THE ANALYST

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

AN Ordinary Meeting of the Society was held on Wednesday, April 2, at the Chemical Society's Rooms, Burlington House. The President, Mr. G. Rudd Thompson, F.I.C., was in the chair.

Certificates were read for the first time in favour of:—Messrs. George William Clough, D.Sc. (Birm.), James Porter Shenton, F.I.C., and William Stanley Wood.

Certificates were read for the second time in favour of:—Messrs. John Joseph Bryant, Edgar Wilfred Deag, Harold Wilton Hewis, B.Sc. (Lond.), A.I.C., Ernest Wilfrid Jackson, F.I.C., Thomas McGrath, and Alfred Scholes, F.I.C.

The following were elected members of the Society:—Messrs. Philip Walter Alloway, Lorentz Oliver Brekke, B.Sc. (Leeds), A.I.C., Alex Munro Cameron, B.Sc., F.I.C., Thomas William Drinkwater, L.R.C.P., L.R.C.S. (Edin.), F.I.C., John Ralph Furlong, Ph.D. (Wurzburg), A.I.C., Ernest Griffiths-Jones, M.Sc. (Manc.), A.I.C., Basil Gordon McLellan, F.I.C., and William Thomas Rigby, F.I.C.

A Report on The World's Dairy Congress held at Washington, D.C., U.S.A., was made by John Golding, D.S.O., F.I.C., who represented this Society, and the following papers were read:—"The Routine Examination of Dairy Products with special reference to the Mojonnier Tester," by L. H. Lampitt, D.Sc., F.I.C., E. B. Hughes, B.Sc., F.I.C., and M. Bogod, B.Sc., A.I.C.; "Experiments on the Absorption of Copper following the Consumption of Vegetables containing Copper Sulphate," by J. C. Drummond, D.Sc., F.I.C., Miss M. G. Palmer, B.Sc., and Miss D. E. Wright, B.Sc.; and "Attempt to extend Mitchell's Colorimetric Method to the Catechol Tannins," by Miss P. H. Price, B.Sc. (work carried out under the Investigation Scheme).

The Composition of Beef and Malt Wine.

By G. D. ELSDON, B.Sc., F.I.C.

(*Read at the Meeting, March* 5, 1924.)

Some little time ago a sample of so-called beef and malt wine was submitted to the writer for analysis which was labelled in the following way:—"Liebig's Beef and Malt Wine. Made with Liebig's Extract of Beef and Malt Extract. Health, Strength, Vigour. A wineglassful may be taken two or three times a day. Non-exciseable." A small label on the neck bore the following words:—"This beverage is prepared in accordance with the requirements of the Food and Drugs Act and contains a small quantity of Salicylic Acid as a preservative."

As a result of the analysis of this sample, three formal samples were taken from the manufacturer in due course. The results of the analyses were so unsatisfactory that the samples were classified as adulterated and the one made the subject of proceedings was certified in the following way:

	Per Cent.
"Water	75·63
Total sugars	21·80
Alcohol	1·50
Other extractive matter	1·00
Salicylic acid	0·07
	100·00

This 'other extractive matter' contains 0·01 per cent. of nitrogen (calculated on the original liquid), which indicates the possible presence of not more than 0·2 per cent. of a mixture of equal parts of meat and malt extracts. This opinion is based on the fact that a mixture of equal parts of meat and malt extracts contains about 5 per cent. of nitrogen.

Observations.

No change had taken place in the article that would interfere with the analysis.

This is not a beef and malt wine. Its composition is similar to that of a flavoured cordial which usually contains up to 0·01 per cent. of nitrogen derived from sources other than beef or malt extracts.

The amount of salicylic acid present is contrary to the declaratory label on the bottle; it is six times that suggested as the maximum by the Departmental Committee on the preservation of food, 1901, in respect of beverages."

The vendors were prosecuted, and were fined the maximum fine in each of three cases, and 20 guineas costs were given against them. The analyses were accepted, the amounts of meat and malt extracts actually added being about half the maximum amount mentioned in the certificate. The contention of the defence was that it was impossible to market a non-alcoholic wine containing larger

proportions of meat and malt extract than those present without the addition of a much higher percentage of preservative. Although the magistrate decided that this substance was not a beef and malt wine, the question arises as to what a beef and malt wine really is, and it may not be without interest to put before the Society the information which the writer collected while dealing with this matter.

Hutchinson, in his *Food and the Principles of Dietetics*, has the following passage:

> "*Medicated Wines* are concoctions, the basis of which is port or sherry, to which has been added extract of beef, extract of malt, peptone, pepsin, coca leaves, cocaine, cinchona, iron, or some other dietetic or medicinal substance. A 'beef and malt wine' may usually be regarded as containing about 1½ ounces of extract of meat and 2 ounces of malt extract in a pint of 'detannated' port or sherry. . . .
>
> The use of these wines can on no grounds be recommended. In the first place, they are not worth the price charged for them, for it is far cheaper and also better for an invalid to get beef or malt extract separately and take along with them, if need be, a definite quantity of sound wine of known antecedents. In the second place, it is open to grave question whether the ferment of malt (diastase) is not much impaired by the action of the alcohol to which it is exposed when dissolved in a fortified wine, such as port or sherry."

This seems a somewhat high standard, and is considerably higher than is given by any other authority that has been consulted. It is important, however, as a medical opinion, and is specially interesting when read alongside the analyses which follow, and the so-called professional opinion which often accompanies the wrappers on bottles of such wines.

Pharmaceutical Formulas, published at the office of the *Chemist and Druggist*, suggests that beef and malt wine should contain 4 ozs. of extract of beef and 8 ozs. of extract of malt per gallon of wine. This is equivalent to 2·5 per cent. and 5 per cent., respectively.

I have approached two manufacturers of beef and malt wine, and they state that they add 2½ per cent. of extract of beef and from 2½ to 5 per cent. of extract of malt; a standard which is practically the same as that given by *Pharmaceutical Formulas* above.

It would appear, therefore, that a beef and malt wine should be prepared by the addition of at least 2½ per cent. of beef extract and 2½ per cent. of malt extract, whilst the opinion of Hutchinson shows that these are most certainly minimum quantities. These quantities are, of course, the proportions which are added. A considerable proportion of these extracts is insoluble in water or is precipitated by the alcohol of the wine, so that the amount of nitrogen and phosphorus contained in the wine as it is finally delivered to the consumer will be substantially less than the amounts calculated from the proportions of material added. Experiments have been made to estimate the amount of nitrogenous matters which is so precipitated, and this matter is considered below when dealing with the question of standard.

For the purpose of estimating the proportion of beef and malt extract present the most valuable evidence is obtained, of course, from the estimation of the total nitrogen and of the phosphorus (P_2O_5) in the ash. These estimations are carried out in the usual way, viz. the total nitrogen by the Kjeldahl process, the phosphorus by precipitation as phospho-molybdate, solution of the precipitate in ammonia and reprecipitation with magnesia mixture. In order that there may be no loss of phosphorus during ignition it is better to add excess of sodium carbonate to the wine before evaporation to dryness, although in practice such loss probably very rarely occurs.

MALT EXTRACTS.—It is necessary to know the figures which are given by commercial meat and malt extracts; I have determined such figures on present-day samples and these are compared below with similar figures contained in the literature on the subject.

Published figures for the composition of malt extract are few. Penn ("Leach," 3rd Edition, p. 729), as a result of the analysis of three samples, gives the albuminoids as 3·1 to 4·9 per cent., the ash as 1·19 to 1·23 per cent., and the phosphoric acid as 0·43 to 0·57 per cent.

Rink (ANALYST, 1904, **29**, 244) gives the ash of six samples as 1·34 to 1·64 per cent.

Harrison and Gair (*Year Book of Pharmacy*, 1906, p. 282), reporting on thirteen samples, give proteins varying from 3·6 to 7·0 per cent., but some of these samples were possibly adulterated.

The writer has examined several samples of commercial extract of malt purchased in the ordinary way by retail. These samples had the following composition, all figures being expressed on the original sample:

No. of Sample	Ash Per Cent.	P_2O_5 in Ash Per Cent.	Nitrogen Per Cent.
1.	1·4	0·57	0·99
2.	1·4	0·55	1·32
3.	0·88	0·36	0·73
4.	1·56	0·60	1·30
5.	1·60	0·70	1·46
6.	1·41	0·50	1·33
7.	1·31	0·34	1·20
8.	1·88	0·69	1·27

Sample No. 3 was a proprietary article specially prepared to obviate the awkward consistence of the ordinary extract; such an article would, of course, not be used in the preparation of a beef and malt wine. From these figures it would appear reasonable to take as averages for extract of malt, 1·3 per cent. of nitrogen and 0·6 per cent. of phosphoric anhydride.

MEAT EXTRACTS.—A number of different types of extract are on the market, but we need only consider the solid extract (originally prepared by Liebig, and of which "Lemco" is a typical example), as this is the material used by manufacturers of beef and malt wine—in fact, the wines are frequently termed "Liebig's

Extract of Meat and Malt Wines." The word "Liebig" in this sense usually bears no relationship to the Liebig's Extract of Meat Company (the original proprietors of "Lemco") and merely signifies an extract of meat made (or alleged to be made) by the process suggested by Liebig.

The following table gives the composition of various extracts according to different authorities.

Authority.	Ash Per Cent.	Phosphoric Acid Per Cent.	Total Nitrogen Per Cent.
Leach, 3rd Edn., p. 242	20·5—31·7	2·29—4·55	6·02—9·07
Hehner (Parry, "Food and Drugs," Vol. 1, 399)	18·8—29·4	5·16—6·95	8·21—9·80
American A.O.A.C.	Not more than 27		At least 8
König & Bomer	—	—	9·28
Elsdon	16·5—23·2	5·11—5·50	8·10—8·85
Villavecchia "Applied Analytical Chemistry," II. 16	17·0—25·0	—	8·5 —9·5

The last author states that not less than 56 per cent. of the extract should be soluble in 80 per cent. alcohol, and that the more usual value is 61 to 64 per cent.; this figure is used later in deciding what standard is reasonable for total nitrogen in a meat and malt wine.

WINES USED AS A BASE.—The wine which is generally used as a base in the preparation of meat and malt wine is a cheap Spanish or Portuguese wine of a "Port" character, but, of course, any cheap wine may be used. The following table gives results obtained for total nitrogen and phosphorus pentoxide by other workers on a variety of wines of different kinds:

Authority.	Ash Per Cent.	P_2O_5 Per Cent.	Total Nitrogen Per Cent.
König, Leach, 3rd Edit., p. 687	0·18—0·74	0·022—0·046	0·019—0·043
Bigelow, U.S. Dept. of Agri. Bur. of Chem. Bul. 59	0·05—0·45	—	0·014—0·147
Parry, "Food & Drugs," 1, 314	0·13—0·61	0·027* / 0·014—0·068	—
Villavecchia, II. 220	0·15—0·45	0·02 —0·06	—

A number of wines of the type usually used by manufacturers—in fact, some of them have been wines actually taken from stocks kept for the purpose—have been examined by the author with the following results in grms. per 100 c.c.:

Sp. Gr.	Alcohol by vol. Per Cent.	Total Solids Per Cent.	Ash Per Cent.	P_2O_5 in Ash Per Cent. on original sample.	Total Nitrogen Per Cent.
—	15·9	—	0·23	0·027	0·021
1·0179	14·3	7·2	0·25	0·016	0·034
1·0172	16·2	8·3	0·32	0·044	0·083
1·0349	13·2	11·7	0·21	0·020	0·038
1·0372	13·5	12·6	0·26	0·013	0·025
1·0178	17·6	8·6	0·22	0·016	0·015
0·9954	9·2	1·6	0·29	0·048	0·024
0·9971	10·9	2·0	0·28	0·038	0·019

* Spanish red wine—average of many samples.

STANDARDS FOR BEEF (MEAT) AND MALT WINE.—From the various analyses given above it will be seen that the average figure for the total nitrogen in a meats' (beef) extract is about 9·0 per cent., and that 8·7 per cent. is certainly not a high figure to take. Malt extract shows about 1·3 per cent. of total nitrogen, so that a mixture of equal parts of meat and malt extracts will contain about 5·0 per cent. of total nitrogen; this is the standard which has been adopted. In those cases where a meat extract containing a lower percentage of nitrogen than 8·7 has been used, the factor based on this percentage will not, of course, give the actual amount of extract added. This would not seem to be important, as it is immaterial to the consumer whether his wine is prepared from a small amount of a good extract or a larger amount of a poor one; it shows, however, the necessity of stating the percentage of nitrogen that has been assumed in order to calculate the meat-extract present.

Assuming, therefore, 5 per cent. of total nitrogen as an average figure for a mixture of equal parts of meat and malt extracts, and a minimum quantity of 5 per cent. of this mixture to be added to the wine, it follows that, assuming all the nitrogen to be retained by the wine, the prepared beef and malt wine will contain at least 0·25 per cent. of nitrogen, without taking into account the amount of this substance (some 0·03 per cent.) natural to the original wine. As mentioned above, however, a proportion of the meat and malt extract is not soluble in the wine, according to Villavecchia, about 40 per cent. being insoluble in 80 per cent. alcohol. Experiments by the writer have shown that about 70 per cent. of the nitrogen added as a mixture of beef and malt extracts remains in solution. It follows, therefore, that a wine sold as a beef and malt wine should contain at least 0·18 per cent. of total nitrogen. That this is not by any means a large amount is shown by the fact that some wines of a port character themselves contain up to 0·08 (or even more) per cent. of total nitrogen.

Treating the percentage of phosphorus pentoxide in the ash in a similar way, it will be seen that a mixture of equal parts of meat and malt extract will contain about 2·8 per cent. Using the same reasoning as for the percentage of total nitrogen, the finished beef and malt wine should contain at least 0·12 per cent. of phosphorus pentoxide, allowing 0·02 per cent. for the amount naturally present in the wine, and assuming (as has been shown by experiment) that about 70 per cent. of the total phosphorus pentoxide of the meat and malt extracts goes into solution.

COMMERCIAL SAMPLES OF MEAT (BEEF) AND MALT WINE.—In order to discover how far various commercial samples could be truthfully described as "Meat and Malt Wine," a number of these have been examined, and the results are given in the following table. Figures obtained from an examination of various proprietary wines which are supposed to be of a beef and malt wine character are also included. Comment is hardly necessary, but it is interesting to observe that sample No. 8 had a label attached which bore the following words:—"The presence of a LARGE PROPORTION OF PHOSPHATES is distinct evidence of the

as esence of both malt and beef, while the IMPORTANT QUANTITY of NITROGENOUS ac XTRACTIVES affords further confirmation."
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No.	Sp. Gr.	Alcohol by volume Per Cent.	Total Solids Per Cent.	Ash Per Cent	P_2O_5 in Ash Per Cent.	Total Nitrogen Per Cent.	Description.
1.	1·0505	18·0	16·7	0·32	0·060	0·082	Liebig's Beef & Malt Wine
2.	1·0257	19·2	10·7	0·53	0·140	0·115	,,
3.	1·0367	16·1	7·4	0·54	0·090	0·110	,,
4.	1·0490	16·3	15·4	0·38	0·094	0·098	,,
5.	1·0494	20·4	16·6	0·32	0·048	0·052	,,
6.	1·0280	19·2	9·6	0·54	0·100	0·040	,,
7.	1·0234	18·0	9·7	0·27	0·018	0·120	Tonic Wine
8.	1·0286	17·6	10·2	0·32	0·047	0·052	——'s "——"
9.	1·0326	17·1	10·5	0·26	0·038	0·040	——'s Wine
10.	1·0341	17·4	11·3	0·33	0·062	0·058	"——"
11.	1·0262	16·0	8·7	0·36	0·060	0·049	"——"
12.	1·0530	18·6	16·2	0·44	0·080	0·080	——'s "——"

The Volumetric Estimation of Columbium.

BY W. R. SCHOELLER, Ph.D., AND E. F. WATERHOUSE.

THE volumetric estimation of columbium is based on the reducibility of columbic acid in solution and the re-oxidation of the lower oxide by permanganate, tantalic acid being unaffected under the same conditions. Assuming quantitative reduction to the sesquioxide, then, according to the equation $Cb_2O_3+2O=Cb_2O_5$, each c.c. of 0·1 *N* permanganate indicates 0·006655 grm. Cb_2O_5 (Cb = 93·1).

Two methods for the volumetric estimation of columbium have recently been published. The first is that of Metzger and Taylor (*J. Soc. Chem. Ind.*, 1909, **28**, 818), in which the mixed oxides of tantalum and columbium are fused with bisulphate; the melt is dissolved in sulphuric acid with addition of succinic acid to prevent hydrolysis, and the hot solution passed through a Jones reductor containing amalgamated zinc. The reduced solution is received in a flask filled with carbon dioxide and titrated with 0·1 *N* permanganate, one c.c. of which equals 0·007052 grm. Cb_2O_5.

The other method, due to Levy (ANALYST, 1915, **40**, 204), is a modification of that of Osborne (*Amer. J. Sci.*, 1885, **30**, 328). The mixed oxides are dissolved in hydrofluoric acid; the bulk of the acid is evaporated, hydrochloric acid is added to the remainder, and the solution reduced with granulated zinc in a flask filled with hydrogen. When all the zinc is dissolved, the liquid is diluted with sodium phosphate solution and sulphuric acid and titrated with 0·1 *N* permanganate. One c.c. = 0·00833 grm. Cb_2O_5 (Osborne's factor is 0·00763).

The main object of the present paper is to call attention to the unreliability of the volumetric estimation of columbium by the methods outlined, the authors' conclusions being drawn partly from their own work, partly from that of Treadwell (*Helv. Chim. Acta*, 1922, **5**, 806; ANALYST, 1922, **47**, 533).

The above factors show that neither process achieves a stoichiometric reduction, and that the sesquioxide stage is much more nearly reached by the powerful action of a zinc reductor than by simple solution of granulated zinc in the acid liquid. The supposition that definite intermediate oxides, such as Cb_3O_5, Cb_8O_{13}, $Cb_{10}O_{17}$, etc., are formed in the reduction may be dismissed as unlikely until their existence can be proved; no doubt the correct view will prove to be that only part of the pentoxide undergoes reduction, the balance remaining unaffected. Now, the extent of such incomplete reaction is not likely to be exactly the same every time, being easily affected by slight changes in the conditions under which it is carried out. Hence it is not surprising that the degree of accuracy attained in either process is admittedly not very great.

A. METZGER AND TAYLOR'S PROCESS.—A satisfactory explanation of the incomplete reduction of columbic acid in this process was given by Treadwell (*loc. cit.*), who found that columbium solutions prepared with addition of succinic acid are unstable, as they deposit a white precipitate on standing, and that their reducibility decreases with increasing age or dilution. On titrating the reduced solutions he obtained irregular results, and arrived at the conclusion that columbic acid is present partly in the colloidal state, only the dissolved portion being acted upon by the zinc. To verify this inference, he titrated columbium solutions in presence of titanic sulphate, ammonium molybdate, or ammonium vanadate in known considerable excess, and obtained at once a more nearly complete and constant reduction. The addition of these salts produced soluble complexes in which the columbic acid was reduced completely; but Treadwell could not calculate the extent of the reduction, as he had no pure columbium pentoxide at his disposal.

A short time prior to the appearance of Treadwell's paper the authors had concluded an investigation, begun in 1920, into the volumetric estimation of columbium. This comprised a repetition of the work of the earlier investigators and the full elaboration of a process suggested in the last paper published by the late W. B. Giles (*Chem. News*, 1909, **99**, 1). In the test analyses, by Metzger and Taylor's method, the fusion of columbium pentoxide with bisulphate was carried out in glass flasks, after addition of 2 to 3 c.c. of sulphuric acid, which obviated the need of using platinum vessels; otherwise the original directions were carefully followed, particularly as regards amalgamation of the zinc. Four titrations carried out with +30-mesh amalgamated zinc (ordinary pure) gave a mean factor of 0·007424 grm. Cb_2O_5 per c.c. of 0·1 *N* permanganate (maximum deviations, +0·77 and −0·89 per cent.), while two tests, in which chemically pure zinc granules (similarly amalgamated) were used, gave 0·008139; we were thus unable to get the same factor as Metzger and Taylor's (0·007052). Our results exemplify the magnitude of the error likely to arise, not only from the indiscriminate use of the same factor by independent workers, but even from a change in the supply of zinc; they satisfied us

as to the remarkable indefiniteness of the reaction of metallic zinc upon columbic acid, to which the disparity between Levy's and Metzger and Taylor's factors had already pointed. Metzger and Taylor found the degree of reduction to vary even according to the extent to which the zinc had been amalgamated, and found it necessary to prescribe definite proportions of zinc and mercury for the amalgamation. Treadwell's comment is that the cause of the influence of the amalgamation on the degree of reduction of the columbic acid is not elucidated by the experimental evidence.

B. OSBORNE AND LEVY'S METHOD.—Treadwell investigated the reduction of columbium fluoride dissolved in strong hydrochloric acid, but not by addition of granulated zinc as directed by Levy; he passed the solution through a cadmium reductor and titrated it with permanganate in presence of manganous sulphate. Proceeding in this manner he obtained concordant results, but could not arrive at a factor as his columbium preparation was contaminated with tantalum. Treadwell observes that if the hydrofluoric acid solution is evaporated to dryness and the residue taken up in strong hydrochloric acid the resulting solution is not always clear, while any excess of hydrofluoric acid not expelled by evaporation prevents complete reduction. We desire to emphasise this point, as it forms one of our arguments against Levy's method.

In our test analyses, ten estimations were made in strict accordance with Levy's directions. Nine titrations gave an average factor of 0·01006 (Levy's factor: 0·00833), with maximum deviations of +3·75 and −5·30 per cent. We ascribe these errors chiefly to the interference of hydrofluoric acid, the removal of which "*nearly* to dryness" (Levy's italics: original, p. 208) is, in our opinion, a great weakness of the method; for "enough hydrofluoric acid must be present to give a perfectly clear solution in hydrochloric acid," but "the quantity must be so small as not to interfere with the reduction" (p. 209). We argue with Treadwell that any quantity of free hydrofluoric acid, however small, prevents the reduction of a corresponding quantity of columbium. We must assume our manipulations to have been made correctly, for in all cases we obtained clear solutions in hydrochloric acid and observed the formation of a bulky white precipitate within two minutes after the end of the titration. Levy says: "If this precipitation does not take place the result obtained should be treated with suspicion, probably being too low" (p. 210).

A source of error not investigated by us was pointed out by Levy himself in the variation of the tantalum present, low results being obtained with low ratios of columbium to tantalum. This can be explained by the formation of fluoride complexes, tantalum fluoride acting like an excess of hydrofluoric acid in preventing the reduction of part of the columbium.

A most serious objection against the three methods investigated is the influence of the physical and chemical state as well as the quantity of the reducing agent. We used the same brand of chemically pure, granulated zinc in the work on Levy's method; his original directions prescribe the use of shot "23 of which usually weighed 10 grms." We conformed as closely as possible to this specification

except in the last test, in which 10 grms. of coarser shot was used, with the result that reduction was now less complete, as indicated by the factor 0·01124. Levy, on the other hand, found less complete reduction to attend the use of very small pieces, which dissolved too quickly; we conclude that the absence of a zinc reductor is an additional point against the method, being the cause of less complete and uniform reduction.

C. NEED OF STOICHIOMETRIC REDUCTION.—The evidence given in the preceding paragraphs shows that the volumetric estimation of columbium in its present form lacks the features of a reliable oxidation process. In the volumetric estimation of iron the reduction may be carried out with hydrogen sulphide, sulphur dioxide, stanuous chloride, or zinc (either in Jones' reductor or added to the solution); the iron and acid concentration may vary within wide limits, and the presence of a large number of other elements is without influence: the same factor is always valid, and it is obtained by stoichiometric calculation. In the case of columbium the factors are quite empirical; not only were we unable to get those obtained by Metzger and Taylor and by Levy, in spite of close adherence to their directions, but every time a slight variation in the working conditions occurred, a serious deviation in the factor was the result. Observations to the same effect were made by Treadwell as well as by the authors of the methods under consideration. We are forced to the conclusion that the two processes must be regarded as unreliable.

Speaking of his method, Levy says: "Reduction does not go as far as in any of the others, which may be an advantage, in that the less one aims at, the more likely one is to obtain that little" (p. 209). With the practical experience gained, we totally disagree with this view, and consider that, in an accurate oxidation method, reduction should always proceed to a definite oxide regardless of slight variations in the working conditions and the amount of tantalum present. In the volumetric estimation of other elements, we know of no reliable method, in use at the present time, that is not based on stoichiometry.

Treadwell points out that the immediate difficulty in the case of columbium consists in obtaining the whole of the element in a state of true molecular solution, and as he has intimated his intention to continue his experiments in that direction, the authors have suspended further work for the present.

A brief consideration of the reduction of the oxides of two allied metals will support the authors' views on the necessity of stoichiometric reduction: tungstic, like columbic acid, does not give true solutions with strong acids. When the solution of tungstic acid in hydrochloric acid is reduced with zinc a colloidal blue precipitate is formed. The re-oxidation of the lower oxide gives erratic results, and there is no satisfactory oxidation method for tungsten. Molybdic acid is readily soluble in strong acids, and after passage through Jones' reductor the molybdenum in this brown solution can be estimated accurately with permanganate, reduction proceeding quantitatively to the sesquioxide. In feebly acid solutions, however, molybdic acid exists in a form more akin to that of tungstic and columbic acids, and such solutions upon reduction furnish colloidal molybdenum

blue. This is of indefinite composition, intermediate between the tri- and the sesquioxide.

D. GILES' PROPOSED METHOD.—Giles (*loc. cit.*) suggests the use of zinc dust as a reducing agent, and effects solution of columbium pentoxide by fusion with potassium carbonate, solution in water, and boiling with excess of phosphoric acid until the solution is quite clear. The last paragraph of his paper gives a short outline of the process he intended to work out; but no further paper was published by him. We desired to ascertain whether the intensely-coloured solution produced by the energetic action of zinc dust upon the phosphoric acid solution of columbium might not contain the whole of that element as sesquioxide. Our results prove that such is not the case; no useful purpose would therefore be served by a detailed account of our method, the numerous tests made, and the elaborate apparatus devised, in which addition of zinc dust, reduction and heating, filtration through a quartz-sand column, washing, and collection in a solution of ferric alum were carried out under complete exclusion of air. The following is a *résumé* of our observations, which all confirm what has already been said concerning the erratic behaviour of columbic acid in contact with zinc:

(1) Eleven consecutive titrations gave a mean factor of 0·006990, with maximum deviations of +1·00 and −1·91 per cent., hence reduction stopped short of the sesquioxide. In these tests 5 grms. of zinc dust were used for 0·25 grm. of columbium pentoxide; the solution was obtained according to Giles' suggestion, no bisulphate being employed.

(2) When the quantity of zinc dust was doubled, reduction was less complete, the increase in zinc being equivalent to a decrease in the acid concentration: the mean factor from six titrations was 0·007261 (maximum deviations, +2·25 and −2·22 per cent.).

(3) If the zinc dust in the above method was replaced by pure zinc powder, filings, or granules, the results were altogether hopeless; the burette readings registered between 10 and 80 per cent. of the reduction attained with zinc dust. Magnesium was also tried: the reduction was almost nil.

(4) In the final series of tests, the columbium pentoxide was fused with bisulphate and sulphuric acid, the cold liquid heated with phosphoric acid, and the solution reduced with zinc dust, etc., as before. Entirely different values were now obtained; for tests in which 5 grms. of zinc dust were used, gave an average factor of 0·008365 (maximum errors, +2·19 and −2·48 per cent.), while two tests with 10 grms. of zinc dust gave 0·009897.

In conclusion, the authors' modification of Giles' method must also be regarded as unreliable; under strictly uniform conditions fairly concordant results may ensue, but discrepancies occur as in the other methods, and a slight change in the working conditions causes serious divergences in the degree of reduction. The factors obtained are non-stoichiometric. The presence of a colloidal phase is, no doubt, the cause of the irregularities.

SUMMARY.—(1) Theoretical considerations and the results of original tests are submitted to prove that the existing methods for the volumetric estimation of columbium are unreliable. These methods furnish empirical factors.

(2) The view is advanced that the criterion of an accurate volumetric method should be a stoichiometric factor, indicating reduction to a definite oxide.

THE SIR JOHN CASS TECHNICAL INSTITUTE,
ALDGATE.

Apparatus for Extraction and Solvent Recovery.

BY S. A. DE LACY, A.I.C.

(*Demonstrated at the Meeting, February* 6, 1924).

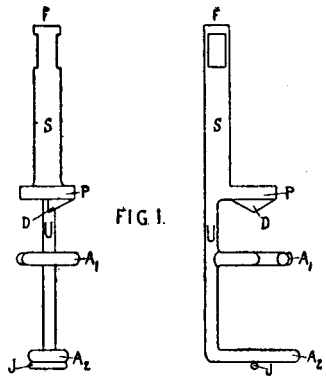
IN systems of extraction by volatile solvents the vessel containing the solution of the extracted material is removed and connected with a condenser, so that the solvent may be recovered by distillation.

This apparatus has been devised to obviate the inconvenience of disconnection with its attendant losses of time, solvent, and heat. In it, the container of the substance to be extracted is suspended in the extraction vessel so that the condensed solvent from a reflux condenser drops immediately into the upper end of the container, percolates through the substance and, charged with extracted material, eventually falls into the boiling liquid.

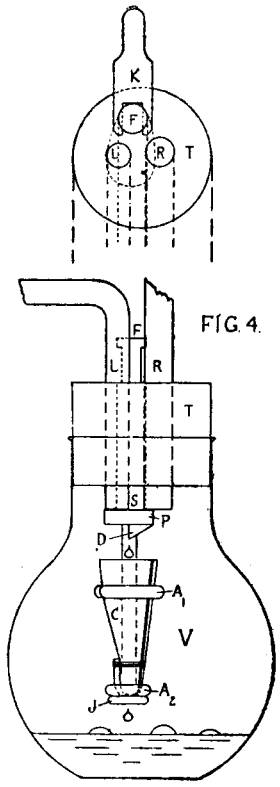
The essential feature of the apparatus consists of a glass frame operated by a key external to the extraction vessel, by which means the reflux tube or distillation tube may be closed. The frame consists of a thick circular rod S, having near its upper end two parallel surfaces cut; to its lower end an oval platform P is attached with a drip point D beneath it. The support for the container of the substance to be extracted is attached to the lower surface of the platform by the upright U so that the apex of the drip point lies over the centre of the container, the junction of the upright with the platform being separate from the drip point, since otherwise solvent tends to run down the upright instead of falling from the drip point.

The support for a folded paper, made by shaping ashless filter paper on a thick piece of plate glass and securing the turned up end, is shown in Fig. 1. This form has two arms, A_1, A_2, attached to U. In the oval arm A_1 there is a gap through which the upper end of the paper is inserted until it fills the arm, when in position; the lower end of the paper drops within the closed rectangular arm A_2 on to the cross bar J.

In Figs. 2 and 3 the distinct forms of support hold the container by a strong glass pin passing through holes placed in its sides so that it is vertical when its weight rests on the pin; they are specially suitable for alundum extraction thimbles. The substance may be covered by a perforated disc of filter paper or some cotton wool, the pin being above this.

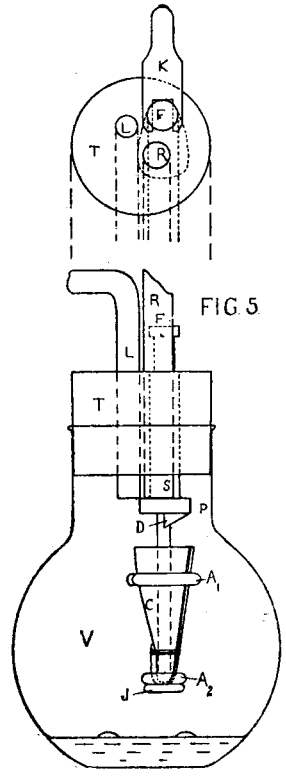


ELEVATION, FRONT AND SIDE.



ELEVATION AND PLAN.

DIAGRAM OF FRAME AND ITS USE.
(Similar letters refer to similar parts throughout).



ELEVATION AND PLAN.

Fig. 2 shows a support having the extremities of its arms projecting inside the container and bent to form vertical slots. In use, the upper end of the container is placed over the extremities, then a pin N is pushed through the holes in the container sides to rest easily in the slots.

Fig. 3 illustrates a support composed of a horizontal semi-circular arm, the ends of which are bent to form vertical slots. In use, the holes in the container sides have a pin pushed through them and its projecting ends are dropped into the slots as the container is placed within the arm. All supports are made of glass rod, making the frame capable of hard wear.

The stopper of the extraction vessel (in this instance of cork) has three holes in it, and the frame end F is pushed through the suitable orifice in the base of the stopper until the flat surfaces are just clear of the upper surface of the stopper when a rectangular key is fitted on them with its lower surface resting on the stopper and its upper surface against the uncut portion of the rod S. A reflux condenser tube and a tube L, to conduct away the vapour of the distilled solvent, are pushed down through the remaining holes until the flat ends just clear the upper surface of the platform. The container is placed in the support and the platform moved into the position for extraction, that is, with the portion above the drip point partially closing the orifice of the reflux condenser tube, while the tube L for distillation is shut off (Fig. 4). The vessel containing the solvent is connected and the solvent boiled, when the condensed vapour runs down the reflux tube on to the platform, over its edge, and falls from the drip point D. After extraction is considered complete the platform is moved to close the tube R while the tube L is left entirely free; thus the solvent is recovered (Fig. 5). No solvent falls from the drip point in this position, so that, on disconnection, the extracted material is in the vessel, and the dry container, with its unextracted contents, can be easily removed. In both the above positions of the frame the condensed solvent on the upper surface of P forms a liquid seal for a completely shut-off tube, and in no intermediate position does P close both tubes; this ensures safety. An indifferent gas may be passed through the apparatus during the operations.

When difficulty in extraction is anticipated the excellent method of Stokes (ANALYST, 1914, 39, 295) may be employed. The platform being as in Fig. 5, sufficient solvent is placed in the extraction vessel to cover the substance; thus the warm liquid asserts its soaking action while the solvent slowly distils. The frame is then turned so that extraction may be finished as in Fig 4, after which the remainder of the solvent is recovered by the return of the key to its original position.

Where no container is held in supports, as in Figs. 2 and 3, it is obvious that the system may be used with advantage for reactions which require boiling under a reflux condenser, followed by the subsequent distillation of unaltered solvent or a resultant product.

The bench and permanent wall-type forms of the apparatus demonstrated were devised and constructed by the author. For modifications and further details see Eng. Pat., 206, 711, Nov. 22, 1922, and addition 28,018, Nov. 7, 1923.

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.

NOTES ON THE LIQUID FROM RIPE COCONUTS.

THE liquid from ripe coconuts varies from nearly clear to distinctly opalescent; in the latter condition, when examined under the microscope, it shows fatty globules, agglomerations of fat and amorphous matter. No starch granules or anything staining blue with iodine are seen. One or two rod bacteria are present.

The liquid from one nut (No. 4) was measured and found to be 122 c.c. It, like the other three examined, had the usual sweet sappy taste, with the characteristic nutty flavour.

Specific Gravity.	Water = 1000.	Solids in grms. per 100 c.c. (Sp. gr. – 1000). ÷4·25.*
Mixed liquid from 2 nuts	1042·1	9·92
Nut 3	1055·5	13·06
Nut 4	1045·9	10·80
Average sp. gr. = 1047·85.	Average solids = 11·26 g. per 100 c.c.	

* *Dry Solids* :—10 c.c. of (Nut 4) dried in a platinum dish in water oven and weighed at intervals till a point of fair constancy was reached before definite browning set in gave 1·08 grms. = 10·8 grms. per 100 c.c. (Sp. gr. 1045·9 – 1000÷10·8 = 4·25 divisor for solids).

Optical rotation of the 3 samples of liquid reading 100 mm. tube Soleil-Ventzke-Scheibler instrument:

	AD 4·25. div.
Nos. 1 & 2	$\alpha = 10·6 = 37·0$
3	$\alpha = 16·1 = 42·7$
4	$\alpha = 13·2 = 42·3$

Average specific rotatory power = 40·7

Further experiments were made with the liquid from Nut 4. The amount of ash obtained was 1·25 grms. per 100 c.c.

It had some reducing effect with Fehling solution, 1 c.c. giving 0·011 CuO = 1·1 grm. per 100 c.c., which brought to invert sugar or dextrose by divisor 2·31 = 0·47 per cent. of reducing sugar.

A dilution of the liquid, 10 c.c. to 100 c.c. with distilled water + 0·5 grm. of citric acid, when digested at boiling temperature for one hour gave a greatly increased reduction with Fehling solution. Thus 2 c.c. gave 0·071 CuO = 17·75 grm. of CuO per 100 c.c. of original liquid, which, corrected for the original reducing power and brought to reducing sugar (invert) by use of the divisor 2·31, gave 7·21 grms. per 100 c.c. of invert sugar = 6·87 original sucrose from which it proceeded.

The change in optical rotation caused in the above experiment was calculated into sucrose and gave 7·3 grm. per 100 c.c.

The coconut liquid (No. 4) proved to be directly fermentable by Burton yeast;* at the end of an active fermentation the difference between the original gravity and the extract gravity was 28 degrees; this divided by 3·86 (the solution divisor for sucrose) and corrected for the 0·47 grm. per 100 c.c. of reducing sugar originally present, gave 7·1 grms. per 100 c.c. fermented, which, in face of the evidence afforded by the citric acid inversion, may be confidently regarded as sucrose. We thus get:

	Grms. per 100 c.c.
Sucrose by inversion and CuO	6·87
,, ,, optical rotation	7·30
,, ,, fermentation	7·10
Average	7·09

When the liquid is warmed coagulation sets in at about 43° C. and becomes more and more pronounced as the temperature rises towards boiling point. The white coagulum was separated by filtration and examined. It gave evidence of containing cellulose in some form, a portion being soluble in ammoniacal cupric oxide, reprecipitable on rendering the solution slightly acid, and it showed nitrogen by Armstrong's test (heating with sodium, etc).

The original liquid tested with paste made from potato starch showed a feeble diastatic power only, the paste becoming somewhat thinner. The approximate P_H value of the original liquid was 6·0

There can be little doubt that the coconut liquid (or milk, as commonly so-called) is a very complex and interesting liquid, containing, as it must do, the substances necessary to the deposition or elaboration of the flesh of the nut. Cellulose matter and fat evidently come from it; and it is a fair deduction that sucrose plays a very important part in the cell metabolism.

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* The fermented liquid had a poor, vapid taste.

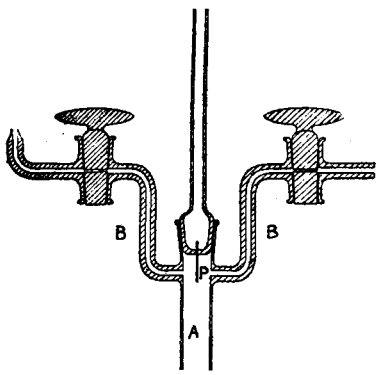
HYDROGEN ELECTRODE FOR USE WITH MEAT PASTES, ETC.

A KNOWLEDGE of the P_H value of meat pastes, potted meats and fish, etc., is sometimes useful as indicating the liability of these products to become infected with food poisoning organisms, more especially *Bacillus botulinus*. This organism is unable to develop in media the acidity of which exceeds a certain point.

With the form of electrode here figured, these determinations may readily be carried out on the meat paste itself, without previous extraction with water.

The central tube A is tightly packed with the meat paste, and, with the aid of a small glass plunger, the upper surface of the paste is levelled off, so as to be just in contact with the tip of the platinum wire P. The lower end of the column of meat paste is flush with the bottom of the tube A. The electrode is then adjusted so that the lower end dips into a beaker containing saturated potassium chloride solution, and the electrical circuit joined up in the usual manner (ANALYST, 1921, **46**, 318). A slow stream of pure hydrogen (preferably electrolytic) is passed continuously through the tubes B.B while the readings are being taken. If the meat paste is tightly packed, its conductivity is quite high enough for sharp readings to be obtained on the bridge.

Several samples of potted meat and fish examined in this way gave the following P_H values:



		P_H
Chicken, ham and tongue	Sample 1	5·33
	,, 2	5·45
Chicken and ham	,, 1	5·58
	,, 2	5·60
Ham and tongue	,, 1	5·60
	,, 2	5·65
Salmon and shrimp	,, 1	5·82
	,, 2	5·82
Salmon and anchovy	,, 1	5·55
	,, 2	5·63
Shrimp	,, 1	6·28
	,, 2	6·43

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TESTS FOR THE PURITY OF CARBON TETRACHLORIDE.

IN view of the recent extensive use of carbon tetrachloride as a remedy for hook-worm, and the occasional reports of serious symptoms arising after its administration, some investigations into the tests for the purity of this drug were undertaken. Most of the tests which could be found in the books available were tried, and those which were found most useful are detailed in this paper, which is offered for publication in the hope that other analysts may be induced to give their experience of such tests, and also their opinion as to the standard of purity to be demanded before any sample of this drug is passed for use as medicine.

The tests proposed are:

I. FOR ADULTERATION: THE DENSITY.—This was determined on three specimens, which all appeared to be pure by other tests, and was found to be, at 30° C.

$$1{\cdot}5744 \pm 0{\cdot}0001$$
$$1{\cdot}5745 \pm 0{\cdot}0005$$
$$1{\cdot}5742 \pm 0{\cdot}0003$$

Between 15° and 30° C. the rate of change of density with temperature was found to be $0{\cdot}00200 \pm 0{\cdot}00005$ per degree, and linear within this error. The density at 15° C. is, therefore, $1{\cdot}6044 \pm 0{\cdot}0007$. These figures are true densities, *i.e.* the mass in grms. of 1 c.c. of the liquid.

II. FOR PHOSGENE, ETC.—Some of the carbon tetrachloride is shaken with water in a separating funnel and the water phase tested for

(1) Neutrality; (2) absence of chlorides; (3) absence of free chlorine.

The benzidine test was also used as for chloroform, but, as benzidine is insoluble in carbon tetrachloride, about 10 per cent. of chloroform was added to bring the benzidine into solution. Obviously a blank test with the chloroform used must be made simultaneously.

III. For Carbon Bisulphide.—One c.c. of the carbon tetrachloride is mixed with 2 c.c. of aniline oil and 2 drops of ammonia solution (0·880). After standing five minutes 1 or 2 c.c. of a freshly made, dilute solution of sodium nitroprusside (about 1 per cent.) are added, and the mixture shaken. A purple colour in the sodium nitroprusside layer indicates the presence of carbon disulphide, and the test made in this way will give a distinct reaction with 0·03 per cent. The purple fades quickly when the carbon disulphide is only present in small quantities, but can generally be reproduced, after standing, by shaking again. A considerable number of experiments was made to determine the best proportions in which to mix the reagents in this test, and it was found that the delicacy of the test was especially affected by the amount and the strength of the ammonia used. In some cases a green colour develops on standing, and this appears to be due to the sodium nitroprusside solution not having been prepared recently enough. It does not seem to be due to any impurities in the carbon tetrachloride. In this laboratory this test has proved more satisfactory than the xanthate test (Lunge, *Technical Methods*, III., 327) or the ammonium thiocyanate test.

IV. For Sulphur Chloride, etc.—A small silver coin (a two anna bit), preferably much worn, is cleaned with emery powder and soap and water, and allowed to lie in the carbon tetrachloride to be tested. In the presence of as little as 0·001 per cent. of sulphur chloride, S_2Cl_2, a darkening of the silver begins to show after ten minutes.

V. For Aldehydes and other Oxidisable Substances.—One c.c. of the carbon tetrachloride is shaken at intervals during one hour with 10 c.c. of a solution containing $N/1000$ potassium permanganate solution and $N/2$ sulphuric acid. The permanganate should not be entirely decolorised. The test can be made quantitative by running a blank and titrating the residual permanganate with $N/1000$ oxalic acid. Schiff's test for aldehydes was also used, the carbon tetrachloride being shaken with the reagent; this will detect 1 part of benzaldehyde in 10,000

VI. For other Organic Substances.—The carbon tetrachloride, when shaken with strong sulphuric acid, should not colour the acid.

The above tests will detect, in the proportions noted, any impurities which seem likely to occur, and good specimens of carbon tetrachloride will pass them all. It is suggested that such a quality be demanded of any specimen of this drug to be used medicinally.

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NOTE ON THE DENIGÈS TEST FOR BUTYRIC ACID.

A method for the detection and estimation of butyric acid is given in an abstract in the Analyst (1918, **43**, 145). A pink colour is developed on treating a dilute solution of butyric acid with hydrogen peroxide in presence of ferrous iron, and, after the removal of the iron, adding sodium nitroprusside and acidifying.

The process has been investigated with a view to its application to the detection and estimation of small quantities of butter in margarine.

It was first noted that the quantity of hydrogen peroxide recommended in the case of the more dilute solutions was insufficient to oxidise the iron, and that the amount of sodium hydroxide mentioned was indefinite; but conditions were

found for the reaction to proceed, and the colour, varying in intensity with the amount of butyric acid present, was regularly obtained.

To determine whether the test was specific for butyric acid, caproic acid was substituted. The colour was again produced, leading to the supposition that the caproic acid (b.pt., 201° to 203°) was contaminated.

Carefully fractionated caprylic acid (b.pt., 234·5° at 754 mm.) reacted similarly, however, the colour intensity indicating a degree of contamination with butyric acid equal to about 20 per cent.

A fraction of higher boiling point was next tried with similar results. Attempts were then made to remove any possible traces of butyric acid from the higher homologue (b.pt., higher than 234° C.) as follows:

(*a*) Repeated washing with hot water in a separating funnel.

(*b*) Fractional salting out of the sodium salts.

(*c*) Recrystallisation of the acid from 500 times its weight of water.

By none of these methods was it possible to detect any diminution in the proportion of butyric acid as indicated by the test.

The Reichert distillate from coconut oil also gave a positive reaction, although this oil is stated to be free from butyrin.

Acetic acid yielded no colour.

It appears to be certain that the fatty acids higher in the series than butyric give a similar reaction, and that the test fails as a method of estimating small quantities of butyric acid.

The writer is indebted for the suggestion of the problem to Mr. G. D. Elsdon, in whose laboratory the tests were carried out.

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THE ESTIMATION OF CHROMIUM.

ALTHOUGH the results given by Mr. Britton in his paper on the estimation of chromium (ANALYST, 1924, 130), go further to prove the accuracy of the alkaline oxidation method, he is mistaken when he says that "Up to the present no one appears to have used sodium peroxide, as such, in the volumetric estimation of chromium."

As a matter of fact, the method has been in common use, at least by leather trade chemists, for the estimation of chromium in chrome tanning liquors, since (I believe) the early days of chrome tanning, and is described in most text books on leather chemistry.

It appears in Prof. Procter's well known Pocket Book, and also in the present writer's *Practical Leather Chemistry*. It has always been considered an accurate method, but iron should be removed by filtration from the alkaline solution of chromate (*cf. Practical Leather Chemistry*, p. 87).

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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.

SAND IN CINNAMON.

On March 24th a tradesman was summoned at Thornaby-on-Tees for the sale of cinnamon adulterated with sand. According to the analyst's certificate the sample contained 3·1 per cent. of silicious matter, instead of only 2·1 per cent., and the additional matter was practically all sand.

For the defence it was urged that, as cinnamon grew in sandy soil, it was practically impossible to keep it free from sand. Moreover, no standard for the permissible amount of silicious matter in cinnamon had been fixed by the British Pharmacopœia, since it was not possible to do so. Exception was also taken to the certificate on the ground that it did not state the component parts of the sample.

The Bench held that the certificate was not adequate and dismissed the case.

CHESHIRE CHEESE.

On March 14th a grocer was summoned at Salford for selling Dutch cheese in place of Cheshire cheese, which was demanded by the purchaser. Evidence was given that the cheese was sold at 1s. 2d. per lb., whereas the price of Cheshire cheese at that time was about 1s. 6d. per lb.

On analysis the sample was found to be deficient in fat to the extent of 26 per cent., and the Public Analyst stated in his certificate that "A genuine Cheshire cheese never contains less than 45 per cent. of fat calculated on the water-free cheese."

Mr. J. O. Garner, Secretary of the National Farmers' Union, stated that Cheshire cheese should be a full cream cheese, containing 28 to 30 per cent. of fat, which would correspond to 47 to 50 per cent. on a dry basis. Notwithstanding the fact that between 40,000 and 50,000 cows had been slaughtered in Cheshire, the competition of the Dutch cheese had caused the demand for Cheshire cheese to be slow.

The defence was that instructions had been given to the assistant not to sell the cheese as Cheshire cheese, that although it was Dutch cheese it looked like Cheshire cheese, and that the sale had been made through carelessness.

The Stipendiary fined the defendant 40s.

FISH MEAL WITH EXCESSIVE OIL AND DEFICIENT PHOSPHATE.

A case was heard before the Justices at the Bromley Police Court on March 24th, 1924, when the Kent County Council prosecuted the sellers of a meal known as "Drikod" Fish Meal, under Section 6, Sub-Section (a) & (b) of the Fertilisers and Feeding Stuffs Act of 1906.

Defendants in this instance had sold their fish meal with a warranty which had combined the albuminoids and oil, and had therefore failed to give the respective

percentages of oil and albuminoids, as required by Section 1 (2) of the Act. Also, whereas 15 per cent. of phosphates had been guaranteed, the fish meal was found to contain only 11·57 per cent.

The offence was admitted by the representative of the defendants.

The Official Agricultural Analyst for the County of Kent, Mr. F. W. F. Arnaud, F.I.C., in his evidence, stated that he had found this meal to contain 15·75 per cent. of oil, 32·75 per cent. of albuminoids, and 11·57 per cent. of phosphates. In his opinion this amount of oil was excessive, and it was important that the oil in fish meal should be low, because it had been ascertained that fish oil was liable to produce a taint in the flesh of an animal to which it was given. Also, as fish meal was given largely for its albuminoid, and partly for its phosphate contents, it was essential that the percentage of albuminoids should be as high as possible. The Association of Fish Meal, Fish Guano and Fish Oil Manufacturers, comprising nearly all the manufacturers of fish meal in Great Britain, had agreed that a good fish meal should contain not less than 55 per cent. of albuminoids, not more than 5 per cent. of oil, and about 16 per cent. of phosphates. As a result of his experience he had found that fish meals of good quality approximately conformed to this composition, and that this was the quality of fish meal required for feeding purposes. The sellers of this particular fish meal had given a combined guarantee of albuminoids and oil of 50 to 55 per cent., and the real composition of the meal was, therefore, not apparent to the purchaser. The composition of this "Drikod" Fish Meal corresponded with the product known as fish guano, which was sold only as a fertiliser, and guanos contained fish and fish wastes which did not enter into the composition of meals used for feeding purposes. Fish guano was of much less value commercially than fish meal.

The Bench, after considering the evidence, fined the defendants £10 for the offence under Section 6 (a) and £5 under Section 6 (b), and allowed the prosecution £6 6s. costs. At the same time they did not wish to state that the defendants were fraudulent.

SPIRITS SOLD WITH NOTICE OF DILUTION.

On March 12th, a publican was summoned at the Hampstead Police Court for selling rum containing 3¼ per cent. of water in excess of that in a rum of 35 per cent. under proof. A similar summons was brought against another Hampstead licensee, for the sale of whisky 38·8 per cent. under proof.

In neither case was there any dispute as to the strength of the spirits sold, but it was urged for the defence that a notice was exhibited in the bar of each of the defendants to the following effect:—"All spirits sold in this establishment are of the same superior quality as heretofore, but, as required by the Food and Drugs Adulteration Act, they are now sold as diluted spirits, no alcoholic strength guaranteed."

The Bench were informed that, whichever way their decision went, it was intended to make an appeal to the High Court, and it was accordingly decided that, pending the result of the appeal, only the first case should be heard.

After hearing the arguments on each side, the Bench decided to convict the defendant on the ground that the notice was extremely ambiguous and misleading, and that it did not convey to the mind of the purchaser the fact that when he asked for rum he was to be supplied with spirits diluted in strength below the statutory limit. The sale was, therefore, prejudicial to the purchaser. As they did not consider the case a bad one, they fined the defendant 20s., with £5 5s. costs.

Notice of appeal was given and the Bench agreed to state a case.

Department of Scientific and Industrial Research.

FUEL RESEARCH BOARD.

METHODS OF ANALYSIS OF COAL. ESTIMATION OF NITROGEN.

EXPERIMENTS made subsequent to the issue of the description of the methods of analysis recommended by the Sampling and Analysis of Coal Committee of the Fuel Research Board (*cf.* ANALYST, 1924, 36), have shown that the particular modification of the Kjeldahl method for the estimation of nitrogen, described on pages 7 and 8 of their interim report*, gives perfectly reliable results; there is a close agreement between the results of this method and those obtained by a proved modification of the Dumas method.†

It has also recently been proved that copper sulphate is *not* a suitable substitute for mercury, as stated in a footnote on page 7 of the interim report, since its use yields, in general, low results.

* Fuel Research Board, Physical and Chemical Survey of the National Coal Resources, No. 2, "Interim Report on Methods of Analysis of Coal," 1923. Published by H.M. Stationery Office. Price 1s. 6d. net.

† "The Estimation of Nitrogen in Coal," by A. Baranov and R. A. Mott, *Fuel*, 1924, **3**, 31, 49.

Collection of Legislative Prescriptions Concerning Cheese.*

UNITED STATES OF AMERICA.—Cheese must not contain less than 50 per cent. of milk fat.† Skim and part-skim milk cheese must be so marked, and in the case of Edam, Roquefort and Camembert cheese quantity of contents of packages must be stated.

ARGENTINE.—Addition of salicylic, boric acid and its salts is prohibited.

AUSTRALIA.—All export cheese must be graded superfine, 1st grade, 2nd grade or 3rd grade, and no foreign matter, except rennet, salt or colouring matter deemed harmless by the Minister, may be added.

AUSTRIA.—Margarine cheese must contain 5 per cent. of sesame oil.

Cream cheese must not contain less than 50 per cent. of fat in the dry substance.

Whole milk cheese not less than						40	,,	,,	,,	,,
¾	,,	,,	,,	,,	,,	,,	30	,,	,,	,,	,,
½	,,	,,	,,	,,	,,	,,	20	,,	,,	,,	,,
¼	,,	,,	,,	,,	,,	,,	10	,,	,,	,,	,,
Skim milk cheese				,,	,,	,,	10	,,	,,	,,	,,

Ratio of fat to protein on the dry substance must be about 1:0·83 for whole milk cheese, and 1:2 for ½ whole milk cheese. Gervais cheese must not contain less than 50 per cent. of fat, Camembert 30 per cent. and Camembert (fat) 40 per cent., and in each case the water percentage must be 62.

BELGIUM.—No mineral substances, other than salt, and no antiseptics may be used.

* Summarised from the book published by Dr. A. J. Swaving, The Hague. (*Cf.* ANALYST, 1924, 207.)

† All Fat percentages are on the dry material.

BRAZIL.—

Cream cheese must contain	45	per	cent.	of	milk	fat in the dry substance.
Whole milk cheese must contain	35	,,	,,	,,	,,	,,
½ fat cheese must contain	25	,,	,,	,,	,,	,,
Skim milk cheese less than	25	,,	,,	,,	,,	,,

CANADA.—Cheese must not contain less than 45 per cent. of milk fat in the dry substance, and cheese for export must be graded for flavour, texture, closeness, colour, and finish.

	Minimum Fat percentage content.	Maximum Water percentage content.
DENMARK.—(A) Hard Cheese.	(stamped on cheese).	
Class I.	45	50
,, II.	30	54
,, III.	20	57
,, IV.	10	59
,, V. Skim milk	—	60
(B) Soft Cheese	(stamped on wrapper).	
,, VI.	45	60
,, VII.	30	60
,, VIII.	20	60
Roquefort	50	52
Camembert	45	60
Gervais	50	60
Guarg	—	65

Emmenthaler Cheddar must be in Class I., Danish Swiss in Classes I. or II., Gouda, Edam, Tilsit, Steppecheese in Classes I., II. or III., and factory cheese in classes III., IV. or V.

(*Note.*—These divisions are likely to be revised in the near future.)

ESTHONIA.—

Cream cheese must not contain less than						40	per	cent.	of	fat in the dry substance.
Whole milk cheese	,,	,,	,,	,,		30	,,	,,	,,	,,
¾ fat	,,	,,	,,	,,	,,	25	,,	,,	,,	,,
½ fat	,,	,,	,,	,,	,,	20	,,	,,	,,	,,
¼ fat	,,	,,	,,	,,	,,	10	,,	,,	,,	,,
Skim milk cheese less than						10	,,	,,	,,	,,

FINLAND.—Dairy factories have agreed that

Whole milk cheese should contain	45	per	cent.	of	fat.		
½ fat cheese	,,	,,	30	,,	,,	,,	,,
Skim milk cheese	,,	,,	15	,,	,,	,,	,,

FRANCE.—Camembert cheese must contain 36 per cent. of fat by weight on the dry substance, and be exclusively made from cows' milk. Further regulations for cheese in general are probable.

GERMANY.—"Proposals for Regulations concerning Foodstuffs, 1913," suggest minimum fat percentages for cream cheese, 50; whole milk cheese, 40; ¾ fat cheese, 30; ½ fat cheese, 20; ¼ fat cheese, 10; skim milk cheese, less than 10 in the dry substance. Modifications may be expected.

IRELAND.—Irish Free State manufacturers have agreed to 45 per cent. fat for whole milk cheese and 25 per cent. for skim milk cheese in the dry substance.

ITALY.—Cheese is considered to be made from margarine if the refractive index of the fat is above 48, and the Reichert-Meissl-Wollny number below 18.

If the latter is between 18 and 24 the cheese is considered suspicious, and if above 24 as genuine. Margarine cheese may not contain added colouring matter.

NETHERLANDS.—Manufacture of cheese is under strict Government control, and whole milk cheese must contain 45 per cent. of fat in the dry substance, and be stamped accordingly. Cheese made from partially skimmed milk must be stamped 40+, 30+, 20+, according to the proportion of fat contained.

NORWAY.—Cheese must be in one of the four following groups and be marked according to the fat content.

I. Whole milk cheese with not less than 45 per cent. of fat in the dry substance.
II. ½ fat ,, ,, ,, ,, ,, ,, 28 ,, ,, ,, ,,
III. ¼ fat ,, ,, ,, ,, ,, ,, 18 ,, ,, ,, ,,
IV. Skim milk cheese with less than 18 ,, ,, ,, ,,

It is likely that the limits for groups II. and III. will be raised.

POLAND.—Whole milk cheese must contain 30–40 per cent. of fat.
½ fat cheese ,, ,, 20–30 ,, ,, ,, ,,
Skim milk cheese ,, ,, 10–20 ,, ,, ,, ,,
and the maximum water content is 50 per cent. in the dry substance.

ROUMANIA.—Cheese from part skimmed cows' milk must not contain more than 75 per cent. of water, and sheep cheese more than 55 per cent., whilst mixed cheese from these milks must not have more than 70 per cent. of water. No other materials than lactic acid, rennet, salt, ferment or seasoning may be used in the manufacture of cheese.

SWITZERLAND.— [substance.
Whole milk cheese must contain not less than 45 per cent. of fat in the dry
¾ fat cheese ,, ,, ,, ,, ,, 35 ,, ,, ,, ,,
½ fat ,, ,, ,, ,, ,, ,, 25 ,, ,, ,, ,,
¼ fat ,, ,, ,, ,, ,, ,, 15 ,, ,, ,, ,,
Skim milk cheese less than 15 ,, ,, ,, ,,
The water content in Emmenthaler whole milk cheese must be between 26 and 36 per cent., and for ¾ fat Emmenthaler between 27 and 38 per cent.

STRAITS SETTLEMENTS.— [substance.
Whole milk cheese must not contain less than 50 per cent. of milk fat in the dry
Skim milk cheese ,, ,, ,, ,, ,, 10 ,, ,, ,, ,, ,,
Cream cheese ,, ,, ,, ,, ,, 60 ,, ,, ,, ,, ,,
No foreign fat, preservative other than salt, or colouring matter other than harmless vegetable colouring matter may be added.

SOUTH AFRICA.—Cheese must be graded into one of 3 grades for export according to flavour and aroma, quality (including body and texture), colour, salting, finish and general appearance.

NEW ZEALAND.—Quality of export cheese is rigorously controlled by Government, and standardised grading is being aimed at.

VENEZUELA.—Starch and gelatin may be added if stated, and all cheese not made with cows' milk must be marked in accordance with its origin, and the rind of artificial cheese must be coloured red. No cheese containing cheese-mites may be sold.

No special standards are in force in the following countries:—Algeria, British India, Bulgaria, Chili, Czecho-Slovakia, Grand Duchy of Luxemburg, Great Britain, Greece, Hungary, Lithuania, Livonia, Portugal, Serbia, Sweden, or Tunis.

D. G. H.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Detection of Nitrates in Milk. M. E. Pozzi-Escot. (*Bull. Soc. Chim.*, **1924, 35, 72**).—The detection of nitrates in milk by means of brucine or diphenylamine necessitates the use of concentrated sulphuric acid, which acts on the proteins and lactose, and is prone to produce colorations similar to that given by a trace of nitrate. This inconvenience may be avoided by precipitating the casein and fatty matter by heating the milk with sulphuric acid, neutralising the whey with ammonia and evaporating it to a syrup or even to dryness, and triturating the residue with cold, slightly diluted sulphuric acid and a little ether. The ether, which will contain the nitric acid of any nitrate present in the milk, is decanted, neutralised with a drop of ammonia solution, and evaporated at a low temperature on the water-bath, being the residue tested for nitric acid by any of the ordinary methods. T. H. P.

Composition of Canned Salmon. O. E. Shostrom, R. W. Clough and E. D. Clark. (*Ind. Eng. Chem.*, 1924, **16**, 283–289.)—The following table gives a summary of the results obtained in an investigation of the variations in the composition of salmon and trout as influenced by species and locality where caught. In the case of the special samples, the tins examined contained approximately the same section of an individual fish; with the exception of the bone content, all the results are calculated on the bone-free substance.

Species of Fish.	Number of Samples.	Bone Per Cent.	Moisture Per Cent.	Fat Per Cent.	Salt-free Ash Per Cent.	Proteins Per Cent.
Special samples.						
Sockeye salmon	126	1·96	67·19	8·58	1·32	21·04
Chinook salmon	216	1·78	63·98	13·51	1·21	19·51
Coho salmon	99	1·88	67·49	8·49	1·24	21·08
Pink salmon	90	2·32	70·05	6·20	1·31	20·56
Chum salmon	108	1·87	70·85	5·15	1·28	21·48
Steelhead trout	20	1·67	57·70	20·09	1·21	19·95
Commercial samples.						
Sockeye salmon	41	2·37	64·79	11·22	1·23	20·80
Chinook salmon	24	2·21	63·17	15·72	1·21	17·67
Pink salmon	9	2·53	69·80	6·99	0·76	21·40
Chum salmon	33	2·33	66·95	10·52	1·02	20·67
Steelhead trout	14	2·40	66·84	8·95	1·21	21·32
Atlantic salmon	6	1·14	64·30	12·49	1·22	21·14

The fat content of different salmon of the same species caught in the same locality varied considerably (from 6·7 to 27·2 per cent.); it also varied greatly between different species, localities, and years, the extreme variation being from 2·65 to 27·26 per cent. W. P. S.

Nature of Corrosion in Canned Fruits. E. F. Kohman and N. H. Sanborn. (*Ind. Eng. Chem.*, 1924, **16**, 290–295.)—The acidity of fruits is not the chief cause of perforations in the tin containers or of hydrogen formation ("blown" tins), since there is no relation between the hydrogen ion concentration of the different fruits and the extent to which they cause trouble. The fact that perforations, etc., are more common in varnished tins than in plain tins indicates that the substance causing the trouble is present in small quantity; its action is concentrated on minute unprotected parts of the varnished tins, but is less noticeable on the entire surface of the plain tins. A small amount of oxygen acts in this way in the case of canned apples, but the removal of oxygen by reduced pressure does not entirely eliminate perforations with all fruits, as it does with apples. Some fruits appear to contain oxidising substances other than oxygen, and it is suggested that these substances may be intermediary respiratory substances (oxyhaemoglobin, or possibly the fruit colours, the anthocyans). The fact that oxygen disappears slowly and hydrogen formation follows soon after its disappearance in varnished tins, whilst in plain tins the oxygen disappears rapidly, but hydrogen formation is delayed, indicates that there is a protection of some kind in the tin against acid alone, and that the combination of acid and oxygen is able to break down this protection and allow the acid to continue its action even after the oxygen has been used. There is no evidence as to the nature of this protection. W. P. S.

Apparatus for the Estimation of Tin in Canned Foods. E. J. B. Willey. (*J. Soc. Chem. Ind.*, 1924, **43**, 70–72 T.)—The ordinary gravimetric estimation of tin in foods suffers from two disadvantages; one, that it is needful to take a large quantity of the sample for the initial wet combustion, and two, that unless the tin is dissolved and re-precipitated it is liable to be contaminated, especially with silica, and so yield high results. The volumetric method as ordinarily employed tends to give low results on account of atmospheric oxidation. An apparatus is described and figured which enables the reduction and titration to be conducted entirely in an atmosphere of carbon dioxide. With the use of this method a smaller quantity of the sample (25 grms.) may be taken for the initial combustion, which is carried out as usual; the tin is precipitated as sulphide, filtered on asbestos, and dissolved in dilute hydrochloric acid with the aid of potassium chlorate. The solution is then boiled to expel chlorine and introduced into the apparatus, reduced by means of zinc foil, and finally titrated with 0·005 *N* iodine solution. H. E. C.

Estimation of Starch and Sugars by the Use of Picric Acid. M. R. Coe and G. L. Bidwell. (*J. Ass. Off. Agric. Chem.*, 1924, **7**, 297–304.)—The picric acid colorimetric method hitherto used in biological work (ANALYST, 1921, 288, 508) may be applied to the estimation of starch or sugar in feeding stuffs and similar materials. In the case of starch, the preliminary hydrolysis cannot be effected by means of taka-diastase, since concordant results are unobtainable in that way; if, however, a cold water extract of malt is used, the results are satisfactory.

When reducing sugars are to be estimated, the colour is developed immediately the sodium carbonate is added to the solution. With sugars that require hydrolysis, however, advantage is taken of the hydrolysing properties of the picric acid, and the alkali is added only after the hydrolysis has taken place.

When the starch or sugar is accompanied by mucilaginous substances such as are found in linseed cake, apple pomace, or pectin pulp, or by polyphenols, aldehydes, ketones, purine bases, the colouring matters present in molasses and the like, this colorimetric method yields high results; such disturbing substances may, however, often be eliminated by special preliminary treatment.

The procedure to be followed in the case of starch and in that of sugars is given in detail. The advantages of the method are its applicability to small samples or to materials containing small proportions of starch or sugar, the saving of time in comparison with the copper reduction method, and the simplicity of the manipulation, preparation of crucibles and the handling of bulky solutions being avoided. T. H. P.

Detection of Fruit Wines (Cider or Perry) in Ordinary Wine. F. Schaffer. (*Mitt. Lebensm. Unters. Hyg.*, 1923, 15; *Ann. Chim. anal.*, 1924, **6**, 88).—The author replies to Widmer's objections to his method for detecting cider in wine, this being based on the diminished reducing power of the wine. The following modified procedure is now recommended:—Forty c.c. of the wine are decolorised by boiling with 3 grms. of pure animal charcoal and filtered, and the filtrate is heated to boiling with excess of precipitated calcium carbonate. After further filtration the cooled liquid is treated, slowly and with shaking, with 8 c.c. of 10 per cent. barium acetate solution, and again filtered. Five c.c. of the neutral filtrate are mixed with 2 drops of 0·2 *N* silver nitrate solution, and, after being made alkaline by addition of 1 c.c. of 0·1 *N* sodium hydroxide solution, are placed in the dark, and the time elapsing before distinct reduction is apparent is noted; the test should be made in duplicate or even in triplicate. With cider the reduction becomes evident in a few minutes, and, with wines containing a marked proportion of cider, in 10 to 20 minutes; in some cases comparison of the suspected wine with the natural wine of known purity is advisable. T. H. P.

Evaluation of Charcoals used for the Decolorisation of Tinted White Wines. X. Rocques. (*Ann. Chim. anal.*, 1924, **6**, 65–72.)—No matter what care is taken to separate the juice rapidly from the stalks prior to fermentation, white wines prepared from grapes with colourless juice but coloured skin often exhibit a slight colour. In France it is permissible to decolorise such wines by means of animal black purified by treatment with hydrochloric acid to dissolve the alkaline earth carbonates and phosphates, and then freed from the excess of acid by washing with water. For the examination of such decolorising carbons, which are supplied mostly as paste (since they lose in value when dried) the following methods are recommended:—The moisture, total ash, and ash insoluble in hydrochloric acid are estimated on 5 grms. of the material. There being no definite correlation between the decolorising power and moisture content, no

maximum value to be allowed for the latter is suggested. The ash soluble in hydrochloric acid should be small in amount, a maximum of 1 per cent., calculated on the material as supplied, being suggested.

Reaction and chlorides:—Ten grms. of the black are shaken with 100 c.c. of distilled water and, after standing for 24 hours, filtered off. The reaction of the filtrate should not be acid towards litmus in comparison with that shown by the original water; further, the filtrate should contain not more than 0·1 per cent. of chlorides calculated as sodium chloride. When 5 grms. of the black are shaken for 15 minutes with 200 c.c. of a 10 per cent. aqueous solution of tartaric acid, and the liquid is filtered, the acidity, estimated by titration with 0·05 *N* sodium hydroxide solution and corrected for the slight dilution involved, should not be diminished by more than 7 or 8 per cent.; moreover, the tartaric acid solution should not dissolve more than 0·04 grm. of iron per 100 grms. of the black.

The decolorising power of the charcoal is tested as follows:—A pink liquid is prepared from red wine and 10 per cent. tartaric acid solution, to match a solution containing 8 c.c. of 0·1 *N* potassium permanganate solution, and 16·8 c.c. of 0·1 *N* potassium dichromate solution per litre. The amount of the black just required to decolorise 200 c.c. of the pink wine when shaken therewith in a half-litre bottle for 15 minutes is then determined. With paste black of good quality 200 grms. are required per hectolitre of the wine. T. H. P.

Chemical Composition of Sesame Oil. G. S. Jamieson and W. F. Baughman. (*J. Amer. Chem. Soc.*, 1924, **46**, 775–778.)—The oil expressed from Chinese yellow sesame seeds has been examined by well-known methods. The saturated and unsaturated acids were separated, and the former were methylated and separated by fractional distillation under reduced pressure, and the usual constants determined on the fractions, from which the fatty acids were subsequently liberated and crystallised. The unsaturated acids yielded no hexabromide, but linolic tetrabromide separated, and calculation from the iodine value showed the presence of oleic acid. The percentage composition of the glycerides is calculated to be as follows:—Oleic, 48·1; linolic, 36·8; palmitic, 7·7; stearic, 4·6; arachidic, 4; lignoceric acid, trace; and unsaponifiable matter, 1·7.

H. E. C.

Comparative Analytical Study of Various Oils in the Chaulmoogra Group. G. A. Perkins. (*Philippine J. Sci.*, 1923, **23**, 543–569.)—The oils from 9 species of authenticated seeds related to Chaulmoogra (*Gynocardia odorata*; *Hydnocarpus alcalae*; *H. anthelmintica*; *H. Hutchinsonii*; *H. subfalcata*; *H. venenata*; *H. Wightiana*; *H. Woodii*; *Pangium edule*) and 15 commercial samples of *Taraktogenos kurzii* oil were examined. Fractional distillation of the ethyl esters of 13 of the oils and crystallisation of the recovered fatty acids from each of four fractions were carried out, and the eight resulting fatty acid fractions were examined for freezing point, iodine absorption, specific rotatory power and saponification values. The results obtained from the whole examination confirm the exclusion of *Gynocardia odorata* from the Chaulmoogra group. A close similarity between the oil

of *Taraktogenos kurzii* and *Hydnocarpus* oils was established, and the only *Hydnocarpus* oil found to differ materially was that of *H. alcalae*, which contained a large proportion of chaulmoogric acid and little or no hydnocarpic acid. From a therapeutic point of view there appears to be no reason for considering Chaulmoogra oil superior to *Hydnocarpus* oils, but clinical confirmation of this point is needed. It appears doubtful whether *Pangium edule* oil contains either chaulmoogric or hydnocarpic acids, although some variable optically active constituent is present in the fatty acid fraction, possibly the above-mentioned acids, together with a destructive enzyme. Nine tables are given in the paper comprising (1) Physical and chemical data already available on Chaulmoogra and related oils, (2) Extraction of the oils, (3) Characteristics of the oils, (4), (5), (6), (7), (8), and (9) Characteristics of the fractions. D. G. H.

Detection of Adulteration of Cacao Butter. A. Koehler. (*Comptes rend.*, 1924, **178**, 940–941.)—Under similar conditions of temperature and of shaking, the volume of ethylacetoacetate which must be added to cacao butter to produce a standard turbidity is a constant dependent upon the purity of the sample, and hence is useful for estimating impurities or adulterants; it is more sensitive than the determination of the usual constants. An adulteration of 15 per cent. with other vegetable butters is detected with certainty. As the number of c.c. of the ester required to produce the standard turbidity is largely influenced by temperature it is best to titrate the sample and one of known purity, for comparison under identical conditions. The method is to run the ester from a burette into a 20 per cent. solution of the cacao butter in chloroform at a temperature of 15–20° C., until the appearance of a turbidity which is not increased by a further drop (an end point quite easily recognised). The number of c.c. of ester required by 2 c.c. of the 20 per cent. solution is called "the turbidity index," and is remarkably constant for pure cacao butters, but is increased by an adulterant. In the case of samples grossly adulterated the ester may precipitate the adulterant; in such cases the solution is diluted with a 20 per cent. solution of a pure cacao butter. H. E. C.

Brucine in Strychnine Nitrate. Reck. (*Chem. Zeit.*, 1924, **48**, 166.)—Strychnine nitrate not infrequently contains brucine. The following modifications of Keller's method is used by Gordin for the quantitative separation of strychnine and brucine:—A quantity of 0·2 grm. of the alkaloid is dissolved in 15 c.c. of 3 per cent. sulphuric acid, and, when cold, treated with 3 c.c. of a cold mixture of equal parts of nitric acid (1·42) and water. Brucine is decomposed, with the formation of oxidation products, which are not soluble in chloroform made alkaline with sodium hydroxide solution. Therefore the mixture is shaken in a separating funnel in the presence of excess of sodium hydroxide solution with successive quantities of chloroform (20 c.c., 10 c.c., and 10 c.c.). The chloroform extracts are evaporated, and the residue of strychnine dried at 110° C. until the weight is constant. A freshly-prepared mixture of 0·1002 grm. of strychnine nitrate and 0·1021 grm. of brucine nitrate gave, on analysis, 0·1004 grm.

of strychnine nitrate. Results of investigations of 6 different commercial samples of strychnine nitrate containing brucine are given. The advisability is discussed of calling the preparations which contain more than 50 per cent. of brucine nitrate, "brucine nitrate containing strychnine," since brucine is very inactive compared with strychnine. P. H. P.

Microcrystalline Reaction for Identifying Traces of Cantharidin. G. Denigès. (*Bull. Soc. Pharm. Bordeaux*, 1923, **61**, 63; *J. Pharm. Chim.*, 1924, vii, **29**, 106.)—As little as 0·1 or 0·05 mgrm. of cantharidin may be identified by adding one or two drops of chloroform to the material on a slide and allowing the solvent to evaporate; the cantharidin is left in tablets or prisms showing characteristic step-like incisions. The sublimed product also exhibits distinctive form. In either case, addition of a drop of benzene to the crystals renders these more distinct. Even 0·001 mgrm. of cantharidin may be detected if it is concentrated in as little solvent as possible before crystallisation. T. H. P.

Biochemical, Bacteriological, etc.

Preservation of Blood by means of Formaldehyde. J. C. Bock. (*J. Biol. Chem.*, 1924, **59**, 73–76.)—It does not seem advisable to use the average formaldehyde as a preservative for specimens of blood intended for blood sugar estimation by the revised Folin-Wu method (*J. Biol. Chem.*, 1920, **41**, 367) as recommended by Denis and Aldrich (*J. Biol. Chem.*, 1920, **44**, 203). They claim that formaldehyde in certain quantities will affect neither the alkaline copper tartrate solution used nor the tungstic acid filtrate. The author describes experiments in which he used six different samples of formaldehyde, and gives a table of results showing the effect of adding varying amounts of the different formaldehydes to glucose solution, tungstic acid filtrate and blood specimens before precipitation. Five out of six samples of the formaldehydes investigated reacted with the alkaline copper tartrate, thereby materially influencing the results. In several cases there was an increase in the amount of reduction, expressed in terms of glucose, of more than 40 per cent. Denis and Aldrich must have investigated an inadequate number of formaldehyde samples. P. H. P.

Enzyme Actions of Beef Tissues. K. G. Falk, H. M. Noyes and K. Sugiura. (*J. Biol. Chem.*, 1924, **59**, 213–223.)—The ester-hydrolysing actions of six beef tissues, brain, kidney, spleen, liver, heart-muscle, and lung were determined on ten purified esters, *viz.* methyl acetate, ethyl acetate, isobutyl acetate, phenyl acetate, benzyl acetate, glyceryl triacetate, methyl butyrate, ethyl butyrate, methyl benzoate and ethyl benzoate, and the protease actions on two protein preparations, peptone and casein (0·1 grm. of each of the latter in each test). The beef (steer) tissues were ground and extracted with water, and toluene was added and was present throughout the tests. The mixtures were kept in an ice-box overnight and filtered through paper the next day. Portions of 5 c.c. each of the filtrates were diluted with water to 15 c.c. and tested, and the residues from

the extraction were also tested. The degree of lipase actions was determined by titration with 0·1 *N* sodium hydroxide solution, with phenolphthalein as indicator. For the protease actions the formaldehyde method (with phenolphthalein and 0·1 *N* sodium hydroxide solution) was used, each test being made in duplicate. The temperature was maintained at 38° C. for 22 hours, and the solutions were all brought to an initial P_H value of 7·0. The results are presented in the form of tables for the absolute actions and curves for the relative actions. The characteristic relations for the various tissues are discussed and compared with those for the corresponding rat tissues. It is possible to plot the relative actions caused by each tissue on a number of different substrates so that a characteristic "picture" of the enzyme action is obtained, as can be done for rat tissues. The "pictures" for the actions of beef kidney, liver and lung are quite characteristic. The three other beef tissues show "pictures" more or less similar to the Flexner-Jobling rat carcinoma "picture." Explanations for similarities and differences will be advanced when data for other animal tissues have been gathered. P. H. P.

Evaluation of Saponins as Frothing Agents. L. Kofler. (*Chem. Zeit.*, 1924, **48**, 165–166.)—The determination of the toxicity of saponins by haemolysis action for deciding whether they should be used as frothing agents in foods is one-sided and often leads to false suppositions. Not many data concerning the frothing power are known. The author gives the following table:

Saponin.	Hæmolytic Index.	Frothing-Number	$\frac{\text{Poison}}{\text{Froth}}$-Quotient.
Guaiacum-Saponin, Merck.	660	28,500	0·023
Sapotoxin. Merck.	30,000	20,000	1·5
Sapindus-Saponin. Hoffmann La Roche	28,600	16,700	1·7
Aphrogen	2,000	800	2·5
Horse-chestnut-Saponin, Merck. ..	10,000	3,300	3·0
Saponin pur. albiss., Merck.	25,000	2,500	10·0

He finds that the haemolytic action of aphrogen towards red blood corpuscles and white mice is smaller than that of other saponins. This confirms the work of Mandelbaums. On the other hand, the frothing power is smaller than that of other commercial saponins, *e.g.* 25 times smaller than that of sapotoxin and 36 times smaller than that of guaiacum-saponin. The frothing number was determined according to the author's method (ANALYST, 1922, **47**, 403). Experimental conditions for a froth one cm. high were found. By comparing the haemolytic action with the frothing power, *i.e.* by dividing the haemolytic index by the frothing number, the (poison: froth) quotient is obtained which without further data gives the usefulness of the saponin as a frothing agent for foods. The smaller the haemolytic index and the greater the frothing number, the smaller is the poison: froth-quotient. By comparing the quotient of aphrogen with that of sapotoxin it is found that, on making lemonades with aphrogen and sapotoxin and adding so much of each saponin that both drinks have the same frothing power, the haemolytic action of the aphrogen-lemonade is about 67 per cent. greater than that of the sapotoxin-lemonade.

Details are given of subcutaneous injections on white mice. The fatal dose for a medium-sized mouse is 0·03 grm. of aphrogen, but 0·00075 grm. of sapotoxin; Therefore, judging by this, aphrogen is 40 times less poisonous than sapotoxin, but since, according to the table, the frothing power of aphrogen is 25 times less than that of sapotoxin, so, from injection experiments, the toxicity of a sapotoxin-lemonade is about 60 per cent. greater than that of an aphrogen-lemonade, when both froth equally. Saponins can only be evaluated as frothing agents by comparing the physiological action with the frothing number and obtaining the poison: froth quotient. P. H. P.

Effect of Storage of Livers on the Vitamin *A* Potency of Cod Liver Oil. A. D. Holmes. (*Ind. Eng. Chem.*, 1924, **16**, 295–297.)—Cod liver oils were rendered from fresh cod livers, from livers which had been stored in ice out of contact with air for six months, and from livers stored for one year under similar conditions. Albino rats suffering from vitamin *A* starvation were fed with the three classes of oil, and the results obtained showed that the oils from the stored livers were as efficient as the oil from the fresh livers. One mgrm. per day of each of the oils contained sufficient vitamin *A* to promote good growth in the rats.

W. P. S.

Amount of Available Insulin in the Pancreas of Domestic Animals. F. Fenger and R. S. Wilson. (*J. Biol. Chem.*, 1924, **59**, 83–90.)—This investigation was carried out for the purpose of obtaining some definite information regarding the amount of insulin present in the pancreas of cattle, hogs and sheep. The method of separating the active substance, a modification of that of Doisy, Somogyi and Shaffer (*J. Biol. Chem.*, 1923, **55**, 31) is described in detail; also the method of standardising the purified insulin. The preparations were tested on rabbits, and a table of results is given. The insulin unit chosen by the authors is the amount of insulin per kilo. of body weight necessary to produce convulsions and coma within 5 hours in from 60 to 70 per cent. of rabbits weighing between 1 and 2 kilos. Larger rabbits respond as promptly to their quota of insulin as do the smaller ones, and the convulsive dose of insulin is in direct proportion to ths body weight within these weight ranges. The authors find that the amount of insulin in the pancreas of domestic animals varies from 1,500 to 2,200 rabbit units per kilo. of fresh glands. The average yield approximates 1,800 units from cattle, hogs and sheep. The domestic animals slaughtered yearly in the United States under Federal Government inspection furnish about 5½ million kilos. of fresh pancreas glands, so that the available supply of raw material is far in excess of any possible demand for insulin. Insulin solution may be sterilised with impunity. Absolutely fresh glands, still warm if possible, are necessary for insulin production, and a grinding process, in addition to the mincing, breaks up the cell walls and renders the active substance available for extraction. An experiment described shows that the presence of acid is required to bring insulin into solution in either water or alcohol. The samples have shown no decrease in strength over a period of several months. There seems to be no doubt that

insulin is of a protein nature, probably a derived and not a native protein. The most active products at the authors' disposal possess the physical and chemical characteristics of complex protein derivatives not lower than the primary proteoses.

P. H. P.

Method of Measuring the Activity of Laccase. P. Fleury. (*Comptes rend.*, 1924, **178**, 814–816.)—A current of air is passed for a definite time through 10 c.c. of guaiacol solution mixed with the laccase to be tested, the solution being placed for the purpose in a special wash-bottle in a thermostat. The guaiaquinone thus formed is extracted by shaking the solution with 10 c.c. of chloroform, and is estimated colorimetrically. The results of a series of tests show that, within certain limits of time and of the concentration of the enzyme, laccase resembles diastase in obeying the law of proportionality between the amount of substrate transformed and both the time and the quantity of enzyme. Within such limits it is possible to obtain a measurement of the activity of a laccase preparation.

T. H. P.

Chemotherapeutic Experiments with Chaulmoogra and Allied Preparations. O. Schöbl. (*Philippine J. Sci.*, 1923, **23**, 533–542; 1924, **24**, 23–27).—I. *The Growth-Inhibiting Activity of Chaulmoogra Oil and its Derivatives toward Bacillus tuberculosis in vitro.*—This was tested by adding measured amounts of the oils to test tubes containing 10 c.c. of 5 per cent. glycerin agar, and it was found that chaulmoogra oil has considerable inhibiting power over *B. tuberculosis*, which is specific, *i.e.* it occurs in dilutions of the oil in which no inhibition of non-acid-fast bacilli can be discerned. Oils from plants related to *Taraktogenos kurzii* behave similarly. *Hydnocarpus Wightiana*, *H. alcalae*, *H. subfalcata*, and *H. venenata* (all containing optically active acids) are decreasingly active in the order given, whilst the oil from *Gynocardia odorata* (containing no optically active acids) is inactive. The sodium salts of chaulmoogra oil acids were found to vary in their activity, that prepared from isolated hydnocarpic acid approaching in activity the soap from the total fatty acids, whilst the sodium salt of chaulmoogric acid was far inferior in its growth-inhibiting effect.

II. *Comparison of the Antiseptic Power of Chaulmoogra Oil with that of other Vegetable and Animal Oils, rare and common.*—Although certain vegetable oils containing optically active fatty acids are found to inhibit the growth of acid-fast bacteria *in vitro*, they do not do so to such a high dilution as Chaulmoogra and Hydnocarpus oils, which contain the optically active fatty acids. A table is given showing the growth-inhibiting "titer" (*i.e.* the amount of oil which, when added to 10 c.c. of agar, still produced inhibition of growth) and growth-inhibiting values (*i.e.* the relative strength of the inhibiting effect) for 42 different oils. It may be noted that certain essential oils and oils containing volatile constituents show very high selective inhibitory action upon acid-fast bacilli. D. G. H.

Viability of Intestinal Pathogenic Bacteria in Fruits and Philippine Foods eaten Raw. A. Vasquez-Colet. (*Philippine J. Sci.*, 1924, **24**, 35–39.)—The foods used in the experiments were bagong (small shrimps pickled in salt),

patis (obtained from bagong by extraction and boiling), both sold in the market and eaten raw, vinegar (obtained by fermentation from the Nipa palm), mango, banana, chico, lanzones, apples, quesco, and guava. It was found that the cholera vibrio will survive from a few hours to at least 6 days, but is less long lived when mixed with human faeces; typhoid bacillus survives from a few minutes to at least 3 days; and dysentery bacillus (Flexner and Shiga types) from a few minutes to at least 5 days. The results show that such articles of food eaten raw may convey infection, but not to a marked extent, and in the case of foods rich in bacteria, such as patis, or in the fermenting state, survival of the cholera vibrio is very limited in duration. D. G. H.

On the Cancer-Producing Factor in Tar. E. L. Kennaway. (*British Med. J.*, 1924 [March 29], 564–567.)—A summary is given of the evidence, from both industrial and experimental sources, which indicates the presence or absence of cancer-producing power in the different fractions obtained from coal tar and in the pure substances isolated from it. Of the different kinds of tar, those which produce cancer are lignite tar, gasworks tar, some forms of producer-gas tar, and probably coke-oven tar, whilst blast furnace tar does not. The chief differences between blast-furnace tar and the ordinary cancer-producing gasworks tar are that the former contains greater quantities of paraffins and of phenols other than carbolic acid, but is deficient in some aromatic compounds found in the latter. The industrial evidence alone shows that the cancer-producing substance in gasworks tar is present in the fractions of higher boiling point, *viz.* creosote oil, anthracene oil (and hence in the green oil and crude anthracene) and pitch; the experimental evidence derived from mice supplements this by showing that the substance is not concentrated in the solids suspended in anthracene oil, and that it is present in the distillate of highest b.pt. obtainable from pitch. Hence it may distil through an interval of temperature extending roughly from 250° C. (the creosote fraction) to above 500° C. (the "pitch distillate"). So far, attempts to find the cancer-producing substance among the known constituents of coal tar have given wholly negative results. Anthracene, phenanthrene, chrysene, picene, retene, truxene, acenaphthene, fluorene, acridine, carbazole, aniline benzene, toluene and xylene have been excluded by experimental tests (several of which are described in detail); and either the experimental or industrial evidence, or both of these, also excludes naphthalene, the acids, bases and other nitrogenous compounds, paraffins, olefines and naphthenes. It now seems not unlikely that the cancer-producing substance is a compound, as yet unknown, which is unstable and present in amounts as small as those of the vitamins in foods; as in the case of some hormones, its identification may be long delayed, even when very concentrated preparations can be obtained.

A long list of references to the chemical and medical literature of the subject is given.

Toxicological and Forensic.

Influence of Blood on Chemical Reactions. G. Beccadelli. (*Arch. Farm. Sper.*, 1923, **36**, 137–144; *Chem. Abstracts*, 1924, **18**, 406).—The presence of blood has a pronounced influence on the reduction of ammoniacal silver nitrate solution by formaldehyde, the colour of the colloidal silver varying with the species of animal from which the blood is derived. For the identification of blood stains on fabrics, about 5 sq. mm. of the material are cut out and macerated for 24 hours in 1 c.c. of water at the ordinary temperature. About 0·5 c.c. of the extract is transferred to a small test tube and treated with 0·5 c.c. each of 38 to 40 per cent. formaldehyde, 0·75 per cent. silver nitrate solution and ammonia solution of sp. gr. 0·925. The tube is inverted two or three times and allowed to stand. In the case of human blood a characteristic amber yellow coloration develops within 16 hours if the stain was not recent, or a turbidity if it was recent. Failure to obtain this coloration may indicate that the stain was old human blood or the blood of a lower animal. The colorations produced by the blood of different animals are as follows:—Rabbit, amber yellow, not very pronounced; goat, amber yellow, still less pronounced; dog, brick red; sow, straw yellow; ox, brass yellow. The differences in colour are attributed to differences in the size of the colloidal silver particles.

Water Analysis.

Soluble Aluminium and the Haematoxylin Test in Filtered Waters. W. D. Hatfield. (*Ind. Eng. Chem.*, 1924, **16**, 233–234.)—In cases where water is treated with aluminium sulphate previous to filtration, the filtered water will, in all probability, contain traces of soluble aluminium if the P_H value of the water is less than 5·7 or more than 7·3, the minimum solubility of aluminium hydroxide in carbonate and bicarbonate solutions lying within these limits. The following method is proposed for the estimation of small quantities of aluminium (*e.g.* 0·1 part per million of water):—It consists in the formation of the haematoxylin-aluminium colour compound in a portion of the water which has been adjusted to P_H 8·2 to 8·3; the mixture is then acidified to P_H 4·5, in order to destroy the lavender coloration given by iron and other metals which may be present. Fifty c.c. of the water to be tested are treated in a Nessler cylinder with 1 c.c. of saturated ammonium carbonate solution (this adjusts the P_H value to 8·2) and 1 c.c. of 0·1 per cent. haematoxylin solution; after fifteen minutes, the mixture is acidified with 1 c.c. of 30 per cent. acetic acid, and the coloration obtained is compared with those given by known amounts of aluminium under the same conditions. If the quantity of aluminium present is more than 0·15 part per million, the colorations are best compared by observation through the sides of the cylinders.

W. P. S.

Organic Analysis.

Estimation of Ketonic Compounds and of β-Hydroxybutyric Acid in Diabetic Urine. H. Bierry and L. Moquet. (*Comptes rend.*, 1924, **178**, 816–819.)—The processes of van Slyke and Hubbard may be applied to the estimation in urine of: (1) acetone present as such, together with that formed from acetylacetic acid during distillation, and (2) β-hydroxybutyric acid, which yields acetone when oxidised by means of sulphuric acid and dichromate. The apparatus used consists of a round-bottomed flask connected by sealed joints with a superposed tapped funnel and a vertical tube widened out like a pipette and bending over to connect with a condenser; this makes a tight joint at the bottom with a thistle funnel, by which the condensate is collected.

To defecate the urine, 25 c.c. are mixed with 100 c.c. of water in a 250 c.c. measuring flask and treated with 50 c.c. of 20 per cent. copper sulphate solution and then, gradually, with 10 per cent. milk of lime until the reaction is distinctly alkaline, this being indicated by the appearance of a blue colour; when filtered, the liquid should not reduce Fehling's solution. Twenty-five c.c. of the filtrate and a little pumice are placed in the flask, into which first 130 c.c. of distilled water and then 20 c.c. of dilute sulphuric acid (1:4 by vol.) are introduced through the funnel. Distillation is continued for 10 to 12 minutes, and 45 to 50 c.c. of distillate (*A*) are collected.

To the residual boiling liquid a mixture of 22 c.c. of sulphuric acid (1:1 by vol.) and 26 c.c. of a 0·15 per cent. solution of potassium dichromate (twice crystallised and fused before use) is added by way of the funnel. At intervals of 10 minutes during the distillation, three further quantities of 50 c.c. of the dichromate mixture are added, the distillation then continued for 15 minutes after the last addition, and 200 to 220 c.c. of distillate (*B*) collected.

The two distillates, which are collected with the precautions necessary to prevent loss of acetone, are mixed with the wash waters and redistilled, separately, in presence of 0·5 grm. of sodium peroxide, and the acetone is estimated iodimetrically in the resulting two distillates. In the case of *B*, the result is increased by 16 per cent. to compensate for loss of acetone under the conditions employed. One c.c. of 0·1 *N* iodine solution corresponds with 0·968 mgrm. of acetone or with 2·069 mgrms. of β-hydroxybutyric acid. T. H. P.

Sulpho-chromic Oxidation of Organic Substances and Coals in presence of Catalysts. Rôle of Mercury. D. Florentin. (*Bull. Soc. Chim.*, 1924, **35**, 228–230.)—When heated with chromic and sulphuric acids in presence of a small quantity of mercury, all organic compounds, including coals, are completely oxidised, the sole gaseous product being mostly carbon dioxide; coke yields, under such conditions, also carbon monoxide, but in extremely small proportion. (*Cf.* ANALYST, 1922, 405, 530; 1923, **37**, 398.) T. H. P.

Oxidation of Coal by Silver Dichromate and Sulphuric Acid. L. J. Simon. (*Comptes rend.*, 1924, **178**, 775–777.)—The results of investigations on the oxidation of coal indicate that coal consists of two components in intimate association. The one, which begins to gasify at a relatively low temperature, behaves towards chromic oxidising agents in the same way as aromatic hydrocarbons, gasification proceeding to completion when silver dichromate and sulphuric acid are used, but being only partial when chromic acid is used. The other constituent shows the behaviour of coke, gasification occurring only at about 100° C. and being incomplete under all the conditions investigated.

T. H. P.

Estimation of Glycerol in Crude Glycerins. Fachini and Somazzi. (*Bull. Ind. Olie e Grassi*, October, 1923; *Chem. Trade J.*, 1923, **73**, 702–703.)—The method originally applied to the estimation of glycerin in distilled glycerins (*Chem. Trade J.*, 1923, **73**, 127), which depended on the measurement of the amount of carbon dioxide produced by oxidation of dichromate, is now worked out for crude glycerins, which must be subjected to a preliminary purification. The most satisfactory preliminary treatment was found to be with basic lead acetate solution prepared by boiling a 10 per cent. solution of the salt with excess of litharge for 1 hour, and filtering under conditions eliminating contamination with carbon dioxide. The lead acetate solution eliminates proteins by coagulation, organic acids as insoluble salts, and alkaline carbonates as insoluble carbonates; and, on cooling to 0° C. for one hour, practically all chlorides as insoluble lead chloride. (Final traces of chlorides can be removed, if necessary, with a slight excess of silver carbonate solution.)

About 20 grms. of lye or 2 grms. of crude glycerin are weighed into a 250 c.c. flask, water added to a volume of 50 c.c., and then 5 c.c. of the basic lead acetate solution. After shaking and leaving for 30 minutes, the further addition of a small quantity of lead acetate solution should not produce any further precipitate, and, if a precipitate does form, the estimation must be begun again with a smaller quantity of sample. The contents of the flask are made up to 250 c.c. with water, and cooled in ice for at least one hour. After filtering, and when a temperature of 15° C. has again been reached, 25 c.c. are transferred to the oxidation flask, and the analysis continued as for pure glycerin, glycerol, and trimethylene-glycol being separately estimated. It has been found that glycerins of animal origin consist almost entirely of glycerol, whilst vegetable glycerins contain varying amounts of trimethylene glycol, and the method should prove useful in determining the origin of glycerins.

D. G. H.

Composition of Linseed Oil. A. Eibner and K. Schmidinger. (*Chem. Umschau.*, 1923, **30**, 293–302.)—A sample of linseed oil of Dutch origin was examined by modifications of the bromination method devised by Hehner and Mitchell (ANALYST, 1898, **23**, 310), and was found to contain the following constituents:—α-Linolenic acid, 21·1; an isomeric linolenic acid, 2·7; α-linolic acid, 17·0; oxy-acids, 0·5; saturated fatty acids, 8·2; glyceryl radical, 4·1; phytosterol,

1·0; unestimated (by difference), 46·2 per cent. Proof of the presence of an isomeric linolenic acid was obtained by brominating an ethereal solution of the oil, separating the insoluble bromide, removing the bromine from the filtrate, evaporating the ether by means of the pump, and treating the residue with petroleum spirit. The chilled solution yielded a deposit of the bromoglyceride of α-linolic acid, which was filtered off, and the filtrate was then debrominated, the esters saponified, and the fatty acids liberated dissolved in ether and again brominated. They now yielded 2·7 per cent. (calculated on the original oil) of a linolenic hexabromide (m.pt., 180° C.) bromine 63·03 per cent.

About 25 per cent. of the bromide of a mixed glyceride was obtained in crystalline form by dissolving the crude bromide (m.pt. 139° to 140° C.) in hot tetralin (tetrahydro-naphthalene, $C_{10}H_{12}$), adding boiling acetone until there were signs of precipitation, and then cooling the solution. The crystalline bromoglyceride melted at 155° to 156°, and contained: Br, 59·39; C, 32·1; and H, 4·6 per cent. This corresponded with the formula, $C_{57}H_{94}O_6Br_{16}$, and it was concluded that the compound was α-dilinolenic-α-linolic bromoglyceride. The other bromoglycerides present were not identified, but about 0·6 per cent. of a mixed glyceride was separated by treating the original linseed oil with nitrous acid; it was identified as di-elaido-palmitin. (*Cf.* Toms, ANALYST, 1924, 77.)

Oxidation of Lumbang and Linseed Oils and of the Principal Compounds in Lumbang Oil. A. P. West and A. I. de Leon. (*Philippine J. Sc.*, 1924, **24**, 123–142.)—Lumbang oil (obtained from *Aleurites moluccana*) had the following composition:—*Unsaturated glycerides*: linolenic, 6·5; linolic, 33·5; oleic, 57·0; *saturated glycerides*, 2·8 per cent. The oil was progressively oxidised by passing measured quantities of dried air through a small wash bottle containing the oil at a temperature of 75° C. and passing the excess of air through various absorption tubes. The operation was stopped at intervals for weighings to be made, and for comparative purposes linseed oil was similarly treated. At first (up to 30 hours) the percentage of apparent oxygen absorption, volatile products evolved and total oxygen absorption were found to be greater for lumbang oil than for linseed oil, but as oxidation continued the position was equalised (30 to 40 hours) and finally reversed. It is suggested that, on oxidation, autocatalysts are formed which tend to accelerate the velocity of oxygen absorption, whilst the saturated glycerides retard it. Since lumbang oil contains a smaller proportion of saturated glycerides than does linseed oil, the velocity of absorption is at first greater. Possibly in the case of linseed oil, after 30 hours a sufficient quantity of autocatalysts accumulates to overcome the retarding influence of the saturated glycerides and to exert a marked autocatalytic influence on the absorption velocity. The oxidised and unoxidised acids formed after blowing for definite periods at 75° C. were separated by Fahrion's method and found to be in the following proportions, oxidised acids being given first in each case:—Twenty hours (400 litres of air used), 7·13, 88·52; 40 hours (781 litres of air), 17·46, 79·07; 60 hours (1276 litres of air), 26·86, 68·66; 80 hours (1652 litres of air), 32·24, 62·12 per cent.

Constants of samples of lumbang and linseed oils blown for various periods of time at 75° C. were determined, and very similar results obtained. The experimental data obtained agree with the supposition that the absorption of oxygen takes place at the double bonds. Linolenic glyceride was oxidised more readily than linolic, and linolic more readily than oleic glyceride. D. G. H.

Chemical Factors Determining the Quality of Tobacco. V. Graham and R. H. Carr. (*J. Amer. Chem. Soc.*, 1924, **46**, 695–702.)—Although the change in the composition of tobacco produced by fertilisers is considerable, it is much less than that produced by different soils and different climates. For the comparative examination of tobacco the extract yielded to solvents affords most information. Six grms. of the samples were extracted with 60 c.c. of petroleum spirit, then with ether and then with alcohol, the nicotine being estimated in each extract by the official (Kissling) method. Cigar tobaccos have a small percentage extract and nicotine content, whilst smoking tobaccos have a high solubility in these three solvents. When the calcium content of the ash is high much of the nicotine is combined in a stable form, and so is insoluble in ether and other solvents; when a sample of powdered tobacco was mixed with calcium salts it was found that its nicotine became so fixed as to be extracted with difficulty, even by the official method. Acid phosphate fertilisers improve the aroma of the plants; much of the irritating effect of certain kinds of tobacco is due to loosely combined nicotine and volatile oils, both of which are easily soluble in petroleum spirit.

H. E. C.

Inorganic Analysis.

Potassium Permanganate as an Iodimetric Standard. J. M. Hendel. (*Zeitsch. anal. Chem.*, 1923, **63**, 321–324.)—The standardisation of thiosulphate solutions against permanganate gave results agreeing within 0·1 per cent. with those obtained by means of dichromate or iodine. About 35 c.c. of standard permanganate solution are run into a glass-stoppered flask containing 50 c.c. of 0·7 *N* hydrochloric or sulphuric acid, and 3 grms. of potassium iodide in 10 c.c. of water. (The iodide solution has been treated with aluminium amalgam to reduce any iodate). The stoppered flask is left in the dark for 2 to 5 minutes; the solution is diluted to 450 c.c. and titrated with thiosulphate in presence of starch. Atmospheric oxidation of the iodide during the titration is counteracted by the great dilution. In standardising against dichromate, 10 c.c. of the iodide solution are added to 200 c.c. of a solution containing about 0·13 grm. of potassium dichromate, and hydrochloric acid at 0·3 *N* concentration; the stoppered flask is left 10 minutes in the dark, and the solution, diluted to 800 c.c., is titrated as before.

W. R. S.

Estimation of Copper as Iodide. L. W. Winkler. (*Zeitsch. anal. Chem.*, 1923, **63**, 324–330.)—The neutral or weakly acid solution (100 c.c.) containing 0·1 to 0·01 grm. of copper is treated with one grm. of ammonium chloride and heated to boiling. The beaker is removed from the heat, and a 10 per cent.

solution of one grm. of potassium or sodium iodide added, followed by 10 per cent. sodium sulphite solution, drop by drop, until the precipitate is pure white; an excess of 5 drops is finally added. The liquid is again boiled for 1 to 2 minutes, and kept overnight in a dark place. The precipitate of cuprous iodide is filtered off, washed with 50 c.c. of cold water, dried for 2 hours at 130° C., and weighed. The following amounts are added to the weight, to allow for the solubility of the precipitate: 0·0013 for 0·4, 0·0011 for 0·2, 0·0010 for 0·1, and 0·0008 grm. for 0·01 grm. of cuprous iodide found. The method separates copper from magnesium, zinc, manganese, cobalt, and nickel. Lead interferes and must be removed as sulphate, and cadmium if present in very large amounts gives slightly low results. The process is recommended for the examination of blue vitriol, the commonest impurity in which is ferrous sulphate. An aliquot part of the solution equivalent to 0·5 grm. of the salt is treated as described above; the only modification consists in adding the slight excess of sodium sulphite and hydrochloric acid first, to be followed by the precipitant. W. R. S.

Schönbein's Reaction applied to the Micro-detection of Copper. H. and R. Imbert and P. Pilgrain. (*Bull. Soc. Chim.*, 1924, **35**, 60–64.)—When 3 or 4 drops of freshly prepared tincture of guaiacum resin and then, through a drawn-out tube, a few drops of 0·15 per cent. potassium cyanide solution are added to 5 to 10 c.c. of a neutral cupric solution, a distinct blue coloration is obtained even when the concentration of the copper is only 1 part in 2,000,000 of solution. Small quantities of copper may be separated from solution also by electrolysis, by means of platinum electrodes, in presence of nitric and sulphuric acids; with 10 c.c. of solution containing 0·001 grm. of copper per litre the copper deposited on the cathode after 45 to 50 minutes gives a slight reduction and blue coloration with molybdic acid reagent. These methods have been applied, with good results, to the detection and estimation of copper in distilled water, beans coloured with copper salts, and blood. T. H. P.

Estimation of Cadmium in Spelter and Zinc Ores. C. E. Barrs. (*J. Soc. Chem. Ind.*, 1924, **43**, 77–78 T.)—Ten grms. of spelter are dissolved in 30 c.c. of nitric acid and 100 c.c. of water, diluted to 250 c.c. and ammonia added until the precipitate of zinc hydroxide re-dissolves. The insoluble matter is filtered off and washed with 5 per cent. solution of ammonia; if it is bulky it is dissolved and re-precipitated. The filtrate is heated, an excess of 50 per cent. solution of sodium sulphide added, and the mixture, after being kept hot for an hour, is filtered; the unwashed precipitate is returned to the beaker and dissolved and the filter paper washed with nitric acid; then sulphuric acid is added and the solution evaporated until while fumes appear, after which it is cooled, diluted, and filtered. The insoluble matter is washed with dilute sulphuric acid, sufficient of which is added to make 20 per cent. in the filtrate, which is then warmed while hydrogen sulphide is passed through it to precipitate most of the copper. The precipitate is removed, washed with dilute sulphuric acid, and the filtrate diluted to at least 250 c.c. and again saturated with hydrogen sulphide to precipitate the

cadmium. The cadmium sulphide is dissolved in a silica crucible with nitric acid, sulphated and weighed; then it is dissolved in dilute nitric acid, and any copper present is titrated by the iodide method, calculated as $CuSO_4$ and subtracted from the weight of the cadmium sulphate. Ores are dissolved in nitro-hydrochloric acid, the solution evaporated to dryness, taken up in hydrochloric acid, and the silica removed, then neutralised with ammonia, acidified with 1 c.c. of hydrochloric acid for each 100 c.c. of liquid, and a current of hydrogen sulphide is passed in. The precipitate is washed with dilute hydrochloric acid saturated with hydrogen sulphide, dissolved in nitric acid, ammonia and sodium sulphide are added and the analysis completed as above. H. E. C.

Separation and Estimation of Bismuth by Hydrolysis. G. Luff. (*Zeitsch. anal. Chem.*, 1923, **63**, 330–348.)—The nitrate solution (about 80 c.c.), free from ammonium nitrate, is neutralised with solid sodium bicarbonate until methyl orange just turns yellow. The liquid is acidified with a drop, and treated with 20 c.c., of *N* nitric acid. If the precipitate formed in the neutralisation does not re-dissolve completely, a little more acid is added. The clear liquid is diluted to 100 c.c. and boiled under reflux for one hour; 200 c.c. of water containing 0·5 c.c. less *N* sodium carbonate solution than the volume of *N* acid previously added is run in, drop by drop, through the condenser during the boiling. The cold liquid is filtered through a Gooch crucible; the precipitate of basic nitrate is washed with cold water, dried, and gently ignited to oxide. The process affords a good separation from lead, copper, and cadmium. W. R. S.

Separation of Zinc from Iron and Aluminium. E. G. R. Ardagh and G. R. Bongard. (*Ind. Eng. Chem.*, 1924, **16**, 297–299.)—Zinc may be separated from iron by means of ammonia and ammonium chloride in one precipitation when the following procedure is adopted. The solution, which may contain as much as 0·2 grm. of zinc and 0·4 grm. of iron, is treated with a few drops of hydrochloric acid and evaporated to a volume of about 5 c.c.; while this liquid residue is still warm 5 grms. of ammonium chloride are stirred into it and 10 c.c. of concentrated ammonia are added. The mixture is then diluted with 25 c.c. of water, the precipitate collected on a filter and washed with about 100 c.c. of ammoniacal ammonium chloride solution (5 grms. of ammonium chloride and 5 c.c. of concentrated ammonia per 100 c.c.) The filtrate is acidified with hydrochloric acid, diluted to 250 c.c., and the zinc titrated with standardised potassium ferrocyanide solution (22 grms. of ferrocyanide and 10 grms. of sodium thiosulphate per litre); uranium nitrate solution is used as indicator. The same method may be employed for the separation of zinc from aluminium, but the aluminium hydroxide requires rather more washing than does the ferric hydroxide. W. P. S.

Analysis of Zinc Ores. Use of Powdered Magnesium for the Removal of Copper and Lead. E. G. R. Ardagh and G. R. Bongard. (*Ind. Eng. Chem.*, 1924, **16**, 300–301.)—The use of powdered magnesium is recommended for the precipitation of copper and lead in hydrochloric acid solution; to ensure

complete separation of zinc which may be present, the precipitated metals should be boiled with hydrochloric acid and re-precipitated with magnesium. The zinc in the filtrate from the copper-lead precipitate may be titrated directly with potassium ferrocyanide solution, since even large quantities of magnesium chloride do not interfere with the titration. If aluminium is used for the precipitation of the copper and lead it is necessary to remove the aluminium salts before the zinc is titrated. If the zinc solution contains not less than 7 c.c. of concentrated hydrochloric acid per 250 c.c. the presence of lead does not affect the titration of the zinc with ferrocyanide solution. W. P. S.

New Volumetric Method for Estimating Ammonium Salts. M. V. Auger. (*Comptes rend.*, 1924, **178**, 1081.)—The brown colour in the Nessler test does not appear till sufficient caustic alkali has been added to saturate the whole of the anion combined with the ammonium radicle. The author has applied this principle to the volumetric estimation of ammonium salts. The reagent is prepared by pouring on to 5 grms. of potassium iodide a saturated solution of mercuric chloride till the permanent precipitate forms. The whole is diluted to 100 c.c. and concentrated potassium iodide solution carefully added, drop by drop, till the precipitate disappears. A table is given showing the best quantity of this reagent to add to the solution of the ammonium compound; after the addition the mixture is titrated with sodium hydroxide solution till a permanent brown colour is produced. The titration has a sharp end-point, and is equally applicable to nitrate, sulphate or chloride. R. F. I.

Simultaneous Estimation of Two Halogens. H. Jahn. (*Chem. Zeit.*, 1924, **48**, 150.)—The neutral solution of, *e.g.* chloride and bromide should occupy as small a bulk as possible; free acid is neutralised carefully with dilute ammonia. After addition of a few drops of chromate solution the liquid is titrated with 0·1 *N* silver solution, and the volume added is noted. An excess of silver solution and 5 c.c. of dilute nitric acid are then added; the solution is boiled, left to settle in the dark, and filtered through a Gooch crucible. The precipitate is washed with 3 per cent. nitric acid, dried at 120° C. in the dark, and weighed. The quantity of silver bromide in the mixed precipitate is given by the expression $4{\cdot}2234\ p - 5{\cdot}6116\ q$, in which p = weight of precipitate, and q = weight of silver used in the titration. W. R. S.

Reaction of Sulphur with Alkali and Alkaline-earth Hydroxides. H. V. Tartar and C. Z. Draves. (*J. Amer. Chem. Soc.*, 1924, **46**, 574–581.)—When sulphur dissolves in aqueous solutions of sodium, potassium or barium hydroxide at the boiling point, polysulphides are first formed; on prolonged boiling these are partly reduced to di-sulphides and possibly mono-sulphides. The complexity of the polysulphide depends on the concentration of the reactants, and on the time and temperature. For the estimation of the mono-sulphide equivalent of the polysulphide in the presence of free alkali the A.O.A.C. method of iodine titration is unsatisfactory, but the following gives good results. The

solution to be analysed is added, with constant stirring, to a mixture containing 50 c.c. of a buffer solution containing 8 grms. of borax and 5·5 grms. of boric acid per 100 c.c. and an excess of 0·1 *N* iodine solution over the mono-sulphide. Further 0·1 *N* iodine solution is added until all thiosulphhate has reacted and an iodine colour is obtained. From the total iodine is subtracted that equivalent to the thiosulphate; this is estimated in the filtrate, after precipitation of sulphides by by ammoniacal zinc solution, in the usual way. H. E. C.

Estimation of Water of Hydration, Water of Constitution, and Carbon Dioxide in Hydraulic Limes. J. A. Muller and E. Peytral. (*Bull. Soc. Chim.*, 1924, **35**, 220–225.)—The loss in weight of a hydraulic lime on calcination corresponds with the water of hydration and constitution, the carbon dioxide of the calcium carbonate, and any other carbon compounds present. It is determined by heating about 1 grm. of the material in a lightly covered platinum crucible gradually to bright redness in about 30 minutes, and then in a blowpipe flame at the highest temperature attainable for about 5 minutes; no further loss should occur when the residue is again heated for 5 minutes in the blowpipe flame.

The water of constitution is composed of the water existing as acidic hydrogen or basic hydroxyl in the hydrosilicates and aluminates which, after the carbonates, the quartz sand, and the silicates unattackable by hydrochloric acid, form the principal components of hydraulic limes. The water of hydration is estimated by heating about 1 grm. of the lime in a platinum crucible at 185–190° C. to constant weight. The water of hydration and the water of constitution together are estimated by heating 4 to 5 grms. of the lime gradually to redness in a quartz glass test-tube about 12 mm. in diameter and 12 cm. in length. The tube, slightly inclined to the horizontal, is connected with a tared tube charged with pumice; its upper part is gently warmed to prevent condensation of the water vapour. After the lime has been heated for 15 minutes, the whole of the water of hydration and constitution is eliminated; the apparatus is then rapidly evacuated and disconnected from the pump, the pumice tube being detached and, when cold, weighed. The increase in weight represents the water of constitution and hydration. Subtraction of this increase from the loss on calcination gives the carbon dioxide, the result thus obtained agreeing closely with that obtained by decomposing the carbonates present in the lime with hydrochloric acid and measuring the carbon dioxide liberated. T. H. P.

Analysis of Gypsum and Gypsum Products. F. C. Welch. (*Ind. Eng. Chem.*, 1924, **16**, 238–241.)—The quantities of raw gypsum ($CaSO_4.2H_2O$), calcined gypum ($CaSO_4.\frac{1}{2}H_2O$) and anhydrite ($CaSO_4$) in a mixture containing any or all of these substances may be estimated as follows:—The sample is ground to pass a No. 48 sieve and the lime (CaO) and sulphuric acid (SO_3) are estimated gravimetrically. Hygroscopic moisture is then estimated by drying a portion of the sample in a current of air having a vapour pressure slightly greater than the dissociation pressure of gypsum at the temperature of the experiment. For instance, at 25° C., air of the required humidity may be obtained by passing it through

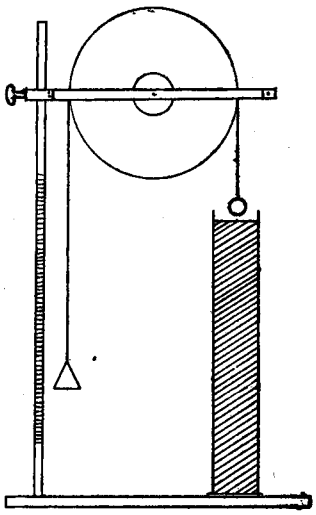
sulphuric acid having a sp. gr. of 1·37 at 23° C. The drying is then completed by heating the sample in a U-tube at 200° C. while a current of dry air is passed through the tube and the water given off is absorbed in concentrated sulphuric acid and weighed. Let this quantity be termed W. The sample is cooled to ordinary temperature and a current of air passed over 25 N sulphuric acid is conducted through the U-tube until a constant weight is obtained. Let the quantity of water thus absorbed by the sample be termed w. If $W = w$, the water lost and gained was all present in calcined gypsum; when W is greater than w, the sample contained both gypsum and calcined gypsum, the amount of gypsum present being 172/27 $(W - w)$, and that of calcined gypsum 145/27 $(4w - W)$. If W is less than w, the sample contained both calcined gypsum and soluble anhydrite; in this case the amount of calcined gypsum is 145/9 (W) and that of soluble anhydrite 136/9 $(w - W)$. W. P. S.

Physical Methods, Apparatus, etc.

Durability Tests of Glass. W. E. S. Turner. (*Pharm. J.*, 1924, **58**, 264–266.)—The action of boiling water on glass discs (average weight of each disc 80 grms.) of varying composition shows that loss in weight of the discs diminishes with the increase of lime present in the glass. Thus, starting with a sodium silicate base of molecular composition $6SiO_2 2Na_2O$, and progressively substituting calcium oxide for sodium oxide, a glass containing, for example, 0·21 per cent. of calcium oxide lost 24000·0 mgrms. per 100 square cm. on boiling in water for 6 hours, whilst with 6·26 per cent. of calcium oxide the loss was 2·5 mgrms., and with 11·68, 0·53 mgrms. Alkaline solutions have a very much more corrosive action on glass than acid. A satisfactory method for testing the suitability of reagent bottles is as follows:—The bottle is first thoroughly cleaned with weak acetic acid and distilled water, and dried. It is then suspended in a boiling water bath and a solution of narcotine hydrochloride (1 part of the salt dissolved in 1000 parts of distilled water in a platinum or good quality resistant glass vessel) added, the mouth plugged with cotton wool, and heating continued. Any alkali dissolved from the glass by the boiling water readily acts on the narcotine hydrochloride, causing precipitation of narcotine in the form of fine needles. With really good bottles an hour may elapse before precipitation begins, and the bottles may usually be regarded as satisfactory if no precipitate forms in 15 minutes. In the case of bottle glasses satisfactory durability is shown when not more than 18 per cent. of sodium oxide is present and less than about 8 per cent. of calcium oxide, but, generally speaking, it is simpler to insist that the glass comply with the durability test rather than to demand a specific composition, for some compositions outside these limits have been found satisfactory. Results of tests on eleven glasses of given composition are detailed. D. G. H.

A Simple Viscometer for Solutions of Resins, etc. K. Albert. (*Chem. Zeit.*, 1924, **38**, 181.)—Defects are pointed out in existing viscometers, especially in cases where the viscosity of unusually thick and dark liquids is required. The

instrument described is on the principle of the falling sphere apparatus. A brass ball of 28·4 mm. diameter and weighing 103·06 grms. hangs over a wheel by means of a very thin thread of gut, at the other end of which is a balance-pan weighing 30·00 grms. The wheel, which has a diameter of 255 mm., is held in a fork which is clamped to a retort stand on which is fixed a cm. scale with two marks. A cylinder containing the liquid to be tested and of a diameter at least three times that of the ball is placed under the ball. To make the determination, suitable weights are placed on the balance-pan depending on the viscosity of the liquid. The two marks on the scale are arranged in such a way that the balance-pan passes the first mark only when the ball has fallen some cm. in the liquid. The time required for the pan to pass between the two marks is measured by means of a stop-watch, and the result expressed in cm. per second. The variation in several readings on castor oil and mixtures of this and resin-oil was not over 7 per cent. A castor oil with an absolute viscosity of 8·16 in Engler's viscometer at 20° C. gave a reading of 7·5 cm./sec. in this test. With this apparatus determinations are quickly made, and the parts can be readily cleaned, since the brass ball and gut only are affected.



R. F. I.

Spectrophotometric Determination of P_H Value and the Apparent Dissociation Constants of Indicators. W. C. Holmes. (*J. Amer. Chem. Soc.*, 1924, **46**, 627–631.)—The estimation of the P_H value of solutions is readily effected by the spectrophotometer by an empirical calibration of two ratios R_1 and R_2 with suitable indicators against known hydrogen electrode values. From the dissociation curves so obtained it is possible to calculate the dissociation constants. With an indicator having only one coloured form the ratio R_1 (which is the percentage colour transformation) is determined by measuring the relative absorption at the desired point and that under conditions of the maximum colour intensity. With two-colour indicators the ratio R_2 is measured; this is the intensity of absorption at two wave-lengths situated near the maxima of the two bands in question. Thus, for example, phenolphthalein has absorption bands at 460$\mu\mu$ and 560$\mu\mu$; the intensity at these wave lengths is measured and their ratio is plotted against known P_H values as determined by the hydrogen electrode. The P_H value of any solution can then be quickly found by measuring the ratio of the absorption at these wave lengths and referring to the curve. H. E. C.

Reviews.

A COMPREHENSIVE TREATISE ON INORGANIC AND THEORETICAL CHEMISTRY. Vol. IV. By J. W. MELLOR, D.Sc. Pp. 1074. LONDON: Longmans & Co. 1923. Price 3 guineas.

In less than two years we are in possession of the fourth volume of this invaluable treatise. The rapidity with which one volume follows another is astounding, and is all the more so when the excellence of the volumes is considered.

The present volume completes the study of the elements of the second group of the periodic classification. The first three chapters—203 pages—are devoted to the structure of matter, radium and radioactivity, and the architecture of the atom. The remaining chapters, extending over 846 pages, deal with the metals of Group IIB. With the exception of zinc and cadmium, the metals receive separate consideration.

The treatment accorded to the theories of the structure of the atom is exceedingly refreshing, especially in these days when so many speculations are rife. It is concise and the various hypotheses have been scrutinised in a rigidly logical manner. One is impressed by the enthusiasm with which Dr. Mellor discusses this subject, and this, no doubt, accounts for the large number of quotations which he has employed. We are reminded that the electronic theories of matter, unlike the older theories, are based on scientific evidence, although, as yet, that evidence scarcely can be regarded as anything other than circumstantial. Before proceeding to the general study of the theories, those subjects which give rise to this circumstantial evidence, namely, the physical properties of matter and radioactivity, are discussed. This section of the volume is concluded with a very apt paragraph, entitled "The Honesty of Science." We are told that although these "speculations probably make the best guess yet made about the ultimate constitution of matter," we are still "confronted with phantasmæ which would be banished at once if we were convinced that they were sterile conjectures and not pregnant hypotheses." In the reviewer's opinion we have in this volume the best general discussion of atomic theories so far written.

The remaining chapters of the volume deal with metals. Mention is made of the controversy which has gone on for a century over the name which should be assigned to the element beryllium. The author regards it as being "largely a question of temperament." This may, perhaps, explain why in papers which the reviewer submitted to two different societies the name was changed by their editors from beryllium to glucinum. Vauquelin, who discovered beryllia, did not assign any particular name, but referred to it as "*la terre du béril.*" He stated that its salts were sweet, and it was on this statement that the editor based the name "*la glucine.*" In reality, the salts are more tart than sweet, due to the

hydrolysed acid, and this property can hardly be held as being peculiar to beryllium compounds. In the text of this volume the term beryllium is used, except in headings, when both names are given.

Dr. Mellor has the tendency to include, in the text, matter which seems to be contrary to general chemical principles. The mention of such substances as zinc cupric oxychloride, the formula of which is given as ($ZnCl_2$.ZnO.2CuO) ($ZnCl_2$.3CuO).6H_2O, and zinc dodecamminooxychloride, 2$ZnCl_2$.ZnO.12NH_3.4H_2O, might well have been omitted (p. 546). The references should, of course, be inserted in the appropriate lists, and they merit only the briefest note, if any. It must be stated, however, that the author points out that there is little to show that these formulæ represent distinct chemical compounds, and that they require systematic study in the light of the phase rule before their individuality as compounds can be established. The hypothetical A, B and C forms of both beryllium and zinc hydroxides are dealt with without comment, although it is extremely dubious whether they really do exist. It is incorrect to say that Bleyer and Kaufmann, as is stated on page 225, obtained analytical data corresponding with the so-called acid hydroxide $H_2Be_2O_3$. Again, some criticism is surely wanting on the remarkable claim of Levi-Malvano that beryllium sulphate hexahydrate always crystallises from those solutions which were made from it, instead of the ordinary stable tetrahydrate (p. 237). Is it possible that at any given temperature the solution of one hydrate is fundamentally different from that prepared from another hydrate, such that the hydrate which separates is determined by the one which was used originally in making the solution, even though that hydrate may not be the stable solid phrase at that temperature ?

A few omissions appear to have been made from the extensive lists given. The reviewer failed to find references to six papers relating to metals discussed.

Apart from these minor points, the volume is well up to the standard of the previous volumes, and will be an indispensable asset to chemists and research students. HUBERT T S. BRITTON.

CONFÉRENCES DE CHIMIE MINÉRALE. MÉTAUX. By M. GUICHARD, Professeur à la Sorbonne. Third Edition. Pp. xxix.+355. Paris: Gauthier-Villars et Cie. 1924.

This text-book contains the author's course of lectures on metals delivered in the University of Paris to first-year students. It is not intended as a work of reference, as it contains a rather bald outline of the chemistry of the metals, only the most important compounds (oxides, hydroxides, and salts) being briefly described. On the other hand, much importance is attached to the application of the phase rule to salt solutions and metallic alloys, and numerous equilibrium diagrams are reproduced. The author has not much use for structural formulæ, and is in favour of using various modes of notation, such as CrO^3H^2O, CrO^4H^2, and $CrO^2(OH)^2$ for chromic acid; but the book seems to indicate a distinct partiality towards the old dualistic notation (*e.g.* AzO^3HAzH^3 for ammonium

nitrate). A good many confusing formulæ appear in which two systems of notation are used without apparent reason, such as

$SO^4H^2.2AzH^3(SO^4)^3Al^2.24H^2O$ (p. 70), and $CO^2UO^3CO^3Na^2$ (p. 227).

In the nomenclature of ammonium salts, the author is still keeping alive the antiquated designation "chlorhydrate d'ammoniaque." The reviewer remembers using Wurtz's *Traité* nearly thirty years ago while at school, and having to insert the term "chlorure d'ammonium" wherever the text-book lapsed into the old nomenclature. Contrary to Werner's proposals, the metallic ammines are termed "amines." The French chemical symbols are gradually drifting away from the international ones. "Az" for nitrogen is an old-established symbol; we are beginning to get familiar with "Tu" for tungsten. The present book uses "St" for strontium; we may live to see "Sb" changed into "An."

The salient feature of the work is its rejection of the Periodic system. The author holds that Mendeleeff's classification cannot be used in the teaching of chemistry, as its principle is too uncertain, and that it is certainly not the best classification. This *ex cathedra* pronouncement will cause the more curious among his students to inquire which classification the *conférencier* regards as the best; but his final opinion on a question that has a decisive bearing on the problem of the Unity of Matter is rendered by the following translation: "In conclusion it may be said that the classification of the elements cannot play a first-class rôle in science, for no very great confusion can result (*on ne peut faire un bien grand désordre*) with the small number of elements known—about a hundred—even if imperfectly classified" (p. 330). The principle of isomorphism as a means of classification appeals to the author, provided such elements only are grouped together as exhibit numerous cases of isomorphism among their compounds, giving what he explicitly terms artificial families (p. 330).

The book gives a table of the elements classified according to the existence, for each element, "of one or two characteristic oxides, that is, such as are easily obtained" (p. 328); while the old-fashioned subdivision into metals and metalloids has been maintained in the sequence in which the elements are described and discussed. The "metalloid metals" comprise the heavier elements of Groups IV. and V., with the exception of lead.

The reviewer is left with the impression that the present volume is hardly a product of the age of isotopes, X-ray spectra, and atomic physics.

W. R. SCHOELLER.

LEAD: ITS OCCURRENCE IN NATURE, THE MODES OF ITS EXTRACTION, ITS PROPERTIES AND USES, WITH SOME ACCOUNT OF ITS PRINCIPAL COMPOUNDS. By J. A. SMYTHE, Ph.D., D.Sc. Pp. 343. London: Longmans & Co. Price 16s. net.

This book is one of the Monographs on Industrial Chemistry edited by Sir Edward Thorpe.

As expressed in the preface, it has been the aim of the author to give, within somewhat restricted limits, a broad outline of the chemistry of lead.

Economy of words is an outstanding feature of the book. The short, well-balanced periods, together with the absence of superfluous presentation of fact, have resulted in an amazing amount of information on lead and the various views held thereon. The whole is contained in a space of 313 pages of well-written matter.

References are drawn from a particularly wide field, and the reader who may already possess an intimate knowledge of any one side of the subject will discover evidence of comprehensive selection from authorities with whom he is himself familiar. In this lies a test of general quality.

The preliminary chapter is aptly devoted to the history of the metal and those compounds known to the ancients.

Next follows a short summary of the occurrence of the various lead-bearing minerals, and in Chapter III. the author opens out the metallurgy of lead with a clear presentation of the chemistry of roasting and smelting. Here the influence of the principal associated minerals at working temperature, the behaviour of furnace gases, the velocity of reactions, and the nature of these phenomena are considered in the light of their bearing on the conditions of the furnace charge, its yield, and the slag production. There is probably little new in the description of blast furnace extraction, but the reader will find a good general survey of modern practice. The softening and desilverising, as well as the refining of the metal, are described as practised, and are considered in conjunction with the equilibrium diagrams of the alloys.

Under the subject of condensation of lead fume there is an excellent description of the Cottrell treatment; finally, so far as the metal is concerned, the most recent views and knowledge concerning its uses, properties, and alloys (including corrosion and the plumbo-solvency of soft waters) are brought within easy compass.

The remainder of the book is devoted to the compounds of lead. As is fitting, prominence is given to the oxides, their preparation, also to the production of white lead, and the theory of the processes. The complexity of this latter subject is well recognised by the author, who has been at pains to present the facts which are known, as well as to indicate the weakness of views which are not based upon the ascertained truth. This chapter is full of interest and is provocative of thought.

Other compounds of lead are dealt with in concise fashion and the book concludes with a chapter on lead-poisoning.

Altogether, the Editor of the series is to be congratulated upon his choice of Author for this Monograph, and the book can be offered with confidence for the use of those who seek knowledge of lead and its compounds.

GEO. R. THOMPSON.
JOHN MYERS.

QUANTITATIVE CHEMICAL ANALYSIS. By F. CLOWES and J. B. COLEMAN. 12th Edition. Pp. xxiv.+576. London: J. & A. Churchill. 1924. Price 18s. net.

The pleasure with which the many chemists whose early training was based on this book will hear of the appearance of yet another edition, will be clouded by the thought that it will be the last to receive the final impress of both authors, for Professor Clowes died before its publication.

When the first edition was published, in 1891, it was immediately recognised that the work had certain distinctive characteristics which made it particularly suitable as an introduction to the general methods of quantitative analysis. The arrangement, the style, the clearness of the directions, and the way in which numerous difficulties were forestalled, soon made the book a favourite both with teachers and students, and it is not surprising that new editions should have followed one another in rapid succession, until now the twelfth has been reached.

It is interesting to compare the first edition with this latest one. The original 409 pages have increased to 576 of a larger size, and, while the general arrangement has been maintained, many new methods of separation and details of analytical processes have been added, and the book now forms a trustworthy work of reference for practising analysts as well as a textbook for students.

In future editions space might, with advantage, be found for a description of colorimetric and electrometric methods of determining hydrogen ion concentration, and for sections on the uses of the microscope and spectroscope in chemical analysis.

By way of criticism it may be pointed out that attention should be called to the fact that the lime method of estimating halogens in organic substances is liable to give too low results.

Again, while a very good outline is given of some of the principal methods used in the examination of oils and fats, the deductions drawn from the constants of typical examples are only partly warranted, and make this branch of analysis seem a much more simple matter than it really is.

The question of terminology also calls for some remark. It is reasonable to restrict the term "estimation" to chemical analyses and "determination" to physical measurements, or to use "determination" for both operations, but there is nothing to be said for the indiscriminate use of the two terms.

Again, the authors state in their preface that the terms "hydrate" and "hydroxide" are used indifferently. This seems a pity, for it is now usual to make a sharp distinction in the application of the two words. This point is of more importance here than in many other cases, for the book is used by hundreds of students in colleges all over the world, and these will afterwards spread the use of the terminology of their student days.

Still, these are minor points which in no way detract from the general excellence of the book. Speaking from many years' experience of its practical value, the reviewer has no hesitation in saying that it is the best introduction to quantitative analysis with which he is acquainted. EDITOR.