicating approximately the quantity to use. After the lapse of 15 minutes, 40 c.c. of water and 10 c.c. of 10 per cent., hydrochloric acid, are added, the liquid boiled for 5 minutes, and the pectic acid filtered off on a qualitative filter-paper and washed with hot water. Filtration should be rapid and should yield a clear filtrate; if it is cloudy or colloidal, the determination should be repeated with addition of more alkali. The pectic acid is washed back into the beaker, the contents of which are adjusted to 40 c.c., cooled to below 25° C., and the saponification and precipitation repeated as described. The liquid is filtered and the pectic acid washed with hot water only until the filtrate shows a negligible amount of acid; more than 500 c.c. of total filtrate should be unnecessary. Finally the pectic acid is washed into a platinum dish and dried on a steam-bath and afterwards in a water-oven to constant weight. It is ignited, and weighed again; the loss in weight represents pectic acid.

T. H. P.

Optical Rotations produced by Lemon Oil and Sweet Orange Oil in Alcoholic Solution. W. W. Randall. (*J. Ass. Off. Agric. Chem.*, 1924, 8, 206-214.)—To find the approximate percentage of lemon oil by volume in an alcoholic extract of lemon, the angular degrees of rotation of the extract at 20° C. in a 200 mm. tube should be divided by 1.12, or the Ventzke degrees by 3.22; for sweet orange extract the corresponding divisors are 1.81 and 5.21 respectively.

T. H. P.

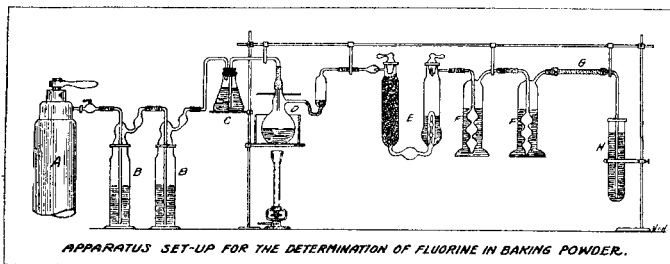
Acids of Whortleberries and Tamarinds. H. Kaiser. (*Chem. Zeit.*, 1924, 48, 700-701.)—The acids of whortleberries and tamarinds have been investigated and, on the assumption that the losses by the individual acids at the completion of the work are always proportionately equal, they were present in the following amounts:—

WHORTLEBERRIES.			TAMARINDS.		
		Per Cent.			Per Cent.
Lactic acid	..	0.81	Lactic acid	..	0.007
Oxalic acid	..	0.16	Oxalic acid	..	0.022
Succinic acid	..	4.87	Succinic acid	..	0.16
Malic acid	..	18.70	Malic acid	..	2.34
Citronic acid	..	72.38	Tartaric acid	..	96.68
Quinic acid	..	2.68(?)	Citronic acid	..	0.04
Unsaturated acids	..	0.40(?)	Unsaturated acids	..	0.75(?)

The method of separation used was based on the difference in solubility of the hydrazides and benzylidene compounds of the respective acids. These esters were prepared after defecation of the juice with lead acetate and separation of pectins by means of alcohol.

P. H. P.

Volatilisation Method for the Determination of Fluorine in Baking Powder. J. K. Morton. (*J. Ass. Off. Agric. Chem.*, 1924, 8, 101-105.)—Twenty grms. of the powder are heated in an 11 cm. porcelain dish in a muffle furnace at a temperature causing no discernible reddening (about 450° C.). The dark ash is powdered, evaporated with 5 c.c. of 10 per cent. magnesium nitrate



solution and again heated as before; this operation is repeated once, and the ash then kept in a desiccator until used. The further procedure, which is a modification of that given by Wagner and Ross (*J. Ind. Eng. Chem.*, 1917, 9, 1116), involves the use of an apparatus comprising a source of air, carbon dioxide, or hydrogen at constant pressure; a gas-drying apparatus; a flask of about 100 c.c. capacity, from which the anhydrous sulphuric acid may be forced by the gas into the digestion flask, which consists of a 4 oz. pyrex distilling flask with a small trap and reflux tube at its outlet; a Schmidt's tube containing glass beads in one arm and, in the other, a 10 per cent. solution of silver sulphate in anhydrous sulphuric acid;

Bowen absorption bulbs containing a saturated solution of dry chromic acid in anhydrous sulphuric acid; a horizontal glass tube, 6 by $\frac{3}{8}$ inches, containing glass wool; a test-tube containing water, into which the silicon fluoride is delivered. The ash, together with 1 grm. of quartz flour, previously digested with concentrated hydrochloric acid, washed free from acid, and dried, and 5 grms. of anhydrous copper sulphate, are introduced into the digestion flask, and mixed thoroughly, the flask being replaced. After 75 c.c. of anhydrous sulphuric acid have been placed in the 100 c.c. flask and 15 c.c. of water in the final test-tube, the gas is turned on very slowly and the pressure allowed to force the acid into the digestion flask; the first portion of the acid is passed into the trap to form a seal. Sufficient flow of air being maintained to prevent back travel through pressure, the digestion flask is heated gradually to about 300° C.; after being kept at this temperature for 2 hours, with frequent shaking, the contents of the flask are boiled for 5 minutes, the flame then removed and the air passed for 30 minutes to remove any silicon tetrafluoride from the system. The contents of the test-tube are boiled gently for 10 minutes with about 250 c.c. of water in a 500 c.c. Erlenmeyer flask, and then cooled to 50° C., the hydrofluosilicic acid being titrated with 0.1 N alkali in presence of phenolphthalein (1 c.c. corresponds with 0.0019 grm. of fluorine). After the titration, the solution is made acid with hydrochloric acid and tested for sulphate; if necessary, the sulphate is determined and the titration result corrected accordingly.

T. H. P.

Gum Tragacanth, W. Peyer. (*Chem. Zeit.*, 1924, 48, 701.)—Gum tragacanth (*Astragalus gummifer*) is not a secretion, but is a pathological formation, probably due to external irritation by insects, etc., and oxidation by way of the injury. According to Tschirch, Hartwig, Rosenthaler and Runne, gum tragacanth does not contain oxidising enzymes, and although all forms of tragacanth give the guaiacum reaction, the author, in common with Oppenheimer, thinks this evidence uncertain; as also that of the pyrogallol reaction described in all the text books. The presence of oxidase in tragacanth mucilage (1:50) is indicated, however, by the addition of eserine, apomorphine, hydroquinone, phenolphthalein, aloin, guaiacum resin tincture, guaiaconic acid solution, guaiacol, α -naphthol, p -phenylenediamine, potassium iodide starch solution, with and without hydrogen peroxide or ethylhydroperoxide solution, α -naphthol- and dimethyl- p -phenylene-diamine. For testing gum tragacanth powder, the following substances are recommended in order of their utility:—Benzidine, adrenaline (1:1000), orcinol, gallic acid, resorcinol and leucomalachite green. In genuine tragacanth mucilage there are to be seen one or more spongy particles, often as large as lentils, which are dyed more or less intensely according to the reagent applied. These may be granules of the gum which become saturated with the solution of the reagent and dyed. Apparently some parts of the gum are richer in oxidase than others. Furfural is produced from the pentoses of tragacanth and reacts with orcinol, probably with the formation of orcein. It is uncertain whether resorufin or resacoin are produced in the resorcinol reaction.

P. H. P.

Japanese Saffron. E. Skarnitzl. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, 48, 213-217.)—Much of the Japanese saffron (*Crocus japonicus*) now on the market has been found to be largely adulterated with coloured barium sulphate and the leaves of the horse-thistle, *Onopordon acanthium*. The botanical and microscopical features of the latter are described in detail and illustrated. The more important diagnostic features are the large unicellular thick-walled bristles or hairs, which are shaped like a canine tooth and measure from 100-160 μ by 30-38 μ , and the short thickened trichome cells of similar dimensions attached to the large sclerenchymatous fibres.

H. E. C.

Biochemical, Bacteriological, etc.

Chemical Constitution and Antiseptic Power. Th. Sabalitschka. (*Chem. Zeit.*, 1924, 48, 703.)—The effect of various concentrations of different substances, with different hydrogen ion concentrations of the nutrient medium, on the development of *Micrococcus candidans*, *Bacillus coli*, *Sarcina flava* and *Penicillium glaucum* was tested, and the connection between chemical constitution and preservation effect was followed. Of the inorganic and aliphatic acids tried, formic acid had the strongest effect, a solution of 0.036 per cent. concentration being sufficient to prevent development of bacteria and fungi. The effect diminished in the following order:—Acetic acid, hydrochloric acid, sulphuric acid, maleic acid, malonic acid, fumaric acid, tricarballic acid, suberic acid, phosphoric acid, malic acid, succinic acid, *d*-tartatic acid and glutaric acid. The inhibiting action of numerous cyclic compounds, such as benzoic acid, hydroxybenzoic acid, salts, esters and other derivatives of these acids, phenols, carbohydrates, etc., was also studied. Phenol, thymol and carvacrol caused the greatest effect, concentrations of 0.014 per cent. inhibiting the development of both bacteria and mould fungi. The methyl esters of benzoic acid, *p*-hydroxybenzoic acid, *m*-hydroxybenzoic acid, anisic acid, and cinnamic acid were effective at a concentration of 0.14 per cent., salicylic acid and metachlorbenzoic acid at 0.21 per cent., and acetylsalicylic acid, and pyrocatechol at 0.28 per cent. Sodium benzoate only prevented development at a concentration of 1.5 per cent., and the sodium salts of the three hydroxybenzoic acids were not effective even at 2 per cent. The antiseptic action is increased by an alkyl hydroxyl group, decreased by a COOH-, HSO₃- and NH₂- group; halogen may either increase or reduce the action. The relative positions of the substituents have also a pronounced effect. Esters have, as a rule, a stronger action than free acids, salts a much weaker one. The pronounced action of the monophenols and benzoic acid depends not so much on the phenolic or carboxylic group as on the whole constitution of the substance.

P. H. P.

Recent Research Work on Saponins. L. Kofler. (*Chem. Zeit.*, 1924, 48, 702.)—An outline of previous work on the saponins is given. It is not clear whether saponins are absorbed through the intact intestinal walls, although this question is of fundamental importance for the explanation of therapeutic effects and for the toxicology of the saponins. An attempt to investigate this question

by examination of the urine was made, but saponin drugs were found not to be serviceable diuretics, no saponin or sapogenin being detected in the urine in any single case. An indirect method of determining the approximate amount of saponins in the blood stream was based on the fact that they increase the amount of cholesterol in the serum. Intravenous injection of small quantities of primulic acid (a saponin from the primula) into rabbits, dogs and human beings produced in every instance an increase in the cholesterol in the blood serum, and this afforded a proof of the absorption of that saponin. Sarsaparilla root, used for centuries as a remedy against bubonic plague, was also found to increase the amount of cholesterol in the serum.

P. H. P.

Quantitative Determination of Menthol Glycuronic Acid in Urine.

A. J. Quick. (*J. Biol. Chem.*, 1924, **61**, 667-677.)—The quantitative methods for free and conjugated glycuronic acids are discussed and their defects pointed out. A new quantitative method proposed for menthol glycuronic acid depends on the extraction of the compound from urine with ether, subsequent hydrolysis of the conjugated acid with a dilute mineral acid, and finally a quantitative determination of the liberated glycuronic acid by means of either the Benedict or the Folin-Wu sugar method. Menthol glycuronic acid was chosen, since it can readily be isolated from urine and prepared pure so as to be suitable for a standard. It has the advantage of being easily soluble in ether and of being readily hydrolysed by dilute acids. It seemed almost impossible to find a general method for all conjugated glycuronic acids, but this method, specific for one, with modifications could probably be applied to others. The preparation of menthol glycuronic acid is described. A modification of the apparatus of the type designed by Clausen (*J. Biol. Chem.*, 1922, **52**, 263) was used for extracting the acid from the urine. A typical analysis is given which was made on a specimen of urine from a rabbit which had been fed with menthol. Modifications of the Benedict method which are necessary to make the method applicable to dilute reducing solutions are described.

P. H. P.

Water Analysis.

Volumetric Determination of Total Carbonic Acid in Dilute Solutions of Calcium Hydrogen Carbonate or in hard Waters. **E. M. Crowther and W. S. Martin.**

(*J. Chem. Soc.*, 1924, **125**, 1937-1939.)—Comparative figures are given for the following six methods of determination:—(1), (2) Addition of barium or calcium hydroxide alone; (3) and (4) of each with the addition of the corresponding chloride; (5) of calcium hydroxide and powdered calcium carbonate (to cause rapid "ageing" of the precipitate, as the freshly formed calcium carbonate is more soluble and more readily hydrolysed); and (6) of calcium hydroxide, calcium chloride and calcium carbonate. After standing for varying periods 0.5 *N* hydrochloric acid was used for the titrations (phenolphthalein as indicator), and the results show that the first three methods are unsatisfactory, the fourth

only approximately accurate after 3 hours' standing, the fifth gives good results after 1 hour, whilst the most satisfactory is the sixth, which gives constant and accurate results after 10 minutes.

D. G. H.

Agricultural Analysis.

Determination of the Ammoniacal Nitrogen in Soils. W. McLean and G. W. Robinson. (*J. Agric. Sci.*, 1924, **14**, 548-554.)—An extension of the Hissink method for the determination of ammoniacal nitrogen is given, which has the advantage of being independent of the water pressure in the laboratory. Twenty-five gm. of the soil, which is ground to pass a 3 mm. sieve, are wetted with 100 c.c. of *N* sodium chloride solution, allowed to stand for half-an-hour, and then filtered, and the soil is further washed with sodium chloride solution by decantation until 500 c.c. have been collected. The filtrate is distilled with magnesium oxide into 0.02 *N* sulphuric acid and titrated in the usual way, preferably with the use of the B.D.H. universal indicator carried to a distinct blue, but methyl red may be employed. Comparison is made with the results of aeration methods, and good results have been obtained, which suggests that the amount of ammoniacal nitrogen in a soil at any given time is definite and is not dependent on the conditions of the determination.

H. E. C.

Determination of Nitrate and Ammonia in Nitrogenous Materials. O. M. Shedd. (*J. Agric. Res.*, 1924, **28**, 527-539.)—Strowd's method (*Soil. Sci.*, 1920, **10**, 333) has been modified by allowing the reaction to proceed for 24 hours in a flask fitted with a Bunsen valve before the final heating and distillation. The method gives good results and is applicable to the determination of small quantities of nitrate, such as occur in tobacco, or for the larger quantities present in fertilisers. The cold-water extract of the material is diluted to 300 c.c., fragments of pumice dust and of paraffin wax are added, and 2.5 to 3 grms. of sodium hydroxide, followed by 2 to 3 grms. of powdered Devarda alloy (Cu, 50; Al, 45; Zn, 5). After standing for 24 hours the liquid is heated slowly in the distillation apparatus for 2 hours, and then distilled into standard acid in the usual way.

H. E. C.

Organic Analysis.

Modified Dichromate Method for the Determination of Glycerol. H. B. Bennett. (*J. Chem. Soc.*, 1924, **125**, 1971-1975.)—In connection with work on the hydration of curd fibres of sodium palmitate the following modification of Hehner's dichromate method for the estimation of glycerol has been worked out, so as to render it applicable to the examination of 0.2 to 0.5 gm. in dilute solution with an accuracy of 0.1 per cent. To a clear solution obtained by warming the 10 grms. of the lye with 28 c.c. of water, 50 c.c. of 50 per cent. sulphuric acid are added, and after well cooling to complete the precipitation of the palmitic acid, a quantity of dried potassium dichromate about 0.1 gm. in excess of that required to oxidise the glycerol is added, and the liquid heated on a boiling water bath

under a reflux condenser for 2 hours. The solution is then cooled and diluted to 1 litre, and 50 c.c. are withdrawn, diluted to about 600 c.c., and titrated with 0.1 N sodium thiosulphate. Glycerol is negatively sorbed by sodium palmitate at room temperatures, even in the absence of any salt. H. E. C.

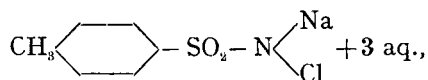
Studies on Pentose Metabolism. I. Colorimetric Method for the Determination of Furfural. (*J. Biol. Chem.*, 1924, **61**, 741-746).—An accurate, simple and rapid colorimetric method is presented for the estimation of furfural, based on the colour produced with aniline and acetic acid. The red colour formed in the reaction readily detects 0.00001 per cent. and 0.00004 per cent. of furfural can be determined quantitatively with an error never greater than 6 per cent. The red colour is not produced if the aldehyde group of the furfural molecule is destroyed, as, for example, by oxidation to pyromucic acid or reduction to furfurol. A table shows the comparison of the colorimetric method for furfural with the gravimetric phloroglucinol (*U.S. Dept. Agric., Bureau of Chem., Bull.* 107 (rev.), 54) method, and Pervier and Gortner's electrometric method (*J. Ind. and Eng. Chem.*, 1923, **15**, 1167, 1255). Full experimental details are given. The reaction of furfural with aniline acetate is specific for furfural, and thus may not only be valuable in studying the decomposition of pentose-containing substances, but, owing to its sensitiveness, is also applicable to the study of the furfural-producing substances occurring in the blood, body fluids and urine. P. H. P.

Inorganic Analysis.

Colour Reactions of Cryogenine with the Heavy Metals. L. Bornet. (*J. Pharm. Chim.*, 1924, **30**, 356-358).—A freshly prepared saturated aqueous solution of cryogenine with a few extra crystals of the solid, is shaken with the solution of the metallic salt; one or two drops of hydrogen peroxide may be added to increase the sensitiveness of the reaction, which should take place in neutral solution. Copper salts give intense red colorations proportional to the amount present. Mercury salts give pink colorations followed by violet precipitates, but the solution must be exactly neutral, and the addition of a few c.c. of hydrogen peroxide should be followed by an acetate; if benzene or ether are then added the precipitate collects as a violet band at the zone of contact of the liquids. With iron salts a red-brown coloration is seen, and in the case of ferric chloride the reaction is as sensitive as with salicylic acid. Strongly (hydrochloric) acid solutions of chromates or of bichromates added to the powdered cryogenine give wine-red colorations.

D. G. H.

Chloramine as a Substitute for Iodine in Analytical Work. A. Noll. (*Chem. Zeit.*, 1924, **139**, 845).—The compound *p*-toluene-sulpho-choramide-sodium



generally known as chloramine, is stable when crystallised from boiling water;

it has the property, when dissolved in cold water, of decomposing potassium iodide solution. In 0.1 *N* solution it keeps well in a dark glass bottle, and can be standardised before use on 0.1 *N* arsenious acid or sodium thiosulphate solutions. Chloramine is much cheaper than iodine, a 0.1 *N* solution costing only about a fortieth of the price of a 0.1 *N* solution of iodine. Tables are given to show the close agreement between results obtained by using chloramine, as compared with iodine, in analysing calcium bisulphite liquors, sodium bisulphite solutions of varying concentrations, and in determining sulphur dioxide in pyrites gases.

R. F. I.

Electrolytic Separation of Lead and Tin. A. Lassieur. (*Compt. rend.*, 1924, 179, 827–829.)—The solution, containing less than 0.15 grm. of tin per 100 c.c., is treated with one-tenth of its bulk of strong hydrochloric acid, one-twentieth of strong hydrofluoric acid, and one grm. of hydroxylamine hydrochloride. It is electrolysed at 60° C. for 10 minutes, then for another 10–15 minutes while the electrolyte is being cooled to room temperature (0.4 volt.). Lead is deposited, whilst tin forms a fluoride complex provided it is present in the stannic condition. If it is not present in that condition, the solution is boiled for a few minutes with 5 c.c. of hydrogen peroxide (12 volumes). After removal of the lead, the solution is treated with 10 grms. of boric acid and 10 grms. of ammonium oxalate, diluted to 300 c.c., warmed, and electrolysed for tin at 0.6 to 0.65 volt with a copper-coated cathode.

W. R. S.

Separation of Rare Earths from Uranium. Alkali Uranyl-salicylates. G. Canneri and L. Fernandes. (*Gazz. Chim. Ital.*, 1924, 54, 770–773.)—The fact that salicylic acid forms the complex uranyl-salicylic acid with uranium, whereas it does not unite with salts of the rare earths to give soluble complexes, furnishes a means of separating these earths from uranium. The hot, faintly acid solution is treated with excess of salicylic acid, and ammonia solution is then added, drop by drop, until the liquid assumes a blue colour, but still remains acid. A slight excess of concentrated ammonium oxalate solution is next added, the liquid being afterwards boiled, left for a few minutes to deposit the precipitate, then filtered, and the precipitate thoroughly washed with boiling ammonium oxalate solution; the rare earth oxalate thus separated is calcined and weighed. The solution containing the uranium is acidified with concentrated hydrochloric acid, allowed to cool, and filtered to eliminate most of the salicylic acid, which is washed with cold water. The filtrate is evaporated to dryness with nitric acid, and the residue calcined on a sand-bath to expel organic matter, and dissolved in hydrochloric acid. Treatment of the solution with ammonia solution, drop by drop, results in the precipitation of ammonium pyrouanate, which is dealt with in the usual manner.

T. H. P.

Sensitive Colour Reaction for Magnesium. F. L. Hahn, H. Wolf and S. Jäger. (*Ber.*, 1924, 57, 1394–1396.)—A sensitive test for magnesium is based upon the fact that it produces a bright blue coloration or blue precipitate in an

alcoholic solution of 1.2.5.8-tetrahydroxy-anthraquinone. The reaction is capable of detecting 0.001 mgrm. of magnesium, but is not so sensitive when ammonium salts or phosphates are present. In applying it to the detection of minute quantities of magnesium in aluminium an excess of sodium hydroxide or of tartrate and alkali is used to keep the aluminium in solution.

Separation of Molybdenum from Tungsten. I. Koppel. (*Chem. Zeit.*, 1924, 48, 801-802.)—The solution of alkaline molybdate and tungstate is almost neutralised with formic acid and treated with a few c.c. of ammonium sulphide followed by 5 c.c. of formic acid, the total bulk being about 100 c.c. The solution is heated for some time on the water-bath to cause coagulation of the precipitated molybdenum sulphide. This is collected on a Gooch crucible, washed with water containing a little formic acid, and weighed as sulphide, or carefully roasted to trioxide. The filtrate is evaporated with sulphuric acid for the precipitation of tungstic acid.
W. R. S.

Precipitation of Tantalum and Niobium by "Cupferron," and their Separation from Iron. H. Pied. (*Comptes rend.*, 1924, 179, 897-899.)—Tantalum and niobium are precipitated quantitatively from their oxalo-tartaric solution containing sulphuric acid by "cupferron," the method of separating iron indicated by Weiss and Landecker (*Zeitsch. anorg. Chem.*, 1909, 64, 69) being thus applicable. The freshly-precipitated hydroxides are dissolved in oxalic acid solution, and the liquid then treated with a little tartaric acid, warmed gently, treated with ammonium hydrosulphide, and left overnight. After removal by filtration of the ferric sulphide, which is quantitatively precipitated, the liquid is treated with excess of sulphuric acid and freed from hydrogen sulphide by boiling, and from sulphur by filtration. The cold solution is mixed with "cupferron" and shaken vigorously, the appearance of a whitish film on the surface indicating the end of the reaction. The precipitate is immediately filtered off with the aid of slight suction, washed with very dilute sulphuric acid, partly dried, and carefully calcined. The tantalum and niobium, and any titanium present, are precipitated completely in this way.

The oxalate solution may be obtained by direct treatment of the fusion mass given when the calcined oxides and many minerals are attacked by pyrosulphate. The rare earths and thorium do not hinder the solution, but form a crystalline precipitate, the extent of which indicates the proportion of these constituents. Further, titanium may be determined in the solution, since oxalic acid has no action in the cold on the coloration given with hydrogen peroxide. T. H. P.

Examination of Sodium Dithionate. F. M. Litterscheid and H. Löwenheim. (*Chem. Zeit.*, 1924, 48, 881-883.)—One gm. of the salt, on being heated in a closed tube, should only give off water and sulphur dioxide without production of a sublimate (sulphur); the residue should remain white. The quantitative analysis may be carried out very simply by heating the finely-powdered salt in a porcelain crucible at 60° C. for 1½ hours, then at 100° C. for

$\frac{1}{2}$ hour. The loss in weight represents water of crystallisation; the salt has the formula $\text{Na}_2\text{S}_2\text{O}_6 \cdot 2\text{H}_2\text{O}$, but a hexahydrate is also known. The dry substance is then gently ignited: $\text{Na}_2\text{S}_2\text{O}_6 = \text{Na}_2\text{SO}_4 + \text{SO}_2$. Decomposition takes place at 250°C . After half-an-hour's heating the residue is left to cool in a desiccator and weighed as Na_2SO_4 , giving SO_2 by difference. The residue may serve for the estimation of SO_3 , or for tests for carbonate, chloride, etc. A process is also described in the paper, in which the dehydrated salt is heated in a glass tube and the evolved sulphur dioxide absorbed in standard iodine solution; it offers no advantage over the simpler method described above.

W. R. S.

Physical Methods, Apparatus, etc.

Measurement of P_{H} Value of Dairy Products by Billmann's Quinhydrone Electrode. V. Lester. (*J. Agric. Sci.*, 1924, 14, 635-641.)—Billmann's quinhydrone electrode (*cf. J. Chem. Soc.*, 1923, 123, 2203) affords a simple and rapid method for the determination of P_{H} in milk, cream and whey, and is of special value because methods depending on dialysis are unreliable and the platinum-hydrogen electrode method is slow and open to many sources of error. The method is applied by adding 50 mgrms. of quinhydrone to 10-20 c.c. of the sample and placing in position three smooth gold electrodes, the connecting link with the standard quinhydrone electrode being the usual potassium chloride bridge. The standard consists of three platinum or gold electrodes in a saturated quinhydrone solution containing 0.01 *N* hydrochloric acid and 0.09 mol. potassium chloride, and a siphon tube from this dips into an ordinary potassium chloride electrode vessel. The potential becomes constant within 5 minutes, and $P_{\text{H}} = 2.03 + \frac{\pi}{0.0577}$, where π is the potential difference which has been measured.

H. E. C.

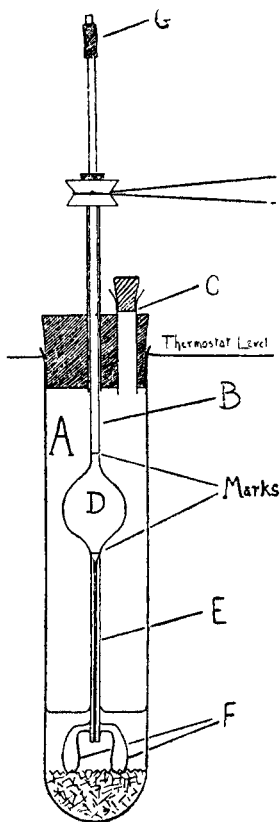
Microscopical Examination of Cacao Products. V. A. Pease. (*J. Ass. Off. Agric. Chem.*, 1924, 8, 176-178.) In the microscopical examination of cacao products, and especially if a slide is to be kept for some time, the standard 1:1 chloral hydrate clearing solution used as mounting medium may be replaced with advantage by Hertwig's solution, composed of 25 c.c. of dilute hydrochloric acid (1:8), 10 c.c. of glycerin, and 45 grms. of chloral hydrate. Care must be taken to use just enough of the solution to fill the space under the cover glass, as the glycerin becomes more fluid when warmed and, if excess is employed, may carry fragments of tissue beyond the edge of the cover-glass. The advantages are that drying of the mount and formation of crystals under the edge of the cover-glass are avoided, and that the procedure is quickened, as the preparation may be made, warmed for about 20 minutes, and counted after standing for only 30 or 45 minutes.

T. H. P.

Apparatus for the Viscometric Determination of Transition Points.

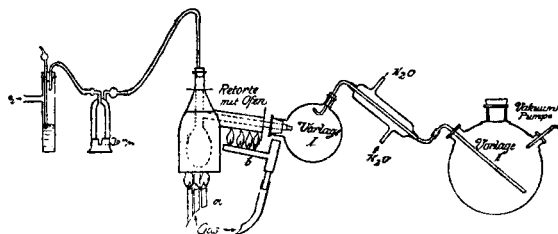
N. H. Hartshorne. (*J. Chem. Soc.*, 1924, 125, 2096-2099.)—The apparatus illustrated enables successive determinations to be made in the same vessel at different temperatures. The tube A is 25×6 cm. and carries the combined viscosimeter and stirrer B and a closed tube C. The bulb D has capacity about 20 c.c. and the capillary E is 10 cm. in length, and of diameter about 0.75 mm. F is a glass paddle sealed on to the capillary. For the determination 80 c.c. of the solution together with excess of the salt in A are immersed in a thermostat and rotated until equilibrium is reached, then the salt is allowed to settle, the dust cap G is removed and the solution is sucked up above the higher mark and the time of flow from the upper to the lower mark is noted as usual. By means of additions or withdrawals through C the quantities of solid and solution can be adjusted.

H. E. C.

**Sublimation Apparatus.** A. Gutbier and T.

Payer. (*Chem. Zeit.*, 1924, 133, 807.)—The apparatus described has the advantage of being suitable for the sublimation of compounds of high volatility, or of those containing water or acid from which it is difficult to separate them, or of those sensitive to decomposition. It consists mainly of a hard glass retort in the form of a distilling flask enclosed in a small fireclay hood, the bottom of the flask being protected by asbestos paper. The side-tube of the retort is closely wrapped in thin platinum foil, passes through

a cylinder of fireclay and delivers into two receivers of glass, I and II, which are connected by a small condenser. Receiver II has tubular openings, one to



facilitate cleaning and one connected with a water-pump which draws air or other gas through the whole apparatus. Where necessary, the joints are made with a cement of asbestos and sodium silicate. The fireclay cylinder around the side-tube only extends 50 mm. (as shown in diagram), its end being provided with an

asbestos disc to protect receiver I from undue heat. The gas supplied to the apparatus during a sublimation passes through a glass vessel which acts as a safety-valve at the desired pressure, thus avoiding liability to "blow-out" as a result of possible blocking of the tubes. The delivery tube into the retort is bent at its end to conform to the side tube, thus tending to remove water vapour and any decomposition-products and to carry them over into receiver II. The internal pressure should be maintained between 30 and 40 mm. of water, the surface of the water in the safety valve being covered with vaseline-oil to prevent water-vapour being carried over. The sublimation of selenium oxide is taken as an example and described in detail. By this method the retort is left quite clean at the end of the process.

R. F. I.

Reviews.

CHEMISTRY IN THE TWENTIETH CENTURY. AN ACCOUNT OF THE ACHIEVEMENT AND THE PRESENT STATE OF KNOWLEDGE IN CHEMICAL SCIENCE. Prepared under the guidance of a Committee with Dr. E. F. ARMSTRONG, F.R.S., as Chairman and Editor. Pp. viii.+281. London: Ernest Benn, Ltd. 1924. Price 15s. net.

As to the value which may accrue from the Chemical Exhibit at the British Empire Exhibition at Wembley there may be differences of opinion. As to the desirability of publishing such a work as the one now in our hands there can be no possible difference of opinion. It may not have been one individual who originated the idea; but whether individual or committee we are thankful that the idea has materialised in such an acceptable form. The essayists are all distinguished men of science who have very judiciously put into the forefront the achievements of British chemists without laying any invidious stress upon nationality, and who have not restricted too narrowly the time limits of their theses. The volume, we are told, has been compiled for those "who have received, or are receiving, a training in science." It was, therefore, important that the writers should have a due regard to the historical setting of their subjects. Most of them have succeeded in furnishing a properly balanced background for their pictures, and will thus capture and hold the attention of the older chemists as well as interest and instruct the newer generation.

As this volume must make an appeal to all the readers of *THE ANALYST*, we think we ought to make them acquainted with the diversity of its contents and the authors of the monographs. These are as follows:—"The Rôle of Chemistry in Physical Science" (Prof. J. I. O. Masson), "The Structure of the Atom" (Dr. E. N. da C. Andrade), "Crystallography" (Sir Hy. A. Miers and Mr. T. V. Barker), "X-ray Analysis of Crystals" (Sir W. H. Bragg), "The Rare Gases of the Atmosphere" (Dr. M. W. Travers), "Chemistry of the Carbon

Compounds" (Prof. J. F. Thorpe), "Milestones in Organic Chemistry" (Prof. H. E. Armstrong), "Chemistry of Colloids" (Dr. Wm. Clayton), "Catalysis" (Dr. T. P. Hilditch), "Fats and Oils" (Mr. John Allan), "Sugars and Carbohydrates" (Dr. J. C. Irvine), "Cellulose" (Mr. C. F. Cross), "Colour in Nature" (Mr. R. Furness), "Coal Tar Colours" (Mr. E. A. Bearder), "Syntheses in the Terpene Series" (Professor G. G. Henderson and Mr. Alex. Robertson), "The Alkaloids" (Prof. F. L. Pyman and Dr. T. A. Henry), "Nitrogenous Constituents of the Living Cell" (Dr. R. H. A. Plimmer), "Biochemistry and Fermentation" (Prof. Arthur Harden), "Chemistry in Agriculture" (Sir John Russell and Mr. H. J. Page), "Alloys" (Prof. C. H. Desch), "Pottery and Refractories" (Mr. Joseph Burton), "Flame, Fuel and Explosion" (Dr. H. F. Coward), "Explosives" (Sir R. Robertson), "Chemistry of Photography" (Sir W. J. Pope and Mr. Walter Clark).

The Introduction has been written by Dr. E. Frankland Armstrong, who directs attention to the clear picture the first few articles give of the development of modern theories of chemistry, as well as to the specialised articles which illustrate, with what he calls "kaleidoscopic rapidity," the progress which has been made in certain industries by the application of the new theories.

The articles by Professor Masson and Dr. Andrade will be read with great interest, because they reveal the great change that has taken place in the relations of the chemist and the physicist. As Dr. Armstrong points out, the atom is no longer hypothetical—it is a real object of definite size and shape, and Professor Masson and Dr. Andrade demonstrate this fact in such a succinct and lucid manner that no chemist can fail to understand its significance. The diagrams representing the orbits of the electrons are very useful.

The history of chemistry acquires from the hand of Dr. Morris Travers a chapter which is of the greatest value. His work, in conjunction with Sir Wm. Ramsay, upon the rare gases of the atmosphere, has drawn together the experiments of Henry Cavendish in the eighteenth century and the discovery of radium with its emanation (niton) at the end of the nineteenth.

Another monograph which will be read in conjunction with those just mentioned is the one by Dr. William Clayton on "The Chemistry of the Colloids." This subject is peculiarly interesting, because the initiation of this branch of chemistry was in Thomas Graham's papers on "Liquid Diffusion," and may be legitimately regarded as of British origin. Dr. Clayton has much to say about the practical side of investigations in colloidal chemistry in respect to their application to soaps, dyes, paints, "smokes," dust explosions, etc., etc.

The two articles on crystallography are specially worthy of notice, for not only are they written by the most eminent authorities in that branch of science, but also, as Sir Henry Miers says, the contributions to it by British investigators have been of the highest importance; moreover, they are full of information and well illustrated.

Space does not permit of particular mention of those essays of a more specialised character. They are necessarily more restricted in their appeal than those named

above, which cannot fail to kindle anew in every chemist the enthusiasm for his science which may have faded somewhat in the course of laboratory practice. Perhaps Dr. Armstrong really meant all that the word "kaleidoscopic" should signify. In any case, the beauty of the ordered progress of the discovery of the constitution of matter shines forth in such a way as to impress the most callous reader. That this country has made so great a contribution to it is a cause for thankfulness. The volume is well printed, well bound and really most moderate in price.

WILLIAM KIRKBY.

MIKROCHEMISCHES PRAKTIKUM. By FRIEDRICH EMICH. Munich: J. F. Bergmann. 1924.

The award of a Nobel prize in 1923 to Prof. Pregl in recognition of his improvements in micro-chemical technique may help to remove much of the prejudice against working with small quantities of material where circumstances do not compel their use. This prejudice is very natural considering the instinctive preference of chemists for methods in which the mechanical difficulties are so much less, and the limits of error more accurately known. However that may be, it is certain that the ingenuity and simplicity of the devices described in the pages of this little book are such that it cannot fail to appeal to a much wider circle, and receive far greater consideration than has been accorded to the great majority of earlier works. It may not be uninteresting to recall in passing that one of the first comprehensive treatises dealing with micro-chemical methods was that of Theodore G. Wormley, which was published as far back as 1867.

Prof. Emich, who is well known as one of the leaders in this branch of work, has produced a book which will certainly prove of considerable value in suggesting ways of avoiding the disappointment of an incomplete investigation by reason of scarcity of material.

An admirable general section describes in considerable detail the manipulative side of the work, giving numerous illustrations of the simple apparatus used, and dealing with the general considerations necessary in the reduction of ordinary laboratory methods to a micro-chemical scale. The application of the methods is dealt with in the second part, and a great many examples are given, chiefly intended as exercises in inorganic and organic analysis and organic preparations.

The author lays the greatest stress on the need for patient practice, and urges the repetition of every typical reaction until positive results are as certain as on the ordinary scale. The need for experience and confidence will be very evident when it is seen that in qualitative tests the amount of material in question may be of the order of one-thousandth milligramme, and that the quantitative methods described are usually sensitive enough to be reliable when working with 2 to 10 milligrammes of material.

The adaptation of the ordinary physical determinations, such as melting and boiling points, refractive indices, molecular weights, etc., where micro-chemical methods are so often called for, is adequately described.

Prof. Emich has written a most helpful and suggestive little book, and one which may be warmly commended to the attention of chemists, the great majority of whom—at one time or another—find themselves compelled to work with very small quantities of material, and who might, perhaps, with advantage, make more frequent incursions into this field of chemical procedure. The book is clearly written, the illustrations are numerous and good, and, unlike many of the works which are being published in Germany at the present time, it is printed on reasonably good paper.

A. CHASTON CHAPMAN.

A SYSTEMATIC HANDBOOK OF VOLUMETRIC ANALYSIS. FRANCIS SUTTON.
Eleventh edition. Edited by W. L. SUTTON, F.I.C., and A. E. JOHNSON,
B.Sc., F.I.C., A.R.C.Sc.I. Pp. xii. + 629. London: J. and A. Churchill.
1924. Price 35s. net.

After a long interval of thirteen years, a new edition of this, the oldest treatise on volumetric analysis in the English language, has made its welcome appearance. Few, if any, British chemists engaged in analytical practice have not, at some time or other, had an opportunity to thank "Sutton" for useful advice on a problem outside their daily routine, or for obtaining reliable information on a volumetric process new to them. Considerable changes have been made in the present work; the section on Gas Analysis has been re-written by Mr. H. Hollings and Dr. J. S. G. Thomas: it occupies 103 pages, and is indexed separately. The editors describe it as "a comprehensive and up-to-date little treatise on the subject such as has not previously existed in English." Part of the section is devoted to information not commonly found in analytical handbooks, *e.g.*, the preparation, graduation, and calibration of gasometric apparatus. The chapter on the calibration of graduated instruments comprises a tabulation of the limits of error allowed by the National Physical Laboratory.

The Section on Indicators is "entirely re-written, with many additions"; it is, therefore, disappointing to find that the question of hydrogen ion concentration, which has been the subject of so much investigation in recent years, has been ignored. No indication is given of the P_H range of indicators; no "universal" or mixed indicators are described. Again, methods of, and apparatus for, potentiometric and conductometric titration find no place in the book. The reviewer believes he is not guilty of harsh criticism when he says that the omission of the above subjects constitutes a failure to keep the book up to date.

In the sections dealing with Applied Methods of Analysis, the text-matter might be enriched here and there with the description of, or, at least, a reference to, some more recently published processes. Thus, in present-day works practice, the favourite method for cerium is Metzger's bismuthate process. An interesting contribution to the volumetric estimation of aluminium has been made by Kraus (ANALYST, 1922, 47, 92). Cadmium—as well as some other divalent metals—can be estimated alkalimetrically as phosphate (ANALYST, 1916, 41, 124). The chapter on arsenic might with advantage include a description of its estimation

in ores by a process of conversion into arsenate and titration of the latter with iodide and thiosulphate (Low, 7th edition, 1914, 45-48). Sodium hydrosulphite (hyposulphite), $\text{Na}_2\text{S}_2\text{O}_4$, is used in large quantities as a bleaching and reducing agent ("hydros"), and its analysis (ANALYST, 1922, 47, 369) should be included in a work of this character. The best method for the removal of tungstic acid in the volumetric assay of tin ores is that of Powell (*J. Soc. Chem. Ind.*, 1918, 285). For the reduction of titanium solutions the use of Jones' reductor is a decided advantage (ANALYST, 1924, 49, 153). Under vanadium, a method for its estimation in ores—including reduction by sulphur dioxide and oxidation by permanganate—might have been added. In most, if not all, Continental zinc smelting works, glazed lead carbonate paper is used as an external indicator in the Schaffner method.

A multiplicity of procedures for the titration of one and the same element is sometimes evident. The use of some of these methods should be discouraged, as they are of doubtful value, and their place has been taken by more accurate ones. For example, the chapter on Copper comprises ten methods, some with modifications. In his moderately wide experience of the metallurgy of copper, the reviewer has seen only the four following methods used, in addition to electrolysis: the iodide, the cyanide, a modification of the thiocyanate, and the colorimetric method. If the other methods are rarely used, they must have been found less convenient or accurate, hence the wisdom of keeping them in the book may be questioned; this applies particularly to Pelouze's process. The text-matter on mercury, arsenic, and antimony presents an almost embarrassing choice of alternatives.

The work appears very free from misprints: the term "Dihydroxytartaric" (for "Dihydroxytartrate") appears in the title on p. 65, and printer's slips were observed in pages 356, 419, and 512. A manifest error in nomenclature has managed to evade revision, one of the solutions used for certain acidimetric titrations being called "Ammonium=Copper Solution" (p. 56). Whatever nomenclature is applied, the compound $\text{CuSO}_4 \cdot 4\text{NH}_3 \cdot \text{H}_2\text{O}$ contained in that solution is certainly not an ammonium salt. Those who still hesitate to employ Werner's nomenclature ("tetramminecupric sulphate") have the alternative of referring to this reagent as "ammoniacal copper solution." The time-honoured appellation "Alkaline Earths" has been changed in this edition to "Alkali Earths" (p. 73); in the reviewer's opinion this is hardly an improvement, as "alkali earth" should, like "alkali waste," refer to some kind of material containing alkali in admixture.

The Atomic Weight Table used in this book is that of 1923, in which the value for antimony is still given as 120.2. The figure 121.8 (ANALYST, 1924, 49, 49, 152) will probably be adopted in the next International Table. Meanwhile it is advisable to standardise solutions used in antimony titrations against the pure metal or, if Sb_2O_3 is required, against the pure oxide.

W. R. SCHOELLER.

QUALITATIVE ORGANIC ANALYSIS. AN ELEMENTARY COURSE IN THE IDENTIFICATION OF ORGANIC COMPOUNDS. By OLIVER KAMM. Pp. 260+vii. New York: John Wiley & Sons, Inc.; London: Chapman & Hall. Price 12s. 6d. net.

In this book, which is based on S. P. Mulliken's "Identification of Pure Organic Compounds," there is developed a system of qualitative organic analysis which is somewhat analogous to the group system which is almost universally used in Inorganic Chemistry. The solubility of compounds in several common solvents, together with the fact of homology form the basis of the scheme.

Part A of the book gives a brief outline of the steps involved in the identification of organic compounds, and later the question of solubility is discussed, and certain approximate rules for the prediction of this property are deduced. This is followed by a very useful section on classification reactions, most of the common types being treated fully.

Part B, entitled "Laboratory Directions," commences with an account of the usual procedure for the examination of a single substance, and a number of exercises on class reactions are worked out. The importance of the tests for characteristic elements and the deductions therefrom are singularly overshadowed by the less helpful solubility discussion, and many an unwary student may be trapped in consequence. In passing, it may be pointed out that the standard nitroprusside test for sulphur is not mentioned in the text. The chapter on derivatives is of special merit, but it is curious that no reference is made to the important art of crystallisation. Lack of skill in this operation surely prevents physical constants from being determined, and hence final identification becomes impossible. The section closes with a chapter on the treatment of mixtures.

Part C concludes the book and contains the physical constants of 2000 or more compounds classified in ascending order of melting point (or boiling point) according to the solubility groups which are the basis of the scheme of analysis.

Many books, most of very limited usefulness, have been written with the object of helping students to identify organic compounds. All that the best books attempt to give is only a series of general hints to enable class assignment to take place, leaving the greater part of the process to the student's knowledge of theoretical chemistry—and to his wits. This is the utmost any book on the subject should do, for the object of organic "spotting" is to get students to increase their knowledge of organic chemistry by continual reference to the literature of the subject. The present book is perhaps too comprehensive, and a slavish adherence to its scheme will take from qualitative organic analysis much of its value, and at the same time render the process rather tedious. Nevertheless, those working for B.Sc. Hons. and A.I.C. (in organic chemistry) will find the book useful, provided that it is regarded merely as a help and not as an infallible guide.

HAROLD TOMS.

THE CHEMISTRY AND PHYSICS OF CLAYS AND OTHER CERAMIC MATERIALS. By ALFRED B. SEARLE. Pp. xiii. + 695. London: Ernest Benn, Limited. 1924. Price 55s.

This book comprises fifteen chapters, the first seven of which deal with the structure, strength and other physical and mechanical properties of ceramic materials and with the changes effected in them by the addition and removal of water. Two chapters are devoted to the chemistry, one to the mineralogy, and one to the physico-chemical reactions of the materials. The remaining chapters embrace thermal, electrical and optical properties.

As pointed out in the preface, although craftsmanship in the ceramic industries had reached a high degree of perfection in very early times, the application of scientific methods to them is comparatively recent, and has been most marked during the last decade. Although this general statement is true, it must not be forgotten that notable attempts to apply scientific methods to the problems of ceramics have been made in the past, as the work of such leaders as Wedgwood, Brongniart and Seger abundantly shows. Further, it is also stated that the volume is not intended to be exhaustive, but to provide a description of the properties of various ceramic materials and the application to them of the more important principles of chemistry and physics, "as will be equally useful to students, manufacturers and users," and the question naturally arises, whether it is possible or even advisable, to attempt to meet the requirements of three such different classes of readers. Be that as it may, much of the information in the book ought to be quite unnecessary, say, for the student of chemistry, and at the same time would be insufficient for the manufacturer to acquire that knowledge of the fundamental principles of the science that he ought to possess in order to follow the subject intelligently.

The book has certain drawbacks. In places the views held by different authorities on some point—views often diametrically opposed—are given as bare statements without any discussion of the arguments in favour of either. As an instance may be mentioned the statement of the views of Rohland, Schurecht, and Bleininger and Fulton on the effect of alkalis and acids on the quantity of water required to develop plasticity (pp. 269–270). Another drawback which seriously hampers the use of the book for reference is that a subject is briefly mentioned, and then for fuller information on it the reader is referred to later pages or chapters. As an illustration of this, colloidal substances are mentioned on p. 7 and p. 9, and on both the same words occur, *viz.* that these substances "are described more fully on page 10 and in Chapter VI." To borrow a simile from the nomenclature of colloids, the book would appear to be an effective dispersion medium. This lack of arrangement leads to much repetition and between pages 400 and 405, two other books by the same author are referred to ten times, apart from other places.

Readers of THE ANALYST will probably be attracted most by the chapters dealing with the chemical constitution and physico-chemical reactions of ceramic

materials. In them chemists will find a good deal with which they are familiar, but unless they are already conversant with the specialised branch of chemistry that deals with silica and its compounds, the book will not be easy reading or prove to be a convenient introduction to the subject. At the same time the chapter contains several interesting phase-diagrams, and the section on corrosion should be useful to those who are unfamiliar with the conditions that influence resistance to corrosive action.

Unfortunately the chapter on the optical properties of ceramic materials is short and hardly lays sufficient emphasis on this valuable but very difficult branch of the work.

Within the compass of 700 pages the author has courageously attempted the survey of a wide field and has brought together a large amount of useful information. There are more references to original papers than in some other books by the same author, but the inclusion of still more would have been a great assistance to the student of the subject. Obviously omissions must occur, but in discussing the evolution of the formulæ that have been proposed to connect chemical composition with refractoriness, the omission of any mention of Richter's Laws seems to spoil the continuity of the argument, and in the chapter on drying there is no reference to the work of Aron or of Jackson and Rich. Unfortunately, in places the wording is rather ambiguous; for instance, on p. 43, corundum and carborundum might be easily confused, and on p. 17 no clear distinction is drawn between the use of sillimanite as a raw material and sillimanite crystals produced by the action of heat in articles made from argillaceous materials.

The book has a copious index, is clearly printed and is in an attractive form, except that, to the writer, the pages seem too wide for easy reading. There are very few misprints; on p. 436, "ammonium cyanite" should of course be "ammonium cyanate," and unfortunately, the author of the well-known book on refractories is spoken of as "Harvard" instead of "Havard."

In conclusion, the perusal of the book leaves the impression that the theme has not been developed as logically as it might have been, and, though an amount of useful information has been presented in a convenient form, it is advisable to preserve a critical attitude with regard to many of the points.

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TECHNICAL WRITING. By T. A. RICKARD. 2nd Edition. Pp. ix. + 337. New York: John Wiley & Sons, Inc.; London: Chapman & Hall. Price 10s. net.

It is not enough to have something to say; even a technical report may be marred by the use of the wrong words or by their bad arrangement. This is essentially the text upon which is based a critical discussion on the writing of technical English. The general scope of the book, which has deservedly reached its second edition, may be gathered from the headings of some of its seventeen

chapters, which include: Naturalness, Clearness, Precision, Superlatives and the Superfluous, Slovenliness, Jargon, The Wrong Word, Construction, Composition, and Style.

The chapter on Punctuation deserves especial mention, for although the author dissents in some respects from the more commonly accepted conventions, he makes out a convincing case for his suggested usage.

In his section on the use of the subjunctive he does not take up the extreme position of the authors of "The King's English," who say: "'Were' is often right and almost always necessary; other subjunctives are never necessary, often dangerous, and in most writers unpleasantly formal." The author of this book, however, remarks "the subjunctive is a mark of scholarship; it is ignored by those who are not particular in such matters, and unfortunately it is a source of perplexity to those unfamiliar with the use of it."

Probably the best rule of guidance over this and similar debatable ground is to use the older forms until general custom makes it pedantic to retain them. Few writers of modern English would now use the expression "it *were* useless to protest"; it has an archaic sound. Some forms of the present subjunctive are nearing the same stage, if they have not already reached it.

The same practical rule of wide acceptance may be applied to the modern extension of the use of an active participle with a passive verb. Although the phrase "speaking generally" and some others have secured the sanction of custom, is there as yet any sufficient justification for the sentence: "The liquid *is titrated, using* phenolphthalein," which is so frequently seen in both English and American chemical journals? What is the objection to "*being used*"?

Some changes in the use of words are inevitable, and in time one must conform to them. It may, perhaps, even be necessary to acquiesce in the local mutilation of words, though one cannot help slyly wondering what reply the author would have made to Archdeacon Hare, who in the fifties of the last century expressed a hope that "such abominations as *honor* and *favor* would henceforth be confined to the cards of the great vulgar."

The author strongly condemns the use of long and abstruse words where the same meaning is conveyed by simpler ones. We are only slowly abandoning this legacy from those who upheld the tradition of Dr. Johnson, and turned *men* into *individuals*, *places* into *localities*, and *homes* into *residences*, and it is not easy to avoid taking our share of it; even the author of this book has not escaped, for more than once he speaks of "aberrances." Possibly that ugly word has a shade of meaning not conveyed by "lapses," but it would have been worth some sacrifice of precision to avoid its use.

In the chapter on Jargon the writer of scientific papers is warned against applying trite phrases such as "the swing of the pendulum" or "a bolt from the blue." This, again, is not always easy, for memory unconsciously reproduces set groups of words. In his remark that "these outworn figures of speech" are

"taboo to the intelligent writer," the author himself approaches very close to his forbidden ground. A well-known writer, long since dead, once gave me the same advice in still more terse and forcible words: "Shun the cliché," he said, "as you would the devil."

Throughout the book numerous quotations are given to show how things should not be written and the reason why. Most of these are drawn from metallurgical and geological sources; had the author referred also to chemical publications he would have found a richer store. Our old friends, "*data is*" and "*phenomena is*" are ridiculed. They are of American origin, but we can match them with "policeman" used as a verb ("policemanning it"); and such gems as "bring to the boil" and "let stand" are shared impartially by chemists in either country.

Our language is the heritage of all the English-speaking peoples. Sometimes Americans take exception to criticisms of innovations that they have brought into it. But, as the author, himself an American, remarks (p. 90): "The official language of the United States, a language that originated in Great Britain, is written in the same way by those who write it well on either side of the Atlantic. I can assure you that Mr. Eliot and Mr. Butler, for example, speak and write the same language as Mr. Asquith and Mr. Balfour*; and if any of us speak or write differently from them, we may be using British or we may be using American, but we are not using English."

One cannot be too grateful for the effective plea on behalf of good English made in this small book. The author is impartial in his comments, which are full of humour and sound sense, and the rules he gives will well repay close study. It is a book to keep ready for constant reference.

EDITOR.

* Uncorrected from 1st Edition, published in 1916. —EDITOR.

Publications Received.

- A SYSTEM OF PHYSICAL CHEMISTRY. Vol. III. Quantum Theory. By W. C. McC. Lewis, D.Sc. Pp. 407. London: Longmans. Price 15s. net.
- SYNTHETIC ORGANIC COMPOUNDS. By S. P. Schotz, D.Sc. Pp. 412. London: Ernest Benn Ltd. Price 45s.
- PHYSICAL CHEMISTRY FOR STUDENTS OF MEDICINE. Pp. 227. London: Longmans. Price 8s. 6d. net.
- CURTIS TESTED SANDS FOR ALL INDUSTRIAL PURPOSES. (Trade Catalogue). Westmoor Laboratory, Chatteris.

Gives in alphabetical order the characteristics (physical and chemical) of 49 different kinds of sands used in various industries.