THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS

AN Ordinary Meeting of the Society was held at the Chemical Society's Rooms, Burlington House, on Friday, March 4th, the President, Dr. J. T. Dunn, being in the chair.

Certificates were read for the first time in favour of:—Edward Bertram Anderson, M.Sc., F.I.C., Edward Foster Eaton, Frank Maudsley, B.Sc., F.I.C., Samuel Gordon Stevenson, B.Sc., B.Pharm., F.I.C., and James William Thom, B.Sc.

Certificates were read for the second time in favour of:—Alan Arthur Douglas Comrie, B.Sc., A.I.C., Edwin William Stanley Press, B.Sc., A.I.C., and Muriel Roberts, B.Sc., F.I.C.

The following were elected Members of the Society:—Albert Green, M.C., M.Sc., Ph.D., F.I.C., John Farrar Hardwick, B.Sc., A.I.C., Ernest Stephen Hawkins, B.Sc., A.R.C.S., F.I.C., Joseph Robert Johnson, F.I.C., M.Inst.M.M., Arthur Pillans Laurie, M.A., D.Sc., F.R.S.E., and John Morgan Tucker, B.Sc., A.I.C.

The Annual General Meeting of the Society then followed, when the President, Dr. J. T. Dunn, delivered his Presidential Address.

The following were elected as Officers and Council for the year 1932:

President.—F. W. F. Arnaud.

Past Presidents, serving on the Council.—E. Richards Bolton, A. Chaston Chapman, J. T. Dunn, Bernard Dyer, Edward Hinks, P. A. Ellis Richards, G. Rudd Thompson, J. Augustus Voelcker.

Vice-Presidents.—H. M. Mason, G. W. Monier-Williams, G. Stubbs.

Hon. Treasurer.—E. B. Hughes.

Hon. Secretary.—G. Roche Lynch.

Other Members of Council.—A. L. Bacharach, H. E. Cox, E. M. Hawkins, A. E. Johnson, W. G. Messenger, H. H. Bagnall, W. T. Burgess, G. D. Elsdon, John Evans, L. Eynon, J. R. Nicholls.

Death.

WITH great regret we record the death, on March 17th, of Sir William Smith, who had been a Member of the Society since 1892.

Anniversary Dinner

THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS held a Dinner at the Trocadero Restaurant, on March 4th, to celebrate the 56th Anniversary of the foundation of the Society.

The members and their guests, numbering over 100, were received by the President, Dr. J. T. Dunn, F.I.C., and Mrs. Dunn, and Dr. Dunn afterwards took the chair at the Dinner.

The guests of the Society included the Rt. Hon. Lord Atkin, P.C., D.C.L. (Lord of Appeal), Councillor J. G. Nixon, J.P. (Lord Mayor of Newcastle-upon-Tyne), Sir Ernley Blackwell, K.C.B. (Assistant Under-Secretary, Home Office), Mr. A. T. A. Dobson, C.V.O., C.B.E. (Assistant Secretary, Ministry of Agriculture and Fisheries), Sir F. Gowland Hopkins, D.Sc., F.I.C., P.R.S., Professor G. T. Morgan, O.B.E., D.Sc., F.I.C., A.R.C.S., F.R.S. (President of the Society of Chemical Industry), Mr. Percy Gates (President of the Institute of Brewing), Mr. George Gray, M.Sc., F.I.C., M.I.Chem.E. (Vice-President of the Institution of Chemical Engineers), Sir Ernest Graham-Little, M.D., F.R.C.P., M.P. for the University of London, Mr. Norman Kendal, C.B.E. (Assistant Commissioner of Police), Mr. A. W. Monro, C.B. (Ministry of Agriculture and Fisheries), and Mr. R. B. Pilcher, O.B.E. (Registrar of the Institute of Chemistry).

After the toasts of His Majesty the King and the Members of the Royal Family had been honoured, the President proposed the health of the Members of H.M. Civil Service. Dr. Dunn emphasised the harmonious relations which existed between those branches of the Civil Service which were intimately associated with the work of Public Analysts and Official Agricultural Analysts, and he described H.M. Civil Service as one of the great causes of the stability of this country, and as the object of envy and respect of other nations.

Sir ERNLEY BLACKWELL, replying to the toast, referred to the connection between the Home Office and the Society, and said that it was almost fifty years since the first two Official Analysts to the Home Office were appointed. He recalled the fact that one of the most distinguished of these analysts was the late Sir Thomas Stevenson, a former President of the Society. He pointed out that the evidence in Court that was based on the facts provided by analysis was, as a rule, accepted by both sides, though the inferences to be drawn from those facts were sometimes challenged.

Mr. A. T. A. DOBSON also replied to the toast, and referred to the friendly relations existing between the Ministry of Agriculture and the Society of Public Analysts. He said that cases in which permission to prosecute was given by the Ministry of Agriculture were seldom the outcome of deliberate fraud. He expressed the hope that farmers would in the future avail themselves even more than in the past of the facilities provided by the new Act to have their fertilisers and feeding stuffs examined by the Official Agricultural Analysts, in order to ensure that their supplies were of the required standard, and that they were receiving value for their money.

Lord ATKIN, in proposing the health of the Society, spoke of the value of the services of Public Analysts to the community, and contrasted the condition of the purity of food at the present time with that which was prevalent before the passing of the first Food and Drugs Act. He expressed the view that definite standards should be fixed for foods, and in particular for milk, and that the onus should be upon the vendor to prove that the article sold conformed to that standard.

The fact that vendors could defy the recognised standard for milk with an explanation about the idiosyncracies of the cow was due, not to legislation, but to a decision of three out of five judges in a certain criminal case. He had always longed for the time when an enterprising municipal authority would really fight this cause in a civil case, and take it, if necessary, through the Court of Appeal to the House of Lords, for the benefit of the community. It would bring great advantage to the public if that former decision could be altered. Lord Atkin also referred to the extension of the original basis of the Society, which now included the chemists of many of the leading industrial firms, and he laid stress upon the benefit to the community from the co-operation between these chemists and Public Analysts.

The PRESIDENT, replying to the toast, said that the income-tax authorities had recently defined a "learned society" as one that existed primarily for the benefit, not of its members, but of the public, and claimed that our Society, through the work it had done in helping to secure better food for the community, was entitled to that designation. The extension of its membership to include "other analytical chemists" gave it a still more authoritative position than formerly, for it could now speak as representing every branch of the profession.

The health of the Kindred Societies was proposed by Mr. A. CHASTON CHAPMAN, who pointed out how the various scientific societies, and particularly those connected with chemistry, were becoming more closely associated, owing to the way in which their work tended to overlap. Although he regarded this as a step in the right direction, he was personally of the opinion that each Society should retain its individual entity.

Sir F. GOWLAND HOPKINS, replying for the Kindred Societies, paid a tribute to the usefulness of the Journal of the Society of Public Analysts (THE ANALYST) to workers in biochemical laboratories.

Mr. E. HINKS proposed the health of the numerous guests, the toast being acknowledged by the LORD MAYOR OF NEWCASTLE and Mr. GEORGE GRAY.

Annual Report of Council

March, 1932

THE Roll of the Society stands at 668, an increase of 7 over the membership of last year.

The Council, with great regret, has had to report the deaths of:

J. Miller.
Gilbert John Alderton.
Meredith Wynter Blyth.
Shelton Gottlieb Agar.
George Craig.
Henry Droop Richmond.
Hubert Taylor.
William Foulkes Lowe.

H. Droop Richmond served several periods on the Council of the Society. He was a Past Vice-President, and occupied the position of Treasurer for two years. ("Obituary," ANALYST, 1931, **56**, 700.)

M. Wynter Blyth was a well-known Analyst, who took an active interest in the affairs of the Society, and was a Member of Council during 1906–1907. ("Obituary," ANALYST, 1931, **56**, 353.)

In W. Foulkes Lowe the Society loses an old member, whose membership dated from 1888. ("Obituary," ANALYST, **1932, 57**, 71.)

During the year, seven meetings of the Society were held, and the following papers were communicated:

"Some Factors Affecting the Solubility of Milk Powder." By L. H. Lampitt, D.Sc., F.I.C., and J. H. Bushill, M.Sc., A.I.C.

(i) "The Determination of the Hydroxyl Content of Organic Compounds: Estimation of Castor Oil." (ii) "The Determination of the Carbonyl and Aldehyde Content of Organic Compounds: Estimation of Phenylhydrazine." By S. Marks, M.Sc., A.I.C., and R. S. Morrell, Ph.D., F.I.C.

"Food Control in Holland." By A. van Raalte, D.Sc., and J. Straub.

"The Determination of Small Quantities of Methane." By H. R. Ambler, B.Sc., F.I.C.

"The Fatty Acids and Component Glycerides of Indian Ghee." By R. Bhattacharya and T. P. Hilditch, D.Sc., F.I.C.

"The Investigation of Japanese Beeswax." By H. Ikuta.

"The Denigès–Oliver Test for Morphine." By J. Bamford.

"Carbon Monoxide Poisoning: Its Detection and the Determination of the Percentage Saturation in Blood by means of the Hartridge Reversion Spectroscope." By R. C. Frederick, A.I.C.

"Experiments on the Hardness of Fats." By H. M. Mason, M.Sc., F.I.C., and G. Walsh, B.Sc., A.I.C.

"A New Process for the Determination of Small Amounts of Bromide in Chloride." By B. S. Evans, M.C., Ph.D., F.I.C.

"The Use of Bromine as a Reagent in the Determination of Alkaloids." By S. G. Walton and R. G. O'Brien.

"A Demonstration of a New Development in Filter Papers." By E. J. Guild.

"The 'Rope' Spore Content of Flour and its Significance." By A. J. Amos, B.Sc., A.I.C., and D. W. Kent-Jones, Ph.D., B.Sc., F.I.C.

*"The Separation of Tin from Tantalum and Niobium." By W. R. Schoeller, Ph.D., and H. W. Webb.

"A New Method for Detecting Decomposition Products in Anaesthetic Chloroform." By N. L. Allport, A.I.C.

"Contaminations in Morphine Deposited in the British Pharmacopoeia Process for the Analysis of Opium." By J. N. Rakshit, F.I.C.

"The Identification of Wood and Wood Charcoal Fragments." By J. Cecil Maby, B.Sc.

"The Examination of Dyed Leather in Cases of Alleged Dermatitis." By T. Callan, M.Sc., Ph.D., F.I.C., and N. Strafford, M.Sc., F.I.C.

"The Determination of Chlorides in Dairy Products and Biological Material." By W. L. Davies, M.Sc., Ph.D., F.I.C.

*"A Reliable Method for the Quantitative Separation of Titanium from Tantalum and Niobium." By W. R. Schoeller, Ph.D., and C. Jahn.

*"The Separation of the Earth Acids from Metals of the Hydrogen Sulphide Group." By E. F. Waterhouse and W. R. Schoeller, Ph.D.

"The Evaluation of the Menthone Content of Peppermint Oil." By J. Reilly, M.A., D.Sc., F.R.C.Sc.I., N. Noonan, M.Sc., and P. J. Drumm, Ph.D.

"The Determination of Vanillin in Chocolate and Cocoa Butter." By D. M. Freeland, A.I.C.

"The Direct Determination of Nitrogen in Gases." By H. R. Ambler, B.Sc., A.I.C.

"A Micro-Method for the Determination of Uronic Anhydride Groups in Pectic Substances." By H. W. Buston, Ph.D., D.I.C.

* Work done under the Analytical Investigation Scheme.

"The Composition of Linseed Oil." By N. E. Cocchinaras, Ph.D.
"Oil from Malayan *Aleurites Montana* and the Properties of Hong Kong Oil." By T. Hedley Barry.
"The Calcium Fluoride Method for the Determination of Fluoride, with Special Reference to the Analysis of Nickel Plating Solutions." By S. G. Clarke, Ph.D., A.I.C., and W. N. Bradshaw, B.Sc.

The following papers were read at meetings of the North of England Section:

*"The Rapid Quantitative Determination of Solid Saturated Fatty Acids." By T. P. Hilditch, F.I.C., and J. Priestman, Ph.D.
"A New Method for the Determination of Solid Unsaturated Fatty Acids." By L. V. Cocks, F.I.C., B. C. Christian, Ph.D., A.I.C., and G. Harding, F.I.C.
"Some Aspects of the Bacteriological Examination of Water." By W. G. Carey, F.I.C.
"River Pollution Prevention Problems." By J. W. H. Johnson, M.Sc., F.I.C.
"Documentary Evidence in Criminal Trials." By C. Ainsworth Mitchell, M.A., D.Sc., F.I.C.
"The Analyst in the Witness-Box." By W. H. Roberts, M.Sc., F.I.C.

A considerable number of other original papers and notes also appeared in THE ANALYST.

The Editor of THE ANALYST, Dr. C. A. Mitchell, reports that the number of pages for the year is the largest in any volume of THE ANALYST so far published. The question of the paper on which THE ANALYST is printed has received the consideration of the Publication Committee. After expert advice, it was decided that the body of the Journal should consist of the same paper as at present, which conforms with the Library Association standard. Owing to the increased demand, the number of copies of THE ANALYST printed was increased at the commencement of the year.

The Honorary Treasurer's statement has been published separately; it shows that under his careful supervision expenses have been successfully met, and that the Society is in a sound financial position.

The Council records its appreciation of the facilities which have been readily afforded to the Society by the Chemical Society and by the Institute of Chemistry in providing accommodation for Committees.

The Standing Committee on Uniformity of Analytical Methods and its Sub-Committees have continued their work, and, though only one finished report has been received during the year, there are several reports nearing completion. The report received, which was the eighth of the series issued by the Essential Oils Sub-Committee, contained recommendations with regard to the determination of cineole in essential oils other than cajuput and eucalyptus oils. A new Sub-Committee has been formed, consisting of representatives of the Society and of interested industrial organisations, under the chairmanship of Professor T. P. Hilditch, D.Sc., F.I.C., to consider the determination of unsaponifiable matter in oils and of unsaponified fats in soaps. The duties of Honorary Secretary of this Sub-Committee have been undertaken by L. V. Cocks, F.I.C. Again the Council expresses its appreciation of the work of the Chairman, E. Hinks, B.Sc., F.I.C., and the members of the Standing Committee, and also of the Chairmen and members of its Sub-Committees. It fully realises the large volume of work entailed by the investigations which have been undertaken.

The Council concurred with the report issued by the "Ad Hoc Committee" appointed by the British Chemical Standardising Body, to explore the possibilities of chemical standardisation. The British Engineering Standards Association has now become the British Standards Institution, and its work is divided into

Sections, one of which is termed "The Chemical Division." The Council has elected E. R. Bolton, F.I.C., to serve as its representative on this division.

During the year, the Ministry of Health set up a Departmental Committee "to enquire into the working of the law as to the composition and description of articles of food other than milk, and to report what alterations, if any, in the law, or its administration, appeared to be desirable." A request was received to hear evidence from the Society, and the Council appointed the President (J. T. Dunn, D.Sc., F.I.C.), E. Hinks, B.Sc., F.I.C., and F. W. F. Arnaud, F.I.C., to represent the Society. The Council of the Society submitted a memorandum of the evidence which it proposed to give, but before the witnesses could be heard, owing to the financial situation of the country demanding measures of economy, the work undertaken by this Committee was suspended. The Council regrets this, while expressing its hope that at the earliest possible moment the enquiry will be re-opened by the Ministry. It is to be noted that, in the terms of reference, milk is excluded from consideration by the Departmental Committee. The Council views with concern the movement that is noticeable in many quarters for the repeal of the Sale of Milk Regulations, and also the character of the legislation which is advocated in this connection. The Council is carefully watching the matter, and will take advantage of every opportunity afforded to put forward the views of the Society.

For many years the Editor, C. A. Mitchell, D.Sc., F.I.C., has represented the Society on the Chemical Society Library Committee. Under the rules of the Chemical Society he must now retire from the Committee. The President and Council have expressed to him their warm appreciation of his services.

The Food Manufacturers' Federation invited the Society to suggest a standard for added flour in shredded suet. A meeting with representative manufacturers of shredded suet took place, and the view of manufacturers was ascertained. The Council considered that the amount of added starch would best be limited by prescribing a minimum content of fat in the shredded suet. It also recommended that this minimum be 83 per cent., and stipulated that any addition to suet should be declared. (*See* ANALYST, **1931**, **56**, 778.)

The question of jam standards again received the attention of the Public Analysts' Committee, and further views with regard to these have been published from time to time. (*See* ANALYST, **1931**, **56**, 391, 701.)

Besides the investigations which were completed during the year under the Analytical Investigation Scheme, there are at present six investigations proceeding. The Secretary would at all times welcome further suggestions for work under the Scheme.

The North of England Section shows continued activity, and, at its last General Meeting, it was stated that five meetings had been held during the year. A Summer Meeting, held at Scarborough, was largely attended by Members of the Section, and a few Members from the South were also present. At this meeting the Chairman—C. J. H. Stock, B.Sc., F.I.C.—moved a resolution, which was unanimously carried, expressing the devotion of the North of England Section to the aims and interests of the parent body. (*See* ANALYST, **1931**, **56**, 497.)

J. T. DUNN, *President.*
F. W. F. ARNAUD, *Honorary Secretary.*

Annual Address of the President

(DR. J. T. DUNN, F.I.C.)

(Delivered at the Annual General Meeting, held on March 4, 1932)

LADIES AND GENTLEMEN,

At the end of another Session we can still congratulate ourselves on being in vigorous life and on increasing our membership and our usefulness. Though we have to lament the removal by death during the year of eight of our members (one of whom, William Foulkes Lowe, had been a member for forty-three years, and another, Henry Droop Richmond, for forty-one), yet our roll of membership holds seven more names than it did at our last Annual Meeting. The number of papers read at our meetings and communicated to our Journal shows no tendency to diminish (indeed, the Editor's difficulty is rather how to accommodate them all, having regard to the desirability of keeping successive monthly issues approximately uniform in size); and a perusal of their titles, as enumerated in the Council's report, will make manifest their variety and the wide extent of ground which they cover. The value and usefulness of our journal are shown by the growing demand for it outside of our own membership—a demand so great that we have had to increase the number of copies that we print. Thanks to the vigilance and care of our Honorary Treasurer, our expenditure has been kept within our income, and we are financially in a position which many kindred Societies would in these difficult times consider enviable.

Our North of England Section continues its vigorous career, and its loyalty to the parent Society is on a par with its activity. It has furnished us with several new members during the year, and has added its contributions to THE ANALYST. It has continued the practice of including in the programme of its meetings, whenever possible, visits to works and manufactories, and it had again last year a Summer Meeting at Scarborough, equally successful with the former one on its serious side, and much more successful in the number of members who attended it, among whom, it is pleasant to remember, were several from the southern portion of the Island. Both the works visits and the Summer Meeting afford opportunities for personal intercourse among the members that are not possible to anything like the same degree during the more formal portions of the meetings, and that do a great deal to foster and develop among them the spirit of comradeship and of loyalty to the Society.

It is pleasant to be able to congratulate our veteran member, Dr. Dyer (and it is also a matter on which the Society is to be congratulated), on the completion of a task which, though it may have been laborious, and at times have given rise to sad and regretful memories, must, on the whole, I think, have been a labour of love and a very pleasant occupation. His "Reminiscences of the First Fifty Years" of our Society is now in paged proof, and we have now only to wait for the completion of Dr. Mitchell's complement to it—"A Review of its Activities." Dr.

Dyer's qualifications for undertaking this work are, of course, unique. We are all under a deep debt of gratitude to him, and it is very pleasant to think that he is still possessed of such health and vigour that he has been able, not only to complete his history, but to come down with regularity to our meetings, and give us the pleasure of his presence and the advantage of his counsel. As one who has had the privilege of reading Dr. Dyer's work in proof, I can say with certainty that when you do read it you will not only unanimously wish, as I know you all do now, that he may long be spared to continue his active association with us, but will regret the improbability that he may be able, when the Society celebrates its centenary, to give us the history of its second fifty years also.

The Society of Chemical Industry, at its December Council meeting, decided to form a "Food Group" in the Society, for those of its members who are mainly interested in the technology of food products, much on the lines of the Chemical Engineering Group, which has existed in the Society for some considerable time. It was recognised that, as our Society deals very largely with the analysis of foods, it was desirable to avoid any overlapping or possible clashing of interests, and to secure, if possible, some measure, not only of co-ordination, but of co-operation; though, as the purview of the members of the Food Group would cover the technological and industrial aspects of food manufacture, rather than the analysis of food products, serious overlapping was not greatly to be feared. Representation of this Society on the Committee of the Food Group was thought to be desirable, but the constitution of the Group as a body of members of the Society of Chemical Industry prevented this. The difficulty was got over, however, by giving the Committee of the Food Group power to co-opt an additional consultative member from outside; and our Treasurer, Mr. Hughes, was so co-opted, and sits on the Committee to form a link between the Group and this Society. There is no reason to think that the procedure of the Food Group will be in any way detrimental to the work or the interests of this Society, and we may heartily welcome its formation and wish it success. The Group is holding its inaugural meeting, jointly with the London Section of the Society of Chemical Industry, in these rooms, on Monday next at eight o'clock, when Dr. Lampitt will give an address on "Science and Food."

Four of last year's papers were accounts of work done under the Analytical Investigation Scheme. This scheme was suggested, I believe, by Mr Chaston Chapman, about 1901. It was referred to in his presidential address, in 1904, by Mr. Fairley, who then said that several investigations were in progress under its auspices. In February, 1908, attention was drawn to the Scheme by publication of its objects and methods of work in THE ANALYST; and Mr. Chapman, in his presidential address of 1915, mentioned that up to that time the results of thirty-seven investigations under the Scheme had been presented to the Society. I mention the Scheme now because one long and important investigation under it has just been brought to practical completion. We may, I think, congratulate Dr. Schoeller and his colleagues on their work. They took up with great courage a piece of work which most chemists would have thought very unpromising, perhaps very uninteresting, and have pursued it with great patience and perseverance over a period of more than ten years; as a result, they have established accurate and workable methods for the determination of tantalum and columbium,

and for their separation from one another, and from tin, titanium, tungsten, hafnium, and zirconium. This work is described in a series of twenty-one papers (one read after the date of Council's Report), all but the first two of which have appeared in THE ANALYST, as results of work undertaken under the Analytical Investigation Scheme.

Of late years the Scheme has to its credit, besides the work of Schoeller and his associates, that of Nicholson and Rhind, Price, Hooper, Fear and A. E. Jones on tannins, and of Hilditch and Priestman on the separation of solid and liquid fatty acids; and there are at present six other investigations proceeding under the Scheme. The work already completed, and that still going on, shows that the Scheme has amply justified its existence; but one could wish that the opportunities it affords for bringing into relation with one another those who have need of trustworthy analytical processes for particular ends, or wish to have the accuracy and trustworthiness of published methods inquired into, on the one hand, and those who have time and opportunity for investigation, and are looking for appropriate objects of inquiry, on the other, were still more widely taken advantage of.

The work of the Analytical Investigation Scheme naturally calls to mind that of the Standing Committee on Uniformity of Analytical Methods. Many may think that the output of this Committee is small, and its work very slow. But, in the nature of things, the analytical processes which the Committee, through its Sub-Committees, is called upon to examine and report upon, are almost all of them, necessarily, processes of difficulty and uncertainty at their present stage, and involve, in overcoming those difficulties, a very large amount of work. This work, too, is, for the most part, team work, and has to be carried out by teams that can only devote a part—sometimes a very small part—of their time to it; teams, too, the members of which are often widely separated geographically, so that collation of results and consultation among the members necessarily take much time. The reports furnished by the Sub-Committees and published in THE ANALYST, give only a faint indication of the amount of labour involved in their preparation. To the members of the Sub-Committees, and, in many instances, to the firms under whom the members work, and who have supplied materials and helped in other ways, not the Council alone, but the whole of the members of the Society, are grateful, and more especially those members who, through the labours of one or other of the Sub-Committees, are furnished with a definite and workable method of analysis for their daily routine, enabling them to reach results comparable with those of other workers, where formerly discrepancy and confusion were only too frequent.

Work, generally similar to that of the Committee on Uniformity of Methods of Analysis, will in future be carried out under the auspices of the new Standards Institution. The recommendations of the Committee appointed by the Conference on Standards of June, 1930, to which I referred a year ago, have been carried into effect; the British Engineering Standards Association has been widened in its scope, and has become the British Standards Institution. It is to work through four divisions, concerned respectively with Engineering, Chemistry, the Building Trades, and the Textile Trades. Each division is controlled by a

Council—that of the Chemical Division consists of thirty-six members, twenty-eight of whom represent various Societies or organisations, of which our Society is one, and the other eight, to be co-opted, will be persons whose services are likely to be of special aid to the Council. The General Council of the Institution consists of forty-one members, nine elected by each of the four Divisions, and one each from the Institution of Civil Engineers, the Board of Trade, the Department of Scientific and Industrial Research, the Federation of British Industries, and the Association of British Chambers of Commerce. The General Council will decide the broad policy of the Institution, and deal with matters of general interest; but, subject to being in accord with that broad policy, each divisional Council will manage its own business without any control or interference from outside. The Divisional Councils are now organising themselves for work, and, in the capable hands of Mr. Bolton, we may be quite sure that the representation of this Society is safe. It may interest members to know that the Joint Committee for the Standardisation of British Chemical Glassware, on which this Society was represented, but the administration of which was conducted through the Institute of Chemistry, has turned over the continuation of its work to the new body.

The Council welcomed the appointment by the Ministry of Health of the Committee to inquire into the working of the Food Laws, and gladly embraced the opportunity of giving evidence before the Committee, in the hope that the Committee's deliberations would result in the removal of anomalies and objectionable features that exist in the present Acts, and in the introduction of greatly-needed reforms. Unfortunately, the sudden recognition of the need for drastic economies in the national expenditure caused the suspension of the Committee; and only a day or two before its first meeting, to which the Society's representatives had been summoned, we had notice that the meeting would not be held, and, shortly afterwards, further notice that the sittings of the Committee would be indefinitely postponed. We can only wait for the restoration of financial stability and the return of some measure of prosperity in the country, and hope that when that is achieved this matter will again receive attention at the hands of the Government of the time; for the urgency of reform will certainly be no less then than now, and most of the desirable alterations in the law will involve little or no cost to carry out.

The first point which we proposed to urge upon the Committee was the need for prescribed standards or limits of composition of articles of food, the prevention of misdescription, and the institution of a Statutory Advisory Committee to devise and recommend such standards or limits, and revise them or add to them from time to time as might become necessary. These matters, of standards or limits, and of a body which should ascertain and prescribe them, have for long been a matter of discussion, and the balance of opinion, at least among Public Analysts, has been overwhelmingly in favour of their introduction. Dr. Dyer tells us that at the first meeting of the Society in 1874 suggestions for standards or limits for certain articles of food were put forward, and referred to the Council for guidance, and I find that at a Conference on Food Adulteration, held at the International Health Exhibition in 1884—nearly fifty years ago—the matter was discussed. Dr. Bell, at that time head of the Somerset House laboratory, deprecated their

wide introduction, on what would now seem, to most of us, the rather curious ground that it might tend to discourage the production and diminish the supply of any article of food; but Dr. Muter went wholeheartedly for it, and declared that there ought to be a permanent Commission appointed by law, consisting of three chemists, nominated respectively by the Government, by the Society of Public Analysts, and by the Chambers of Commerce, to examine all articles of food, and lay down limits. In 1893, in consequence of a Bill promulgated by Dr. Cameron to amend the Food and Drugs laws, which contained some objectionable clauses (but which was afterwards withdrawn), this Society called a conference to discuss the general question. At this conference a proposal was made that there should be constituted a chemical department of the Local Government Board, with which the Public Analysts, as officers of the Board, should be placed in direct relation. Consultation with this Department would, in effect, it was thought, result in the fixing of limits. Possibly as the result of this conference, or of the public attention which the conference attracted, a Select Committee was this year appointed to inquire into the working of the Acts, and the Society, for the consideration of the Committee, formulated a draft bill, which suggested, among other things, the creation of a Board of Reference, to consist of the Chief Chemical Officer of the Inland Revenue Laboratory, a nominee of the General Medical Council, three nominees (all to be Public Analysts) of the Local Government Board, and one nominee of the Board of Agriculture, who were to examine and report on the composition of various articles of food and drugs, describe, investigate, and advise regarding new or improved methods for their examination, set forth definitions and exceptions, and fix limits and standards of quality and purity. They were to meet from time to time, to issue new or revise old limits or standards. The Committee, in its report, adopted many of the suggestions of the Society, but the resultant legislation, which was not effected till 1899, contained no reference to standards or limits, nor to an advisory or prescribing body. Meanwhile, successive Presidents, in their addresses, referred to the matter. Sir Charles Cameron, in 1895, said: "Every effort should be made to induce the Committee to recommend the fixing of standards for all articles that admit it"; and Dr. Stevenson, in 1896: "Our earnest endeavour in the near future must be to secure limits below which articles of food and drugs must not be allowed to fall; limits fixed by some competent and authoritative body, on which Public Analysts must, of course, be adequately represented."

So far as colouring matters and preservatives are concerned, the Departmental Committee on Preservatives of 1901 expressed the same view; they recommended "That means be provided, either by the establishment of a separate court of reference or by the imposition of a more direct obligation on the Local Government Board, to exercise supervision over the use of preservatives and colouring matters in foods"; and the Royal Commission on Arsenical Poisoning of 1903 was still more definite in its proposals, its report reading: "We consider that the Local Government Board (under advice as indicated in this report) should be the authority to prescribe, and from time to time to vary, standards for the purposes of the Food and Drugs Acts. Obviously, account would need to be taken of sundry medical, physiological,

chemical, and administrative questions in fixing such standards . . . and manufacturers should be fairly dealt with. The Committee on Food Products Adulteration (1893), and, more recently, the Committee on Preservatives, alike came to the conclusion that food standards in certain instances were essential to efficient administration. Both Committees realised the impossibility of satisfactory standards being fixed by the central authority in the absence of full preliminary inquiry, and they recommended the establishment of a Board (Court, Permanent Commission or Standing Committee) of Reference, which should consist of a small number of scientific men, nominated by the Crown or departmentally, as the authority to advise on points arising in connection with the Sale of Food and Drugs Acts, and to prescribe the standards which should be fixed for the purposes of those Acts. We are of opinion that if a Government Department, the Local Government Board, or the Board of Agriculture, is to impose standards for the purposes of the Food and Drugs Acts, it is essential that its action should be based upon the advice of a scientific body of this nature."

Mr. Fairley, in his 1905 address, spoke strongly on the matter, quoting the words of the Council in 1897 as being still applicable to the situation, and Mr. Tatlock, in his presidential address in 1909, again referred to the matter, and expressed the hope that it might be made the subject of discussion at a "field-night" of the Society.

In 1913 Mr. John Burns introduced a Bill, which, among other provisions, proposed to give power to the Local Government Board, "after such inquiry as they may think necessary," to make regulations defining an article of food in any matter affecting its nature, substance, or quality. To place such a power in the hands of the Local Government Board, with no further assurance that interested and qualified opinion would first have been obtained than "after such inquiry as the Board may think necessary," would hardly have satisfied our Society, and probably would still less have satisfied the manufacturers of, and dealers in, articles of food; it was, in fact, very strongly deprecated by Mr. Archbutt in his address in 1914, in which he urged the need of defining clearly the machinery by which such regulations were to be made; but the Bill was withdrawn, and the necessity for opposing or modifying its proposals disappeared.

No further step was taken till the Food and Drugs Act of 1928 was passed, which, as we all know, was only a consolidating Act, and introduced no new features; especially was there no part of it dealing in any way with the general setting up of standards or limits of composition. In 1929 our Society addressed a letter to the Committee of Civil Research, pointing out the difficulties which frequently arise in the administration of the Food and Drugs Acts, showing that many of these difficulties would be overcome if there were recognised standards of composition for a variety of foods, and urging the desirability of appointing a body to examine into the necessity for this, and the provision of a scheme to make funds available for the cost of investigations that might be involved.

In our memorandum for the Committee of last year we brought all this forward, and laid stress on the unanimity of qualified opinion on the point coming from so many different sources; and we shall, I hope, urge the matter no less strenuously when the opportunity for so doing arrives. It would serve no good

purpose, and would take more time than we have at our disposal, to go over *seriatim* the whole of the points which we were prepared to urge upon the Committee, but I thought a brief summary of the history of opinion upon this important matter of "Standards and how to fix them," would, perhaps, be interesting and useful.

Now, ladies and gentlemen, this is the last occasion on which I shall occupy this chair, save for a few moments when I come down in April to hand over the position to my successor. I have endeavoured to carry out the President's duties to the best of my ability, but I claim no credit or virtue on that ground, for the work, and all that it has involved, have been a source of unmixed pleasure and enjoyment to me. I am glad to think that I leave the Society in at least as flourishing a condition as when I took over the chair; but neither do I claim any credit for that—for I have had the help and guidance of a Council and officers devoted to their work, assiduous in their attendance at meetings, and so varied in their attainments and their special directions of thought, that the very best advice and counsel has been at my disposal in every contingency. I name none of them, for I should have to name them all; and I am grateful, not only for their work as officers and members of Council, but for their unbroken kindness to me, and for the pleasure which personal intercourse with them has been to me.

I have attended every meeting of the Society since you elected me. I wish I could continue to attend every meeting in the future, but I recognise that that is an ideal to which I can hardly hope to attain. I leave the chair with none but happy memories, and have but one regret, which is, that the pleasant personal intercourse with my fellow-members cannot continue except to a diminished extent. There is, I think—I have said it to the Northern Section, and I say it here again—something in this Society, a spirit of some sort, which inspires its members with affection for the Society to an extent greater than I find in any similar Society that I know. I do not seek to define it or explain it: I am content to feel it and know that it exists. Long may it continue: and as long as it does continue, presidents may come and presidents may go—but the Society of Public Analysts and other Analytical Chemists will go on for ever.

A Micro-Method for the Determination of Uronic Anhydride Groups in Pectic Substances

By HAROLD WILLIAM BUSTON, Ph.D., D.I.C.

(*Read at the Meeting, December* 2, 1931)

Since the pectin molecule was shown by Nanji, Paton and Ling (1925) to be based upon a combination of sugar residues and residues of "uronic acids," the determination of the "uronic anhydride" content of pectic substances has become part of the regular technique. The method devised by these workers for the determination, and subsequently generally adopted, was based on the observation that, on distillation with 12 per cent. hydrochloric acid, the uronic anhydride residues yield carbon dioxide quantitatively, according to the equation:

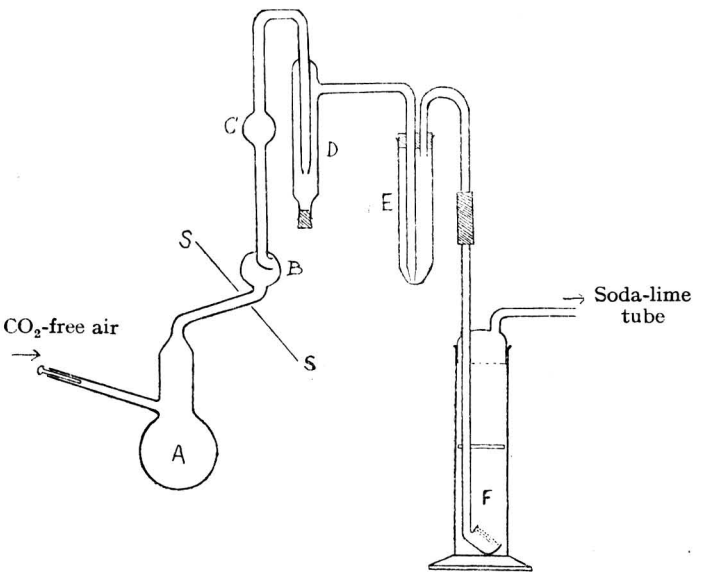
$$\underset{\text{uronic anhydride residue.}}{-C_6H_8O_6-} = \underset{\text{furfural.}}{C_5H_4O_2} + 2H_2O + CO_2.$$

The determination thus involved the estimation of the carbon dioxide produced under certain given conditions. When dealing with a substance such as calcium pectate, which is regarded as having a definite composition, the results obtained were only approximately equal to those calculated from the accepted formula. The error lay chiefly in the titration of the standard barium hydroxide, in which the carbon dioxide was absorbed. Working with the type of apparatus most frequently used, three absorption towers in series are found necessary to trap all the carbon dioxide; each tower contains 100 c.c. of $N/20$ barium hydroxide, of which 20 c.c. samples are withdrawn at the end of the operation, and titrated. Thus a small titration error may be magnified considerably in calculating the result. Apart from this drawback, the apparatus necessary is very cumbrous, large amounts of reagents are required, and the determination, which involves 3 hours' distillation, takes a considerable time to carry out. This seemed a case, therefore, where the use of a micro-method could be adopted with advantage. The familiar advantages of micro-methods—the use of much smaller amounts of material and reagents, and the curtailing of the time involved—were enhanced by the fact that it was found possible to use only one absorption tower, and thus to reduce the error arising from inaccurate titration.

Apparatus.—The preliminary experiments were carried out with the micro-Zeisel apparatus described by Pregl (1924). Some difficulties were met with in using this apparatus, but the early results obtained gave sufficient promise to lead to a continuation of the experiments.

A modified form of the micro-Zeisel apparatus was designed, and is shown in the diagram. The bulb (A) in which hydrolysis of the pectin takes place

has a capacity of about 8 c.c.; the vertical tube which serves as a reflux condenser is displaced some 3 cm. to the right, so that it is removed from the column of heated air rising from the bulb. Thus the refluxing process is made more efficient, and is further improved by placing a screen (SS) of asbestos paper slantwise across the tube below the bulb (B), and keeping the lower part of this tube cool by means of a moistened filter paper. The bulb (B) serves to prevent loss of material in the event of the liquid in A bumping during the hydrolysis, while the upper bulb (C) breaks the column of liquid which tends to collect in the tube, and prevents any



liquid from being carried over into D. The length of the vertical tube (reflux) is 15 cm., and the two bulbs have a diameter of about 1 cm. The trap (D) is empty, but has a loose plug of cotton-wool at the entrance to the side tube. The bubbler (E) contains a few drops of saturated silver sulphate solution, into which the delivery tube just dips; above this, the tube is packed for a distance of 2–3 cm. with glass wool, soaked in a paste of silver sulphate. Thus the gases passing through are brought thoroughly into contact with silver sulphate, without the use of relatively large amounts of water in which carbon dioxide might be retained. A plug of cotton wool in the upper part of E prevents any spray of silver sulphate from being carried over into the absorption tower.

The gas tower (F), which contains the barium hydroxide, is one of a pattern now standardised by Messrs. Schott of Jena. It has a capacity of 20 c.c., and the delivery tube is closed with a disc of sintered glass, of a coarse grade of porosity, which breaks up the passing gas into a fine mist of bubbles. It was the use of this type of bubbler that enabled absorption to be carried out in the one tower. When the gas is passing, some amount of splashing takes place up the sides of the tower; this is minimised by fitting a disc of rubber across the tower, just above

the surface of the liquid, as indicated in the diagram. A soda-lime guard-tube closes the exit from the absorption tower. The whole apparatus was designed to have the smallest internal volume practicable, in order to facilitate thorough removal of carbon dioxide by the current of air.

METHOD.—In carrying out the determination, a current of air is passed through the apparatus during the experiment. This is supplied most conveniently from a small cylinder of compressed air, with a finely adjustable valve. In the experiments quoted below, the air taken into the compressor was passed through caustic soda; any carbon dioxide escaping absorption at this stage was removed by passing the air from the cylinder first through 10 per cent. caustic soda solution, using a delivery tube with a sintered glass disc of the same type described above, and then through a short soda-lime tube. The air is passed at a rate of about 4 c.c. per minute. Four c.c. of 13 per cent. HCl, 90 per cent. saturated with sodium chloride, are placed in the bulb A, and the substance under examination (6–10 mgrm.), weighed out into a small cup of thin tinfoil (see Pregl, *loc. cit.*), is introduced through the side tube. [The use of the HCl–NaCl mixture follows the recommendation of Kullgren and Tydén,* who used a saturated solution of sodium chloride in 13·15 per cent. hydrochloric acid for furfural distillations; it was found that this mixture bumped severely during the heating, but this could be avoided by using a little less sodium chloride.] In the opening of the side tube rests a loosely-fitting glass pin, as in the micro-Zeisel apparatus. The absorption tower is charged with 10 c.c. of $N/50$ barium hydroxide solution, measured out by means of an automatic pipette.

After air has been passed through the apparatus for a few minutes, the tower F is connected, and the acid in A gently boiled over a micro-burner; a few chips of quartz in A promote smooth boiling. In 10–15 minutes, barium carbonate begins to appear in F. The amount rapidly increases at first, but the distillation is continued for 60–70 minutes, since experiment has shown that the process is virtually complete in this time (see below). If the boiling is gentle, no acid escapes into D, the refluxing being quite efficient. At the end of the distillation, F is disconnected, closed with a rubber stopper, the contents mixed, and the barium carbonate allowed to settle. Five c.c. of the liquid are withdrawn by means of an Ostwald pipette, having a rubber bulb attached, and titrated with standard $N/100$ oxalic acid, phenolphthalein being used as indicator.

Before carrying out any estimations, tests were made to examine any possible sources of error, and "blanks" performed. No indication was found that the air used introduced any carbon dioxide, nor that any acid was carried over into the absorption tower. The completeness of absorption of carbon dioxide in the tower was also carefully tested. The pipettes and micro-burette used were accurately calibrated, and the normality of the barium hydroxide and oxalic acid checked from time to time.

The accompanying table summarises the results of a number of determinations of uronic anhydride content of samples of pectic substances. The results obtained indicated that the micro-method is capable of giving results of the same order of accuracy as the macro-method.

* "Über die Bestimmung der Pentosanen," *Ing. Vetenskaps. Akad. Stockholm*, 1929, Handl. 94, p. 19.

TABLE I

Substance.	Time of distillation (minutes)	Percentage of uronic anhydride: Micro-method	Macro-method	Calc. from formula
Calcium pectate ($C_{35}H_{46}O_{33}Ca_2$)	30	42·88	—	
	50	60·28; 61·32	—	
Sample I	60	62·92; 63·08	—	65·54
	70	64·12	—	
	100	64·56	63·43*	
Sample II	70	65·98	—	
Sodium pectate ($C_{35}H_{46}O_{33}Na_4$)	70	65·76	66·50*	64·80
Pectin (methoxyl=8·2 per cent.)	60	66·85	—	67·66†

* Time of distillation, 180 min.

† Calculated for tri-methoxy pectic acid, $-OCH_3 = 8·98$ per cent.

LITERATURE

Kullgren and Tydén, *Ing. Vetenskaps. Akad. Stockholm*, 1929, Handl. 94.
Nanji, Paton and Ling, *J. Soc. Chem. Ind.*, 1925, **44**, 253T.
Pregl (1924), *Quantitative Organic Micro-analysis*, p. 150 *et seq.*

BIOCHEMICAL LABORATORY,
IMPERIAL COLLEGE OF SCIENCE AND TECHNOLOGY,
LONDON, S.W.

DISCUSSION

Dr. H. E. COX enquired whether there were any other sugars which yielded carbon dioxide on oxidation by the method, and whether in the case of jam it was necessary as a preliminary to isolate pure calcium pectate, or could the process be carried out on the more or less crude pectin? What relationship existed between the uronic anhydride groups and furfural?

Mr. T. RENDLE asked if the author could say whether the volume of carbon dioxide varied with pectin from different fruits. The method would probably be very useful if this were the case. He would also like to know if all pectinous bodies were "pectin," so far as this test was concerned.

Dr. BUSTON, replying, said that the sugars themselves did not yield carbon dioxide under the conditions of the experiment, but in natural fruit juices, etc., the pectins were always associated with substances of the hemicellulose type. The method was designed to assist in the investigation of the constitution of pectic substances and hemicelluloses themselves, and its application to the analysis of jams was not contemplated. It was generally accepted that the furfural yield from such groups approximated 16·7 per cent.

The amount of carbon dioxide obtained from the pectins of different fruits would vary with the degree of esterification of the pectic acid present, and also with the amount of contaminating hemicellulose. For pure pectic substances the carbon dioxide yield lay between 17·64 per cent. (for pectic acid) and 16·9 per cent. (tetra-methyl ester), irrespective of the source from which the pectin was prepared.

ADDENDUM.—I am indebted to Mr. Bacharach for the suggestion that the use of achromatic indicators, such as those described by Lester Smith (*Quart. J. Pharm.*, 1930, **3**, 499), would render the end-point in micro-titrations sharper. Opportunity has not been found to examine the matter in detail, but preliminary tests indicate that with a mixed methyl red- methylene blue- phenolphthalein indicator (2:1:100) a very satisfactory end-point is in fact obtained. It is surprising that achromatic indicators have not been more widely used in micro-titrations, where they offer considerable advantages.

Mohler's Test for Benzoic Acid

By EDWARD T. ILLING, B.Sc., F.I.C.

PART I

AN INVESTIGATION OF GROSSFELD'S MODIFICATION OF THE TEST

Mohler's test does not appear to have given satisfactory results in the hands of many workers; but, as fatty acids in moderate amount do not interfere with the production of the colour, due to *m*-diamino-benzoic acid, it was thought that a little study might well be devoted to the essential points.

Grossfeld's modified test is as follows:*—"The residue of benzoic acid is heated for 20 minutes in a steam-bath with 1 c.c. of concentrated sulphuric acid and 0·1 grm. of potassium nitrate. The mixture is cooled, 2 c.c. of water added, cooled again, and treated with 10 c.c. of 15 per cent. ammonia solution and 2 c.c. of 2 per cent. hydroxylamine hydrochloride solution. The colour develops slowly. It may be hastened by warming on the steam-bath, but attains its maximum on subsequent cooling. It may be matched with a solution of 0·86 grm. of ammonium iron alum in 1 litre of water, added to a 2 per cent. solution of potassium thiocyanate in the following amounts." The amounts of iron alum solution equivalent to varying quantities of benzoic acid are given.

Tests carried out by this method gave very inconsistent results. The essential details for success do not appear to have been worked out. The following points are worthy of notice, as bearing upon the quantitative aspect of the test:

(1) Nitration.—This must be carried out under specific conditions. Temperature and time are important factors, as is also the quantity of sulphuric acid.

The best results were obtained by the use of a boiling tube immersed in boiling water for 20 minutes; but continuation of the nitration for a further 5 minutes has no effect upon the final result. It is obvious that if the nitration is carried out in a beaker either on a steam-bath, or even immersed in boiling water, the temperature throughout the liquid is not as uniform as it is when the containing vessel is a comparatively narrow tube immersed in boiling water. It is better to add first the potassium nitrate, and then the concentrated sulphuric acid.

(2) Reduction.—The reduction of the *m*-dinitro-benzoic acid must also be carried out under strictly standard conditions. The two essential conditions are again temperature and time.

The temperature must be carefully regulated, as the amino-compound is decomposed if it is heated at too high a temperature.

* Ministry of Health Reports on Public Health and Medical Subjects, No. 39, January, 1927, by G. Monier-Williams, p. 33.

It was found that the amino-compound showed signs of decomposition (the reddish-brown colour was diminished and a yellow colour began to develop) when heated for 20 minutes at 70° C., but decomposition could not be detected when the duration of the heating was from 3 to 10 minutes. At a temperature appreciably below 60° C. (about 55° C.), the reduction was not complete in 20 minutes, but at 65° C. only 5 minutes were required for the maximum development of colour. The desired conditions are, therefore, 5 minutes at 65° C. A variation of 3 to 4 degrees either way during the heating has no appreciable effect. It should be noted that the temperature of the water in a beaker may fall about 5 to 10 degrees on introducing 6 to 8 tubes, so that it is advisable to have the water at about 72° C. at the beginning of the reduction.

(3) REACTION BETWEEN FERRIC IRON AND POTASSIUM THIOCYANATE.—The reaction between ferric iron and potassium thiocyanate is a reversible one, and, as neither of the reacting ions is coloured, the non-ionised ferric thiocyanate is evidently responsible for the colour. It is, therefore, apparent that the quantity of thiocyanate used is as important as that of the ferric iron solution. The colour is intensified by the increased concentration of either.

A solution of ammonium iron alum containing 1 grm., instead of 0·86 grm., per litre, has been used throughout these tests.

METHOD.—The method is as follows:—The benzoic acid residue (as the sodium salt*) is introduced into a boiling tube (6″ × ¾″), and the tube is heated in a beaker of boiling *brine* until the liquid is driven off, and all the drops of condensation water have disappeared. It is cooled, and 0·1 grm. of potassium nitrate and 1 c.c. of concentrated sulphuric acid are added. The tube is placed in boiling *water* for 20 minutes, then cooled, and 2 c.c. of water are added. The tube is held under running water, and 10 c.c. of 15 per cent. ammonia solution are *carefully* added, followed by 2 c.c. of a 2 per cent. hydroxylamine hydrochloride solution, and the contents are well mixed. It is next placed in a beaker of water at 65° C. for 5 to 6 minutes, then cooled, and the colour matched with that developed by mixing the amounts of iron ammonium alum and potassium thiocyanate solutions given below for varying quantities of benzoic acid.

Benzoic acid	Iron ammonium alum solution (1 grm. per litre)	Potassium thiocyanate solution (2 per cent.)	Volume made up to
Mgrms.	c.c.	c.c.	c.c.
0·1	0·2	0·2	15
0·25	0·3	0·3	15
0·5	0·5	0·5	15
0·75	0·7	0·6	15
1·0	1·0	0·8	15
2·0	1·5	1·5	15
3·0	2·0	1·5	15
4·0	2·0	2·3	15
5·0	3·0	2·5	15
6·0	4·0	3·5	50
7·0	4·0	4·5	50
8·0	4·5	3·5	50
9·0	5·0	4·0	50
10·0	5·5	5·5	50

* Ammonium benzoate is volatile at 100° C.

The standards of 15 c.c. volume were made up in a graduated cylinder by adding the requisite amounts of the two constituents, and making up to 15 c.c., which is the volume of the solution obtained at the completion of the test. The colours were matched in 50 c.c. Nessler tubes. For 6 mgrms. or more of benzoic acid it is better to dilute to 50 c.c. and to use 100 c.c. Nessler tubes. These standards cannot be diluted, as the colours are not proportional in other dilutions.

The above test will detect 0·025 mgrm. of benzoic acid, and amounts of this acid differing by 0·025 mgrm. can be distinguished when working with 0·1 mgrm. or less, but with increasing depth of colour in more concentrated solutions of benzoic acid the sensitiveness of the test decreases.

Salicylic acid, phenolphthalein, saccharin, and vanillin all give yellow colours only. Fatty acids give no colour at all.

Cinnamic acid, when treated by the above process, gives a brownish colour, which can be distinguished from that given by benzoic acid. The test will detect 0·5 mgrm. of the acid, which represents 25 parts per million when working with 20 grms. of food, and thus in the presence of cinnamic acid the amount of benzoic acid indicated would be high. A mixture of 0·5 mgrm. of cinnamic acid and 0·5 mgrm. of benzoic acid gave a colour equal to that given by 1·5 mgrm. of benzoic acid. This quantity of cinnamic acid would have a very slight effect on the determination of the amounts of benzoic acid generally found in 20 grms. of food.

The depth of colour given by a mixture of 3·5 mgrms. of benzoic acid and 0·5 mgrm. of cinnamic acid could hardly be distinguished from that given by 3·5 mgrms. of the former acid.

Phenyl-acetic acid gives a brownish-purple colour. The test will detect 0·5 mgrm., but this amount would not prevent the detection of benzoic acid in a mixture of 0·5 mgrm. of each acid.

Experience has shown that the dilution of a solution containing a known amount of *m*-diamino-benzoic acid is unsatisfactory, and it is better to find the approximate amount of benzoic acid present to within 1 mgrm. by the method of comparison described, and then to take such a quantity of the solution as will contain between 1 and 3 mgrms. Selected standards, containing known amounts of benzoic acid (differing by 0·25 mgrm.), are tested simultaneously with the unknown substance. In this way the quantity of benzoic acid present can be estimated to within 0·125 mgrm.

PART II

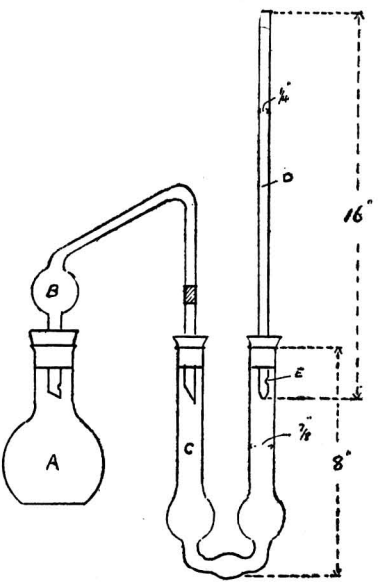
THE APPLICATION OF THE TEST TO THE DETECTION AND DETERMINATION OF BENZOIC ACID IN FOODSTUFFS

APPARATUS.—The apparatus used in this laboratory* is shown in the figure. A is a 300 c.c. flask with a stillhead, B, passing through the rubber stopper in the neck of the flask. C is an ordinary calcium chloride tube which is connected with the stillhead as shown. A straight tube, D, passes through a rubber stopper in the outlet of the tube, C. This tube is drawn out a little at its lower end, and has a hole

* This apparatus was used some time prior to the publication of Leather's paper (ANALYST, 1931, **56**, 299).

in its side at E. The flask, A, is heated on a wire gauze, and the lower limb of C is immersed in boiling water during the distillation.

The food under examination is placed in A, together with the diluting water, acid, and the required amount of salt to make a saturated solution. A little pumice is also added. A seal is made in the tube, C, by means of *N*/1 sodium hydroxide solution. At the end of the distillation, the contents of the tube, together with the washings of C and D, are either evaporated in a porcelain basin, or made up to a known volume and an aliquot part is taken for evaporation, according to the object of the test. The residue is transferred to a boiling tube by means of water, 2 or 3 c.c. only being required, and Mohler's test is carried out.



QUALITATIVE APPLICATION.—Foodstuffs may be tested for absence of benzoic acid by taking 5 grms. in the flask, A, and adding 50 c.c. of water containing 5 c.c. of dilute sulphuric acid, and 20 grms. of salt. One c.c. of *N* sodium hydroxide solution is introduced into C, with sufficient water to form a seal. (In the case of foodstuffs containing much volatile acid, *e.g.* vinegar, an increased amount of sodium hydroxide must be used. For 5 c.c. of vinegar, 0·5 c.c. of 50 per cent. sodium hydroxide solution is taken.) The duration of the distillation is four minutes. The contents of the tube, C, together with washings, are evaporated and transferred to a boiling tube.

Actual results obtained were as follows:

	Benzoic acid.	
	Added. Mgrm.	Found. Mgrm.
Lime juice	0·5	a little less than 0·5
Coffee extract	0·5	more than 0·25
Butter	0·25	more than 0·2
Brawn (decomposed)	0·5	more than 0·25
Sausages	0·5	a little more than 0·25
Jam	0·5	0·5
Cheese	0·5	a little less than 0·5
Milk (8 minutes' distillation)	0·5 0·25	0·5 0·25
Canned cream	0·25	a little less than 0·25
Cider	0·25	0·25
Bottled fruit	0·5	0·5
Essence of ginger (1 per cent. aqueous solution)	0·5	0·5
Candied peel	0·5	more than 0·25

The above process has to be modified in the following manner when applied to articles of food containing much volatile acid, *e.g.* pickles, sauces, vinegar, etc.:

After the distillation, the contents of tube, C, are transferred to a separator, made acid, and extracted with three portions of 15 c.c. of a mixture of equal parts

of ether and petroleum spirit. The combined extracts are washed three times with 1 c.c. of water, and the benzoic acid is then extracted three times with $N/10$ sodium hydroxide solution, 1 c.c. being used for each extraction. The alkaline extracts are collected in a boiling tube and the remainder of the test carried out as described.

	Benzoic acid.	
	Added.	Found.
	Mgrm.	Mgrm.
Vinegar (malt)	0·5	0·5
Sauce (7 minutes' distillation)	0·25	0·25

It is clear from the above results that 50 to 100 parts of benzoic acid per million are easily detected by this process; and should benzoic acid be found, an approximate estimate of the amount present can be obtained.

The presence of salicylic acid (or of any of those substances which give a yellow colour in the test) will not prevent the detection of benzoic acid. The yellow colour becomes orange to deep orange, according to the quantity of benzoic acid present.

Three tests may be quoted:

	Benzoic acid. Mgrms.	Salicylic acid. Mgrms.	Result.
1.	0·5	0·5	Easily detected; colour, orange
2.	2·0	2·0	Easily detected; colour, deep orange
3.	2·0	6·0	Easily detected; colour, orange

These colours cannot be mistaken; once having seen the marked difference between the colour given by benzoic acid and the yellow of salicylic acid, one could never fail to detect the former when present. It is extremely unlikely that excess of salicylic acid would be present.

QUANTITATIVE DETERMINATION.—*General Method.*—The approximate amount of benzoic acid must be known, and, in the majority of cases, the qualitative examination will give this accurately enough for the purpose; but it is suggested that, when dealing with those foods in which benzoic acid is allowed by the Public Health (Preservatives, etc., in Food) Regulations, a combination of the qualitative and quantitative processes be made (*vide infra*).

The approximate amount of benzoic acid having been found, such a quantity of the foodstuff is taken as will contain not more than the amount of benzoic acid given in the following table for the particular article of food under examination. It is better to work with less than the maximum amount.

Maximum amount in mgrms. of benzoic acid that should be distilled.

7 mgrms. for	5 mgrms. for	3 mgrms. for
Lime juice	Marmalade	Vinegar
Cider	Jam	Fats (butter)
Mineral water	Sauce	Margarine, etc.
Non-alcoholic wine	Pickles	Canned cream
Brewed ginger-beer	Brawn	Milk
	Potted meat	Cheese (not more than 2 mgrms.)
	Coffee extract	Cream
	Preserved fruit	All foods containing much volatile fatty acids
		Sausages
		Fish paste

ต้นฉบับไม่มีหน้า 229 - 260

NO THIS PAGE IN ORIGINAL

removed in a stream of nitrogen until the reaction was neutral to litmus. The liquid (about 10 litres) was then decanted from the solid portion, and 4 c.c. of 25 per cent. phosphoric acid were added. As a result of preliminary experiments with numerous solvents and inorganic precipitants the procedure finally adopted was to evaporate the solution to 2 litres and to shake the resulting liquid with a 4-fold volume of acetone, and, after 16 hours at 0° C., to remove the solvent by distillation in carbon dioxide; these operations were repeated 5 times, in the last instance, after evaporation of the juice to 20 c.c. The resulting crystalline precipitates were collected and re-precipitated with acetone from a solution in methyl alcohol and water, and stored in nitrogen at 0° C. A solution of the solid was then neutralised with a solution of barium hydroxide in methyl alcohol, and 2 *N* sulphuric acid was added, followed by 75 c.c. of 10 per cent. lead acetate solution, the excess of which was removed by precipitation with hydrogen sulphide, any residual gas being expelled by boiling under reduced pressure. The solution was finally extracted with ether for 6 hours, the residue left on evaporation of the resulting liquid was extracted with acetone, which was then removed by evaporation, and a solution of the residue in water was used for subsequent experiments. The principal stages of the process were controlled by titration of the reduction-capacity with 2·6-dichlorophenol indophenol as indicator (*cf.* ANALYST, 1929, **54**, 176). This dyestuff, which changes from red to violet between *p*H 4 and 5, is blue at *p*H 7. The usual procedure was followed (*loc. cit.*), a slightly acid solution of the dye (to litmus) being added to the solution under investigation until a blue colour permanent for 1 minute was obtained. The solution was standardised against 0·01 *N* titanous chloride solution by addition of the latter to 20 c.c. of the former, containing a drop of dilute acetic acid and sufficient sodium acetate just to produce a blue colour which is destroyed by the titanium solution at the end-point. The titanous chloride was standardised against ferric ammonium sulphate in the usual way (Knecht and Hibbert). Standardisation of the indicator directly against the iron salt may also be used, but appears to be less satisfactory. The final purified preparation from the lemon juice was thus found to have an "apparent equivalent weight" of 500 (*e.g.* 10 mgrms. of dry substance, requiring 10 c.c. of 0·001 *N* indicator solution corresponds with an "apparent equivalent weight" of 1000). The preparation was slightly acidic, soluble in acetone, water or in methyl or ethyl alcohols, but insoluble in chloroform, benzene or ether, and gave a pale-blue colour in ultra-violet light similar to that from the original juice. The Molisch carbohydrate reaction was strongly positive, but the phenol and tannin tests were negative (*cf.* Agopian and Bezssonoff, *Eng. Pat.*, 1921, 168,903; *French Pat.*, 1925, 595,537; Rygh, Rygh and Laland, ANALYST, 1932, **57**, March issue; Zilva, *id.*). The substance contained a little nitrogen, and gave a positive xanthoprotein reaction, but other protein reactions (ninhydrin, biuret and precipitation tests) were negative. Biological experiments on guinea-pigs indicated an appreciable antiscorbutic power in a daily dose of the preparation corresponding with 1 c.c. of fresh juice, but both the juice and preparation lose this power after oxidation by chlorine water, the effect of hydrogen peroxide being less marked. The results indicate that the reduction-capacity is a measure of the antiscorbutic properties, and that the reducing substance is the carrier of vitamin *C*. J. G.

Toxicological and Forensic

Toxicity of Thiophen. F. Flury and F. Zernik. (*Chem. Ztg.*, 1932, **56**, 149.)—According to Rambousek's experiments (*Gewerbliche Vergiftungen*, 1911, p. 259), the vapour of thiophen is somewhat less poisonous than that of benzene. Experiments made by the authors, in conjunction with Thieme, show that the action of the vapour of pure thiophen is qualitatively similar to that of pure benzene vapour, but is distinctly the more poisonous. With mice, breathing of air containing 0·01 grm. of thiophen per litre produced collapse after 0·75 to 2·5 hours, and, in some cases, subsequent death, which was never caused by benzene in the same concentration. With 0·03 grm. of thiophen vapour per litre of air, death always occurred within 20 to 80 minutes, whereas a corresponding proportion of benzene vapour rarely proved fatal. It is possible that the toxicity of benzene may sometimes be greatly increased by the presence, as impurities, of unsaturated sulphur compounds. Thus, treatment of benzene containing ethylene with carbon disulphide may give rise to dichlorodiethyl sulphide ("mustard gas"). (*Cf.* Ditmar, *Chem. Ztg.*, 1931, **55**, 770.) T. H. P.

Group-specific Substances in Forensic Medicine. R. B. Lloyd. (*Brit. Med. J.*, 1932, 284–285.)—There are four blood groups dependent upon isoagglution reactions, the constitution and inheritance of which is best explained by Bernstein's hypothesis of three multiple allelomorphic factors A, B, and O.

Moss Group I = A + B
II = A + B or A + O
III = B + B or B + O
IV = O + O

The blood groups which contain the A factor are distinguished by the presence in the tissues and body fluids of Forssman's antigen. The serum of rabbits injected with this antigen becomes lytic for sheep red cells, owing to Forssman's heterophile antibody. Such a serum is prepared by injecting rabbits with washed Group II corpuscles. This anti-A serum is decomplemented, and by absorption with Group IV corpuscles anti-human precipitin is removed. This serum, still containing Forssman antibody, has been used by Schiff* to determine whether or not human material is derived from the A-containing groups. The test is carried out shortly as follows:—*Stage* 1—Incubate the extract or fluid, etc., with the test serum; *Stage* 2—Add sheep cells and complement. *Result*—If the extract contains the "A substance," there will be no haemolysis and *vice-versa*.

The test should prove useful, as it enables one to examine material other than blood, *e.g.* semen, saliva, and the organs of a cadaver. Further, the antigenic substance is a very stable body, and is often active in high dilution. But the widespread occurrence of animal species which contain Forssman's antigen limits the applicability of the test in medico-legal work to material the origin of which is unquestionably human, and uncontaminated in any way by such animal matter. Finally, it is not possible to immunise any animal against the B or O substances, which again limits the usefulness of the test. D. M. P.

* *Über die gruppenspezifischen Substanzen des menschlichen Korpers.* By F. Schiff. (1931.) Gustav Fischer, Jena.

Bacteriological

New Autotrophic Bacterium which Oxidises Ammonia directly to Nitrate and Decomposes Petroleum. C. B. Lipman and L. Greenberg. (*Nature*, 1932, **129**, 204–205.)—A coccus or cocco-bacillus isolated from petroleum from a well over 8700 ft. deep, variable in size and somewhat so in shape, was found to grow well under strictly autotrophic conditions in an inorganic salt medium with ammonium sulphate or potassium nitrate as the source of nitrogen. It was found to oxidise the ammonia directly to nitrate, and this power was apparent much more quickly than with the nitrifying bacteria. It was also able to decompose petroleum without apparent gas formation, except for the end-product carbon dioxide, and it is apparently a facultative aerobe. D. G. H.

Chemistry of White Rots of Wood. II. W. G. Campbell. (*Biochem. J.* 1931, **25**, 2023–2027).—In a previous communication Campbell (*Biochem J.*, 1930, **24**, 1235) pointed out that comparatively little experimental work has been done on the chemistry of the particular type of wood decay in which lignin is decomposed, and, further, that it is unknown whether such decomposition is invariably accompanied by simultaneous decomposition of the wood carbohydrates. He showed that in the decay of wood by *Polystictus versicolor* (Linn.) Fr. carbohydrates are depleted as well as lignin. Falck (*Ber.*, 1927, **60**, 225) has concluded that in wood decayed by *Fomes annosus* cellulose is decomposed in the advanced stages. The study of the so-called white rots has now been continued by examination of the respective effects of *Armillaria mellea* (Vahl.) Fr. on beech wood, *Polyporus hispidus* (Bull.) Fr. on ash heartwood, and *Stereum hirsutum* Fr. on oak sapwood. It is concluded that the three samples of white rots examined have only one feature in common, and that this is shared by the white rot caused by *Polystictus versicolor*. In no case is there marked increase in the total alkali-solubility of the major components of wood substance, and, in the case of *Polystictus versicolor* and *A. mellea*, this is due to the fact that one component—namely, the pentosans not in the cellulose—becomes less soluble in 1 per cent. sodium hydroxide solution as decay proceeds. The white rots in question are thus sharply differentiated from the brown rots, for, in some of the latter, even in very advanced stages of decay, the residual wood substance is much more soluble in alkali than the original sound wood. Whilst it has been established that the detailed chemical effect on wood substance of both *Polyporus hispidus* and *Stereum hirsutum* is essentially similar in all respects, this effect can in turn be differentiated from that of *Polystictus versicolor* when the manner in which pentosans are decomposed is considered. *Polystictus versicolor* attacks the pentosans not in the cellulose to a greater extent than the pentosans which are associated with the cellulose, whereas *Polyporus hispidus* and *Stereum hirsutum* attack the pentosans in the cellulose, and have little or no effect on the pentosans not in the cellulose, at the stages of decay which were examined. In view of the effect on the alkali-solubility of wood substance of the decay caused by *Armillaria mellea*, and the fact that the decayed wood possesses to some extent the visual characteristics of a white rot, it must be concluded that in this type of rot a stage can exist in which lignin is apparently unaffected by the

fungus; at the stage examined, decay by *Armillaria mellea* possesses chemical characteristics of both brown and white rots, but it may, with reason, be described as a variety of white rot. It has long been considered by biologists that in certain white rots of wood, where patches or pockets of a white material are found to occur, the fungi concerned have a selective action on lignin, and that the white residue consists entirely of cellulose. In the case of the "pocket" rot caused by *Stereum hirsutum*, it is concluded that this fungus cannot be credited with a selective action on lignin. The white patches were found to represent localities in a decaying mass in which decomposition has progressed to a greater extent than in the surrounding material. In these localities cellulose is decomposed as well as lignin, and, even after the development of the white colour, some lignin remains.

P. H. P.

Organic Analysis

Sensitive Means of Detecting Reducing Carbohydrates. P. K. Bose. (*Z. anal. Chem.*, 1932, **87**, 110–114.)—When 2 c.c. of 25 per cent. sodium carbonate solution are treated with one drop of a 1 per cent. solution of *o*-dinitrobenzene in alcohol and a small quantity of an aqueous solution of a reducing sugar, and the mixture is heated for not quite one minute over a small flame, a deep violet coloration is produced. The smallest amount of sugar thus detectable in 1 c.c. of solution is 0·000006 grm. of dextrose, laevulose, galactose, mannose, rhamnose (hydrate), or lactose, 0·000003 grm. of arabinose, and 0·00001 grm. of maltose. In all cases, the colour appeared after the heating had lasted for 15 or 20 seconds, but it vanished if the heating was continued or if the liquid was left for longer than one minute. The test gave positive results with the ordinary reducing sugars and with glyceraldehyde, 4-β-glucosido-α-mannose, 4-β-galactosido-α-mannose, α-galacturonic acid, and penta-acetyl-β-glucose. Negative results were obtained with the ordinary non-reducing sugars and polysaccharides, glycerol, sorbitol, mannitol, dulcitol, α-methylglucoside, α-methylgalactoside, α-methylmannoside (α-methylmannopyranoside), 2:3:4:6-tetramethylglucose, 3:4:6-trimethylmannonic lactone, salicin, amygdalin, and lactic acid.

Hence, the reaction is shown only in presence of the grouping,

$$-CH(OH)\cdot CH(OH)\cdot O-,$$

and is negative with compounds containing the group

$$-CH(OH)\cdot CH(OR)\cdot O-,\quad -CH(OH)\cdot CH_2\cdot OH,\quad -CH(OH)\cdot CO_2H,\quad \text{or}\quad -CH(OCH_3)\cdot CH(OH)\cdot O-.$$

Penta-acetyl-β-glucose appears to be an exception, but the positive reaction obtained is probably due to liberation of glucose under the hydrolysing action of the alkali. The only aldehyde, either aliphatic or aromatic, which gives the reaction is formaldehyde in relatively high concentration; 5 drops of 0·58 per cent. formaldehyde solution fail to give the coloration under the conditions given above.

T. H. P.

Colour Reaction of Japanese Acid Clay upon Carotene in Palm Oil. K. Kobayashi, K. Yamamoto and J. Abe. (*J. Soc. Chem. Ind. Japan*, 1932, **35**, 35B.)—The palm oil used was obtained from the Eastern Archipelago, and

had the following constants:—Sp. gr. at 30° C., 0·9110; n_D^{30}, 1·477; acid value, 50·3; saponification value, 213·3; iodine value, 50·3. Japanese acid clay gives the deep bluish-green colour much more intensely than ordinary clays, kaolin, silica gels, etc. The best media to use are benzene, petroleum spirit, carbon disulphide, carbon tetrachloride, or chloroform. Ether, methyl or ethyl alcohols and acetone give no coloration. Colour reactions similar to the above are obtained with sulphuric acid, anhydrous zinc chloride, anhydrous aluminium chloride, phosphorus pentoxide, anhydrous ferric chloride, and anhydrous antimony chloride. It is considered that, in the case of Japanese acid clay, the colour is caused by polymerisation, rather than by condensation or dehydration. A study has been made of the absorption bands shown by the pigment extracted from palm oil by alcohol and by carotene from carrot, and, from the results, the authors conclude that the carotenes from these two sources are the same. R. F. I.

Horse Fat. J. Pritzker and R. Jungkunz. (*Z. Unters. Lebensm.*, 1932, **63**, 30–37.) The following data were obtained from fats prepared by the authors from 4 horses (10 to 21 years old):—Sp. gr. (15° C.), 0·9125 to 0·9198; refractometer reading, 51·5 to 53·1; acid value, 0·4 to 1·5; ester value, 194·2 to 196·0; saponification value, 195·6 to 196·7; iodine (Hanus), 74·4 to 78·5; Reichert–Meissl value, 0·33; Polenske value, 0·8; unsaponifiable matter, 0·31 to 0·50 per cent. (yellow-green in colour); cholesterol, 0·1 per cent. (m.pt. of twice-crystallised acetate, 111° C.); solid fatty acids, 24·3 to 27·0 per cent.; *iso*-oleic acid (*cf.* Grossfeld and Simmer, ANALYST, 1930, **55**, 451), 0·17 to 0·23 per cent.; arachidic acid, nil; Kreis rancidity reaction, negative; Bellier's reaction, red to orange-yellow; nitric acid reaction (fat layer), dark-brown. The colours of the fats were yellowish-brown, the consistence at 20° C. was oily, and the odour and taste similar to those of rape-seed oil. The fatty acids had refractometer reading, 38·6 to 40·3; neutralisation value, 203·8 to 206·2; mean molecular weight, 272·1 to 275·2; m.pt., 34·5° to 38·8° C. These results are compared with those of previous workers, and it is concluded that the maximum value of 69 for the refractometer reading, frequently given in the literature arises from a misprint for 59 in the early sources from which the data were taken (*cf.* König, *Chem. der menschlichen Nahr. und Genussm.*, 1910, **3**, 420). Fractional precipitation of the lead salts of the fatty acids (Kreis and Roth, *Z. Unters. Lebensm.*, 1913, **25**, 81) enables horse fat to be distinguished from lard, but will not detect 20 per cent. of the former in the latter. J. G.

A Hydrocarbon in Ishinagi Liver Oil. M. Tsujimoto. (*Bull. Chem. Soc. Japan*, 1931, **6**, 237–239.)—A hydrocarbon obtained from the liver oil of ishinagi (*Stereolepsis ischinagi*, Hilgendorf) has been subjected to a preliminary examination. The liver oil had the following characteristics:—Sp. gr. 15°/4° C., 0·9358; $n_D^{20°}$, 1·5070; saponification value, 146·2; iodine value (Hanus), 155·5; acid value, 11·8; unsaponifiable matter, 22·36 per cent. The unsaponifiable matter was treated with methanol, the precipitate deposited from the solution on standing was repeatedly washed, and a yellow-orange viscous liquid was obtained, retaining its colour after treatment of its ethereal solution with animal charcoal. The still impure substance had a high specific gravity (0·942 at 17°/4° C.) and $n_D^{17°}$, 1·54;

it was highly unsaturated (iodine value, 290·8), and formed bromine and hydrogen chloride addition compounds, the former containing 67·07 per cent. of bromine and the latter 30·85 per cent. of chlorine. D. G. H.

Unsaponifiable Matter of Calamary Oil. M. Tsujimoto. (*Bull. Chem. Soc. Japan*, 1931, **6**, 289–293.)—The calamary oil obtained from the liver of *Ommastrephes Sloani pacificus* (Steentrap) (*J. Soc. Chem. Ind. Japan*, 1927, **38**, 865) had the following characteristics:—Sp. gr. 15°/4°, 0·9298; $n_D^{20°}$, 1·4828; saponification value, 175·8; iodine value, 184·1; acid value, 20·5; unsaponifiable matter, 4·5 per cent. The methanol solution of the unsaponifiable matter was cooled, separated from the crystals, the mother liquid was concentrated, and, eventually, two solid and one liquid parts were obtained, which were examined in detail. The presence of cetyl, batyl, and selachyl alcohols was confirmed, oleyl and chimyl alcohols were probably present, together with a small amount of more highly unsaturated alcohols, and 48 per cent. of cholesterol was found. D. G. H.

Dibasic Acids in Sumach Berry Waxes [Japan Wax]. M. Tsujimoto. (*Bull. Chem. Soc. Japan*, 1931, **6**, 337–341.)—The waxes, or, more correctly, fats, of the berries of four species of Japanese sumach trees have been examined, and were found to have the following characteristics:

Fat.	*Rhus vernicifera* DC.	*Rhus sylvestris* S. et Z.	*Rhus trichocarpa* Miq.	*Rhus toxicodendron* L.
Colour:	Brownish yellow.	Brownish black.	Dark brown.	Brownish black.
Sp. gr. at 100°/4° C.	0·8653	0·8679	0·8639	0·8895
M.pt.	52·5–53° C.	51–52° C.	49–50° C.	38–39° C.
Acid value	3·1	6·2	14·1	—
Saponif. value	209·5	202·9	205·2	208(?)
Iodine value (Wijs)	12·9	24·9	16·8	82(?)
Unsaponif. matter, per cent.	0·62	—	0·78	—
Fatty Acids.				
M.pt.	62° C.	56–57° C.	54–55° C.	ca. 51–52° C.
Neutralisation value ..	212·9	214·1	212·6	212(?)
Iodine value (Wijs)	12·8	14·6	17·1	26
Petroleum spirit insoluble substance (crude dibasic acids), per cent.	6·3	1·6	1·9	6·3

The fatty acids were treated by the lead salt precipitation method, and the crude dibasic acids were recrystallised. In urushi wax (*R. vernicifera*) these amounted to 6·3 per cent. of the fatty acids, and appeared to consist chiefly of eicosane dicarboxylic acid, $C_{22}H_{42}O_4$. In Yama-hazé wax (*R. sylvestris*) the dibasic acids amounted to 1·6 per cent., and in tsuta-urushi wax (*R. toxicodendron*) to 6·3 per cent., consisting mainly of heneicosane dicarboxylic acid, $C_{23}H_{44}O_4$. In the case of the Yama-urushi (*R. trichocarpa*) wax the nature of the substance isolated (m.pt. 102° C.) was not determined. D. G. H.

Dibasic Acids in Japan Wax. M. Tsujimoto. (*Bull. Chem. Soc. Japan*, 1931, **6**, 325–337.)—Japan wax is obtained in Japan only from the mesocarp of

the berries of *Rhus succedanea*, L. (Haze-noki), which grows chiefly in the south-west of the country. The waxes used in the investigation had iodine values ranging from about 17·5 to 20·2, owing to a certain admixture of kernel oil, and it should be noted that, during bleaching, changes in composition occur and figures recorded outside Japan usually refer to such compounds, and are not comparable with those of the genuine wax. The percentage of dibasic acids (dissolving with difficulty in petroleum spirit) in the fatty acids varied from 5·2 to 6·1 per cent. The acids can be separated by fractional precipitation with a calculated quantity of lead acetate in alcoholic solution, or by distillation of the esters, and an investigation of their composition showed the main constituent to be heneicosane dicarboxylic acid, $C_{23}H_{44}O_4$, although the acid $C_{22}H_{42}O_4$ (eicosane dicarboxylic acid) also appeared to be present in fair proportion. The dibasic acids have the property of imparting a fineness of structure and opaqueness, involving coherency and tenacity, to the fatty acids, while the characteristic property of Japan wax—that it can be kneaded without adhering to the fingers—is due to the presence of glycerides of the dibasic acids. The term "japanic acid" of Geitel and van der Want (*Z. prakt. Chem.*, 1900, **61**, 151) is to be avoided, since this is not the only dibasic acid in Japan wax. D. G. H.

Determination of *o*-Cresol. F. M. Potter and H. B. Williams. (*J. Soc. Chem. Ind.*, 1932, **51**, 59–60T.)—The method depends on the relation between the m.pt. and composition of mixtures of cresylic acid and cineole, and is, therefore, the reverse of that proposed by Cocking for the determination of cineole in eucalyptus oils (ANALYST, 1920, **45**, 370). A standard curve was drawn from the following data, which were obtained from mixtures of pure *o*-cresol (cryst. pt. 30·95° C.) and cineole (cryst. pt. 1° C.; b.pt., 175·5 to 177·4° C.):—One hundred per cent. of *o*-cresol, cryst. pt. of *o*-cresol-cineole complex 55·7° C.; 95, 54·2; 89·85, 52·5; 80, 49·2; 63·5, 42·8; 50, 36·3; 40, 30·3; 12·5 per cent., approx. 5° C. The apparatus consisted of a test-tube (0·75 inch diameter) containing the mixture, surrounded by a larger tube (1 inch diameter), which served as an air-jacket, and was itself immersed in a 400 c.c. beaker (2·5 inch in diameter), containing water at 5° to 10° C. below the cryst. pt. (determined from a preliminary trial). A quantity of sample (*e.g.* 2·80 grms. of cresylic acid) was mixed with cineole (*e.g.* 4·00 grms.), in such a proportion as to obtain the molecular ratio required by the cresol-cineole compound, and the mixture was then heated to about 15° C. above the crystallisation point, and the temperature read to 0·1° C. every 30 seconds during cooling, the mixture being stirred gently for 5 seconds before each reading. The true crystallisation point, independent of supercooling, was obtained by the method of Bell and Herty (*J. Ind. Eng. Chem.*, 1919, **11**, 1124), *i.e.* the readings were taken at noted intervals for 5 minutes after the temperature-rise due to crystallisation, and the portion of the temperature-time curve obtained after this rise was extended backwards until it intersected the portion of the curve preceding the rise at a point which corresponds with the true crystallisation point. A rise in temperature of 1° C. should be regarded as the maximum permissible, and supercooling, which is most pronounced when the *o*-cresol content islow, should be avoided by preliminary rapid cooling, or by seeding. Water should be avoided

or removed by distillation, since (*e.g.*) 10 per cent. depresses the crystallisation point by nearly 10° C. Trial experiments on mixtures of pure *o*-cresol with the isomeric cresols, phenol, xylenols, neutral oils or guaiacol, gave results within 0·5 per cent. of the true value; since the optimum accuracy is obtained when over 30 per cent. of cresol is present, known weights of the pure substance should be added to the sample, if necessary, to give at least this quantity. J. G.

Inorganic Analysis

Analysis of Red Lead and Lead Dioxide. N. Busvold. (*Chem. Ztg.* 1931, **56**, 106–107.)—As an equally accurate alternative to the iodimetric method, which, owing to the consumption of iodide, is considered too costly for routine works' purposes, the following volumetric method is proposed for the evaluation of red lead and lead peroxide. It depends on the reduction of lead dioxide by hydrogen peroxide. A 5-grm. sample, contained in a tall 250 c.c. beaker, is mixed with 20 c.c. of nitric acid (sp. gr. 1·2) by means of a glass rod, and 50 c.c. of water are added. The suspension is titrated, very slowly and with constant stirring, with dilute hydrogen peroxide solution (10 c.c. of 30 per cent. hydrogen peroxide diluted to 250 c.c., standardised with permanganate) until practically all of the solid material has dissolved, and a brown turbid solution is obtained; the liquid is then heated on a water-bath until the solution becomes clear. (In the test experiments about 1 mgrm. of insoluble residue remained, which was neglected, since it did not contain lead peroxide). The small excess of hydrogen peroxide remaining in the solution is back-titrated with permanganate. The amount of lead peroxide is calculated from the equation $PbO_2+H_2O_2=H_2O+O_2+PbO$. Good agreement between the results given by this method and the usual iodide method was obtained. In the course of the preliminary work the author tested Lux's method, in which red lead is dissolved in dilute nitric acid with an excess of standard oxalic acid, the dissolved lead is precipitated by sulphuric acid, and the excess of oxalic acid remaining unoxidised is determined by titration with permanganate. This method was found to yield inaccurate results, owing to occlusion of oxalic acid in the lead sulphate precipitate. S. G. C.

Chemical Corrosion of Lead in Presence of Phenol. E. Da Fano. (*Giorn. Chim. Ind. Appl.*, 1932, **14**, 18–21.)—Not every tar is suitable for coating underground telephone cables as a means of protecting the leaden sheath of the cable from the corrosive action of the soil. The tar used must answer certain requirements. If it contains too much oil, this is partly carried away by the soil-water, which is enabled to penetrate to the lead and exert its corrosive effect. On the other hand, an excessive proportion of dry pitch in the tar may lead to cracking of the coating at the low temperatures encountered during transport, laying, or use. It seems, moreover, that phenol may accelerate the corrosion of lead, as it takes part in a cyclic process in which it is constantly regenerated. Hence, phenol should be present in the tar only in the smallest amount possible, although, since it corrodes lead only when water and carbon dioxide are also present, it is harmless as long as the tar coating remains continuous and impervious to water. The best service in this direction is given by specially-prepared tar having a dropping point (Ubbelohde) between 35° and 42° C. T. H. P.

Analysis of Silver Plating Solutions. R. M. Wick. (*Bur. of Standards J. Research*, 1931, 7, 913–933.)—Tests have been made of feasible methods for the determination of the constituents and impurities present in alkaline cyanide silver-plating solutions, and recommended methods are given for determining free cyanide, total cyanide, total effective cyanide, silver, carbonate, chloride, iron, copper, mercury, and ammonia. *Free cyanide* (*i.e.* the excess of alkali cyanide above the amount required to form complex cyanides of the metals present in solution):—To 20 c.c. of the plating solution is added 0·1 to 0·2 grm. of potassium iodide, the liquid is diluted to 250 c.c. and titrated with 0·1 *N* silver nitrate solution. The end-point is the first appearance of opalescence, and is best observed against a black background; 1 c.c. 0·1 *N* $AgNO_3 \equiv 0{\cdot}00980$ grm. NaCN or 0·01302 grm. KCN. *Total cyanide.*—Ten c.c. of the solution are diluted to 250 c.c. in a 500 c.c. flask of a distillation apparatus; 10 c.c. of dilute sulphuric acid (1:1) are added by means of a dropping funnel. The liquid is distilled down to half its original volume, the distillate being received in a flask containing 100 c.c. of sodium hydroxide solution (2 per cent.). The total cyanide, now present in the distillate as sodium cyanide, is determined by the silver nitrate titration described above. *Total effective cyanide* (*i.e.* the total cyanide, exclusive of ferrocyanide).—Fifty c.c. of the solution are diluted to 500 c.c.; to an aliquot part of this (25 c.c.), 50 c.c. of an ammoniacal sodium chloride solution (water containing 10 per cent. of sodium chloride and 0·2 per cent. of concentrated ammonia) are added, the liquid is titrated with 0·1 *N* iodine solution to the first visible turbidity, diluted to one litre, 5 c.c. of starch indicator (0·5 per cent.) added, and the titration continued until a permanent blue colour forms; 1 c.c. 0·1 *N* $I_2 \equiv 0{\cdot}00245$ grm. NaCN or 0·00326 grm. KCN. *Silver.*—The silver is determined by the Volhard thiocyanate titration method after evaporation "to fumes" with sulphuric acid to remove cyanide. If the presence of mercury is suspected the silver should be separated as silver chloride. *Carbonate.*—To the solution remaining after the determination of *free cyanide*, phenolphthalein indicator is added, and the liquid is titrated with 0·5 *N* hydrochloric acid until the pink colour is completely discharged; 1 c.c. of 0·5 *N* HCl ≡ 0·0530 grm. of Na_2CO_3 or 0·0691 grm. of K_2CO_3. *Chloride.*—Ten c.c. of the solution are heated with 20 c.c. of concentrated nitric acid (if the solution contains a high proportion of chloride, an excess of silver nitrate should be added) until the precipitate which forms becomes nearly white, and then for a further period of half-an-hour, the volume being kept constant by the addition of nitric acid; the liquid is diluted to 75 c.c., rendered alkaline with ammonia, and any precipitate of ferric hydroxide is filtered off, dissolved in nitric acid, and re-precipitated. The chloride in the combined filtrates is precipitated in the usual manner as silver chloride, which is weighed. *Ammonia.*—Fifty c.c. of the solution are titrated with 0·5 *N* silver nitrate solutions as described above for the determination of free cyanide. The liquid is diluted to 250 c.c., 10 c.c. of sodium hydroxide solution (10 per cent.) are added, and the ammonia is distilled and titrated as in the ordinary Kjeldahl method. *Iron, Copper, Mercury.*—Normal methods are employed, the cyanide being first expelled by heating with sulphuric acid. S. G. C.

Determination of Beryllium in Alloy Steels. H. Eckstein. (*Z. anal. Chem.*, 1932, **87**, 268–273.)—The following simple technical method for aluminium-free steel is based on the solubility of beryllium hydroxide in alkali. The borings (0·5 grm.) are dissolved in warm, strong hydrochloric acid, the solution oxidised with nitric acid (3 c.c.), and evaporated for the precipitation of tungstic acid and silica. The chloride filtrate is evaporated with 15 c.c. of sulphuric acid (1:1) till heavy fumes are evolved; after addition of a little water, the evaporation is repeated, and the mass taken up in 250 c.c. of water. The hot solution is treated with a little silver nitrate and ammonium persulphate, boiled for 10 minutes, and made faintly ammoniacal. The precipitate is collected, washed, dissolved in a little sulphuric acid and 5 c.c. of saturated ammonium persulphate solution, and the precipitation repeated. The precipitate is dissolved in dilute hydrochloric acid, and the solution poured into 200 c.c. of 20 per cent. carbonate-free caustic potash solution, which is vigorously stirred during addition and for 2 minutes afterwards. After an hour's standing, the precipitate is filtered off and washed, and the filtrate carefully acidified with hydrochloric acid (1:1), treated with a few drops of sulphurous acid and a slight excess of ammonia, and left overnight. Mechanical agitation for half-an-hour is stated to bring about quantitative precipitation of the hydroxide. The precipitate is collected, washed with water containing a few drops of ammonia, and ignited to constant weight over a blast burner. As it always contains a little iron, it is dissolved in hydrochloric acid, the iron determined volumetrically, and the calculated amount of Fe_2O_3 subtracted.

Ferroberyllium.—The solution of the metal in *aqua regia* is evaporated to dryness, the silica made insoluble by two evaporations with hydrochloric acid, and filtered off. An aliquot part of the filtrate (equivalent to 0·2 grm.) is precipitated with ammonia, and the precipitate ignited to constant weight as before. It is dissolved in hydrochloric acid, the iron determined volumetrically (method not stated), and the ferric oxide thus found is deducted. W. R. S.

Determination of Beryllium. L. Fresenius and M. Frommes. (*Z. anal. Chem.*, 1932, **87**, 273–285.)—A painstaking critical investigation of the published processes, with test determinations of beryllium in complex alloy steels. The authors conclude that the five processes tested give concordant results provided all sources of error are eliminated, as in the following directions:

The drillings (5 grms.) are treated with 100 c.c. of strong hydrochloric acid and enough nitric acid to oxidise tungsten; the solution is diluted, boiled, and filtered. The filtrate is evaporated to a syrup and extracted with ether in the usual manner; the extracted aqueous solution is evaporated with sulphuric acid to the appearance of white fumes, the acid diluted, treated with a few grms. of solid persulphate and a little silver nitrate, and boiled until the chromium has been converted to chromate. The solution is precipitated with a faint excess of ammonia, the precipitate collected and washed with dilute ammonium nitrate solution, and dissolved in sulphuric acid. Persulphate treatment, precipitation, and filtration are repeated (precipitate = *A*).

(1) *Hydroxyquinoline Method* (ANALYST, 1928, **53**, 508).—For steel containing aluminium. The precipitate *A* is dissolved in a small excess of hydrochloric

acid, the solution approximately neutralised with ammonia, treated with 0·2 grm. of oxalic acid, and warmed to 60° C.; the reagent (a 5 per cent. solution of the base in 2 *N* acetic acid) is added during stirring, followed by 2 *N* ammonium acetate solution (25 c.c. in excess of the volume required to form a permanent precipitate). The precipitate is collected in a porous crucible and washed with hot water until the washings are colourless. The beryllium in the filtrate is precipitated with a slight excess of ammonia. The precipitate is collected, washed with ammonium nitrate solution just alkaline to phenolphthalein, dried, ignited, and heated with hydrofluoric and sulphuric acids. The residual sulphate is decomposed by heating at 1100° C. and the beryllia ignited to constant weight.

Test for Impurities.—The oxide is heated with a little hydrofluoric and sulphuric acids; after some fuming the sulphuric acid is cooled, diluted with a little water, and heating to the fuming stage is repeated. The sulphate is dissolved in 100 c.c. of water; 20 c.c. are tested with molybdate mixture (P_2O_5: *a*), 20 with hydrogen peroxide (V_2O_5: *b*), 10 with persulphate and silver nitrate (Mn_3O_4: *c*); 40 c.c. are precipitated with hydroxyquinoline as before, and the precipitate ignited, weighed, and tested colorimetrically for vanadium if any appreciable quantity was found in *b*; the difference between the weight of the hydroxyquinoline precipitate and the V_2O_5 found by colorimetry, *plus* $(a+b+c)$, is subtracted from the weight of BeO found. (The correction amounted to 0·02 to 0·04 per cent., the steel assaying 0·96 per cent. of beryllium.)

(2) *Caustic Potash Method.*—For steel rich in manganese (*cf.* preceding abstract). The precipitate *A* is dissolved in a minimum of hydrochloric acid, and the solution poured very gradually into a platinum capsule containing 200 c.c. of warm, 20 per cent. caustic potash solution, as free as possible from carbonate. The liquid is boiled and filtered, the filtrate acidified with hydrochloric acid, concentrated, and made faintly ammoniacal; if the precipitate is yellow, it is re-dissolved and re-precipitated with addition of hydrogen peroxide. The liquid is then stirred for 5 minutes and left to clear. The precipitate is collected and washed with faintly ammoniacal ammonium nitrate solution, and the ammonia precipitation (with addition of peroxide if necessary) repeated. The precipitate is ignited to constant weight and tested for impurities as before.

(3) *Basic Acetate Method.*—The precipitate *A* is dissolved in hydrochloric acid; the solution is approximately neutralised with ammonia, diluted to 200 c.c., treated with a few drops of dilute acid and with ammonium acetate (quantity not stated), boiled, and stirred for some minutes. The precipitate is left to settle and filtered off, the filtrate concentrated to 100 c.c. and precipitated with ammonia. The precipitate is ignited, weighed, and tested for impurities (*supra*); phosphorus need not be considered, as it is precipitated with the basic acetate. The latter may be dissolved and re-precipitated, but it never yielded more than 0·01 per cent. Be.

(4) *Tannin Method* (ANALYST, 1928, **53**, 401). (For steel rich in vanadium).—The precipitate *A* is dissolved in dilute sulphuric acid, the solution neutralised with ammonia till faintly turbid, cleared with a drop of acid, treated with 30 grms. of ammonium acetate and 25 grms. of ammonium nitrate, and diluted to 500 c.c. After addition of 8 c.c. of glacial acid and a few drops of hydrogen peroxide the solution is

boiled and a strong excess of 10 per cent. tannin solution added all at once. The precipitate is collected and washed with dilute ammonium sulphate solution: the filtrate should be yellowish-brown, not mauve. The precipitate is dissolved in hot dilute sulphuric acid, the liquid nearly neutralised as before, and the precipitation repeated. The combined filtrates are evaporated with excess of strong nitric acid, and the residue gently heated for removal of ammonium salts, and then dissolved in water. If green, the solution is treated with ammonium acetate and hydrogen sulphide, and the nickel sulphide filtered off. The beryllium is precipitated, as before, with ammonia; the weighed beryllia is tested for impurities other than vanadium, which is precipitated by tannin.

(5) *Ammonium Sulphide Method.*—The precipitate *A* is dissolved in dilute hydrochloric acid, the solution treated with 20 c.c. of 20 per cent. tartaric acid solution, then with hydrogen sulphide, made ammoniacal, and ammonium sulphide is added. The filtrate from the sulphide precipitate is evaporated with excess of sodium carbonate and with potassium nitrate in a platinum dish; the residue is fused, and the melt taken up in water and acidified with hydrochloric acid. The solution is precipitated with ammonia, after addition of hydrogen peroxide if vanadium is present. The precipitation is repeated, the precipitate being ignited, weighed, and tested for impurities as before. W. R. S.

Use of Zinc Oxide in the Determination of Cobalt and Manganese. J. I. Hoffman. (*Bur. of Standards J. Research*, 1931, **7**, 833–892.)—Tests have been made to determine the degree of completeness of the separations of cobalt and manganese from other metals by the zinc oxide method, particularly from the standpoint of steel analysis. The general method of precipitation adopted was to add to the weakly acid solution of the steel in hydrochloric or sulphuric acid (oxidised with nitric acid), contained in a 500 c.c. graduated flask, a suspension of finely divided zinc oxide (50 grms. of zinc oxide in 300 c.c. of water) in small portions at a time, with frequent shaking, until an excess was shown either by the initially brown precipitate becoming lighter in colour, or by a milky appearance in the liquid after allowing the precipitate to settle; the liquid was made up to the mark and an aliquot part of the liquid was filtered through a dry paper, the precipitate not being washed. The precipitate from a steel was found to contain all the iron, tungsten, vanadium, chromium, uranium, zirconium, titanium, aluminium, phosphorus, arsenic and tin; copper, molybdenum and silicon are not, however, completely precipitated. The separation of cobalt from iron is slightly incomplete if only a single precipitation with zinc oxide is made; the results obtained with steels containing from 3·65 to 9·66 per cent. of cobalt were low to the extent of 0·1 to 0·3 per cent.; for special work, therefore, the small quantity of cobalt co-precipitated with the iron should be recovered by dissolving the iron precipitate in hydrochloric acid and repeating the precipitation with zinc oxide. In the case of manganese the separation was found to be complete with one precipitation, no more than a few thousandths of one per cent. being co-precipitated with the iron, even with a steel containing 3·57 per cent. of manganese. The temperature (20° to 100° C.) at which the precipitation with zinc oxide was made had very little influence on the results. The separation of iron from nickel is not very satisfactory, owing to a tendency

for nickel to be retained by the iron hydroxide precipitate, even after two precipitations with zinc oxide, *e.g.* with a high nickel steel (8·44 per cent.) there was a loss of 0·1 per cent. of nickel. Small amounts of sodium carbonate, which might be present as an impurity in the zinc oxide, are not objectionable if only a slight excess of the reagent is added. S. G. C.

Determination of Small Amounts of Sodium by the Magnesium Uranyl Acetate Method. E. R. Caley. (*J. Amer. Chem. Soc.*, 1932, **54**, 432–437.)—The lower limit in the amount of sodium which can be determined by the magnesium uranyl acetate method, as described in *J. Amer. Chem. Soc.*, 1929, **51**, 1664, *et seq.*, has been found to be 0·2 mgrm. Attempts have been made to adapt the method to smaller quantities of sodium by (*a*) using a more concentrated reagent, (*b*) using a more concentrated solution of the sample and less reagent. These modifications, whilst giving some improvement in permitting the determination of 0·1 mgrm. of sodium in pure sodium chloride solutions, were found to be, generally, of less use than the normal method, since other constituents of a sample, *e.g.* sulphates, ammonium salts, potassium salts, showed a greater tendency to interfere, and difficulties were experienced through constituents of the reagent itself crystallising out. S. G. C.

Preparation of Iodine-Free Bromine. G. M. Karns and H. C. Donaldson. (*J. Amer. Chem. Soc.*, 1932, **54**, 442–444.)—A method is described for the preparation of iodine-free bromine for use, *e.g.* in the micro-analytical determination of iodine. The impure bromine is washed by shaking, in a mechanical shaker, with water, into which the iodine passes as iodic acid; several successive quantities of water and long shaking are required to remove the last traces of iodine. The effectiveness of the method was tested by adding 0·1 per cent. of iodine to 453 grms. of bromine, and shaking this with six successive quantities of 500 c.c. of water for a total period of 44 hours. Analysis of the washings showed that the bulk of the iodine was removed in one washing for 4 hours; 100 grms. of the total of 246 grms. of purified bromine finally obtained were found to contain less than 1·5 thousandths of a grm. of iodine by a method in which the bromine was allowed to evaporate through a layer of water containing a small amount of sodium hypobromite; the aqueous liquid was treated with acidified sodium sulphite solution, evaporated to dryness after the addition of potassium carbonate, the residue extracted with alcohol, and the iodine finally determined volumetrically after conversion into iodic acid by bromine water prepared from the purified bromine. S. G. C.

Microchemical

Micro-Determination of Carbon by the Wet Method. (Part I). H. Lieb and H. G. Krainick. (*Mikrochem.*, 1931, **9**, 367–384.)—A micro-determination of carbon by wet oxidation is modified from the Nicloux–Boivin method (*Bull. Soc. chim. biol.*, 1927, **9**, 639–758; 1928, **10**, 1271; 1929, **11**, 1269). In the Nicloux–Boivin method about 16 mgrms. of substance were necessary, and the carbon determinations showed errors of 1 to 2 per cent. In the new method only 2 to 5 mgrms. of substance are used, containing 1 to 3 mgrms. of carbon, and the error

is reduced to 0·1 to 0·3 per cent. The method is suitable for all organic compounds, and particularly biological material, which is often difficult to burn by the dry combustion process. The determination takes about an hour, but does not require continuous attention. The carbon is oxidised by heating it for 30 minutes at 130° to 140° C. with a mixture of silver dichromate and potassium dichromate in concentrated sulphuric acid. The silver dichromate accelerates the reaction, and renders the oxidation complete. The combustion gases are passed in a stream of oxygen through a tube containing a glowing platinum contact (as used in the Pregl halogen determination) behind which is a wad of silver wool to retain any halogens. The gases are then passed into the absorption chamber, containing $N/10$ baryta solution, through a bubbler of fritted glass (Schott, Jena, size of pore G.1), which makes the bubbles sufficiently small for complete absorption when the fritted glass is moistened with a drop of alcohol before use. The excess of baryta in the absorption chamber is then titrated in the cold with $N/20$ hydrochloric acid, with phenolphthalein as indicator. The titration is carried out in the absorption vessel without opening it, the tips of the burettes being fitted through holes in the stopper. In this way contact with the outer air is prevented, and the determination is carried out in a closed system. *Reagents.*—The oxidation mixture, which is purified so as to give no blank value, is prepared from 20 grms. of silver dichromate, 10 grms. of potassium dichromate and 200 c.c. of pure anhydrous sulphuric acid (sp. gr. 1·84). The mixture is heated at 120–130° C. for an hour in a stream of dry oxygen, passing through at the rate of 10 c.c. per minute, and is shaken at intervals. After cooling to 80° C., the mixture is transferred to a bottle with a narrow curved neck to prevent the entrance of dust. The solution contains 150 mgrms. of chromate salts per c.c. *Standard Solutions.*—(1) $N/20$ hydrochloric acid, containing 3 per cent. of barium chloride, dissolved in water (free from carbon dioxide) and standardised with sodium carbonate. (2) $N/10$ baryta solution, containing 1 per cent. of barium chloride, dissolved in thoroughly boiled water, and filtered if necessary. The standard solutions are kept in bottles attached to the micro-burettes, as used by Pregl in the micro-Kjeldahl method (Pregl, *Quantitative Organic Micro-analysis*, 2nd Ed., p. 115).

Oxidation is effected in a glass apparatus with a neck opening into a funnel, with a protecting cap. The chromic acid mixture is poured down the funnel, and the gas inlet tube, passing down the middle, has a ground-glass portion, which acts as the stopper of the oxidising vessel. With this device, on slightly lifting the gas inlet tube, the acid can be added gradually, and the sulphuric acid acts as an effective tap lubricant and prevents any leaks. The oxygen is used at a pressure of 5 to 5·5 cm. of mercury, and, if it contains organic impurities, it should be passed through a tube containing platinum asbestos heated to 600° C. The carbon dioxide and water are absorbed, as in the Pregl micro-combustion process in a bubble counter containing 50 per cent. potash, and a guard-tube containing ascarite and calcium chloride. The absorption apparatus is attached to a Marriotte flask to adjust the velocity and measure the volume of gases. Between the combustion tube and the absorption vessel is a long-handled glass stopcock.

Method.—The absorption vessel and inlet of fritted glass are carefully cleaned, and then 10 drops of neutral alcohol and two drops of 1 per cent. phenolphthalein

are placed in the absorption vessel. The substance to be determined is weighed into a small open capillary tube with a solid handle, and is placed in the oxidising flask. The air is swept out of the apparatus with 40 c.c. of oxygen, at the rate of 6 c.c. per minute. The platinum contacts are heated, and the gas is turned off for a moment while 3 to 4 c.c. of the chromate oxidising mixture are added from the funnel. The oxygen is turned on again, and, while the long-handled stopcock is turned off, 8 c.c. of $N/10$ baryta are added to the absorption vessel from the micro-burette. The oxidation vessel is then immersed in the heating bath already at 70° to 80° C., the stopcock is adjusted to give a flow of 2 to 2·5 c.c. of gas per minute, and the temperature of the bath is raised to 130° to 135° C., and held constant. After about 10 minutes, when the first cloudiness is observed in the absorption vessel, 100 c.c. of oxygen are passed through the apparatus to drive out all the carbon dioxide. The last 50 c.c. may be passed through at the rate of 3 to 3·5 c.c. per minute. The titration is carried out in a stream of oxygen of 3·5 to 4·5 c.c. per minute to stir the liquid. One c.c. of $N/20$ baryta $\equiv$ 0·3 mgrm. of carbon. Fifteen different substances were tested, and excellent results were obtained. J. W. B.

Micro-gravimetric Determinations with Minute Quantities of Material, using the Electro-magnetic Micro-balance. E. Wiesenberger. (*Mikrochem.*, 1932, **10**, 10–26.)—An electro-magnetic micro-balance of the Angström type, as improved by Emich (Abderhalden, *Handbuch der biochem. Arbeitsmethoden*, Abt. 1, [iii], p. 259), is used for the determinations. This can be used for weighing amounts down to 0·015γ. Details of the construction of the balance are given, including the making of the quartz threads and hooks and the fixing of the suspension and pointer. The balance is enclosed in a five-sided beech-wood case, the fifth side of which is made of aluminium, and fixed with wood at a distance of 1·5 cm. from the wall of the room, so as to give an enclosed pocket of air. A small window in the third wall of the case is made of "Robon glass" (Schott, Jena), which absorbs heat rays, and through this the light from a lamp is directed on to a mirror which throws the rays into a telescope, through which the movements of the pointer are observed. During a weighing the magnetised pointer is restored to its original position by altering the current through an electro-magnet, and the weights are compared from the readings on a millivoltmeter. The null point of the balance remains very constant, though it is disturbed by thunderstorms.

The balance is used for the determinations of metals in salts by conversion into the sulphate, and for the electrical determination of copper. The weight used for each determination is 1 to 12γ or 50 to 100 times less than by the Pregl method. The substance is weighed out into a small platinum crucible (diameter of 3 mm.), made of foil, 0·003 mm. thick, with a hook handle of fine platinum wire; the weight is about 5 mgrms. For the determination of salts a small drop of sulphuric acid is placed on the substance by means of a glass thread as fine as hair, on the tip of which a minute drop has been placed with a hair-fine capillary. The micro-crucible is then placed in a porcelain crucible, suspended from a quartz hook and gently heated with the pilot flame of a Bunsen burner. After a few minutes the heating can be increased. Determinations on lead formate, potassium

tartrate and sodium chloride were made, with maximum errors of less than 1 per cent. Electrometric determinations of copper were carried out, using the micro-crucible itself as the cathode on which the copper was deposited as follows:—The copper salt is weighed out and dissolved in 1000 times its quantity of dilute sulphuric acid, so that the liquid almost fills the crucible; this is suspended and supported by the wire which is connected with the source of current. The platinum anode is carefully dipped into the solution, the poles are connected with a single-cell lead electrode, and the solution is electrolysed for 30 minutes at a potential of 2 volts. The crucible is washed with alcohol, and then with water, while the current is still running; it is then disconnected, dipped first into water, and then into alcohol, dried at 120° C. for 5 to 10 minutes, and re-weighed. The results with 7 to 12 mgrms. of copper sulphate and copper rubidium sulphate differed from the theoretical amounts by 0·05 to 0·3 per cent. J. W. B.

Physical Methods, Apparatus, etc.

Compensator for Constant-Volume Gas Burettes. H. R. Ambler. (*J. Scientific Instruments*, 1931, **8**, 374–376.)—The constant-volume gas analysis apparatus, described in ANALYST, 1929, **54**, 517, has been fitted with a compensator for variations in temperature and barometric pressure. The device is found to save time and increase accuracy, and is adaptable to other instruments, of both the constant-volume and constant-pressure types. S. G. C.

Portable Apparatus for Precise Gas Analysis. H. R. Ambler. (*J. Scientific Instruments*, 1931, **8**, 369–373.)—A portable form of the apparatus described in ANALYST, 1929, **54**, 517, has been developed. The size of the apparatus, complete in case, is 15 by 12 by 4½ inches, the weight 8½ lbs., and the attainable accuracy 0·1 per cent. S. G. C.

Interpretation of Photomicrographs (of Leather Fibres). D. Jordan Lloyd and R. H. Marriott. (*J. Int. Soc. Leather Trades Chem.*, 1932, **16**, 57.)—The fibre-bundles of skin are composed of fibres, and these consist of still finer elements, the fibrils. The fibre-bundles are interwoven in all directions, but, for purposes of classification, the types of weave can be defined as vertical, high angle, medium angle (about 45° to the grain surface), low angle and horizontal. Regularity of weave pattern is always found in first-class leathers. The fibre-bundles can be loosely or compactly woven together. A loose weave indicates a soft, and, except in the case of glove leathers, an undesirable leather. The fibres can be thick or thin, and by the thickness of the outline of the fibres, although some fibres may be naturally thin, such fibres can be differentiated from fibres in which the thinness is caused by errors in process, provided that the adjustment of the microscope is standardised and the refractive index of the mounting medium is constant. The thickness of the outline is also dependent on the closeness of the packing together of the fibres, but the causes of the variations in the thickness of the outline of fibres can easily be distinguished. The various liquors used in the tannery can affect the separation not only of the fibres, but also of the fibrils from one

another. Where the fibres possess longitudinal striations, and the cross section of the fibre-bundles is full of fine markings, indicating the presence of the fibrils, the fibres are said to be "split." Where the "splitting" is so pronounced that the fibrils are separated from each other, and the fibre-bundle loses its definite outline, the fibre-structure is said to show "separation." In general, "splitting" is desirable if flexibility be desired, but "separation" generally indicates a soft, flabby, porous leather. Examples of all these types of structure are given by means of photomicrographs.

Reviews

EXAMINATION OF WATER, CHEMICAL AND BACTERIOLOGICAL. By WILLIAM P. MASON. Sixth Edition, revised by ARTHUR M. BUSWELL. Pp. xi+224. New York: John Wiley & Sons; London: Chapman & Hall. 1931. Price 18s.

Five editions of this work have appeared at intervals from the pen of Professor Mason; the first was apparently published in 1899, the fifth in 1917. The present edition has been revised by Professor Buswell alone. It is obvious that the book has met a demand, at least in the country of its origin. Its purpose, we are told, "has always been to supply the needs of the undergraduate student rather than those of the routine analyst." This edition appears in an entirely new format, with few alterations or excisions, but with extensive additions.

Chapters I and II (to p. 102) are chiefly concerned with the chemical, and Chapter V (pp. 146–162) with the bacteriological examination of water as to its suitability for drinking and general domestic purposes. These chapters contain practically all of the previous edition; additions include some account of the determination of the *p*H value, and information concerning the chemical substances found in natural water, and the waste of soap caused by hardness. In connection with the last matter, the erroneous statement is made that "one gallon per capita per day is usually taken as the amount of water used for washing." The analytical procedures detailed are substantially in accordance with the Standard Methods of the American Public Health Association (first published in 1905), which have been so generally adopted in America. They present no new features of importance. The low results obtained for nitrates in presence of high chlorides with the phenolsulphonic acid process (if the method given for the removal of chlorides is not utilised) would not be prevented by adding sodium chloride to the "standards," as the loss occurs during the reaction with the phenolsulphonic acid; no mention is made of the simple modification of the process which eliminates the error due to this cause. Nessler solution does not improve with age. The method given for examination for poisonous metals does not distinguish between lead and copper; it will occasion some surprise that it is considered that this is not commonly necessary. In the determination of free and albuminoid ammonia, Wanklyn, who devised the process (in 1867), returned the results in terms of NH_3, and this notation

has been generally followed; in the Standard Methods, to which reference has been made, the ammonia results are returned in terms of N. By changing the strength of the standard ammonium chloride solution (in the fifth edition) the author brought his book into line with American practice, but, in doing so, failed to realise that the scores of analytical results of these determinations transcribed from the earlier to the present edition are misleading to the extent of the gross error represented by the difference between 3·82 and 3·15.

With regard to the interpretation of the results of an analysis, the author says: "The intention is simply to place before the reader sundry data and the opinions of various authorities, and absolutely disclaim any desire to set boundaries to the free use of the analyst's good judgment." It is, however, rather difficult to understand how the student just commencing a study of the subject is competent to exercise such judgment. The probability of correct conclusions being reached would have been increased if the author had devoted some of the space given to the opinions expressed in the literature of the '80's and '90's of last century to an extended exposition of the findings in his own experience.

Chapter III (pp. 103–135) deals with routine laboratory methods for the determination of mineral constituents. For the control of water-softening processes the Standard Methods of the American Railway Engineering Association are reprinted, and for examination of water as to suitability for boiler purposes, the methods of the Illinois State Water Survey (of which the present author is chief) are detailed. For the continually increasing pressure at which boilers are now being worked, phosphate is being employed in increasing quantity for treatment of feed water, and Straub's method (1930) for this determination is given; one of the two standards referred to appears to have been omitted in the description of the process. This section concludes with an account of the methods used by the Illinois State Water Survey in the examination of mineral (medicinal) waters.

Chapter IV (pp. 136–145) appears to be a direct reprint of class instruction sheets for laboratory exercises in clarifying, softening, and chlorinating of water; the strength of the chlorine solution specified works out as ten parts, and not one part per million, as stated. The information given is, presumably, intended to be supplemented by verbal instruction.

There is an extended appendix (pp. 163–217) which contains much of interest, but which could have been drastically condensed without disadvantage. The most important section is a reprint of the U.S. Treasury Department Standards for drinking and culinary water supplied by common carriers in interstate commerce, which, though this is not stated, have been extensively adopted for public supplies in general.

This edition, especially when compared with the preceding one published fourteen years previously, exhibits evidence of what is usually termed "hasty" revision. Examples of this are (with page numbers) repetition of definition of Continental degrees of hardness (24, 42); sulphonic instead of sulphanilic uncorrected (50); designation of "recent" retained (54, 87); purpose of ammonium hydroxide is explained (54), but potassium hydroxide is specified (56); repetition to decolorise (56, 57); Welsbach burner recommended if daylight not available (67); albuminoid ammonia changed to albuminoid nitrogen, but only in some

cases; indiscriminate absence of 0 before the point in decimal figures less than unity; dichromate (95) reverts to bichromate (97). There are about a dozen typographical errors, none of them serious. ROBERT C. FREDERICK.

THE BIOCHEMISTRY OF MUSCLE. By DOROTHY MOYLE NEEDHAM, M.A., Ph.D., Biochemical Laboratory, Cambridge. Pp. viii+166. London: Methuen & Co., Ltd. 1932. Price 5s. net.

Methuen's Monographs on Biological Subjects, of which Dr. Needham's work is the fourth, aim "to give brief but authoritative accounts of the present state of knowledge in various departments of Biology." The biochemistry of muscle has just emerged from a serious but very interesting crisis, and a publication on this subject from the pen of an authority who has been actively engaged in research on these lines, is both opportune and welcome. Until 1898, when Fletcher commenced his far-reaching investigations on muscular energy, it was generally believed that this form of energy depended on a sudden production of an unstable compound, which contained oxygen ready for the production of energy by oxidation. These purely theoretical speculations have now been replaced by well-established facts, the logical sequence of which is well developed in the work under review. Dr. Needham traces the problem with precision, from lactic acid metabolism by spontaneous oxidation to the concept of a muscle-machine, and throughout weighs with care the different views and experimental data, often contradictory amongst themselves, which have been put forward to account for the phenomena of muscular energy. *The Biochemistry of Muscle* is a well-written and welcome addition to biochemical literature, and we are indebted to Dr. Needham for having collected and sifted this material.

In conclusion, one feels inclined to suggest that when the second edition is considered it would perhaps be advisable to omit the "Glossary" (pp. 153–155). It adds little to the value of the book, and perhaps lowers its standard. After all, who does not know that "aldose" is "a sugar containing the aldehyde (CHO) group"? M. NIERENSTEIN.

CLINICAL CHEMISTRY IN PRACTICAL MEDICINE. By C. P. STEWART, M.Sc. (Dunelm.), Ph.D. (Edin.), Lecturer in Biochemistry, University of Edinburgh; Senior Biochemist, Royal Infirmary, Edinburgh, and D. M. DUNLOP, B.A. (Oxon.), M.D., M.R.C.P. (Edin.), Assistant, Department of Therapeutics, University of Edinburgh. Pp. x+246+2 for Notes. Edinburgh: E. & S. Livingstone. Price 7s. 6d. net.

Laboratory workers are apt to be over-enthusiastic, and they tend to underestimate the value of bedside observations. To be reminded that biochemistry is not the philosopher's stone of medicine is a healthy warning to all concerned with the usual routine in a laboratory attached to an infirmary. Valuable as biochemical investigation is, it must always be considered in conjunction with clinical examination, a point clearly demonstrated by Drs. Stewart and Dunlop, whose book will be found not only of practical use, but also stimulating. There have been many works written on chemical routine in connection with medicine,

but we know of no other book of its size where clinical experience has been so well tempered with laboratory routine. Few chemists have such opportunities as are to be found at the Royal Infirmary at Edinburgh.

It is beyond the limits of a review to discuss in detail and to evaluate the different methods, and the preferences for some of the processes described, as, for example, the collection and preservation of samples, the study of basal metabolism and of the mechanism of neutrality in the living organism, and the many other topics so vital to laboratory routine. Each of the twelve chapters is permeated with practical laboratory experience, and at the same time with clinical understanding. Obviously, only those who use the book are in the position to judge its practical application, but, meanwhile, one can say, with confidence, that it deserves the serious attention of those who require a manual for use in the clinical laboratory.

M. NIERENSTEIN.

REVIEW OF THE EFFECTS OF ALCOHOL ON MAN. Edited by K. KITCHIN and D. H. KITCHIN. Pp. 300. London: Victor Gollancz, Ltd. 1931. Price 8s. 6d.

An enquiry into the medical, social and economic aspects and dimensions of the alcohol problem in Great Britain was initiated by a group of persons of wide public interests, *viz.* Lord Buckmaster, Lord Balfour of Burleigh, Field-Marshal Lord Methuen, the Hon. Mrs. Alfred Lyttelton, the Right Hon. Philip (now Lord) Snowden, and Mr. W. D. Hitchens.

This enquiry was instituted before there was any sign of the appointment of the recent Royal Commission on the subject of alcohol.

Two Committees were appointed, one to deal with the social and economic side of the problem, the other with the medical. The present work is the Report of the Medical Committee.

The various aspects of the subject are dealt with in separate chapters by authors who have made a special study of the particular branch of the problem on which they write, and all are recognised authorities. It would have been difficult to have chosen a better team.

Dr. Kathleen Kitchin and Mr. D. Harcourt Kitchin have carefully edited the various contributions and, without altering their context, have, by elimination of unnecessary repetition, made a compact and harmonious whole of the isolated chapters.

Chapter I, by Dr. Howard Florey, gives an admirable summary of the physiological actions of alcohol which is worthy of very close study. The physiological action on the circulatory, respiratory, digestive and nervous systems are considered in detail, and the conclusion is arrived at that the action of alcohol on the highest parts of the central nervous system is narcotic and not stimulant.

This conclusion is in agreement with that of the other writers, who deal with the subject from different angles. It is shown that alcohol can be oxidised in the body for the production of useful work, when given in amounts producing physiological rather than pathological conditions.

Dr. G. Roche Lynch, in Chapter II, gives a very valuable review of the toxicology of alcohol. The recent work on the absorption and excretion of alcohol is summarised, and the various percentages of alcohol in the blood are compared with the corresponding clinical symptoms caused thereby.

The curves illustrating the rates of excretion of alcohol, when taken in the form of whisky or stout, are produced, and a most interesting series of drawings, made by the subject of this experiment, illustrates the effect of alcohol on the co-ordinating action of the nervous system.

Acute and chronic alcoholic poisoning are fully dealt with from the toxicological aspect, and also the influence of such factors as the kind of beverage, the relationship of the taking of food with alcohol, idiosyncrasy, tolerance, and special acquired susceptibility.

The addiction habit of alcohol is compared and contrasted with that of drug addiction (morphine).

Chapter III relates to the effect of alcohol on the body resistance to disease, and in it Professor F. S. Langmead and Dr. T. C. Hunt show that, while profound excess markedly reduces the body resistance, there is no evidence proving that in the physiological amounts taken by the temperate drinker any effect is produced on the essential processes of immunity and protection.

Dr. Bedford Pierce, who is so well qualified to deal with this aspect of the subject, gives in Chapter IV a most interesting summary of the mental effects of alcohol.

Chapter V, by Dr. Isabel G. Wilson, deals with alcohol and mental disorder in an admirably fair and well-balanced summary of this much-disputed question.

The morbid changes caused by alcohol are very carefully and critically reviewed in Chapter VI by Dr. W. D. Newcomb, whose great post-mortem experience entitles him to speak with authority. He points out that alcohol has been credited as being the aetiological factor in a number of diseases, such as arterio-sclerosis, nephritis, etc., where the scientific evidence fails to support the past conclusions. In some diseases, such as cirrhosis of the liver, while alcohol may often play an important causative part, it is clearly shown that other factors are also at work.

Professor F. S. Langmead and Dr. T. C. Hunt, in Chapter VII, give an admirable account of the use of alcohol in the practice of medicine. They point out that, while the use of alcohol as a therapeutic remedy is very much less common than in the past, yet the real deciding factor is the clinical effects produced by it in certain diseased conditions. They state that alcohol is a valuable drug, but there are limitations to its actions, and its prescription requires skill and discretion.

Chapter VIII, by Dr. F. A. Crew, gives a most interesting biological study of the racial effects of alcohol. The subject is dealt with in a most instructive and novel manner, and the influence of alcohol is impartially and clearly reviewed.

The Appendix deals with the various forms of alcohol other than ethyl alcohol, and gives a very useful table of the various alcoholic beverages, with their source, descriptive remarks, and respective alcoholic content.

This work gives the most concise and accurate review of the medical aspects of alcohol which has been yet published. It should be in the possession of every

analytical chemist who is concerned with the analysis of alcoholic beverages. It is of the utmost value to medical men and chemists who are confronted with the medico-legal aspects of the effects of alcohol. It should be included in every medical and chemical library.

W. H. WILLCOX.

Publications Received

ALLEN'S COMMERCIAL ORGANIC ANALYSIS. Fifth Edition. Vol. IX. Edited by C. AINSWORTH MITCHELL. (*Proteins of Plants*, by D. Jordan Lloyd; *Proteins of Milk*, by G. D. Elsdon; *Milk*, by J. Golding; *Milk Products*, by E. R. Bolton; *Meat and Meat Products*, by C. R. Moulton.) London: J. & A. Churchill. Price 32s.

APPLIED PHARMACOLOGY. By A. J. CLARK. London: J. & A. Churchill. Price 17s.

STRUCTURE AND COMPOSITION OF FOODS. By A. L. WINTON and K. B. WINTON. London: Chapman & Hall. Price 53s. net.

MICROBES AND ULTRAMICROBES. By A. D. GARDNER. London: Methuen & Co. Price 3s. 6d.

MAKERS OF CHEMISTRY. By E. J. HOLMYARD. London: Oxford University Press. Price 7s. 6d. net.

WATER DIVINERS AND THEIR METHODS. By HENRI MAGER. Translated by A. H. BELL. London: Bell & Sons. Price 16s. net.

HYDROGEN IONS. By H. T. S. BRITTON. London: Chapman & Hall. Price 25s. net.

THE PRACTICE OF ABSORPTION SPECTROPHOTOMETRY. Published by Adam Hilger, Ltd. Price 5s. net.

RECENT APPLICATIONS OF ABSORPTION SPECTROPHOTOMETRY. Published by Adam Hilger, Ltd. Price 3s. 6d. net.

ANNUAL REPORTS ON THE PROGRESS OF APPLIED CHEMISTRY. Vol. XVI. Society of Chemical Industry. Price 7s. 6d. to Members; 12s. 6d. to non-Members.

ALCOHOLIC FERMENTATION. By A. HARDEN. London: Longmans, Green & Co., Ltd. Price 15s. net.

BRITISH CHEMICALS AND THEIR MANUFACTURERS. 1931. The Association of British Chemical Manufacturers. Supplied gratis to users of chemicals.