

THE ANALYST

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

ANNUAL GENERAL MEETING AND ORDINARY MEETING, HELD
FEBRUARY 6, 1924.

THE Annual General Meeting of the Society was held at the Chemical Society's Rooms, Burlington House, W.1. The President, Mr. P. A. Ellis Richards, F.I.C., was in the chair.

The Minutes of the previous General Meeting were read and confirmed.

Messrs. H. E. Burgess and T. McLachlan were appointed scrutators of the ballot papers for the election of Officers and Council for 1924.

The Hon. Treasurer presented the accounts of the Society for 1923, which were adopted, and votes of thanks were passed to the Hon. Treasurer and the Hon. Secretaries.

Messrs. Marreco, Houseman and Brandon, chartered accountants, were appointed auditors of the Society's accounts for 1924.

The President delivered his Annual Address. Dr. J. A. Voelcker proposed that a hearty vote of thanks be accorded to the President for his address and for his services in the chair, and that his permission be asked to print the address in *THE ANALYST*. This was seconded by Mr. E. M. Hawkins, and the motion was carried.

The Scrutators having reported the results of their examination of the ballot papers, the President announced that the Officers and Council for 1924 had been elected in accordance with the Council's nominations, as follows:—

President.—G. Rudd Thompson, F.I.C.

Past-Presidents serving on the Council (limited by the Society's Articles of Association to 8 in number).—Leonard Archbutt, F.I.C., A. Chaston Chapman, F.R.S., F.I.C., Bernard Dyer, D.Sc., F.I.C., Otto Hehner, F.I.C., P. A. Ellis Richards, F.I.C., Alfred Smetham, F.I.C., E. W. Voelcker, A.R.S.M., F.I.C. and J. Augustus Voelcker, M.A., B.Sc., Ph.D., F.I.C.

Vice-Presidents.—S. F. Burford, F.I.C., F. H. Carr, C.B.E., F.I.C., and Andrew More, A.R.C.Sc., F.I.C.

Hon. Treasurer.—Edward Hinks, M.B.E., B.Sc., F.I.C.

Hon. Secretary.—E. Richards Bolton, F.I.C.

Hon. Assistant Secretary.—R. G. Pelly, F.I.C.

Other Members of the Council.—F. W. F. Arnaud, F.I.C., H. Ballantyne, F.I.C., E. T. Brewis, F.I.C., R. T. Colgate, D.Sc., F.I.C., R. L. Collett, M.A., F.I.C., J. C. Drummond, D.Sc., F.I.C., S. Elliott, F.I.C., E. M. Hawkins, F.I.C., H. F. E. Hulton, F.I.C., A. E. Parkes, F.I.C., A. R. Tankard, F.I.C., and J. F. Tocher, D.Sc., F.I.C.

An Ordinary Meeting followed the Annual Meeting, the newly-elected President, Mr. G. Rudd Thompson, F.I.C., being in the chair.

Certificates were read for the first time in favour of:—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. (Manch.), A.I.C., Basil Gordon McLellan, F.I.C., and William Thomas Rigby, F.I.C.

Certificates were read for the second time in favour of:—Messrs. Hugh Browning Brown, A.I.C., Sidney Augustus de Lacy, A.I.C., A.M.I.Ch.E. Joseph Henry Lane, F.I.C., B.Sc. (Lond.), Leslie Herbert Lampitt, D.Sc. (Birm.), F.I.C., Reginald Francis Moon, B.Sc. (Bristol), Maximilian Nierenstein, D.Sc. (Geneva), Ph.D. (Berne), William Simpson Shaw, M.Sc., A.I.C., Robert Norman Wright, A.R.C.S., B.Sc. (Lond.), A.I.C., Misses Phyllis Honor Price, B.Sc. (Bristol), and Mabel Suzanne Lavinia Snelus, A.I.C.

The following were elected Members of the Society:—Messrs. Thomas Francis Doyle, George Hogan, F.I.C., Frank Knowles, Archibald Knox, A.I.C., Charles Roger Middleton, B.Sc. (Lond.), A.R.C.S., D.I.C., A.I.C., and Harold Richard Read, A.I.C., A.R.C.Sc. (Dublin).

The following paper was read:—"Osmium Tetroxide as a Reagent for the Estimation of Tannins and their Derivatives," by C. Ainsworth Mitchell, M.A., F.I.C.; and An Apparatus for Fat Extraction and Solvent Recovery was demonstrated by S. A. de Lacy, A.I.C.

STANDARDISATION OF ANALYTICAL METHODS.

The decision of the Council on this question is given in the President's Address (p. 123).—EDITOR.

Annual Address of the Retiring President.

SINCE I had the honour of addressing you in February last the Society has had a prosperous and successful year. Our members are now 465, as we have lost by death and by resignation 23 members, whilst 32 new ordinary members have been elected.

The names of the deceased members are:—George Embrey, Frederick James Lloyd, John Edward Stead, William Thompson, and John Matthew Wilkie.

George Embrey, who passed away on March 10th of last year at the age of eighty-three, was one of our oldest members. Born in Birmingham in 1840, he was educated at King Edward's Grammar School in that city, and as a student was trained at the Birmingham and Midland Institute and, afterwards, at the Royal College of Chemistry. He commenced his professional work as a consulting and analytical chemist in connection with the electro-plating and glass-making trades, and later moved to Gloucester, being appointed County Analyst in January, 1885, and City Analyst in September of the same year. He served as Member of Council of this Society for two periods, later becoming Vice-President, and in 1916 was elected President. His agreeable and genial nature endeared him to all his colleagues and his many friends deeply regret his loss.

Frederick James Lloyd was a member of this Society for many years, and also served on our Council. His early education was carried out at Bristol Grammar School, and in 1870 he entered Dr. Augustus Voelcker's laboratory, where he first studied agricultural chemistry. A little later he became assistant to Dr. Thomas Stevenson, and then senior assistant in the laboratory of the Royal Agricultural Society, finally starting practice on his own account in 1884. He will be long remembered for the special researches he carried out on the subject of Cheddar cheese-making and the chemistry of cider, whilst his loss will be felt not only by his colleagues in agricultural chemistry, but also by all members of the dairying industry, to whom his name was a household word.

William Thomson, who died very suddenly in his laboratory on October 4th, was for many years a member of this Society. Born in Glasgow in 1851, he commenced his professional career as assistant in the Royal Institution Laboratory at Manchester in 1869, becoming, four years later, a partner in the firm of Crace-Calvert and Thomson, and also Public Analyst for Stockport, an appointment he held until his death. He will be chiefly remembered for his work on the phosphorescent sulphides of calcium and barium, the detection of arsenic in beer, and for his efforts to diminish the soot pollution of the atmosphere of Manchester. His loss will be deeply felt by the many colleagues with whom he was closely associated.

John Edward Stead, another of our oldest members, who passed away at the age of seventy-two, was for long associated with analytical processes in relation to steel. In early days he studied under the late Mr. John Pattinson, and later

joined him as partner. He devoted much time to microscopical metallography, and his great skill in this subject was widely recognised. In the past he was frequently present at our meetings, although, of late years, distance from London and his advancing age prevented his attendance. He was elected a Fellow of the Royal Society in 1903 and President of the Iron and Steel Institute in 1920.

John Matthew Wilkie commenced his career as a pharmaceutical chemist, and in 1900 entered the analytical laboratory of Messrs. Boots at Nottingham. For some years past he held the appointment there of deputy chief analyst, and, when the joint meeting of this Society and the Nottingham Section of the Society of Chemical Industry was held in that town last year, he took a very active part in the proceedings, and contributed a valuable paper on the estimation of minute amounts of arsenic. He passed away on November 29th after a severe operation.

During the year 1923 thirty papers have been read at our meetings, and the following have already been published in the pages of THE ANALYST:--

"The Colorimetric Estimation of Pyrogallol, Gallotannin and Gallic Acid." By C. Ainsworth Mitchell, M.A., F.I.C.

"The Estimation of Codeine." By H. E. Annett, D.Sc., F.I.C., and R. R. Sanghi.

"The Estimation of Narcotine and Papaverine in Opium." By H. E. Annett, D.Sc., F.I.C., and M. B. Bose, M.A.

"Note on the Sulphuric Acid Test for Fish Liver Oils." By Norman Evers, B.Sc., F.I.C., and H. J. Foster.

"Sliding Scales for the Convenient Titration of Strong Liquids by Dilution and the Use of Aliquot Parts." By C. H. D. Clark, B.Sc., D.I.C., A.I.C.

"The Estimation of Formaldehyde and Acetaldehyde." By E. W. Blair, B.Sc., D.I.C., A.I.C., and T. Sherlock Wheeler, B.Sc., A.R.C.Sc.I., A.I.C.

"Electrolytic Apparatus for the Estimation of Arsenic." By G. W. Monier-Williams, M.A., Ph.D., F.I.C.

"Some Observations with regard to the Unsaponifiable Matter and Sterols of Edible Fats." By D. W. Steuart, B.Sc.

"Notes on the Presence of Sulphur Dioxide in Cattle Foodstuffs after Fumigation." By H. Alan Peacock, B.Sc.

"The Examination of Firearms and Projectiles in Forensic Cases." By A. Lucas, O.B.E., F.I.C.

"Further Notes on the Estimation of Potassium by the Perchlorate and Cobaltinitrite Methods, and on the Removal of Sulphates." By R. Leitch Morris, F.I.C.

"The Action of Potassium Carbonate on Lead Glass." By H. Droop Richmond, F.I.C.

"Physiological Standardisation." By J. Stanley White, M.R.C.S., L.R.C.P.

"The Presence of Barium and Strontium in Natural Brines." By A. G. Francis, B.Sc., F.I.C.

"Titanium in Nile Silt." By E. Griffiths-Jones.

"An Investigation into the Chemistry of the Reinsch Test for Arsenic and Antimony, and its extension to Bismuth." By B. S. Evans, M.C., M.B.E., Ph.D., F.I.C.

"Determination of the Purity of Vanillin." By S. B. Phillips, A.I.C.

"The Quantitative Estimation of Hemp and Wood Fibres in Paper Pulp." By W. Dickson, F.I.C.

- "The Estimation of Boric Acid in 'Liquid Eggs' and other Foodstuffs." By G. W. Monier-Williams, M.A., Ph.D., F.I.C.
- "Notes on the Examination of Preserved Meats, etc." By Osman Jones, F.I.C.
- "The Absorption of Atmospheric Gases by Water." By J. H. Coste, F.I.C.
- "Tables for Sugar Analysis." By G. D. Elsdon, B.Sc., F.I.C.
- "A Quantitative Study of the Limitations of the Reaction between Ammonia and Sodium Hypobromite." By D. R. Nanji and W. S. Shaw.
- "Notes on a Method for Testing the Accuracy of Babcock Cream and Milk Test Bottles." By B. J. Smit, B.A.
- "The Estimation of Lactose by the Polarimetric and the Gravimetric Methods." By A. L. Bacharach, B.A., A.I.C.
- "Estimation of Fat, Lactose and Moisture in Dried Milks." By H. Jephcott, M.Sc., F.I.C.
- "The Melting Point and Iodine Value of Refined Natural *d*-Camphor." By Maurice S. Salamon, B.Sc.
- "Preservatives in Food: Hull Statistics for the ten years 1913 to 1922." By A. R. Tankard, F.I.C.
- "The Sampling of Coal: The General Problem and some Experiments." By J. H. Coste, F.I.C., E. R. Andrews, F.I.C., and W. E. F. Powney, F.I.C.
- "The Volumetric Estimation of Vanadium in Steel." By A. T. Etheridge, B.Sc., F.I.C., M.B.E.
- "A New Test for Distinguishing Castor Oil from other Oils and Determining its Purity." By H. B. Stocks, F.I.C.

Our Journal, under the able editorship of Mr. C. A. Mitchell, continues as interesting and useful as ever. The last volume contains 634 pages of printed matter, compared with 544 pages for the preceding year, whilst, in addition to the original papers already referred to, we have published 20 notes, 521 abstracts, 57 reviews, and 32 legal and Parliamentary notes. Certain public analysts are sending us their reports regularly, and more of these would be welcomed. Government analysts overseas are co-operating in this regard, so that our Journal forms a connecting link between the work of those at home and those abroad.

Sixteen of the papers were concerned with the analysis of food and drugs, two with organic analysis, six with inorganic analysis, and three with apparatus and methods; there was one on bio-chemistry, one on forensic chemistry, and one on physiological methods of standardisation.

The Ministry of Health and other Government Departments continue to send us their Orders and Regulations, and these, or abstracts of them so far as they touch upon our work, are printed in *THE ANALYST*. A notable instance was the Report on the Loch Maree case of botulinus poisoning.

May I repeat the wish expressed last year that all papers on food analysis, toxicology, and bio-chemical work in connection with food products be contributed to our Society with a view to their publication in our Journal, the recognised medium for such communications.

Under the Analytical Investigation Scheme two papers have been read at our meetings during this session, one by Messrs. J. H. Coste, E. R. Andrews and W. E. F. Powney on "The Sampling of Coal," and the other by Mr. Harold Toms on "The Crystalline Bromides of Linseed Oil." Other investigations are proceeding, notably on fibre estimations required in paper analysis and on methods of

estimating tannins. I hope that during the ensuing year some more of our members will find time to carry out further researches under this scheme, and, with this object in view, will put themselves in touch with our Honorary Secretaries.

Although the work was not undertaken in connection with the scheme, some interesting and useful tables for employment in sugar analysis have been drawn up by Mr. G. D. Elsdon and published in the September number.

A matter of considerable moment to both Public Analysts and consulting analysts generally has been the passing into law of the Milk and Dairies (Amendment) Act, 1922.

This Act definitely prohibits the addition of colouring matter or water or any dried or condensed milk, skimmed or separated milk, to ordinary milk intended for sale; and for the purposes of this section, except as regards the addition of skimmed or separated milk, milk includes skimmed and separated milk. This section is definite and helpful.

The Act further deals with milk sold under various designations, *i.e.* "Certified," "Grade A," and "Pasteurised," and licenses for the sale of milk are granted subject to certain terms and conditions prescribed by Orders made under this Act. In certain of these Orders, issued by the Ministry of Health, bacteriological standards for the various classes of graded milk have been prescribed. To secure compliance with these requirements, arrangements have been made for milk of these types to be systematically sampled by inspectors and examined bacteriologically. The Ministry expected to develop this work on a considerable scale, and hoped that public analysts would be prepared to undertake the necessary examinations. Since the latter are largely concerned with the control of the milk supply, the bacteriological examination would appear to many a natural extension of their work. But such examinations involve considerable time, labour, specialised knowledge and expense for materials and apparatus. For a suitable remuneration I have no doubt that many of us would have been prepared to undertake the work, but when, on enquiry, we found that the Ministry of Health had suggested that the fee should be five shillings per sample, the Joint Committee of this Society and the Institute of Chemistry were almost incredulous.

After careful consideration a joint deputation from the Institute and the Society was appointed to interview the Ministry on the matter. The results, however, were far from satisfactory, as the representatives of the latter stated very definitely that the scheme must be self-supporting, the only funds available being those derived from payments for licenses. The members of our deputation pointed out that the fee proposed would in no sense meet even the actual cost of the bacteriological examination, and, consequently, that no one of us would be prepared to undertake the work. We further made it clear that, in our opinion, an inferior rough-and-ready bacteriological examination was of no use at all, and was most unfair, not only to the general public, but also to the members of the dairy trade whose milk would be taken for testing purposes, as any lack of care or skill during the examination could only lead to an increase in the apparent bacterial content of the samples.

In conclusion we were informed that certain laboratories, the majority state- or rate-aided institutions, had undertaken to carry out the work at the suggested fee, but the Ministry would give very careful consideration to the views we had expressed.

In my address of last year I referred to Circular 325 issued by the Ministry of Health suggesting that it was undesirable that a prosecution should be based upon the results of an isolated test showing a deficiency of fat in the case of a particular milk supply. It is satisfactory to note that, following the joint letter of protest written by the Presidents of the Institute of Chemistry and this Society, the Ministry, recognising the unfortunate results produced by this suggestion, issued last May a further Circular withdrawing the former one.

The Ministry have issued Regulations as to the labelling and composition of condensed milk and of dried milk. Minimum percentages are fixed by the Regulations for fat and milk solids in full-cream condensed milk and for milk-solids in condensed skimmed milk. Similar minima are also fixed for full, three-quarter, half and quarter cream dried milk. It is also required in all cases that the instructions as to dilution or preparation given on a tin or package of condensed or dried milk shall be quantitatively accurate.

The Regulations in regard to condensed milk have already come into force, whilst those for dried milk are to operate as from May 1st, 1924.

Article 2, section 2, of the Regulations in respect of dried milk reads as follows:

"These Regulations apply to dried milk to which no other substance has been added, and to the dried milk contained in any powder or solid of which not less than 70 per cent. consists of dried milk."

Whether this section is intended to apply to sweetened dried milk only, or whether it is to cover such substances as infant's and invalid's foods is not clear.

Both of these Regulations have already appeared in our Journal, and so I have not attempted to give a *précis* of their requirements. The latter, valuable as they undoubtedly are in the public interest, necessitate considerably more work on the part of public analysts, but one regrets to see that the Ministry has made no suggestion for increased remuneration to these officers on this account.

In many counties and boroughs throughout the country it has been the custom to test the accuracy of the dispensing of medicines, the Food and Drugs Acts inspectors being instructed to submit definite prescriptions to pharmacists for this purpose. The contents of the medicine bottle are then divided into three parts, and one portion submitted to the public analyst in the ordinary way. Whether or no such samples come legitimately under the provisions of the Sale of Food and Drugs Acts is perhaps a moot point, but, assuming that they do, various troublesome facts arise that have to be dealt with by the analyst and taken into account by him when drafting his report.

In the first place many prescriptions usually given by medical men are not at all suitable for quantitative analysis. Again, the bottles in which the medicines are put up are notoriously very far from accurate, both as regards their actual

capacity when full, and also as regards their sub-division into ounces, tablespoonfuls, and what not. It would appear to be a very general practice on the part of pharmacists, after weighing or measuring the required amounts of the various drugs, to make up to the total amount in the bottle rather than in a graduated measure. Consequently the final volume may be either greater or less than is supposed, depending on the actual capacity of the bottle. These and other points that I need not refer to here become of considerable importance when a question of prosecution arises.

In the spring of last year your Council was approached by the Council of the Pharmaceutical Society who proposed a joint conference on this subject. We welcomed this suggestion, and, as a result, a meeting took place at the rooms of the Institute of Chemistry between representatives of our Society, of the Pharmaceutical Society, and of the Retail Pharmacists Union. We had a very friendly, and, I think, useful meeting, the views of both sides being carefully considered and discussed. As an outcome, your Council suggested to the Ministry of Health that the latter should issue a Circular to Local Authorities recommending that the following procedure should be adopted in taking samples of prescriptions:

That the Analyst and the Medical Officer of Health should both be consulted as to the type of prescription it is desirable to use for the purpose of checking the accuracy of dispensing, and that, in the event of any substantial inaccuracies being disclosed by analysis, both these officers be consulted as to the desirability of instituting proceedings.

Further, that the inspector be instructed, prior to dividing the sample into three parts, to mark, in the presence of the vendor, the height that the contents reached in the bottle in which the medicine is originally supplied to him by the vendor.

Also that the bottle so marked be submitted to the analyst in order that he may determine the total quantity of medicine supplied.

The latter suggestion was made on account of the definite statement of the pharmaceutical representatives that the medical man, in writing out his prescription, intends that an aliquot portion of the total volume should be taken by the patient in each dose, rather than an accurate ounce, or half-ounce, etc., as the case may be. I am glad to say that the Ministry adopted the suggestions we made, and has issued to Local Authorities a Circular on the lines recommended.

With the letter already referred to, your Council sent another to the Ministry of Health pointing out that the analysis of dispensed medicines was never contemplated in the Act of Parliament, and that the fee customarily paid for analyses of samples taken under the Sale of Food and Drugs Acts cannot be considered a reasonable one in such cases. They urged the Ministry to use such means as they thought fit to ensure that the public analyst should receive adequate remuneration for the difficult work entailed by the analysis of such medicines. They further reminded the Ministry that, year by year, increased demands have been made upon the public analyst without any corresponding increase in his fee, and that to require work of this kind is putting an unjustifiable burden upon him.

I am sorry to say that the Ministry have not accepted this suggestion, and it remains for us to consider what further steps we should take in the matter.

This year I have to refer again to the entirely inadequate remuneration so frequently offered when a vacancy occurs in an official position under the Sale of Food and Drugs Acts. In the early part of last year the County and City of Gloucester advertised for a Public Analyst in the place of the late Mr. George Embrey. The number of samples proposed to be taken for analysis was not mentioned in the original notice, but enquiry elicited the fact that they were out of all proportion to the remuneration offered, and a protest was sent by the Joint Committee of the Institute and our Society to the Clerk of the Gloucestershire Council. In spite of this, the appointment was made on the terms proposed, but the successful candidate was not a member of our Society or of the Institute of Chemistry. The Ministry of Agriculture and Fisheries and the Ministry of Health have since informed the Gloucestershire County Council that they were not prepared to approve this appointment. A sequel to this is that the County Council is now exercising its legal right to send the work to the Public Analyst of another district, and it is understood that it intends to follow this course for such time as would afford the original candidate an opportunity of qualifying for the post.

Amongst other public analystships that became vacant during the past year were those for the City of London and the Borough of Islington. Your Council and that of the Institute of Chemistry regarded the terms offered in both of these cases as inadequate, and the joint committee again brought pressure to bear upon the authorities concerned with a view to improving them, but again the new appointments were made at the old rate of payment.

During the year our special branch of science has been brought somewhat more prominently than usual before the notice of the general public by the holding of various conferences and meetings in various parts of the country.

The Fourth International Chemical Conference took place at Cambridge under the presidency of Sir William Pope, and was a well attended and successful meeting.

In September the British Association met at Liverpool, and the brilliant address of the President, Sir Ernest Rutherford, on the electrical structure of matter has been received with world-wide interest, whilst many other important and valuable papers were contributed to the Chemistry Section.

Upon the invitation of the Liverpool and North-Western Section of the Institute of Chemistry a Conference of Fellows and Associates was also held there in October last. At this meeting several interesting discussions took place on matters of considerable moment to ourselves, in which many chemists, some of them members of our Society, took part. Amongst the subjects discussed were the questions of the formation of a national federation of men of science and the desirability or otherwise of a publicity campaign on the service to the community rendered by chemists.

In the same month a Chemists' Dinner, which was widely attended by the members of the various Societies, took place in London. This was in the nature

of an innovation, as no dinner on exactly similar lines had been organised previously, and I am sure that all those who were present will agree that it was a most successful function, and one that should tend to increase the harmony and friendship already existing between those of us who are working in the many different branches of our profession.

During the year the Minister of Health has appointed a Committee to enquire into the use of preservatives and colouring matters in food and to report:

(1) Whether the use of such materials, or any of them, for the preservation and colouring of food is injurious to health, and if so in what quantities does their use become injurious;

(2) Whether it should be required that the presence of such materials and the quantities present in food offered or exposed for sale should be declared.

As originally constituted, this Committee included amongst its members as chemical representatives one of our past Presidents, Mr. Otto Hehner; one of our honorary members, Dr. F. Gowland Hopkins; and Mr. George Stubbs. Owing to the regretted departure of Mr. Hehner for South Africa, in November last, I was asked to fill the vacant seat on the Committee.

The matters referred to us cover a wide area, and yet in some respects, perhaps, the area is hardly wide enough, since the contamination of food with such substances as tin, lead, arsenic, etc., scarcely comes within our purview, although it is a matter of considerable importance to everyone.

Much evidence covering both the scientific and trade points of view has been and is still being heard, and it is to be hoped, for the sake of the public, and also especially for members of this Society, that when the Report of the Committee is issued, in due course, it will help to put the question of the use of preservatives and colouring matters in food on a more satisfactory basis than it is on at the present time.

With the state of the law now existing the procedure adopted by Local Authorities in regard to preservatives varies throughout the kingdom. In some districts no samples are taken with a view to the detection of preservatives, whilst in other places the vendors of foods so treated are prosecuted in almost all cases. This means, of course, that the public in places coming under the first category have no protection at all in this respect, whilst elsewhere they may be called on to pay the costs of actions rendered unsuccessful by the lack of standards and regulations.

Another point that requires very careful consideration is the importation into this country of food that, from necessity or not, as the case may be, comes in chemically preserved. Whether this treatment should be permitted, or what limitations should be placed upon it, needs a thoughtful and wise decision.

In this connection I may remind you that last month we held an extra meeting of this Society at the Institute of Chemistry, and devoted it to a discussion on preservatives in food. The debate was opened by Sir William Willcox and Dr.

Roche Lynch from the physiological, and by Mr. Hinks from the analytical standpoint. We had a very large audience of members and their friends, there was a good discussion, and the meeting proved a most interesting one.

The question of the advisability of standardisation of analytical methods has been under consideration by your Council for some time past, and the matter was referred finally to a sub-committee, who in due course made two recommendations to the Council, which the latter adopted. They are as follows:

(1) The formation by the Society of a Committee to deal with the question of standard analytical methods in general is not considered practicable owing to the very large amount of specialised work which would be involved. The Committee regret their inability to advise the Society to undertake the responsibility of recommending standard methods.

(2) The Committee recommend that chemists and analysts interested in particular industries be invited to confer among themselves upon cases where divergent results have occurred owing to the use of different methods of analysis, and to submit reports to appropriate committees of the Society; these reports, after consideration, to be published in *THE ANALYST*; the Society, however, taking no responsibility for recommending methods of analysis, but merely acting as a "clearing house."

Following this, a paper on "The Plea for Standardisation" was contributed by Mr. M. S. Salamon to the December meeting of the Society, and an interesting discussion ensued.

I believe I have referred to all the chief events that have occurred in connection with, or are of interest to, the Society during the past year, and must now bring this address to an end. It has, I fear, of necessity, been a little disjointed, as so many varied topics have had to be touched upon.

To-day marks the conclusion of my eighteen years official connection with the Society, first as Honorary Secretary, and for the last two years as your President. It is a somewhat long period, upon which I can look back with great pleasure on account of the uniform kindness and consideration extended to me by all members of the Society, and more particularly by the Officers and Council, with whom my duties have brought me most in contact. As President, my work has been comparatively light on account of the able assistance of the Officers, Messrs. Mitchell, Hinks, Bolton and Pelly, to all of whom I tender my most hearty thanks.

Before I resume my seat I desire to welcome most cordially as my successor my old friend, Mr. G. Rudd Thompson. In Mr. Thompson you will have as President one who has the interests of the Society closely at heart, who has been long connected with it, and who will, I am sure, occupy the chair with credit and distinction to the Society and himself.

The Estimation of Lead in Potable Waters, and in Urine.

By JOHN C. THRESH, M.D., D.Sc., F.I.C.

(Read at the Meeting, November 7, 1923.)

IN a previous paper, published in the Journal of the Society for July, 1921 (ANALYST, 1921, 45, 270), a method was described for estimating small amounts of lead in water, and it was pointed out that variations in the acidity caused a variation in the colour produced by colloidal lead sulphide. Further use of the method confirms the opinion expressed of its reliability, but it seems preferable to make the gelatin acid solution as follows:

Acetic acid B.P., 30 c.c.; distilled water, 70 c.c.; and gold leaf gelatin, 0.1 gm. Dissolve the gelatin in the dilute acid at a temperature below the boiling point. The amount used in each experiment is 1 c.c. to 50 c.c. of the water to be examined.

Unfortunately most moorland waters are coloured, some markedly so, and this colour interferes with the estimation of the lead. At first the difficulty seemed to be surmounted by using the same water, or the same dilution with distilled water, in making the standard solutions, but, under certain circumstances, it is not always as satisfactory as could be desired, especially when the amount of lead to be estimated is very small. Also, it soon became obvious that different waters gave different depths of colour when containing equal quantities of lead, and further investigations shewed that different distilled waters gave variable results. The following table exhibits the extent of this variation and the extent of the error which would result therefrom: All the water used contained 0.2 mgrm. of lead in 100 c.c.

	Colour.	Lead sulphide indicated. Mgrm.
(1) The purest distilled water, glass condensed ..	1.0	0.2
(2) The purest distilled water condensed in tinned copper	0.3 to 0.9	0.06 to 0.18
(3) Commercial distilled waters	0.6 to 1.0	0.12 to 0.2
(4) Moorland water, Durham	0.7	0.14
(5) ,, ,, Central Wales	0.8	0.16
(6) ,, ,, Cumberland	1.0	0.2
(7) Infusion of heather rootlets (very dilute) ..	1.0	0.2
(8) ,, of bog moss (very dilute)	0.85	0.17
(9) ,, bilberry rootlets (very dilute)	0.8	0.16

It will be observed that moorland waters do not vary nearly so much as distilled waters, and it was a long time before the cause of the variation was

discovered; in fact, it was long before it was discovered that the nature of the condensing apparatus was chiefly responsible. This delay was due to the extraordinarily small amount of impurity which can produce a marked effect. On connecting a boiler with two condensers, one of glass and the other of tinned copper, it was found that the water collected from the former was uniform and satisfactory, whilst that collected from the latter varied considerably, and the more slowly the water was distilled the worse the result. Finally it was found that copper was the interfering substance. With hydrogen sulphide copper gives a much paler colour than the same amount of lead.

Colour given by

H ₂ S with 0.2 mgrm. of lead in 100,000	Colour	1.0
„ „ 0.2 mgrm. copper in 100,000	„	0.3
„ „ 0.2 mgrm. copper + 0.2 mgrm. lead + 0.2 mgrm. copper		„	0.5

Hence, instead of the copper increasing the depth of tint, it reduces it fifty per cent.

The effect of still smaller quantities is shewn in the following table, where different quantities of copper were added to water containing 0.2 mgrm. of lead in 100,000.

Copper added per 100 c.c. Mgrm.	Colour corresponded to distilled water + lead. Mgrm.	Colour corresponded to Cumberland water + lead. Mgrm.
0.1	0.1	—
0.05	0.06	—
0.025	0.06	—
0.01	0.9	—
0.005	0.12	0.14
0.0025	0.14	0.16
0.0015	0.19	—
0.001	—	0.1
0.000	0.2	0.2

The presence of one part of copper in 100 millions has a distinct effect.

Copper was found in the distilled waters giving too little colour with lead sulphide, but it has only been found in one of the samples of moorland water examined. This was from South Wales, and contained 1 part in 100 millions.

This amount has a decided effect, but if metallic tin is immersed in a water containing a trace of copper the copper seems to be removed, as the water then gives a normal reaction with lead sulphide. Silver, on the other hand, acts very much like copper. A silver spoon left all night in a pure distilled water has a marked effect in reducing the colour of colloidal lead sulphide. The above results, however, did not explain the variations with moorland waters free from copper. Further investigation shewed that the interfering substance was not of organic nature, but simply iron in solution. This is not so powerful as copper, but it explains all discrepancies, since every coloured moorland water examined contained easily detectable traces of this metal. The effect of small quantities is

shewn in the results obtained with various dilutions in distilled water containing 0·2 mgrm. of lead in 100,000.

Iron present. Mgrm.	Colour.	Lead.
0·1	0·3 =	0·06
0·05	0·3 =	0·06
0·02	0·35 =	0·07
0·01	0·7 =	0·14
0·005	1·0 =	0·2

Hence, in the case of a water which, undiluted, gives any colour with the potassium thiocyanate test for iron, the iron present will appreciably effect the estimation of the lead.

It is obvious, therefore, that, in estimating the amount of lead in any potable water, a sample of the same water free from lead should be obtained for making the standards for comparison.

The importance of the standard and water examined having the same degree of acidity and acid of identical character may again be emphasised, especially as it has a marked bearing upon the estimation of lead in urine. The subjoined table will impress this fact upon the mind.

Lead in each solution 2 mgrms. in 100 c.c.

Acid added to 100 c.c.	Colour.
2 c.c. Gel. Acetic acid	1·0
4 c.c. " " " "	0·85
2 c.c. 0·1 <i>N</i> hydrochloric acid	0·75
2 c.c. <i>N</i> hydrochloric acid	0·0

With 2 c.c. of 0·1 *N* hydrochloric acid the colour is different from that produced by the gelatin acetic acid, and in a very few minutes the water becomes opalescent, and accurate comparison is impossible. The colouring matter of peaty waters always contains iron, and apparently the colour is always reduced by acidification, and increased (to a less extent) by addition of an alkali. A number of experiments have been made comparing this colour with that given by dilute infusions of *Sphagnum* and of rootlets of heather and bilberry. The colour apparently comes from the two latter (often chief constituents of peat), but some of the free acid is derived from the *Sphagnum*, and these acids are probably quinic acid and some product of the oxidation of tannin. This question, however, is left over for further investigation.

From what has been already said it is obvious that the detection of very minute quantities of lead in coloured liquids must be a matter of some difficulty. In connection with suspected cases of lead poisoning in a town using a moorland water, samples of urine from the patients under observation were submitted to examination. It was found that the addition of acid and hydrogen sulphide to the urine never produced any obvious change in colour, whereas a change could be detected if the urine contained 1 part of lead in 2 millions; the amount present,

if any, must have been less than this. No satisfactory test could be found in the text books, and, accordingly, a series of experiments was made to devise one. At first, advantage was taken of the fact that water or urine containing lead gives this metal up very rapidly, even in the presence of dilute acetic acid, to specially prepared charcoal, but it was found that the charcoal contained a trace of iron and retained the lead somewhat obstinately. Finally its use was abandoned, and the following method was devised, which gives excellent results in skilled hands:

Heat a measured quantity of the urine (not less than 100 c.c.) nearly to boiling point, add *N* sodium carbonate solution until the liquid becomes turbid, and the precipitate flocculates on gentle agitation (1 c.c. per 100 c.c. of urine suffices in most cases). Then allow the whole to stand until the supernatant liquid is clear. This can be poured off or passed through a small filter paper, upon which the whole precipitate is collected, washed with distilled water made faintly alkaline with sodium carbonate, and finally dissolved in about 2 c.c. of B.P. acetic acid, plus 10 c.c. of hot water. The liquid and washings need not exceed 25 c.c. Then add 0.5 c.c. of gelatin acetic acid and hydrogen sulphide. The colour then produced must be estimated from a solution of corresponding strength of acid.

No one should attempt this estimation until he can get absolutely negative results with lead-free urine treated in the above manner, but, if this cannot be done, the amount found in the check examination may be deducted from that found in the suspected urine. After assuring myself that the chemicals and glass ware used contained no lead, positive results were still obtained. Finally the filter paper was suspected and found to contain lead. Filter paper which has been thoroughly exhausted with acid must therefore be used.

I have had submitted to me the analyses of 3 kinds of filter paper usually employed for qualitative work.

	Ash. Per Cent.	Pb. Per Cent.	Cu.
1. English make	0.17	0.0078	None.
2. A continental make	0.41	0.0106	Very slight trace.
3. Another continental make	0.53	0.0095	ditto.

The following results were obtained with urine to which lead had been added. The first series of analyses was made by myself, the second by a friend, who could not get a negative result with another urine.

SERIES 1		SERIES 2.	
Lead added. per 100 c.c. Mgrm.	Lead found. Mgrm.	Lead added. Mgrm.	Lead found. Mgrm.
0.1	0.105	0.00 (check sample)	0.03
0.075	0.08	—	—
0.05	0.035	0.04	0.07
0.05	0.058	Diff.	0.04
0.04	0.043	0.00 (check sample)	0.02
0.02	0.03	0.03	0.05
0.02	0.021	Diff.	0.03
0.01	A trace.	—	—

When urine, not distinctly acid, containing a trace of lead, is passed through filter paper, most, if not all, of the metal is retained by the fibres of the filter, and can be dissolved out with acid, but, as a method of detecting and estimating lead, the results obtained were not sufficiently concordant to render the process available.

LEAD IN BEER AND CIDER.—In attempting to estimate small quantities of lead in beer and cider by the process used for urine, it was found that the precipitate produced carried down so much colouring matter that the process was not applicable. By using precipitated chalk, instead of sodium carbonate, the precipitate is very pale in colour, and the acetic and solution, though not colourless, can be used for nesslerising, a dilute solution of burnt sugar being used for the standard. In this way a beer to which 1 part of lead per million had been added gave a most distinct reaction with hydrogen sulphide, and the estimated amount of lead was 0.08. With some slight modification, it is obvious that this method may be useful.

DISCUSSION.

Mr. W. T. BURGESS said that the author's paper made us wonder whether our colorimetric estimations were correct. He was surprised to find that if one had a little copper in the presence of lead the colour was only the same as when lead alone was present. Anyone with experience of water knew that peaty water changed its colour when its reaction was altered by the addition of acid or alkali. He called attention to the organic matter in the reagent used by the author; the gelatin put into the test was 200 parts per 100,000. He doubted whether these tests would come out in this way when hydrogen sulphide alone was used.

Dr. Thresh had asked him whether he had suspected lead in the filter paper; he had not done so, but he had found it useless to filter water containing traces of lead through filter paper, owing to the adsorption of the lead by the filter paper.

Mr. RAYMOND ROSS said that he would like to know why it was necessary to estimate the lead as sulphide and not chromate; with the latter, he considered, it would be easier to judge the opalescence. If water was being passed through a mechanical filter it was always best to filter it on the acid side, because, if one allowed it to become at all alkaline, when passing through the filter, it would absorb colour from the filter; if it was acid, one could add lime water, after filtration, without altering the colour.

Mr. E. M. HAWKINS, referring to the sulphide test, said that the reaction was much affected by a very minute quantity of acid.

Mr. E. J. GUILD, referring to the peaty water of Edinburgh, said that he agreed with the author as to the difficulty of estimating lead in the presence of iron; as regards filter papers, they had actually estimated the amount of lead by ashing and precipitation of the lead as sulphate. He emphasised the importance of making blanks, especially in the case of a delicate test.

The Colorimetric Estimation of Lead in Cream of Tartar.

BY R. L. ANDREW.

(Read at the Meeting, December 5, 1923.)

IN connection with the administration of the Sale of Food and Drugs Act all importations of cream of tartar during 1921-1922 were examined for lead.

This resulted in the carrying out of a large number of estimations, and involved a critical examination of methods.

The B.P. method, with the use of primary and auxiliary solutions containing seven and two grms. of tartrate respectively, is cumbersome, and has the further objection, that, in the case of samples carrying large amounts of lead, the colorations produced are so intense that accurate comparisons are difficult.

A more satisfactory method is to take two grms. of the cream of tartar, add 1 c.c. of 5 per cent. potassium cyanide solution, 1 c.c. of ammonia, and 40 c.c. of water, and dissolve by warming and shaking. The solution is cooled and passed through a dry filter.

Standards are prepared in a similar manner from two grms. of lead-free cream of tartar, with the addition of requisite amounts of a lead nitrate solution containing 0.01 per cent. of lead. Any tint in the solution is then matched by the addition of very dilute caramel to the standards.

A few drops of ten per cent. sodium sulphide solution are added, the solutions made up to the mark, and the amount of lead estimated by comparison with the standards.

The results so obtained agree with those of the B.P. method, using 7 and 2 grms. of cream of tartar, shewing that the different concentrations of tartrate do not affect the depth of colour of the lead sulphide.

If, however, an aqueous solution containing lead is used for the standard, the colour is only about half the depth of that produced by an equal amount of lead in a tartrate solution, and a serious error is introduced.

The effect of iron in both the ferric and ferrous states was investigated.

R. R. Tatlock and R. T. Thomson (*ANALYST*, 1908, **33**, 176) say that the iron should not exceed 0.25 mgrm. in 50 c.c. of the solution under examination; otherwise the results may be on the high side.

It was found that amounts of iron not in excess of 0.4 mgrm. have no effect.

Contrary to Wilkie's findings (*J. Soc. Chem. Ind.*, 1909, **28**, 636), it makes little, if any, difference whether the iron is in the ferric or ferrous state.

An amazing error was found in the method given for the detection of lead in cream of tartar, in Allen's *Commercial Organic Analysis*, 4th edition, Vol. I., p. 549, where it is recommended that "if the solution is coloured it must be treated

with purified animal charcoal and filtered." Such treatment completely removes any lead that may be present. Experiments shewed that 0.5 grm. of animal charcoal will, under the conditions of the estimation, remove 1 mgrm. of lead from solution.

Of 389 samples of cream of tartar examined during 1921 and 1922, only 26 contained more than one-seventh of a grain of lead to the pound.

Many of the samples were of a very high degree of purity, some failing to shew a trace of lead in ten grms., and in no case was there sufficient iron present to affect the estimation.

No originality is claimed for these observations, but experience has shewn that sometimes sufficient attention is not paid to possible errors, particularly to that produced by the use of an aqueous solution of lead as a standard.

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Notes on the Estimation of Chromium.

BY HUBERT T. S. BRITTON, M.Sc., F.I.C.

(Read at the Meeting, December 5, 1923.)

I. GRAVIMETRIC ESTIMATION.—The gravimetric estimation of chromium by precipitation as the hydroxide and subsequent ignition to the oxide does not give quantitative results. They are always too high, usually about one or two per cent. Prolonged heating of the oxide at a high temperature appears to be one of the conditions necessary to eliminate this error. The reason for these high results is to be found in the fact that the ignited oxide invariably contains appreciable amounts of chromic anhydride, as may be seen when the ash is extracted with water and the solution tested for chromate by the ordinary reagents.

This observation is by no means new. There appears, however, to have been some controversy extending over the last sixty years concerning the magnitude of the error involved. Treadwell, on page 102 of the 1919 English edition of his *Quantitative Analysis* (trans. by Hall), states that "the results obtained are always a few tenths of a per cent. too high on account of the formation of small amounts of alkali chromate, even though the entire operation takes place in platinum vessels. The alkali comes from the reagents." When every precaution is taken to use only the purest reagents and salts, the ignited oxide is still found to contain some extractable chromate. This was also the experience of Rothaug. (*Zeitsch. anorg. Chem.*, 1914, **84**, 165).

Souchay (*Zeitsch. anal. Chem.*, 1865, **4**, 66) was the first to observe that gravimetric analysis gave high values, but, as he happened to obtain figures 2.62 per cent. too high when the precipitation was carried out in glass vessels, as compared with 1 per cent. in platinum vessels, he was led to conclude that the

arger increase was due to some extraction from the glass having taken place. Similar views were held by Wilm (*Ber.*, 1879, 12, 2172 and 2223), whose results showed errors up to 6 per cent. On the other hand, Treadwell (*Ber.*, 1882, 15, 1392) obtained accurate results, no matter in what vessels the estimations were made, although he mentions that the ash always contained some chromic anhydride. Later, Stock and Massacui (*Ber.*, 1901, 33, 467) apparently obtained accurate results when the precipitation was carried out by means of a mixture of potassium iodide and iodate. In 1914 Rothaug (*loc. cit.*) published a very exhaustive paper on the estimation of chromium as oxide. He investigated the methods in which the chromium is precipitated with each of the following reagents: Ammonium hydroxide, ammonium sulphide, and a mixture of potassium iodide and iodate. With ammonia his results show an average error of 0.75 per cent.; with ammonium sulphide, 1.6 per cent.; and with the iodide-iodate method 1.02 per cent. After heating the oxide in an electric furnace for two hours the error was much less, averaging 0.17 per cent. The only procedure which gave him accurate results was ignition of the oxide in a stream of hydrogen. Analyses of aqueous extracts of the ash showed, on ignition, some chromium chromate— $\text{Cr}_2(\text{CrO}_4)_3$ —was formed, and was more or less decomposed by heating at a higher temperature, and completely reduced to chromium oxide when heated in an atmosphere of hydrogen. Hence, it will be inferred that as the ash on ignition tends to form coherent particles, thereby enveloping some chromate, the total decomposition of the higher oxide will be an extremely slow process.

As these observations happened to be in accord with the author's experience, the errors being much greater than would be gathered from the remarks of Treadwell, it was resolved to direct attention to volumetric methods.

II. VOLUMETRIC ESTIMATION.—Several methods of oxidation of chromic salts to chromic acid have been put forward, but they involve the use of such reagents as hydrogen peroxide and persulphates. Thus W. J. Sell (*Chem. News*, 1886, 54, 299) precipitated the chromium with potassium hydroxide and then added more alkali in amount just sufficient to redissolve the precipitate. To this solution an excess of hydrogen peroxide was added, and the liquid then boiled to complete the oxidation, and also to decompose the excess peroxide. Afterwards the chromic acid was estimated iodimetrically. This method of oxidation was examined by Bourion and Sénéchal (*Comptes rend.*, 1913, 157, 1528), who found that a considerable excess of hydrogen peroxide is necessary to ensure complete oxidation, and that the excess of alkali is without much effect on the decomposition of the excess peroxide. They estimated the chromic acid thus formed by allowing it to oxidise ferrous ammonium sulphate and titrating the unoxidised iron with potassium permanganate solution.

Up to the present, no one appears to have used sodium peroxide, as such, in the volumetric estimation of chromium, although less common and more troublesome substances have been used.

The following is a description of some work done in which the conditions for the accurate volumetric determination of chromium were established. Solutions

of sodium peroxide of about 1 per cent. concentration are decomposed quantitatively after boiling for ten minutes; the amount of iodine which is liberated from potassium iodide after the solution has been rendered strongly acid in the cold is beyond the limits of titration. Again, chromium salts are readily oxidised to chromates by means of sodium peroxide in alkaline solution, but in acid solutions the addition of peroxide brings about the formation of perchromic acid and the subsequent reduction to the original condition. Thus, all that is necessary is to oxidise in alkaline solution, decompose the excess of peroxide by boiling, cool and then acidify with either moderately concentrated hydrochloric or sulphuric acid, add potassium iodide and, after dilution, titrate the liberated iodine with sodium thiosulphate.

The following procedure was found to yield readily accurate results: The acid present in a solution of the chromium salt is roughly neutralised, and an excess of sodium peroxide (about 1 gm. is generally sufficient) is added. The solution is diluted to about 100 c.c. and boiled for ten minutes, in which time the chromium is completely converted into chromate and the excess of peroxide decomposed. A precipitate sometimes appears during boiling, but re-dissolves as the oxidation proceeds. After cooling, the solution is acidified, and the chromic acid estimated iodimetrically. If the solution has been sufficiently cooled, it does not matter whether hydrochloric acid or sulphuric acid is used; but if the solution is at all warm there is a distinct tendency to obtain low results with hydrochloric acid, on account of the oxidation of the hydrochloric acid by the chromic acid.

RESULTS.—The following are some results which were obtained by this method, the chromium having been estimated in varying quantities of pure chrome alum:

Weight of chrome alum taken. Grm.	Equivalent to Cr_2O_3 taken. Grm.	N/20 $\text{Na}_2\text{S}_2\text{O}_3$ required. c.c.	Cr_2O_3 found. Grm.
0·1108	0·0153	12·10	0·0153
0·2246	0·0342	27·10	0·0343
0·2288	0·0348	27·60	0·0350
0·2230	0·0339	26·85	0·0340
0·4000	0·0609	47·93	0·0608
0·6993	0·1064	83·85	0·1062

The following typical results (as Cr_2O_3) were obtained gravimetrically by precipitation with ammonium hydroxide and by the more accurate volumetric method: Gravimetric, 0·1096; volumetric, 0·1074 gm. Here the gravimetric result is 2·04 per cent. too high.

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NOTE.—Since writing the above, my attention has been directed to a paper by J. Clark (*J. Chem. Soc.*, 1893, **63**, 1083) on the use of sodium peroxide as an analytical reagent. In the estimation of chromium in chrome steel, he obtained

satisfactory results only when the oxidation was carried out by the usual fusion process. He stated, however, that the oxidation may be effected in solution by adding a freshly prepared solution of sodium peroxide to the chromium solution until it had become alkaline, and then boiling. He stated that this method yielded low results. This was evidently due to the fact that he had also much iron present in the solution, thereby causing some chromic acid to be occluded by the ferric hydroxide which was precipitated. He did not test the accuracy of the method when there were no disturbing influences present in the solution.

THE APPLICATION OF THE IODIMETRIC METHOD TO THE ANALYSIS OF SUGAR PRODUCTS.

BY C. L. HINTON, F.I.C., AND T. MACARA, F.I.C.

ERRATA.—Delete "C" after the polarisation degrees in the following places in the January issue:—P. 6, l. 8 and 9; p. 7, l. 29; p. 12, l. 24, 28, 30, 31.

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.

ESTIMATION OF BORIC ACID IN MILK, ETC.

SPECIAL experiments on Thomson's method (ANALYST, 1896, 21, 64) proved that: (1) In the presence of excess of alkali boric acid is not volatilised at a red heat. (2) It is better to treat the ash with water and hydrochloric acid successively. (3) If the solution is not dilute, alkali will precipitate calcium borate. (4) The amount of calcium chloride recommended by Thomson (=7 c.c. of a *N* solution for 70 c.c. of milk) is too much, and the addition of lime water increases the precipitation of calcium borate. (5) The alternate addition of calcium chloride and sodium hydroxide in the presence of phenolphthalein, as recommended by Shrewsbury (ANALYST, 1907, 32, 12) is a satisfactory indication of the correct amount of calcium chloride to be added in the absence of carbon dioxide. If carbon dioxide is not removed the end reaction is indefinite, and phosphate remains in solution. (6) The acid solution can be boiled for 5 minutes in an open flask without loss of boric acid.

The following is the method recommended:—Evaporate 70 c.c. of milk in a platinum dish on a water bath with 7 c.c. of 3 *N* sodium hydroxide solution for about four hours. Apply a flame to the side of the dish, taking care that the contents do not froth over. Complete the ignition at a high temperature until the contents are white, or nearly so. Add 35 c.c. of water and filter after standing. Add 5 c.c. of 3 *N* hydrochloric acid to the dish and 15 c.c. of water. Filter through

the same filter, and wash dish and filter with 4 c.c. of 3 N hydrochloric acid mixed with 16 c.c. of water. Boil for 5 minutes, cool, and add 6 c.c. 0.5 N calcium chloride solution, phenolphthalein and N sodium hydroxide solution to make the liquid a permanent faint pink. Add another c.c. of calcium chloride solution and more sodium hydroxide if the colour is destroyed. Repeat this addition, if necessary. Dilute to 100 c.c. and filter through a dry filter. To 80 c.c. of filtrate add methyl-orange and 0.1 N hydrochloric acid to slight excess. Boil for 5 minutes, cool, neutralise to methyl-orange and titrate with 0.1 N sodium hydroxide solution and 30 c.c. of glycerin. Subtract the acidity of 30 c.c. of glycerin.

$$\begin{aligned} \text{W/v boric acid} &= \text{c.c. } 0.1 \text{ N sodium hydroxide solution} \times 0.011. \\ \text{Grains per gallon} &= \text{c.c. } 0.1 \text{ N} \quad \quad \quad \quad \quad \quad \quad \quad \times 7.75. \end{aligned}$$

If more than 5 c.c. of sodium hydroxide solution are used, calcium borate may be precipitated, and the precipitate should be re-dissolved and re-precipitated as before.

In the test analysis 5 c.c. of 0.1 N boric acid were added to 70 c.c. of milk, and 4.95 c.c. of 0.1 N sodium hydroxide solution were required, but when 10 c.c. of boric acid were used 9.7 c.c. were found, indicating that with this quantity re-precipitation is necessary. Genuine milk treated as above requires no 0.1 N sodium hydroxide solution.

The same method may be applied to preserved cream and cake, 10 grms. being taken, or more if the proportion of boric acid is small. With liquid eggs it is convenient to heat gently about 2 grms. with 50 c.c. of 1.5 N alcoholic sodium hydroxide in a covered dish for 2 hours, then to evaporate to dryness and ignite as before. Sausages may be treated similarly, about 10 grms. being used.

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J. F. LIVERSEGE.
H. H. BAGNALL.

BORIC ACID IN CAKE.

IN reference to the Circular, No. 381, by the Ministry of Health (20-3-23), "Boric Acid in Cake," I would like to draw the attention of those of our members who are interested in the question to a somewhat startling experience which we have had recently arising out of the Circular in question, for, as is known, the Bakery and Allied Traders' Association gave a definite undertaking as to the boric acid content of imported whole egg and/or yolk; hence, on examining certain cakes, the boric acid found rather pointed to a breach of the above undertaking.

The cakes in question were various varieties of so-called fruit cakes, but, on picking out the currants, sultanas, raisins, dried cherries, etc., and separately testing them, we were much perturbed at the boric acid content of these dried fruits.

Further purchases of these articles, *per se*, in the ordinary shops, have shewn boric acid contents which would rather cause me to believe that we are dealing with something more than a natural constituent. This is purely a preliminary note to a detailed paper upon the subject, which is well in hand, and which we hope to present in due course to the Society, as it certainly opens up a rather wide field in (possibly) a hitherto unexplored region.

G. RUDD THOMPSON.

NEWPORT, MON.

THE RATE OF LOSS OF STRENGTH OF SPIRITS KEPT EXPOSED TO THE AIR.

In many cases when proceedings are taken for selling diluted spirits the defendants plead that the loss of alcohol is due to evaporation owing to the spirit being left exposed to the air.

In a recent case where this excuse was pleaded, the spirit had been kept in a small china barrel, the bung hole being covered with a piece of cardboard. The sample was found to be 37·5° under proof at 15·5° C. With a view to testing how rapid such loss of alcohol might be, 250 c.c. of whisky, of exactly 34·1° U.P. strength, were kept in a flask, with the mouth loosely covered with a cardboard cover, at room temperature, which did not exceed 15·5° C. throughout. The following results were obtained:

Length of time Days.					Strength of spirit at 15·5° C.
0	34·1° U.P.
1	34·5° "
2	34·9° "
3	34·9° "
4	35·1° "
21	37·1° "

In 21 days the spirit had fallen in strength 3°, an average of about 0·15° per day. The loss, no doubt, would vary with temperature and humidity of the atmosphere, but the above figures give some indication of what may be expected under normal conditions of temperature.

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DEVICE FOR STOPPING A LEAK IN A WATER JACKET.

With the Haldane apparatus for air analysis, trouble is sometimes experienced on account of the water jacket developing a leak. The jacket consists of a glass cylinder closed at the bottom with a cork. Even a very marked leak can be immediately stopped by pouring in mercury to a depth of about $\frac{3}{8}$ " ; this can be done without obscuring the graduations of the air burette. Presumably this method would be equally effective with any similar water jacket.

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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.

LEAD STANDARDS FOR CREAM OF TARTAR.

ON December 14 the Birkenhead and District Co-operative Society were summoned for selling cream of tartar not of the nature, substance and quality demanded, since it contained 44 parts of lead per million.

The Inspector admitted, in cross-examination, that he had asked for and received cream of tartar.

Mr. H. E. Davies, the Public Analyst, said that he always worked upon the B.P. standard.

Mr. N. G. Goldie, for the defence, contended that there were two standards for cream of tartar,—namely, one for the purified product (obtainable from Pharmacists), and the other for the ordinary commercial article, which could be purchased from grocers for use in cooking and other purposes.

Mr. W. H. Roberts, City Analyst for Liverpool, stated that there was a distinct difference between the two qualities. Genuine cream of tartar was supplied in this case, and the amount of lead was not injurious to health. He would not condemn a sample containing less than 100 parts per million as one likely to cause injury to health.

The Bench held that the inspector had received, not what he had expected, but what he had asked for. As they had been asked by the prosecution to fix a standard, they considered that cream of tartar containing up to 50 parts of lead per million was safe for ordinary commercial purposes.

The case was dismissed.

LEAD IN CREAM OF TARTAR.

It will be generally admitted that the advance in methods of manufacture, as well as in our knowledge of physiological chemistry, makes it desirable that standards of purity for articles used in food should be, at least, maintained—certainly not lowered. The decision of the Birkenhead Bench in a recent case of lead in cream of tartar is regrettable, in that a lax standard of purity is set up for this article as the result of divergence of evidence by two Public Analysts.

In the discussion on Tatlock and Thomson's paper (*ANALYST*, 1908, 33, 173), C. A. Hill pointed out that 0.002 per cent. was quite a reasonable maximum figure, and that no difficulty was found in obtaining that degree of purity. This is in agreement with the writer's experience in the examination of a large number of trade samples. Even second-grade, 98 per cent. cream of tartar usually only reached 0.002 or 0.0025 per cent. This was 16 years ago, and methods of manufacture are surely not worse to-day.

The standard suggested in Dr. MacFadden's Report to the Local Government Board in 1907 is that arrived at years before by agreement between manufacturers, users and analysts, namely, 0.002 per cent., and this is for the commercial article. It is noticeable that the same figure was adopted in the B.P. 1914 for "purified cream of tartar," so that as far, at least, as lead is concerned,

no distinction is to be drawn between the purified article and the commercial product. This is only reasonable, in view of the fact that cream of tartar is used almost exclusively for human consumption, either in food or medicine.

Apart from the merits of any particular case, it is unfortunate that a standard should be set up which differs from the well-known and widely-accepted figures suggested by official reports, without the hearing of evidence from disinterested parties, manufacturers, users and chemists.

H. E. Cox.

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11, BILLITER SQUARE, E.C.3.

GOLDEN SYRUP WARRANTY.

CULVER *v.* SANDERS.

ON January 16th the Divisional Court (the Lord Chief Justice and Justices Avory and Greer) heard an appeal by the respondents against their conviction by the Croydon justices (*cf.* ANALYST, 1923, 48, 544).

Mr. Levy, for the appellants, said that it was admitted that the article sold was not golden syrup, and no doubt contained more glucose and less cane sugar than it should have done. It was sold in tins on which were the words "Amolco, an ideal food for all uses for which sugar is used . . . ; is a nutritious and delicious golden syrup containing a high percentage of sugar."

The tins were supplied from the American packers under a contract contained in a sale note "We have this day sold you 100 cases each containing 36 tins Amolco brand pure cane syrup."

Counsel pointed out that pure cane syrup was a trade name for, and was synonymous with, golden syrup, and he contended that the sale note and label contained a written warranty that the tin contained golden syrup, and that therefore the defendants ought not to have been convicted.

The Court dismissed the appeal without calling on counsel for the respondents. Mr. Justice Avory said that the sale note and label did not agree with each other. The sale note was a sale of tins labelled "Amolco" brand pure cane syrup, and that could not be treated as a warranty that what was being sold was pure golden syrup. As there was no warranty under the Act, the justices were right in the conclusion to which they came.

Department of Scientific and Industrial Research.

REPORT OF THE FOOD INVESTIGATION BOARD FOR THE YEAR 1922.*

THE outstanding event of the year was the beginning of work at the Low Temperature Research Station at Cambridge, and already this station has begun to fulfil its double purpose of acting as an instrument of research for the various Committees of the Board and of taking its part in the general scientific work of the University of Cambridge.

* Copies of the Report can be obtained from H.M. Stationery Office, Imperial House, Kingsway, W.C.2. Price 1s. 6d. net.

As a result of apples in this country exhibiting "brown heart" disease a small expedition was requested to go to Australia, and left early in 1923 to investigate the gases in ship's holds in which apples were stored.

The several Committees (I-VII) have continued their work during the year, and the Report deals with the results obtained in some detail.

SECTION I.:—THE THEORY OF THE FREEZING OF TISSUES.—An examination into the critical rate of cooling for beef and certain fish was interrupted by the discovery that in the case of eggs preservation does not depend upon the critical rate of cooling being exceeded, but upon the rate falling within certain limits, and these experiments have necessitated a new outlook on the whole question, and have completely upset the accepted theory of freezing of tissues.

SECTION II.:—FISH PRESERVATION COMMITTEE.—Work was chiefly directed to the investigation of certain superficial defects developed by fish on storage with brine freezing, such as the appearance of a central opaque white patch in the eye and a brown colour in the gills. Further points to which attention was directed were the brine freezing of white fish and the effect of the composition of the brine upon the storage properties of the fish.

SECTION III.:—MEAT COMMITTEE.—Work on carbohydrate and nitrogenous autolysis was carried on at "chilling" and cold storage temperatures. The question of the origin of small quantities of succinic acid in muscle is being investigated and a method for the estimation of the succinic acid has been evolved, there being some indication that during storage the quantity of acid rises to a maximum and then falls off. The study of the colour changes developing when meat is frozen in brine was continued and the conditions governing the change of oxy-hæmoglobin to methæmoglobin ascertained. A full report on work carried out on the conditions governing the growth of bacteria will shortly be ready.

SECTION IV.:—ENGINEERING COMMITTEE.—Two problems received from the Fruit and Vegetables Committee received attention. (1) The question of adequate cooling of stores suitable for the small grower, and (2) the leakage of carbon dioxide through the walls of stores in which the method of gas storage is employed. In connection with these problems the porosity of a large number of materials was tested, and tests are still being carried out. The work on the thermal properties of various insulating materials has been continued and a report for ethyl chloride prepared. The "time lag" of thermometers under cold store conditions has been investigated and the testing of a combined hair hygrometer and dew point apparatus has led to the conclusion that the dew point apparatus is an efficient check on the hair hygrometer (precautions being taken to guard against draughts), and that the latter will keep its calibration for several months to within 2 to 3 per cent., provided changes of temperature and humidity are not too great. Experiments on the relationship of density of packing of slag wool, and heat conductivity established a well-defined minimum value for the thermal conductivity.

SECTION V.:—FRUIT AND VEGETABLE COMMITTEE.—Experiments on the cold storage of home-grown apples were continued; amongst other results the effect of the type of soil in which the trees were grown upon the storage life of the apples was confirmed; the "age of tree" factor was found to be unimportant; oiled wrappers were successfully used; 1° C. was again found to be the most satisfactory cold store temperature. Strawberries and loganberries can be held in cold store in good condition for market for at least 10 days, and in the case of apples in the "gas" store at Histon the average gross sale price after storage was 2.34 times the gross cost of the apples when received for storage. In this store a great variation in the keeping qualities of apples from different localities was noticeable, and the use of oil wrappers was found to be essential.

Work on the respiration of apples is being directed to correlating the respiration-age relationship and soil effect upon storage life. Knowledge of the various apple diseases has been considerably advanced, and there is evidence that the openings of the minute tubes containing air (utilised in the respiration of the fruit) are the points where many infective diseases begin. The chemistry of ripening continued to receive attention, particularly as regards the sugar content of apple juice, its acidity, the development of pectin on storage, and the parasitic fungi of stored apples.

SECTION VI.:—OILS AND FATS.—Since, with the exception of the *a*-methyl ether, the methyl ethers of glycerol have not been characterised, and they were needed as reference compounds in the study of the products of fat hydrolysis, their preparation was proceeded with. Work on oleic acid was continued, and attempts were made to synthesise naturally occurring higher unsaturated acids (oleic, tariric) by methods allowing of no ambiguity as to the position of double bonds. At the Lister Institute the study of conditions affecting the formation of fat by the yeast cell has been continued, and information as to the condition in which fat occurs in the vegetable cell has been gained by seeing how far the fat could be removed without hydrolysing the cell substances.

SECTION VII.:—CANNED FOODS COMMITTEE.—The method used for estimating the amines produced by the action of bacteria upon fish depends upon the action of sodium nitrite in acid solution, the titration being carried out in the presence of alcohol. Inorganic phosphate in aqueous extracts of herring was estimated by a preliminary precipitation of protein with dilute hydrochloric acid followed by the magnesium citrate method applied to both filtrate and washings; or by addition of magnesium citrate mixture, solution of the precipitate of magnesium phosphate with part of the protein in hydrochloric acid, addition of alcohol to the extent of 80 per cent., filtration of the re-precipitated protein, and application of the magnesium citrate method to the filtrate and washings.

A preliminary study of the changes occurring during cooking and storing of herrings seems to show that the presence of tin does accelerate the production of simple breakdown products, especially during cooking.

An appendix gives the list of publications during 1922-23, comprising Special Reports and Papers published in Scientific Journals.

D. G. H.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Proteins of Milk-Whey. Their Separation by the Acetone Method.

M. Piettre. (*Comptes rend.*, 1924, 178, 333-335.)—The acetone method, previously applied to blood serum (*Comptes rend.*, 1920, 170, 1466; 1924, 178, 91), is employed to separate the proteins of whey as follows: The casein is precipitated in fine flocks by shaking the whey vigorously at about 35° C., and adding, drop by drop, a moderately concentrated solution of commercial rennet. After filtration by means of suction, the whey is treated with 0.7 c.c. of *N*/100 hydrochloric acid

per c.c. and is then carefully exhausted with ether without excessive emulsification of the mixture. The clear, decanted liquid is then treated at about 0° C. with 1.35 times its volume of cooled acetone and almost immediately filtered with the aid of the pump. The residue on the filter is moistened and removed to a dish with a volume of water equal to about one-tenth of that of the whey treated. After 12 hours any lumps are crushed and the well-cooled mixture rendered homogeneous by shaking it with ether. Centrifugation then separates a clear, pale liquid, containing the lactalbumin, from the whitish insoluble protein, which may be washed by centrifugation once or twice with water saturated in the cold with ether. The white flocculent matter, occurring to the extent of about 2.3 grms. per litre of milk, resembles the globulins in its physical characteristics and its chemical reactions, although, like casein, it always contains inorganic matter, largely calcium phosphate; in alkaline solution its specific rotation is -31.2° .

The lactalbumin is purified by either complete or fractional precipitation with 96 per cent. alcohol, 3.2 grms. being obtained per litre of milk; it has $[\alpha]_D -41.23^\circ$, and coagulation commences sharply at 67–70° C. and extends to 76–78° C.

T. H. P.

Estimation of Alcohol in Bread. T. Sundberg. (*Ann. Falsificat.*, 1923, 181, 531–534.)—About 200 to 300 grms. of bread are cut into small cubes and immediately placed in a two-litre round-bottomed flask connected with a condenser and standing in a calciumchloride bath. The temperature of the bath is raised to 100°–105° C., and a current of steam is passed through the flask till about 100 c.c. have been collected. Another receiver is placed in position, the calcium chloride solution heated to 110°–115° C., and a further 100 c.c. distilled. The specific gravity of both distillates is determined and the alcohol content deduced from the table of K. Windisch. If the distillates show separation of a thin film of oil, they are filtered through a damp filter-paper. If they are at all acid, they are neutralised with 0.1 *N* sodium hydroxide solution (solid phenolphthalein as indicator), and redistilled through a fractionating column. Any acetaldehyde counts as alcohol, but this error is counteracted by the presence of small amounts of furfuraldehyde. Dry bread contains practically no alcohol, and of the numerous varieties of fresh bread mentioned, Stockholm French rolls contained 0.1 to 0.2 per cent., malt bread 0.2 to 0.5 per cent., and samples of "pain aigret de Kalmar" 0.6 per cent. of alcohol by weight.

R. F. I.

Use of Essential Oil of Mustard as a Preservative for Wine. P. Malvezin and G. Bidart. (*Ann. Falsificat.*, 1923, 181, 534.)—The essential oil of black mustard seed (b.pt. 150.7° C.) is soluble in alcohol and ether, but in water only to the extent of 1.16 grms. per litre at 15° C. Its antiseptic power, when used in wines, is such that one c.c. of a one per cent. solution added to 1 litre of wine is efficient, but the amount required varies with certain factors, such as the amounts of sugar and sulphurous acid in the wine, the quantity and vitality of any yeasts present, and the temperature. The souring of red wines, which generally contain no sugar, can be stopped by the addition of 0.8 c.c. of the one

per cent. solution above mentioned. Essential oil of mustard is about 200 times as active as sulphurous acid and does not affect the odour or flavour of the treated wines. It does not volatilise from the wine, and therefore does not require to be renewed. It has no action on the colouring matters of red wines, fruit juices, caramel, etc.

It is estimated by heating 5 c.c. of the preservative on a water bath for one hour with 10 c.c. of ammonia solution and 55 c.c. 0.1 N silver nitrate solution in a graduated 100 c.c. flask connected with a reflux condenser. The solution is cooled, diluted to the mark, filtered from the precipitate, and the silver estimated volumetrically in 50 c.c. of the filtrate.

R. F. I.

Protopectin and some other Constituents of Lemon Peel. R. Sucharipa. (*J. Amer. Chem. Soc.*, 1924, 46, 145–156.)—The so-called pectins of fruits are of three kinds, *viz.* free pectin, which is water-soluble and of great jelly-making power, protopectin a pectin-cellulose compound insoluble in water, and pectic acid which is the product of the hydrolysis of the other two. Free pectin is the fully methylated ester of pectic acid. Albedo, the white material from lemon peel, after extraction of the essential oils, resins, etc., with petroleum spirit, is composed almost entirely of the three pectins with hemicelluloses and cellulose. After extraction with alcohol and ether, the free pectin in the dry powdered residue is extracted with water, the cellulose is removed by repeated treatment with Schweitzer's reagent, and the residue, after washing out the copper with dilute acetic acid, is then protopectin. It is suggested that in protopectin the cellulose is linked with the pectic acid by carboxyl groups. This complex is split up by heat, the cellulose being replaced by methoxyl groups; this is essentially the ripening process. The heat of the sun hydrolyses the protopectin, forming water-soluble pectin, which renders the whole of the tissue soft.

H. E. C.

Estimation of the Extract from Tea. R. E. Andrew. (*J. Assoc. Off. Agric. Chemists*, 1923, 7, 154–157.)—The following procedure has been favourably reported on by a number of analysts, and is recommended for adoption as the official method for estimating the water-extract in tea: Two grms. of the ground sample are treated with 200 c.c. of hot water in a 500 c.c. measuring flask closed with a rubber stopper, through which passes a glass tube 30 inches long as condenser. The liquid is boiled gently, so that no steam escapes from the tube, for an hour, with occasional rotation of the flask. The cooled liquid is afterwards made up to volume, mixed, and filtered through a dry filter paper, 50 c.c. of the filtrate being evaporated to dryness over a steam-bath and the residue dried in an oven at 100° C. for an hour, cooled, and weighed.

T. H. P.

Composition of Commercial Mustard Brans with Special Reference to the Detection of Added Mustard Bran in Prepared Mustard. R. Hertwig and J. I. Palmore. (*J. Assoc. Off. Agric. Chemists*, 1923, 7, 170–173.)—The figures given below show that the compositions of commercial mustard brans, comparatively free from inside seed materials, differ markedly from those of whole

seed; such difference is accentuated by the ratios given. The proposed ratios between nitrogen, crude fibre, total phosphoric acid, calcium oxide, and magnesium oxide should be of valuable assistance in the detection of small quantities of added mustard bran in prepared mustard in the many instances when the crude fibre alone does not show its presence.

The limiting percentages and ratio values, calculated on moisture-free and fat-free material, are: (1) For the seeds: (a) Nitrogen, 6.42-8.35; (b) crude fibre, 7.20-11.10; (c) total P_2O_5 , 2.28-4.40; (d) CaO, 0.734-1.734; (e) MgO, 0.616-1.326. The ratio, (f) N: crude fibre, 0.56-1.00; (g) P_2O_5 : crude fibre, 0.28-0.49; (h) MgO: crude fibre, 0.07-0.16; (i) CaO: MgO, 0.79-1.71; (k) CaO \times crude fibre: $P_2O_5 \times N$, 0.26-0.78; (l) CaO \times crude fibre: $P_2O_5 \times N \times MgO$, 0.21-0.88. (2) For mustard brans, the corresponding values in order have been found to be: (a) 2.70-5.92; (b) 14.1-29.2; (c) 0.40-2.42; (d) 0.862-1.910; (e) 0.266-0.792; (f) 0.09-0.42; (g) 0.01-0.17; (h) 0.01-0.06; (i) 1.2-7.2; (k) 1.3-52.3; (l) 1.6-19.66. T. H. P.

Salad Dressing. A. E. Paul. (*J. Assoc. Off. Agric. Chemists*, 1923, 7, 138-140.)—A salad dressing composed of cottonseed, arachis and olive oils, vinegar, mustard, sugar, salt, egg yolks and boric acid was analysed by four analysts, estimations being made of the total solids, the reducing sugars, before and after inversion, sucrose, total and volatile acids, oil and lecithin- P_2O_5 , by the methods given by Lepper (*ibid.*, 1921, 5, 248). The approximate egg-content was calculated from Jack's figure 0.1316 gm., for the amount of lecithin- P_2O_5 in the yolk of an average egg. The results obtained by the four analysts show that the methods used are quite satisfactory. T. H. P.

Baudouin's Test for Sesame Oil. C. O. Gravenhorst. (*J. Ind. Eng. Chem.*, 1924, 16, 47-48.)—The coloration given by genuine sesame oil in the Baudouin test depends, to some extent, on the origin of the oil; the intensity of the coloration is affected considerably by a decrease in the strength of the hydrochloric acid used, the strongest reaction being obtained with an acid of sp. gr. 1.190 to 1.197. The presence of a moderate amount of free chlorine in the acid is without influence on the test. Whilst rancid sesame oil will give a normal reaction with the test, a mixture of sesame oil with arachis oil or butter ceases to give a coloration when it has become badly rancid. If, however, the rancid mixture is washed with warm 5 per cent. sodium hydroxide solution and then dried, it will yield a normal coloration when subjected to the test. W. P. S.

Constituents of Pecan Oil. P. D. Boone. (*J. Ind. Eng. Chem.*, 1924, 16, 54-55.)—Pecan oil is obtained from the edible nuts of a hickory tree (*Carya olivoeformis*) growing in Florida, Georgia and Alabama; the oil has a light yellow colour and a pleasant odour and taste, and its characteristics are as follows:—Sp. gr. at 20°/20° C., 0.9118; saponification value, 191.5; acid value, 0.80; iodine value (Hübl), 97.1; refractive index at 20°/20° C., 1.470. The composition of the oil is approximately:—Oleic acid (as glycerides), 80; linolic acid (as glycerides), 16; palmitic and stearic acids, 4 per cent. Phytosterol, m.p. 134° C., is also present. W. P. S.

Glycerides of Lard. C. Amberger and A. Wieseahn. (*Zeitsch. Unters. Nahr. Genussm.*, 1923, 46, 276-291, 291-299.)—I. The fatty acids of lard, prepared from different parts of the pig, were first separated in the usual way into solid and liquid portions, and found to consist of stearic acid, 7·8; palmitic acid, 32·2; and oleic acid, 60·0 per cent. Then, in order to find the constitution of the glycerides, their separation was effected by repeated fractional crystallisation. The first stage consisted in solution in warm ether, followed by cooling and separation of the soluble and insoluble portions, which were then fractionally crystallised from alcohol, the progress being followed by the estimation of iodine value and m.p. These fractions were collected into four distinct groups, which were again separated by dissolving them in acetone and cooling the solution to 0° C., when two layers form, the upper one containing the more unsaturated glycerides. From the constants of the various fractions and comparison of them with those of known glycerides it is deduced that the unsaturated glycerides present in pure lard fat include oleodistearin of m.p. 42° C., oleopalmitostearin of m.p. 41° C., and palmitodiolein, which is a colourless oil at summer temperature, and that triolein may be present, although not in all samples. The percentage composition of fat from the omentum was calculated to be: Palmitodistearin, 3; steardipalmitin, 2; oleodistearin, 2; oleopalmitostearin, 11; and palmitodiolein, 82.

II. Pure glycerides prepared by known chemical methods, the details of which are given, were compared as to their properties and constants, and those of some of their products of hydrogenation, with the glycerides separated from lard by the foregoing methods. From such comparison it is concluded that the configuration of the glycerides is:— β -palmito- α - α -distearin, β -stearo- α - α -dipalmitin, α -oleo- α - β -distearin, α -palmito- α - β -diolein, and probably β -oleo- α -palmito- α -stearin.

H. E. C.

Acids of Beeswax. A. Gascard and G. Damoy. (*Comptes rend.*, 1923, 177, 1222-1224.)—In view of the confusion existing in the literature with reference to the acids present in beeswax, the authors have investigated this question by saponifying the wax, exhausting the calcium salts of the acids with boiling alcohol, and separating the free acids by fractional crystallisation from alcohol of different strengths at definite temperatures, the solutions being filtered at each temperature. This procedure yields four acids:—(1) Neocerotic acid, $C_{25}H_{50}O_2$, melting at 77·8° C. (*cf. Marie, Bull. Soc. Chim.*, 1896, 15, 510); (2) cerotic acid, $C_{27}H_{54}O_2$, melting at 82·5° C., identical with that furnished by Chinese wax; (3) montanic acid, $C_{29}H_{58}O_2$, melting at 86·8° C., apparently identical with the acid of this name extracted from montan wax (*cf. Tropsch and Kreutzer, Brennstoff-Chem.*, 1922, 3, 49, 177, 193, 212); (4) melissic acid, $C_{31}H_{62}O_2$, melting at 90° C.

T. H. P.

Alcohols and Hydrocarbons of Beeswax. A. Gascard and G. Damoy. (*Comptes rend.*, 1923, 177, 1442-1443.)—By prolonged saponification of beeswax with excess of potassium hydroxide, the authors have obtained the following alcohols and hydrocarbons, corresponding with the acids obtained (preceding abstract). *Alcohols*:—Neoceryl alcohol, $C_{25}H_{52}O$, melting at 75·5° C.; ceryl

alcohol, $C_{27}H_{56}O$, melting at $80^{\circ} C.$, already found in Chinese wax; montanyl alcohol, $C_{29}H_{60}O$, melting at $84^{\circ} C.$, and myricyl alcohol, $C_{31}H_{64}O$, melting at $87^{\circ} C.$; the second and fourth of these alcohols are obtained in largest proportions. *Hydrocarbons*:—Pentacosane, melting at $54-54.5^{\circ} C.$; heptacosane, melting at $59.2-59.5^{\circ} C.$; nonacosane, melting at $63.5^{\circ} C.$; and hentriacontane, melting at $68.4-69^{\circ} C.$

T. H. P.

Note on Podophyllin. D. B. Dott. (*Pharm. J.*, 1923, 57, 661.)—In view of the fact that the resins of *Podophyllum peltatum* and *Podophyllum emodi* have been stated to possess similar pharmacological properties, and both to be soluble in B.P. ammonium hydroxide solution, their behaviour with 25 c.c. of such a solution was examined by macerating 0.25 gm. of each resin for half an hour, filtering, washing the residues with 25 c.c. of water and weighing. In the case of *Podophyllum peltatum* there remained 3.6 per cent. of residue, and of *P. emodi*, 40.4 per cent. Half a gm. of two other samples, when treated, respectively, for 15 minutes each with 15 c.c. of ammonium hydroxide solution diluted with 15 c.c. of water, filtered, and the residues washed with 30 c.c. of water, gave the following amounts of insoluble matter:—*P. peltatum*, 6.0; and *P. emodi*, 47.2 per cent. Treatment with ammonium hydroxide solution thus affords a rapid method for distinguishing the two resins, but an examination of a large number of genuine samples would be necessary before limits of solubility could be definitely stated. On treating 0.5 gm. of each of the two resins for 10 minutes with 10 c.c. of acetone, and washing the filtered residue with a further 10 c.c., 8.2 per cent. of insoluble matter remained in the case of *P. peltatum*, and only 2.0 per cent. in that of *P. emodi*. It would hardly appear likely that substances differing so substantially in their properties would have a similar therapeutic value.

D. G. H.

Colour Test for Chloroform and Chloral Hydrate. J. H. Ross. (*J. Biol. Chem.*, 1923, 58, 641-642.)—The red colour produced on heating chloroform, bromoform, iodoform and chloral hydrate with aqueous sodium hydroxide and pyridine may be used to identify these compounds; and, further, to identify substances which produce these compounds under given conditions and in amounts inferior to 0.005 mgrm. Piperidine and quinoline, when used instead of pyridine, do not produce this colour. The test may apply only to substances which contain the grouping $R-CHlg_3$ ($Hlg_3 = \text{halogen}$). The method is as follows:—Three to 5 c.c. of concentrated (17 to 25 per cent.) sodium hydroxide solution in a test-tube are covered with a layer 2 mm. thick of pyridine. A small quantity of the substance or a drop of the solution to be tested is added, and the contents of the tube are raised to the boiling point, with shaking to avoid bumping. If the colour has not appeared in a few seconds after boiling, the tube is shaken vigorously, and held until the pyridine layer has risen to the surface. The presence of chloroform, etc., colours the pyridine pink or deep red. P. H. P.

Chemical Examination of some Commercial Brands of Carbon Tetrachloride. L. E. Warren. (*Amer. J. Pharm.*, 1923, 95, 864-874.)—Carbon tetrachloride is now being extensively used for the treatment of hook-

worm, and it is important to ascertain the purity of the drug used. Since carbon tetrachloride is usually prepared by chlorinating carbon disulphide, free chlorine and carbon disulphide are likely impurities. The presence of free chlorine, however, is rare, but carbon disulphide, which possesses toxic properties, is frequently present and may be estimated as follows:—Five grms. of the sample are heated beneath a reflux condenser for 30 minutes with 20 c.c. of 0.5 *N* alcoholic potassium hydroxide solution; 50 c.c. of water and 5 c.c. of 20 per cent. alcoholic potassium hydroxide solution are then added, the mixture warmed till the carbon tetrachloride and alcohol have been removed, and an excess of bromine water gradually added. After warming for 15 minutes, an excess of barium chloride solution is added, and the barium sulphate formed filtered off, and estimated in the usual way. ($\text{BaSO}_4 \times 0.16308 = \text{CS}_2$.) Of 9 brands of carbon tetrachloride examined one contained no carbon disulphide, and the amount present in the others varied from 0.015 to 0.27 per cent. The specific gravity at 25°/25° C. varied between 1.5879 and 1.5897, and the boiling points lay between 75.9 and 76.4° C. The Council on Pharmacy and Chemistry for New and Non-Official Remedies require that medicinal carbon tetrachloride should be clear, colourless and mobile, of specific gravity not less than 1.588 at 25° C., and that it should boil between 76 and 77° C. On shaking 10 c.c. with 10 c.c. of water the aqueous layer should be neutral to litmus, give no opalescence with silver nitrate solution (*chlorides*), and, on addition of a few drops of potassium iodide solution, should not be coloured violet in 5 minutes (*free chlorine*). On warming 10 c.c. of medicinal carbon tetrachloride with 10 c.c. of 25 per cent. potassium hydroxide solution no yellow or brown colour should develop (*aldehydes*), and on mixing 10 c.c. with 10 c.c. of sulphuric acid and shaking occasionally for 5 minutes, not more than a barely perceptible trace of colour should develop in either layer (*organic impurities*). On evaporating 25 c.c. almost to dryness and leaving the remainder to evaporate spontaneously, the residue, if any, should be odourless and should not weigh, on drying, more than 0.001 gm. Not more than 0.1 per cent. of carbon disulphide, estimated as described above, should be present.

D. G. H.

Biochemical, Bacteriological, etc.

Solubility of Copper in Milk. F. E. Rice and J. Miscall. (*Inter. Rev. Sci. Pract. Agric.*, 1923, 1, 1095–1096.)—This work was carried out to find to what extent milk can act as a solvent of copper, since this metal was found in cows' milk. Some milk, after being left in contact with a strip of pure copper in a glass vessel, was evaporated to dryness, the residue was burnt, and the ash taken up with nitric acid. The copper that had passed into solution was estimated either electrolytically or by the ethyl-xantho-potassic method. If copper vessels are used in dairy work, they should be closed, since air and oxygen enormously increase the corrosive action of milk on copper; carbon dioxide does not affect it. A film

of oxide on the copper also increases the solubility; dairy utensils should be clean and not disinfected with chlorine compounds. Boiling appears to destroy or transform the factor upon which the solvent action of milk depends, and thus copper is least easily attacked by boiling milk. The addition of sugar (to about 18 per cent.) does not increase the action, nor does a slight amount of acid. Experiments with cream and whey showed that the metal is chiefly dissolved by the water. Theoretically and experimentally it appears that copper in solution in milk can be displaced by tin; therefore, condensed or evaporated milk kept in iron receptacles lined with tin will contain less copper than the original product as made in unlined copper utensils.

P. H. P.

Action of Alkaline Compounds on Insulin. E. J. Witzemann and L. Livshis. (*J. Biol. Chem.*, 1923, 58, 463-474.)—Tables of results are given showing the lability of insulin in the presence of alkali. Preparations were injected into rabbits that had been made to fast during the 18 hours previous to the injection. The blood sugar concentrations were determined before, and again 2 hours after, injection, unless the rabbits suffered convulsions, in which case blood was taken when convulsions were first observed. Results show that insulin is more or less completely inactivated at room temperature by 0.5 or 0.7 *N* ammonium hydroxide in the course of some days, and that its original activity is usually quickly restored on acidifying the ammoniacal solution with hydrochloric acid. Sodium and potassium hydroxides, even at 0.1 *N* concentration, inactivate insulin irreversibly at room temperature. Sodium carbonate and bicarbonate and disodium phosphate had hardly any action upon insulin. Exploratory experiments on the effects of mixed alkalis upon insulin indicate that the above reactions are subject to secondary accelerative and retarding effects depending on the mixture used. The nature of the chemical action of alkaline compounds and of ammonium hydroxide upon insulin are discussed. The reversible effect of ammonium hydroxide suggests a tautomeric re-arrangement, but nothing definite can yet be said as to the nature of the groups that would be involved in this re-arrangement.

P. H. P.

Vitamin C in Canned Foods. W. H. Eddy and E. F. Kohman. (*J. Ind. Eng. Chem.*, 1924, 16, 52-53.)—To determine the extent to which vitamin C is destroyed in the process of canning foods, fresh cabbages were packed in tins, the tins were then filled with boiling water, the air was exhausted, and the tins sealed and heated at 100° to 126° C. for periods varying from fifteen to sixty minutes. The results of subsequent feeding experiments with this canned cabbage indicated that the destruction of vitamin C is dependent on some factor other than temperature and time of heating and that, in any case, the process is less destructive than cooking in open vessels. Protection against scurvy was afforded by 4 or 5 grms. of the canned products when given to guinea-pigs, and, with smaller doses, the product heated at 115° C. was more effective than that which had been heated at 100° C. for the same length of time.

W. P. S.

Fat-Soluble Vitamins, XVI. H. Steenbock, J. H. Jones and E. B. Hart. (*J. Biol. Chem.*, 1923, 58, 383-393.)—The authors describe the preparation of ether extracts of saponified cod liver oil. When rats were fed with this material it was found that the vitamin *A* content had not been sacrificed, nor had the anti-rachitic vitamin been destroyed by the saponification and extraction. Some dogs were kept on a basal ration and, after several weeks, deformation occurred, and they had difficulty in walking, owing to maintenance of weight with growth in size and gradual decalcification of the skeleton putting great strain upon the skeleton. This deformation usually occurs with animals afflicted with rickets late in the course of the disease. Where the basal ration was supplemented with cod liver oil, saponified or untreated, growth was very uniform, and continued so until the experiment was terminated. Figures are shown of sections, taken through the thorax to include the fifth rib, at the end of the experiment. The sections of the dogs fed on the basal ration alone show several partly repaired fractures and enlarged costochondral junctures. Analyses for ash carried out showed that the growth itself of these dogs had actually been interfered with. A lowering also of the calcium and phosphorus content of the blood occurred in the basal animals. These dogs had severe tetanic convulsions, but these have been observed in dogs afflicted with rickets that had a normal calcium content of the blood. Apparently a decreased calcium content cannot *per se* be the cause of these convulsions. Therefore, from the standpoint of growth, calcification of bones and maintenance of normal calcium and phosphorus content of the blood, the ether extract of saponified cod liver oil is as efficacious as the untreated oil.

P. H. P.

Bacteriological Examination of Ground Beef, I. R. E. Hoffstadt. (*Amer. J. Hygiene*, 1924, 4, 33-42).—Different samples of meat were purchased from public markets (open and closed stalls) and from shops. Some were ground for the purchaser and others were ready for sale. They were examined by St. John's method as cited by Tanner (*Bacteriology and Mycology of Food*. Chapter XIII.), which was modified by using the mechanical shaker. The meat was shaken with sterile glass beads in sterile water and plated in ordinary media. The bacterial count of market samples showed a wider range than that of shop samples. The bacterial count of all samples showed much variation in relation to sanitary conditions, *i.e.* the condition of floor, counter and ice-boxes, and the organoleptic test. The total aerobic count or count of dextrose fermenters is unreliable as a standard for meat analysis. The presence of an aerobic form or group cannot be taken as an index to the freshness of the meat. There was no relation between initial counts and counts in subsequent spoilage of samples, and there was no relation between the percent-age of proteolytic aerobes and the type of spoilage. Colour and odour were two of the points considered when grading the meat as good or poor.

P. H. P.

Bacteriological Examination of Ground Beef, II. R. E. Hoffstadt. (*Amer. J. Hygiene*, 1924, 4, 43-51.)—From the results obtained in this work it may be concluded that the presence of proteolytic anaerobes affords a definite indication.

as to the keeping qualities of meat. The technique described for indicating their presence can be adapted easily for laboratory use. Anaerobiosis can be successfully obtained by layering with vaseline and the McIntosh and Fildes jar (*Lancet*, 1916, 1, 768) for incubation can be dispensed with. The method used in isolating the organisms is given, with full descriptions of six different types of anaerobic species represented by 110 cultures which were identified, *viz.*, 2 saccharolytic, 2 moderately proteolytic and 2 strongly proteolytic types. A definite type of spoilage indicates the presence of each group. There is no relation between the anaerobe present, the original organoleptic test, the sanitary conditions and the aerobic count, although each type of spoilage represents a special type of anaerobe.

P. H. P.

Bile Acids in Urine. C. L. A. Schmidt and J. A. Merrill. (*J. Biol. Chem.*, 1923, 58, 601-609.)—Results of repeated investigations on this subject are not in harmony. It is thought that bile acids are eliminated in the urine only in small quantities, and the question is raised as to the fate of these substances. To study the subject an accurate method for the quantitative estimation of the bile acids in urine is a necessary pre-requisite. The principle of the method of Foster and Hooper (*J. Biol. Chem.*, 1919, 38, 355), modified by Schmidt and Dart (*J. Biol. Chem.*, 1920-21, 45, 415) in conjunction with an observation made by Tengström (*Z. Physiol. Chem.*, 1904, 41, 210), has been used by the authors for their estimations. Their results indicate a recovery of 97 per cent. of the bile acid nitrogen from ox bile by precipitation of the bile acids by the addition of magnesium sulphate. They added a solution of the bile acids to urine and carried out experiments to determine the recovery of the acids. Whole human and ox bile were added in varying amounts to specimens of urine, and always more than 90 per cent. of the bile acids were recovered. They found that hippuric acid does not interfere with the estimations carried out by their method. An allowance for an error of 5 per cent. in the estimations should be made. Details and precautions are given. The bile acid output in the urine was estimated in some cases of jaundice; the values were extremely small. There was no constancy in the amounts eliminated by a particular person. The authors do not consider that the amounts in the urine represent the total production of the bile acids in the system.

P. H. P.

Organic Analysis.

Refraction Method for Estimating Oil in Paraffin Wax. R. E. Wilson and R. E. Wilkin. (*J. Ind. Eng. Chem.*, 1924, 16, 9-12.)—Five grms. of the paraffin wax are dissolved in 75 c.c. of ethylene dichloride, the solution is cooled to -18° C. and filtered at this temperature. The insoluble wax is washed with 75 c.c. of ethylene dichloride, cooled previously to -18° C., and the filtrate and washings are evaporated. The residue thus obtained, containing all the oil and a trace of wax, is transferred to a weighing bottle with the aid of a small quantity of ethylene dichloride, and the solvent is evaporated completely. A known quantity (about 0.2 c.c.) of a mixture of "mineral seal" and "ligature" oils

is then added to dissolve the wax, and the refractive index of the solution is determined at 25° C. Reference to a graph gives the percentage of oil in this solution and the quantity in the original wax is calculated. The mixture of mineral seal and ligature oils is prepared by mixing 45 per cent. of the former with 55 per cent. of the latter; the mixture has the same refractive index (1.4470) as paraffin wax, and any change in this value is due to the oil present in the residue. Six samples of refined paraffin wax examined contained from 0.08 to 0.28 per cent. of oil.

W. P. S.

Composition of Whale Oil. C. H. Milligan, C. A. Knuth and A. S. Richardson. (*J. Amer. Chem. Soc.*, 1924, 46, 157-166).—The composition of whale oil has been investigated by separating the solid and liquid fatty acids by Twitchell's modification of the lead salt and ether method (*ANALYST*, 1921, 46, 466), esterifying the separated acids, and then fractionally distilling the methyl esters. From the fractionation it is calculated that the esters of the solid acids were composed of myristic acid, 13.6; palmitic, 68.0; stearic, 16.8; and higher unsaturated acids, 1.6 per cent. The composition of the esters of the liquid acids corresponded to the following percentages:

Carbon content of fatty acid.	Per Cent.	Carbon content of fatty acid.	Per Cent.
14	2.7	20	20.2
16	15.4	22	15.4
18	42.7	24	2.8
		Unsaponifiable	0.7

From this and other data the approximate composition of whale oil fatty acids is calculated to be:

	Per Cent.		Per Cent.
C ₁₄ , Myristic	4.5	C ₂₀ , Unsaturated	16
C ₁₆ , Palmitic	11.5	C ₂₂ , Unsaturated	10
Palmitoleic	17.0	C ₂₄ , Unsaturated	1.5
C ₁₈ , Stearic	2.5	Unsaponifiable	0.7
Unsaturated (nearly all oleic)	36.5		

It is remarked that the mean molecular weight of the raw fatty acids was 277; complete hydrogenation should theoretically increase this only to 279.7, but in practice it was increased to 285.5 by hydrogenation at 217° C.

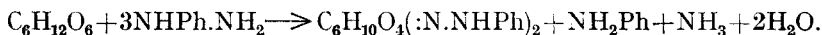
H. E. C.

Action of Catalysts with Non-drying Oils. A. G. Hill. (*J. Ind. Eng. Chem.*, 1924, 16, 23-24).—The presence of small quantities of iron salts in olive oil or "red oil" (commercial oleic acid) causes the oils to become so heated that they may be dangerous in textile factories. Similar effects are also produced by sodium, magnesium, zinc, and other salts, the most energetic action being produced by those metals of which the lower oxide is more stable than the higher oxide. A method of testing oils which may contain traces of metals consists,

briefly, in mixing 14 grms. of the oil with 7 grms. of ordinary bleached cotton wadding, and packing the mixture in a wire-gauze cylinder which is placed in a cylindrical copper tube surrounded by a hot water jacket. A current of air is drawn through the apparatus, and water in the jacket is kept boiling. A thermometer is inserted in the oil-wadding mixture, and any oil which shows a temperature exceeding 100° C. after one hour's heating, or 200° C. after two hours, should be regarded as dangerous and liable to produce spontaneous combustion. Of the metallic salts investigated, only those of aluminium, which forms but a single oxide, failed to act as catalysts in the reaction. In the case of zinc, the second oxide has been discovered comparatively recently. (*Cf. Mackey, J. Soc. Chem. Ind., 1896, 15, 90.*)

W. P. S.

Interaction of Glucose and Phenylhydrazine. E. Knecht and F. P. Thompson. (*J. Chem. Soc., 1924, 125, 222-226.*)—Quantitative study of this reaction showed a maximum yield of phenylglucosazone of 84 per cent.; this is obtained when 3.6 grms. of glucose, 6.48 grms. of phenylhydrazine and 20 c.c. of glacial acetic acid in a total volume of 100 c.c. are heated on the water bath for 3 hours. Analysis of the resulting mixture shows that the reaction proceeds in accordance with the empirical equation:—



For the analysis the uncombined phenylhydrazine in the mixture was estimated by collecting the nitrogen liberated on boiling with Fehling's solution (*Strache, Monatsh., 1892, 13, 299*), and the ammonia by distillation and titration with standard hydrochloric acid, litmus being used as indicator, because with it aniline salts react as free acid.

H. E. C.

Analysis of Naphthalenesulphonic Acids and Naphthalene. W. S. Calcott, F. L. English and F. B. Downing. (*J. Ind. Eng. Chem., 1924, 16, 27-30.*)—In the methods described the naphthalene residue is estimated by oxidising the sulphonic acids to derivatives of phthalic acid by means of an excess of vanadic acid dissolved in 70 per cent. sulphuric acid; the naphthalene is then calculated from the amount of potassium permanganate required to re-oxidise the reduced vanadium. Each molecule of sulphonic acid requires (for oxidation to sulphophthalic acids) nine atoms of oxygen, irrespective of the number and arrangement of the sulphonic acid groups. The sulphonic acid groups may be estimated by boiling a mixture of the free acids and sulphuric acid with barium carbonate, which converts the sulphuric acid into barium sulphate and the sulphonic acids into soluble barium sulphonates; the mixture is filtered and the barium in the filtrate is precipitated with dilute sulphuric acid. The resulting barium sulphate is collected and weighed, and the sulphonic acid sulphur is calculated from the result obtained. The relation between the naphthalene residue and the sulphonic acid sulphur gives the degree of sulphonation in the sample under examination. For the estimation of naphthalene in crude naphthalene, 12 grms. of the sample are

placed, together with 100 c.c. of 5 per cent. sodium hydroxide solution, in a round-bottomed 500 c.c. flask which is connected with a similar flask containing 100 c.c. of 10 per cent. sulphuric acid; this second flask is, in turn, connected with a 1 litre receiver, over which a stream of cold water is run, and with a 500 c.c. receiver surrounded by an ice bath. The mixture is steam-distilled for about twenty minutes, the distillation flask and the sulphuric acid flask being heated meanwhile to maintain the volumes of the solutions constant; the first receiver is then removed and another substituted, and the distillation is continued until nothing but oil separates in the receiver or until the receiver fills with a white acrid-smelling vapour. The naphthalene in the receivers is collected on a filter, the receivers are rinsed with 10 c.c. of alcohol; this is diluted with 200 c.c. of water and also passed through the filter; the naphthalene is washed with water from the filter into a beaker, the lumps are crushed, and the substance is again collected on a filter and pressed to remove as much water as possible. The naphthalene is then transferred to a thick-walled pyrex tube, of 110 c.c. capacity, 25 c.c. of concentrated sulphuric acid and 30 c.c. of 20 per cent. "oleum" are added, and the tube is closed tightly with a rubber stopper and heated at 100° C. for one hour. After cooling, the mixture is diluted with 250 c.c. of water, boiled for twenty minutes to expel sulphur dioxide, cooled, and diluted to 1 litre. Twenty-five c.c. of this solution are then oxidised with vanadic acid as described previously.

A method for the estimation of water in naphthalene depends on the change in the critical solution temperature of a standard toluene-water mixture after the addition of the moist sample.

W. P. S.

Estimation of Ferrocyanides. W. M. Cumming. (*J. Chem. Soc.*, 1924, 125, 240-243.)—The estimation of ferrocyanides by precipitation with copper or iron salts is unreliable, different results being obtained according as sodium, potassium or calcium ferrocyanide is used. A method which has been found to give accurate results with all three is based on the precipitation of benzidine hydroferrocyanide, $3C_{12}H_{12}N_2H_4FeC_6N_6.H_2O$, by benzidine hydrochloride in neutral solutions of the ferrocyanides. The method may be applied gravimetrically or volumetrically; in the former case the precipitate is washed, dried, and ignited to Fe_2O_3 , the factors 4.599, 3.799, and 3.649 being used to convert Fe_2O_3 into anhydrous potassium, sodium, or calcium ferrocyanide respectively. (The true formula of the crystalline form of the last-named salt includes 11 H_2O not $11\frac{1}{2}$ or 12 as quoted in text books.) The volumetric modification of the above process consists in titrating the ferrocyanide with a benzidine solution of known strength, using as indicator bromine water just decolorised by sodium carbonate and applied as follows:—When the reaction is nearly complete a drop of the mixture is spotted on filter paper, and a drop of the bromine water placed near by so that, on spreading, it touches the other ring; a yellow coloration at the juncture denotes the end-point. It is important to note that this colour is produced at the edge, as at the centre there will always be a yellow colour, owing to decomposition of the precipitate in the air. A satisfactory end-point cannot be obtained on titration in the reverse direction.

H. E. C.

Inorganic Analysis.

Use of Hypophosphorous Acid in Gravimetric Analysis. L. Moser and M. Niessner. (*Zeitsch. anal. Chem.*, 1923, 63, 240–252.)—*Mercury.* Solutions of mercuric chloride free from nitric acid are treated with an excess of 0.5 molar solution of hypophosphorous acid and 10 c.c. of strong hydrochloric acid, and heated on the waterbath. The metallic precipitate is left to settle, filtered off on tared paper, washed once with dilute hydrochloric acid, then with hot water, alcohol, and ether, dried at 30° C., and weighed in a closed weighing-bottle. Mercurous chloride and sulphate are suspended in water and heated with hypophosphorous acid; the nitrate must be reduced without addition of hydrochloric acid, otherwise some metal would be re-dissolved on heating. Mercury may be separated in this manner from iron, lead, cadmium, and zinc. *Gold.*—The chloride solution is treated with 2 grms. of sodium chloride, heated to incipient boiling, then treated with 0.25 molar hypophosphorous acid. The hot solution is allowed to stand till quite clear before filtration, addition of a little filter pulp being recommended. The precipitate is washed with one per cent. acetic acid till quite free from chlorine, and ignited. *Palladium.*—The feebly acid, boiling chloride solution is treated with an excess of the reducing agent and kept gently boiling for 5 minutes in the covered beaker. When flocculation is complete the hot liquid is filtered, and the precipitate washed with hot water and ignited. The estimation of mercury, gold, and palladium is very accurate. *Platinum* is not precipitated, and may be separated quantitatively from gold; the results are accurate with widely varying proportions of the two metals. On the other hand, the separation of platinum from palladium cannot be accomplished by means of hypophosphorous acid, as palladium catalytically induces the precipitation of platinum. The estimation of *copper* is unreliable; *bismuth* nitrate solution yields a precipitate consisting largely of phosphate, whilst a chloride solution is incompletely precipitated. W. R. S.

Volumetric Estimation of Antimony by means of Permanganate. O. Collenberg and G. Bakke. (*Zeitsch. anal. Chem.*, 1923, 63, 229–240.)—This method was submitted to a critical investigation; it was found to give correct results, equal to those of the bromate method, provided the acidity is kept within the limits of 10 to 19 c.c. of hydrochloric acid (sp. gr. 1.19) per 100 c.c. of solution. The permanganate solution is standardised against sodium oxalate. Agreement with gravimetric methods is attained only when the new atomic weight—Sb = 121.8—is used in the calculation (*cf.* ANALYST, 1924, 49). Petriccioli and Reuter's modification (dilution of the antimony solution with water until oxychloride is precipitated and solution of the precipitate in a minimum of hydrochloric acid, previous to titration) gives negative errors of 1.2 to 1.5 per cent.

W. R. S.

Interference of Cobalt in the Bismuthate Method for Manganese. G. E. F. Lundell. (*J. Amer. Chem. Soc.*, 1923, 45, 2600–2603.)—Cobaltous salts are oxidised by sodium bismuthate, and the oxidised compound reacts with both

ferrous sulphate and permanganate. The action on the latter does not start until some permanganate has been decomposed, after which the reaction proceeds rapidly; the action on the ferrous salt is much slower. The net result is that a low manganese value is obtained. Manganese can be estimated satisfactorily in presence of cobalt by oxidation with persulphate in acid solution, followed by titration with sodium arsenite.

W. R. S.

Estimation of Small Amounts of Molybdenum. Application to Ammonium Phosphomolybdate for the Indirect Titration of Phosphorus.

A. Vila. (*Comptes rend.*, 1923, 177, 1219–1221.)—Phosphorus may be rapidly estimated, even in amounts below 0.1 mgrm., by estimating volumetrically the molybdenum in ammonium phosphomolybdate. This, or other molybdenum compound, is reduced by means of hydrogen, this reaction yielding either metallic molybdenum or its dioxide, which, in presence of excess of molybdic acid, is converted quantitatively into the blue saline oxide, MoO_2 , 4MoO_3 . The ammoniacal solution of molybdic acid or ammonium phosphomolybdate is evaporated to dryness in a transparent silica test-tube; the deposit should cover not more than one-third of the length of the tube. A stream of pure hydrogen is passed through the tube, which is closed and introduced into an electric furnace heated to about 700°C . After the lapse of 15 minutes, the tube is withdrawn and allowed to cool with the hydrogen passing. For converting the residual metal or dioxide into the blue oxide, use is made of a solution prepared by dissolving 5 grms. of pure molybdic acid in 10 c.c. of 60 per cent. phosphoric acid, decolorising the solution exactly with potassium permanganate, and making up to 50 c.c. with water. This reagent is introduced in quantities of 2 c.c. into the tube, which is carefully heated, the molybdenum blue at first formed giving place to a more or less deep green syrupy liquid. This is allowed to cool and is then treated with water and boiled, the resulting molybdenum blue being titrated with $N/400$ potassium permanganate solution until the liquid is completely decolorised: 1 c.c. of this permanganate corresponds with 0.200 mgrm. Mo, 0.300 mgrm. MoO_3 , 0.00538 mgrm. P, or 0.0123 mgrm. P_2O_5 .

T. H. P.

Volumetric Estimation of Titanium. G. E. F. Lundell and H. B. Knowles. (*J. Amer. Chem. Soc.*, 1923, 45, 2620–2623.)—The authors find that, contrary to the generally accepted idea, solutions of titanous sulphate are easily and quantitatively reduced to the trivalent stage by the use of Jones' reductor (19 mm. bore, 43 cm. amalgamated zinc-column). The reductor was washed immediately before use with dilute sulphuric acid, and the receiver emptied and rinsed and filled with ferric sulphate solution (from 3 to 5 times in excess of the quantity required for the oxidation). There were passed through the reductor 25 to 50 c.c. of 3 to 5 per cent. sulphuric acid, 150 c.c. of titanium solution in dilute acid of the same concentration, another 100 c.c. of dilute acid, and, finally, 100 c.c. of water. The reduction was effected at a speed of 100 c.c. per minute. The reduced iron was titrated with 0.1 N permanganate standardised against

sodium oxalate. The errors averaged not more than one drop of standard solution. The reduction can be done at any temperature between 25° and 100° C., and it is unnecessary to expel air from the solutions used.

W. R. S.

New Reagent for Potassium. H. and W. Davies. (*J. Chem. Soc.*, 1923, 124, 2976-2982.)—Sodium 6-chloro-5-nitro-*m*-toluenesulphonate is proposed as a reagent for potassium. The solubility of the potassium salt, which forms characteristic voluminous scales, is 0.235 gm. in 100 grms. of water at 0°, and about 0.40 at 20° C. The test can be carried out in cold, strongly alkaline or in dilute mineral acid solution, and potassium can be detected in presence of large quantities of sodium and magnesium. Ammonium must be previously removed by boiling with sodium hydroxide. The reagent will detect one part of potassium in about 2500 of water. The reaction can be applied to quantitative analysis; the results of the analysis of potassium salts were within 0.5 per cent. of theory. The reaction is carried out in aqueous solution dilute enough to allow the potassium sulphonate compound to dissolve completely at boiling heat; the precipitate formed on cooling and standing is washed with a little water, and weighed; a considerable excess of reagent and very small volume of solution are aimed at. Sulphates and moderate amounts of aluminium, sodium, and magnesium, are not detrimental; but alkaline earths interfere, and must be eliminated. The potassium factor is 0.1347.

W. R. S.

Detection of Halogens. W. B. S. Bishop. (*J. Soc. Chem. Ind.*, 1924, 43, 23T.)—In testing for the presence of ethyl iodide in ether a piece of well oxidised copper gauze of such a size as to absorb about one c.c. is dipped into it. The gauze is then placed in a Bunsen flame, which becomes green if halogen is present. This test was found to detect 0.00004 gm. of iodine in one c.c. of ether, and is said to be more delicate than either that of Rübke or Lassaing. It will also detect 0.0001 gm. of bromine in one c.c. of ether.

R. F. I.

Apparatus.

Substitute for Platinum in Electrolysis. M. Bleesen. (*Zeitsch. anal. Chem.*, 1923, 63, 209-228.)—The author submits the results of test analyses in which cathodes of Borchers' alloy were used (Ni, 65-60; Cr, 30-35; Mo, 2-5; Ag, 0.2-1 per cent.). This alloy is rendered passive by immersion in strong nitric acid; the surface is made dull by sand-blasting so that adherent deposits are obtained. The best form of cathode is wire-netting or gauze. The deposit is removed from the cathode by a suitable acid in the cold; the loss in weight of the cathode hardly exceeded 0.0004 gm., and was generally smaller. The results submitted are satisfactory.

W. R. S.

Reviews.

ALLEN'S COMMERCIAL ORGANIC ANALYSIS. Fifth Edition, Vol. I. Pp. 796.
London: J. & A. Churchill. 1924. Price 30s. net.

The appearance of the first volume of a new edition of *Allen's Commercial Organic Analysis* (on this occasion the fifth), is an event. There is no other book in the English language that covers the ground covered by this book, or, for which, it may be added, analysts have so great an affection. It must be allowed that there is no chemist capable, by himself, of covering this large ground adequately, and that this is so is a compliment to the activities of chemists, not a slight on their industry. Consequently, this edition, as was the case with the last, is written by a number of experts in the various sections concerned, under the general editorship of S. S. Sadtler and E. C. Lathrop, as American editors, and C. A. Mitchell, as English editor.

The volume now under review (Vol. I.) deals with the same matters as did the corresponding volume of the 4th edition, published fifteen years ago.

The first section, named "Introduction," is word for word the same as it was in the previous edition (even to the mis-spelling of carbonyl chloride on p. 84); the only addition is a footnote concerning the xylene and petroleum distillation methods for the estimation of water content.

That this section has not been re-written is much to be regretted, for, during the past fifteen years, a number of important advances have been made in physical apparatus and the application of physical means to analysis, and, even when the last edition was published, this section was not particularly serviceable. Colorimeters and immersion refractometers of extended range fail to gain recognition, and there is no reference to hydrogen ion determination or to the large range of modern indicators and the use to which they can be put. Solvents would apparently stand in the same position as they did twenty years ago, which is far from the case.

If reasons of space have led to serious omissions from this section (the volume shews an increase of 200 pages over that of the 4th edition), the section could have been remodelled in order to make room for more useful matter. The chemist who uses "Allen" does not require minute directions to "turn a knob T," and so forth, in the use of a polarimeter, and a page devoted to the use, and a picture, of a separating funnel is too ample an allowance. There is much in this section, in fact, suitable only for a junior student, whilst much that is wanted by a practising chemist is omitted.

Later sections have, to a great extent, been re-written, and in them have been incorporated the later information and data appearing in the Supplementary Volume (Vol. IX.) of the last edition.

The section on the alcohols, revised by L. M. Burghart, has been extended, and deals mainly with ethyl and methyl alcohols. Acetone is included in this section, and certain higher aliphatic alcohols are briefly treated. The ethyl alcohol tables are those of the American Bureau of Standards.

It is agreeable to note that sections on Malt and Malt Liquors, on Wines, Cider, and Potable Spirits, are still permissible in "Allen." These are by J. L. Baker and L. Eynon respectively. They are largely re-written and include much new matter, new, that is, to this book. The former author contributes much from his own work. Particular wines and spirits are but lightly dealt with, this section being devoted chiefly to analytical methods of general applicability. A "British Government Laboratory Method" for the estimation of higher alcohols in spirits is given without any reference.

A short section on yeast by E. Schlichting follows.

Neutral alcoholic derivatives form another section, by H. Leffmann. Aldehydes, ether, esters, chloroform, chloral and so forth, are herein treated.

The important section on sugars has been revised by L. Eynon; sugars and some sugar products are dealt with at length, and glucosides, urine analysis as regards sugar, and pentoses receive a short treatment.

The starch section is by T. H. Pope, and deals with starches, cellulose, gums and cereals. Considering the importance of wheaten flour, the space given to it seems inadequate, especially as the flour industry is becoming so largely a chemical one.

E. Sutermeister contributes a section on paper and wood pulp, and the volume closes with a section on aliphatic acids by H. Schlatter. The acids herein treated comprise acetic acid (including vinegar), and certain homologues, oxalic, succinic, citric, tartaric and malic acids.

The index is, perhaps, adequate, though barely so; it is certainly not a generous one. This fault is partly compensated for by the fact that each section, in a way, provides its own index by means of headings in distinctive type. Short bibliographies are supplied at the end of each section.

A perusal of this volume leads to the conclusion that, in most respects, it is a better production, and a more useful one, than the corresponding volume in the last edition. Criticism, no doubt, there will be, criticisms which are partly an index of the great interest that this book has for English analysts. In these days of complexity it is a matter of difficulty to make a judicious selection of the matter that should be included or excluded, a difficulty that is increased by the endeavour to provide for the needs of analyst in both America and England. Commercial organic analysis, as distinct from "pure" chemistry, has often to be approached from different points of view in the two countries. English readers have cause for regret that this book is no longer an English production in its entirety, but, with that reservation, will welcome this new edition and be grateful for the enterprise that has led to it.

E. HINKS.

THE DETERMINATION OF HYDROGEN IONS. By W. MANSFIELD CLARK, M.A., Ph.D., Baltimore, U.S.A. Williams & Wilkins Company. Second Edition. 1923. Price \$5.50.

Since the publication of the first edition of this book (reviewed in *THE ANALYST*, 1921, 46, 70), it has become generally recognised that hydrogen ion concentration is an important factor in nearly all chemical reactions, and that, most particularly in the biochemical field, it is as essential that the P_H value should be controlled as that the temperature should be controlled. An increasing number of chemists have found it necessary to make themselves familiar with the fundamental theory of the subject, and to employ one or other of the methods of determination. For this purpose chemists cannot do better than go to this book, which gives a comprehensive description of the theory of hydrogen ion concentration, and treats fully of indicator and electrometric methods of determination. The theoretical part is intelligible to the ordinary reader, considering the present state of flux of the ion question. The new concept of hydrogen ion "activity" put forward by Lewis is mentioned, but the author wisely has made no attempt to introduce this into his general treatment of the subject—a decision which will be welcomed by the ordinary worker, who has no wish to change his standards of reference until something more definite has evolved out of the present confusion. The great expansion in the literature dealing with hydrogen ion concentration is shown by a comparison of the excellent bibliography at the end of the book with that in the first edition, and some idea of the width of the field in which hydrogen ion concentration has been studied may be gained from the chapter on "Applications." This expansion has necessitated a considerable increase in the size of the second edition, which now contains 480 pages as compared with 317 in the first edition. Many chapters have been practically re-written and new ones have been included.

One is glad to see the colour chart of indicators in its original form. Taking into account the difficulties in printing such a chart, it is remarkably well done, and of great value for rough indicator determinations of P_H in the laboratory.

The book is admirably printed, and the reviewer has not observed a misprint. It remains the best book in the English language dealing with hydrogen ion concentration.

NORMAN EVERS.

A TESTED METHOD OF LABORATORY ORGANISATION. By SEYMOUR PILE, M.A. (Cantab.), and REGINALD G. JOHNSTON. Pp. 96. London: H. F. & G. Witherby. Price 7s 6d.

Organisation, as applied to scientific work, has hitherto received but little attention in this country, and a brief account of a laboratory which had to be designed, furnished, staffed and organised, at express speed and during one of the most critical years of the war, should be both interesting and informing to the reader.

The writers, however, do not limit their attention to the merely mechanical details of equipment, methods of work and financial problems which arise in

connection with them. They treat also the psychological side of the chemist, the kind of being he is, or ought to be, the influences by which he is moulded, his virtues and failings, his relations to his colleagues, and the difficulties with which he has to contend when in contact with the lay mind.

After an introduction and preface, there follow four chapters dealing with the laboratory, the personnel, the treatment of the sample, and the methods of costing, after which, in three final sections, the writers "let themselves go" and discourse generally on, to use their own words, "the scientist in relation to this, that and the other."

All who own or use a laboratory cannot fail to be interested in the ideas which Messrs. Pile and Johnston have set forth, whether they agree or, as is equally likely, do not agree, as to details. Such points as the use of acid-proof paints, methods of ventilation and fume cupboards, sinks and drains, the width of benches and the illumination of balances, are all matters of interest and of contention.

The personnel of a large laboratory is a subject which gives rise to many problems which have hitherto received but scant attention. Here, too, the authors have ideas of their own which cannot be accused of being either academic or conventional.

Perhaps one of their most revolutionary suggestions is that the members of the staff should be of equal social standing, and this involves the abolition of the "bottle washer" and the "laboratory boy," who, say the authors, "break more glassware than their salaries will pay for." "The staff should clean up its own apparatus."

Greatly daring, they go further, and suggest that "all special knowledge," after a good secondary education, can be acquired in the laboratory and, while they do not minimise the value of academic training and degrees, they consider that the special training for the industrial scientific worker should be given in the works' laboratory itself.

It is impossible to touch upon the many other questions which the authors discuss, but it is safe to say that all readers of this book, whether they possess or control, or only work in a laboratory, will at least be brought to think about these problems, and cannot fail to be interested and stimulated by the clear and outspoken views set forth.

CECIL H. CRIBB.

ALCOHOLIC FERMENTATION. By ARTHUR HARDEN, Ph.D., D.Sc., F.R.S. London: Longmans, Green & Co. 1923. Price 6s. 6d. net.

The third edition of this work constitutes a most useful epitome of a subject of singular fascination alike to the investigator and the technologist. Before discussing Prof. Harden's contribution, the reviewer feels that he may be permitted to express the thanks of chemists to the author of the valuable series of Monographs on Biochemistry and to the publishers for issuing volumes at a reasonable price with a necessarily restricted sale. In these days of specialisation

the sub-dividing of bio-chemistry into branches and the publishing of monographs thereon is applicable not only to other fields of our science, but also to the technical side.

Prof. Harden provides an interesting historical introduction which, however, as in the first edition (1911), concludes with the general effect of Buchner's work on researches associated with the mechanism of fermentation. In a future edition this chapter might with advantage be amplified, and some indication given of how the author's own researches, together with those of C. Neuberg, H. Euler and A. Fernbach, have added to the historical structure.

A good account is given of zymase and its properties, and of the manner in which the co-enzyme and phosphorus fits into the machinery of fermentation. The rôle played by the yeast enzyme, carboxylase, which the reader will remember decomposes certain hydroxy and keto acids, among them being pyruvic acid, is lucidly described, and from this the author passes on to an account of the chemical changes involved in fermentation and to a discussion of the different theories which have been enunciated in recent years.

The chapter on by-products of fermentation will be read with interest by those chemists who have to deal with the analysis of potable spirits. The bearing of Ehrlich's classical work on the alcoholic fermentation of amino acids is of great importance to the technologist, for, as this chemist points out, "the great variety of the bouquets of wine and aromas of brandy, cognac, arrack, rum, etc., may be very simply referred to the manifold variety of the proteins of the raw materials (grapes, corn, rice, sugar, cane, etc.) from which they are derived."

In concluding his remarks on the mechanism of fermentation the author writes that "Buchner's discovery of zymase has opened a chapter in the history of alcoholic fermentation which is yet far from being completed. In every direction fresh problems present themselves, and it cannot be doubted that, as in the past, the investigation of the action of the yeast-cell will still prove to be of fundamental importance for our knowledge of the mode in which chemical change is brought about by living organisms."

A very full bibliography, which also functions as a name index, is not the least useful feature of the volume. Biochemists are indeed indebted to Prof. Harden for writing this monograph. It supplies a want, for the literature on the subject of fermentation is bewildering even to those zymotechnologists who are investigators in a modest manner.

JULIAN L. BAKER.

THE CHEMISTRY OF RUBBER. By B. D. W. LUFF, F.I.C. Pp. viii. +242. London: Ernest Benn, Ltd. 1923. Price 25s. net.

In his preface the author, who is the chief research chemist of the North British Rubber Co., Ltd., states that his aim has been to deal as fully as possible with the chemical principles involved in the cultivation, the collection and the manufacture of rubber; at the same time "consideration has been given to the

concomitant changes in physical properties accompanying the process of vulcanisation.”

During the past decade the study of the chemistry and physics of rubber and of the processes employed in the rubber industry has made rapid strides, and the literature of the subject has grown to such an extent that any work dealing more especially with modern principles and practice is to be welcomed, provided that the compilation has been carried out on sound lines. In the latter respect Mr. Luff may be regarded as having achieved success. His review of recent literature is distinctly good, and carries us almost up to the date of his preface—October, 1923.

The first seven Chapters (90 pages) of the book (if we except the opening historical review) deal with the sources, composition and properties of rubber latex and of crude rubber. Chapters VIII. to X. describe the vulcanisation process, the factors affecting vulcanisation and the properties of vulcanised rubber. Chapters XI. on “Compounding Ingredients” and XII. on “Accelerators,” will probably, by specialists, be regarded as extremely useful, for it is around these materials and their application that a large proportion of recent research has developed. The penultimate section constitutes a brief sketch of the manufacturing process, and the final Chapter (XIV.) is headed “Methods of Analysis.” To most analysts the latter will probably prove to be somewhat disappointing; to the expert, because Mr. Luff scarcely touches upon the important subject of the interpretation of the analytical and physical data obtained in the course of commercial work, and to the general analyst, because of its somewhat attenuated nature. The analysis of crude rubber is dealt with at rather disproportionate length, while the examination of vulcanised rubber, compounding ingredients and the like is treated very briefly. Notwithstanding such criticism of points of detail, Mr. Luff may be congratulated on having produced a work modern in its outlook and scientific in the handling; a book well worthy of taking its place in the library of all those interested in rubber. In conclusion, it may be stated that the printing and binding are exceptionally good, and that the 16 full plate illustrations are not only well chosen, but admirably reproduced.

PHILIP SCHIDROWITZ.

Publications Received.

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- PHARMACEUTICAL AND FOOD ANALYSIS. By A. THURSTON. London: Chapman & Hall. 1923. Price 21s. net.
- THE VEGETABLE PROTEINS. By T. B. OSBORNE. London: Longmans, Green & Co. 2nd Edition. 1924. Price 9s. net.
- THE CHEMISTS' YEAR BOOK, 1924. Edited by F. W. ATACK. Manchester: Sherratt & Hughes. Price 21s.