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THE ANALYST

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THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS

THE Annual General Meeting of the Society was held at 3.15 p.m. on Wednesday, March 3, 1943, at The Chemical Society's Rooms, Burlington House, London, W.1. The chair was occupied by the President, Dr. E. B. Hughes. The Financial Statement for 1942 was presented by the Hon. Treasurer and approved and Auditors for 1943 were appointed. The Report of the Council was submitted by the Hon. Secretary and adopted. The following were elected Officers and Council for the coming year:

President.—S. Ernest Melling, F.I.C.

Past Presidents serving on the Council.—F. W. F. Arnaud, Bernard Dyer, John Evans, Edward Hinks, E. B. Hughes, G. Roche Lynch, W. H. Roberts and G. Rudd Thompson.

Vice-Presidents.—H. E. Cox, G. Hogan, T. Rendle and, *ex officio*, W. Gordon Carey (Chairman, North of England Section) and A. R. Jamieson (Chairman, Scottish Section).

Hon. Treasurer.—George Taylor.

Hon. Secretary.—Lewis Eynon.

Other Members of Council.—J. W. Corran, R. H. Ellis, J. G. A. Griffiths, D. W. Kent-Jones, R. Lessing, H. M. Mason, B. G. McLellan, M. Pearson, W. H. Simmons, R. W. Sutton, E. Voelcker, K. A. Williams and, *ex officio*, Arnold Lees (Hon. Secretary, North of England Section) and R. S. Watson (Hon. Secretary, Scottish Section).

A vote of thanks was accorded to the retiring President, Dr. E. B. Hughes, for his valuable services in that office during three years of exceptional difficulty, and a miniature replica of the Presidential Badge was presented to him by Mr. S. E. Melling on behalf of the Society.

The meeting was followed by an Address by Dr. C. Ainsworth Mitchell, M.A., F.I.C., on "Ink in Relation to Crime" (see p. 103).

NORTH OF ENGLAND SECTION

THE Eighteenth Annual General Meeting of the Section was held at Manchester on Saturday, January 30, 1943. The Chairman (Mr. J. R. Stubbs) presided over an attendance of thirty-two. The Hon. Secretary presented the Report and Financial Statement, which were adopted.

Appointments were made as follow[illegible]-W. Gordon Carey; *Vice-Chairman*—E. Gabriel Jones; *Committee*—A. A. D. Comrie, W. F. Elvidge, H. M. Mason, S. E. Melling, J. R. Stubbs, G. H. Walker; *Hon. Auditors*—U. A. Coates, J. R. Walmsley; *Hon. Secretary and Treasurer*—Arnold Lees.

The following papers were read and discussed:—"The Resazurin Test for Milk," by G. Sykes, M.Sc., A.I.C.; "The Determination of Arsenic in Glass," by H. N. Wilson, F.I.C.

SCOTTISH SECTION

THE Eighth Annual General Meeting of the Section was held in the Rhul Restaurant, 123, Sauchiehall Street, Glasgow, on Friday, January 28, 1943. Mr. J. W. Hawley presided.

The following office bearers were elected:—*Chairman.*—A. R. Jamieson. *Vice-Chairman.*—J. B. McKean. *Members of Committee.*—A. Andrew, A. M. Cameron, H. Dryerre, J. W. Hawley, J. E. Ritchie, and J. Sword. *Hon. Secretary and Treasurer.*—R. S. Watson. *Hon. Auditors.*—A. R. Campbell and J. A. MacNair.

NEW MEMBERS

THE following have been elected members of the Society:—Harold Edwin Brookes, B.Sc. (Lond.),* John Walter Flint, B.A. (Cantab.), F.I.C., Jack Leslie Forsdike, B.Pharm. (Lond.), A.I.C., Ph.C.,* Paul Frederic Holt, Ph.D. (Lond.), D.I.C., F.I.C., Robert Heaton Horrocks, B.Sc. (Lond.), A.I.C.,* George Kenny, B.Sc. (Manc.), A.I.C.,*

* Through the North of England Section.

Edgar Roberts Ling, M.Sc. (Lond.), A.R.C.S., F.I.C.,* Gordon Frederick Lothian, M.A. (Oxon.), F.Inst.P., Martin Lovett, B.Sc. (Lond.), F.I.C.,* William Joseph Lowe, M.Sc. (Liv.), A.I.C.,* Miss Christina Cruickshank Miller, Ph.D., D.Sc. (Edin.), Robert Laurence Okell, Ph.D. (Liv.),* Raymond John Michael Pollitt, M.Sc. (Lond.), A.I.C., Leslie Gordon Lovett Unstead-Joss, B.Sc. (Lond.), A.I.C., Sidney Watkinson,* Gordon Hilliar Wyatt, B.Sc., Ph.D. (Lond.), F.I.C.*

Annual Report of Council: March, 1943

THE roll of the Society numbers 1003, an increase of 43 over the membership a year ago.

The Council regrets to have to record the death of the following members:

H. A. Bromley	F. W. Harbord	J. W. H. Johnson	C. H. Rosier
A. R. Buchanan	J. B. P. Harrison	H. W. Lawrence	G. A. Stokes
C. Crocker	G. G. Henderson	F. O'Brien	F. G. H. Tate

Bromley was for many years head of the laboratories of H.M. Stationery Office. He was the author of an excellent text-book on the chemical, microscopical and mechanical examination of stationery materials.

Buchanan, who died at the age of 72, was for over fifty years associated with, and latterly Director of, Messrs. John Buchanan & Co., Manufacturers of Confectionery and Preserves.

Crocker, who died in his 88th year, had been a member of the Society for 46 years. He was for a period of about 20 years with Dr. W. Morgan, Borough Analyst for Swansea; he then went into partnership with Dr. R. P. Charles. During the war of 1914–18 he was chief chemist at Brand's Spelter Works, Irvine.

Harbord, who died at the age of 80, was elected a member of the Society in 1908 and served on the Council in 1910–11. He was a distinguished metallurgist and had been President of the Iron and Steel Institute and of the Institution of Mining and Metallurgy.

Henderson, who died in his 81st year, was elected an Honorary Member of the Society in 1925. He was President of the Society of Chemical Industry in 1914–15, of the Institute of Chemistry in 1924–27 and of the Chemical Society in 1931–33 and Professor of Chemistry at the Royal Technical College, Glasgow, from 1892 to 1919, and at Glasgow University from 1919 to 1937, the date of his retirement.

Johnson, whose death occurred in his 66th year, was a member of the North of England Section and served on its Committee. He was in the service of the West Riding Rivers Board from 1898 until his retirement in 1940. He published a number of papers on the analysis and treatment of waters, sewage and effluents. (Obituary, ANALYST, 1942, **67**, 248.)

Lawrence, who died at the age of 77, was connected with agricultural chemistry during the whole of his professional career. He worked, first in the laboratory of the Royal Agricultural Society, then at the Rothamsted Experimental Station and, in 1901, he went to New Zealand where, after serving for some years in the Department of Agriculture, he became a consulting chemist. (Obituary, ANALYST, 1942, **67**, 345.)

O'Brien, who died in his 69th year, had been a member of the Society since 1929. In 1907 he joined the late Mr. C. J. Waterfall in a consulting practice in Bristol, having previously been research chemist to the Western Tanning Co., Ltd., for whom he continued to act as analyst. After Mr. Waterfall's death he carried on the practice until his retirement in 1940. He served at various times on Committees appointed by the International Society of Leather Trades Chemists.

Rosier, who was 61 at the time of his death, worked for six years as assistant to Mr. H. Droop Richmond when the latter was Chemist to the Aylesbury Dairy Co. Since the formation of United Dairies, Ltd., he was in charge of the Control Laboratory at one of the large pasteurising plants, first at Forest Hill and since 1936, at Vauxhall.

Tate, who died in his 64th year, was elected a member of the Society in 1921. He served as a member of Council in 1938–39 and as a Vice-President in 1940–41. On leaving the Royal College of Science in 1902 he joined the staff of the Government Laboratory and at the time of his retirement he held the position of Superintending Chemist. He made numerous contributions to technical literature. (Obituary, ANALYST, 1943, **68**, 1.)

* Through the North of England Section.

ORDINARY MEETINGS.—In the course of the year five meetings were held and the following papers were communicated:

"The Biological Estimation of Vitamin P Activity." By A. L. Bacharach, M.A., F.I.C., and M. E. Coates, Ph.C.

"A New Absorptiometric Method for the Estimation of Blood Sugar." By R. F. Milton, B.Sc.

"A Modified Hilger Vitameter." By R. J. Taylor, B.Sc.

"The Gravimetric Micro-Determination of Magnesium." By P. F. Holt, Ph.D., F.I.C.

"The Analysis of Foods containing Meat, Cereal Filler, and Soya Bean Meal." By E. T. Illing, B.Sc., F.I.C., and E. G. Whittle, B.Sc., F.I.C.

"The Iodine Value as an Index of the Relative Firmness of Pig Back Depôt Fat." By W. Bolton, M.Sc., and R. G. Baskett, M.Sc., A.I.C.

"The Micro-Determination of Calcium in Serum." By P. F. Holt, Ph.D., F.I.C.

"The Estimation of Iron in Blood, Haemoglobin, and Haemin and its Application to the Standardisation of Haemoglobin." By G. E. Delorey, M.Sc.

"The Histology of Mammalian Hair." By J. L. Stoves, Ph.D.

"The Estimation of Added Calcium Carbonate (*Creta Praeparata*) in National Flour." By E. N. Greer, B.Sc., A.I.C., J. D. Mounfield, M.Sc., Ph.D., A.I.C., and W. J. S. Pringle.

"The Spectrophotometric Determination of Vitamin A in Fish Liver Oils." By J. I. M. Jones, M.Sc., F.I.C., and R. T. M. Haines, M.A.

"The Determination of Sulphur Dioxide in Citrus Juices." By A. H. Bennett, B.Sc., and F. K. Donovan, B.Sc.

"The Estimation of the Narcotic Power of Hemp Drugs by a Colorimetric Method." By B. K. Mukhopadhyay, M.Sc., K. S. Subramanian, M.Sc., A.I.C., and H. B. Dunnicliff, I.E.S., C.I.E., M.A., Sc.D., F.I.C.

"A New Colorimetric Method for the Determination of Boron." By D. Dickinson, M.Sc., F.I.C.

"Methods of Analysis for the Purposes of the Cake and Flour Confectionery (Control and Maximum Prices) Order, S.R. & O., No. 2103, of 1942."

"The Photometric Estimation of Potassium by a Modification of the Jacobs–Hoffman Method." By A. Eden, M.A., Ph.D., F.I.C.

The December meeting was a Joint Meeting with the Food Group of the Society of Chemical Industry. The subject was "Flavours in Food," and the following papers were read and discussed:

"The Naturally Occurring Flavours of Foodstuffs." By W. A. Waygood, B.Sc., A.R.C.S., F.I.C.

"Some Aspects of the Chemistry of Flavours." By T. F. West, M.Sc., Ph.D., F.I.C.

At the Annual General Meeting Professor J. W. Munro, M.A., D.Sc., gave an Address on "The Entomology of Commerce: Insect Pests and their Control."

INFORMAL DINNER.—Following the Annual General Meeting an Informal Dinner of the Society was held at the Trocadero Restaurant. The function was very successful, although, owing to war conditions, accommodation could not be obtained for all who wished to attend.

THE ANALYST.—The paper for THE ANALYST has now been restricted to 30% by weight of the pre-war supply. To comply with the Regulation, it has been necessary to print on thinner paper than before and to give increased attention to verbal condensation, including the use of a number of approved abbreviations. By these means it has been possible to reduce the number of pages from 541 in 1941 to 408 without sacrificing anything of analytical importance. For instance, as many papers (31) were published as in 1941 and more notes (30) than in any of the preceding three years and the balance of interest has been maintained. Thus, the subject-matter of the papers was distributed as follows:—food and drugs, 12; biochemical, 4; organic, 2; inorganic, 9; microchemical, 3; physical, 1; similarly with the notes:—food and drugs, 10; biochemical, 1; organic, 6; inorganic, 8; microchemical, 1; toxicological, 2; apparatus, 2.

The Ministry of Food made use of THE ANALYST for the publication of a method of assaying aneurine in flour. We have also received from the Ministry the numerous Food Orders as published, and the contents of those with an analytical bearing have been summarised in our journal.

With the aim of standardising nomenclature, the Publication Committee approved the publication of a Report on Spectrophotometric Terms drawn up by Mr. B. S. Cooper with the assistance of a panel of specialists.

Once more we have welcomed the Reports of Government Analysts in the Dominions and India, which have provided valuable analytical information.

HON. TREASURER'S REPORT.—The Hon. Treasurer reports that he anticipates that the financial position of the Society will prove satisfactory, as it has been for many years.

WAR GASES.—Arrangements, similar to those for Mustard Gas and Lewisite, have been made for the distribution of D.M. Arsenical Smoke to Public Analysts and other chemists concerned in the examination of gas-contaminated food.

The Ministry of Food has issued a description of the S.D. Test for Mustard Gas vapour and a form of application for field testing kit and protective clothing to Public Analysts, etc.

SAMPLING OF FOOD ORDER, S.R. & O. 531 of 1942.—Following the issue of this Order, the Ministry of Food issued a Circular, FIG/ENF/48, according to which samples of food taken by Enforcement Officers are to be submitted only to the Government Chemist and not to either the Government Chemist or a Public Analyst as prescribed in the original Order.

As a result of a protest made by the Council, a communication was received stating that the Ministry of Food is prepared to except from the general arrangements, under which samples are dealt with by the Government Chemist, those foodstuffs, the standards for which are prescribed in Statutory Rules and Orders made by the Ministry, in so far as such samples are analysed with the object of determining whether they conform with those standards. These samples will be analysed, as heretofore, by Public Analysts.

ANALYTICAL METHODS COMMITTEE.—Most of the Sub-Committees engaged on various analytical problems have now suspended work owing to difficulties created by the War. One Report, however, from the Poisons Sub-Committee, on the Assay of Aconite, has been published (ANALYST, 1942, **67**, 289).

The appointment of a Sub-Committee to investigate chemical methods of estimating Vitamin B_1, referred to in last year's Annual Report, was found to be unnecessary as the Medical Research Council had already initiated work on the subject in which members of the Society have participated. The Report on this is shortly to be published. An investigation, under the same auspices, on the Determination of Crude Fibre in National 85% Flour, has also been undertaken by several members of the Society and a report on their recommendations will be available at an early date.

The Committee has been asked by the Technical Sub-Committee of the Federation of Wholesale and Multiple Bakers to formulate methods for the determination of Fat and Sugar in products submitted under the Cake and Flour Confectionery Order, 1942, but it was decided to postpone any action until after the reading of a paper on the subject at the February meeting of the Society.

The Committee profoundly regrets the resignation, owing to ill-health, of its Chairman, Mr. E. Hinks, who has occupied the chair of the Committee since its formation nearly 19 years ago. The Council has tendered its cordial thanks to Mr. Hinks for the signal service he has given to the Committee during his long tenure of the Chairmanship. Dr. E. B. Hughes has been appointed by the Council as Mr. Hinks's successor in the chair and Mr. R. C. Chirnside has also been appointed a member of the Committee.

NORTH OF ENGLAND SECTION.—Four meetings have been held during the year and the following papers have been read:

"Absorption Spectrophotometry in Analytical Practice." By Dr. W. F. Elvidge, B.Sc., F.I.C.

"The Determination of Oil in Extract of Malt with Cod Liver Oil and in other Pharmaceutical Emulsions." By C. R. Bond, M.Sc., F.I.C., and S. Druce, B.Sc., A.I.C.

"Adulteration through the Ages." By J. R. Nicholls, D.Sc., F.I.C.

"Notes on the Composition of Some Varieties of Onions." By J. G. Sherratt, B.Sc., F.I.C.

A discussion on "Food Substitutes" was opened by C. H. Manley, M.A., F.I.C.

There have been good attendances at the meetings.

The Section now numbers 147 members, an increase of 5 on the previous year.

The Honorary Secretary wishes to express his appreciation of the loyal support and help accorded to him by the Chairman and members of the Committee during the year.

SCOTTISH SECTION.—Three meetings were held in the year, at which the following papers, by members of the Section, were read and discussed:

"Methods of Soil Analysis." By A. M. Smith, Ph.D., D.Sc.

"An Investigation relative to the Milk of an Abnormal Herd of Cows." By A. Scott Dodd, B.Sc., Ph.D., F.I.C., and R. Cowan.

"The Estimation of Fibre in National Wheatmeal—The Estimation of the Proportion of White Flour used in the Manufacture of a Purchased Loaf." By James Sword, M.A., B.Sc., Ph.D., F.I.C.

"Note on the Conversion of Calcium Oxalate to Oxide." By James Sandilands, F.I.C.

The Committee record, with regret, the death of two members, Professor G. G. Henderson and Mr. A. R. Buchanan.

Forty-nine subscriptions were received, being three more than in the previous year. Two new members joined the Parent Society through the Section and two members resigned on taking up residence outside the Scottish Area.

PARLIAMENTARY AND SCIENTIFIC COMMITTEE.—The President was re-appointed to represent the Society on the Executive Committee of the Parliamentary and Scientific Committee.

INTER-DEPARTMENTAL COMMITTEE ON FOOD STANDARDS.—On the invitation of the Ministry of Food to nominate two members of the Society to serve on a Provisional Inter-Departmental Committee to advise the Ministry on proposals with respect to food standards, Professor W. H. Roberts and Mr. G. Taylor were nominated.

NATIONAL COMMITTEE FOR CHEMISTRY.—Mr. F. W. F. Arnaud was appointed as representative of the Society on the National Committee for Chemistry in place of Professor W. H. Roberts, whose term of office expired on December 31st, 1942.

Despite the increased difficulties of travelling, a high standard of attendance at Council and Committee meetings has been maintained, and thanks are especially due to country members whose attendance has often involved great inconvenience and sacrifice of time. The Council also desires to record its thanks to organisations and members of the Society for accommodation and hospitality to the Committees.

E. B. HUGHES, *President.*

LEWIS EYNON, *Hon. Secretary.*

Ink in Relation to Crime

AN ADDRESS BY C. AINSWORTH MITCHELL, D.Sc., M.A., F.I.C.

(*Delivered at the Annual General Meeting, March* 3, 1943)

IN this address Dr. Mitchell gave an account of some of the numerous trials in which he has appeared as witness for the Crown. He outlined only briefly the general principles involved in his scientific evidence on ink, for detailed particulars of several of the cases have already been published, and he dwelt rather on the points of human interest disclosed and on incidents worth recording because they have a bearing on legal procedure.

SCOPE OF MEDICO-LEGAL EVIDENCE.—Up to the early part of last century a doctor was commonly accepted as the embodiment of all scientific knowledge, and it was not uncommon for medical men to give evidence on chemical subjects with which they had little acquaintance. For example, Taylor in his "*Manual of Medical Jurisprudence* (1844),"[1a] records a case in which a doctor gave evidence that ink in writing on a forged cheque had been bleached.* At the present time such specialised chemical work would not be undertaken by the medico-legal expert. In 1907, Sir Thomas Stevenson, the Official Analyst to the Home Office (President of the Society in 1895), was asked by the Treasury if he could give evidence upon the identity of an ink upon a will, but he advised the Director of Public Prosecutions to refer the matter to a chemist who had studied the subject.[2] In this case (*Rex* v. *Brinkley*)[2,3,4] the accused had attempted to poison a man named Parker whose signature appeared as that of a witness on a disputed will, but who said

* In this case (*Rex* v. *Hart*), tried at the Central Criminal Court in December, 1833, the prisoner was charged with having removed "2" and substituted "5" in a bank acceptance for £200. The witness stated that some acid, probably muriatic or oxalic, had been used to remove the "2," but that he had been unable to ascertain its nature.

that he had been induced by misrepresentation to sign a paper which he did not know purported to be a will. Cyanide was put into a bottle of stout and left in Parker's lodgings, where it was found and drunk by the landlord and his wife, both of whom died. Dr. Ingleby Oddie (then junior counsel to the Treasury), in his recent book "*Inquest*,"[3] mentions this case as a classical example of the law that if a man intends to kill one person, but unintentionally kills someone else, he is none the less guilty of murder. The prosecution required corroboration of Parker's statement that the ink at the public house where he said that he signed a petition for an outing agreed with that in his signature on the will. The President of the Probate Court refused permission for chemical tests to be applied to the will, and so evidence of the identity of the ink (Mordan's *Azuryte*) had to be based on optical tests by means of the tintometer, microscope and comparison colour bands, as described in THE ANALYST.[6] This was the first occasion on which evidence of the identity of modern inks was given in an English court of law.

ADMISSIBILITY OF NOTE BOOKS IN EVIDENCE.—At the trial of Brinkley at the Guildford Assizes, Mr. (afterwards Sir) Richard Muir led for the prosecution, and Mr. Walter Frampton, subsequently well known to Public Analysts, defended the accused. The medical and toxicological evidence was given by Sir Thomas Stevenson and R. Bodmer. In the course of this evidence the question was raised whether it was permissible for Bodmer to read the results of the analysis of the poisoned stout from the entries in his laboratory notebook. The Judge, Mr. Justice Bigham (afterwards Lord Mersey) held that, provided that the entries were the actual ones made at the time of the analysis and not copied into the book at a later date, the witness might read them out. In the lecturer's experience, judges have followed this rule in many later cases and have even allowed a witness to refer to the proof of his evidence for details of an examination which would be burdensome to remember.

Referring to this question, Taylor[1b] (p. 30) mentions a case (prior to 1844) in which the analyst referred to notes of previous experiments made by him. When challenged, he admitted that he had entirely forgotten these results, and counsel thereupon objected to their production. The Judge upheld the objection and rejected the evidence. Taylor's comment on this is: "Notes can only be used for the purpose of remembering a fact indistinctly remembered. The most eminent legal authorities have laid it down that if there be any single point in the notes which the witness does not recollect, except that he finds it there written, such point is not evidence. Notes are only allowed to assist recollection, not to convey information."

PHOTOGRAPHING AND SKETCHING IN COURT.—At the time of the Brinkley case no objection was taken to photographing scenes in court even when proceedings were actually in progress, but a few years later photography was prohibited anywhere within the precincts of a court. Several photographs of the scene within the court and of Brinkley in the witness box were taken and published in the press [the lecturer showed some of these on the screen].

In the notorious trial of *Rex* v. *Wood*, at the Old Bailey,[4] in which a young artist was acquitted of the brutal murder of a woman, Emily Dymock, the prisoner appeared the most unconcerned person in court, and in the intervals of his trial was constantly sketching. Even while waiting for the verdict of the jury, he made sketches [shown on the screen] of his counsel, Marshall Hall, and of some of the leading celebrities—actors, actresses and novelists—who came each day to the court to study emotional sensations. Sketching in court is now also prohibited.

In this case evidence was given that the pigment of a copying ink pencil found on the accused (a Swan pencil) corresponded with that in the writing on a burnt fragment found in the grate of the room of the murdered woman. Acting on the advice of his counsel, Marshall Hall, the defendant at last confessed that he had written the words on this fragment and on a postcard found under the lining paper in a drawer, because he recognised that they proved that he had recently been in the company of Emily Dymock.[4,7] Details of the scientific facts of the identification of the copying ink pencil pigment are given elsewhere.[6] This case was remarkable for an exhibition of hysterical mass enthusiasm for the prisoner. A dense crowd waited outside the Old Bailey for an artist's model who, against her will, had identified Wood's writing, and she had to be smuggled away, disguised as a charwoman, to escape the vengeance of the crowd.

PRESENCE OF EXPERT WITNESSES IN COURT.—This question was raised in the case of *Rex* v. *Pilcher* (1911),[4] in which for the first time chemical evidence of the age of a modern ink was given. The evidence, based on the formation of a resinous tannate as ink becomes oxidised,[8] proved that the writing in blue-black ink on a will could not be as old as it purported to be. The President of the Probate Court had previously given permission for these tests to be made.

At the outset of the case it was held that there was no objection to the expert witness remaining in court throughout the proceedings. It is usually customary for counsel for the prosecution to ask counsel for the defence if he minds the expert being present. If counsel agree, the judge will usually not object either in High Court civil actions or in criminal trials. In Privy Council appeals, however, all witnesses, including experts, are excluded from the court both before and after giving evidence (*e.g.*, *Wakeford* v. *The Bishop of Lincoln*), and sometimes the clerks in local magistrates' courts will advise the bench to exclude expert witnesses until they give their evidence; but this is now unusual in England, for it is generally recognised that the evidence of an expert witness is not affected by the evidence of other witnesses. In the Scottish courts permission for the expert to remain may be given if specially requested.

EXPERT EVIDENCE BY A SOLICITOR.—This question arose in the case of *Rex* v. *Rogers*,[4,9] in which the accused had sold to an old-age pensioner two 17th-century parchments bearing forged signatures of William Penn the Quaker. The old ink had been imitated by the use of a silver salt solution, and the signature had been written with a steel nib.* The prisoner was found guilty of uttering the document, knowing it to be forged. Between the hearing of the case at the Slough Police Court and the trial at the Aylesbury Assizes the defendant's solicitor had received a letter purporting to have been sent by the victim of the fraud, Mrs. Field, and exonerating Rogers. When shown this letter she said: "Yes, that is my writing,

* Wise's pens, sold in 1803, consisted of a metal tube bent round but with edges unjoined, leaving a slit; the tube was cut away to form a point. In Donkin's patent (No. 3118 of 1808) claim was made for the manufacture of metal nibs with a slit point. William Penn died in 1720.

but I never wrote it." The Judge (Mr. Justice Acton) accepted Dr. Mitchell's statement that without close study of a great deal of the genuine writing of an illiterate woman it would be difficult to express any opinion on the subject. Then the solicitor went into the box with the intention of saying that he recognised Mrs. Field's writing; but the Judge asked him if he had made a special study of handwriting, and when he replied "No," the Judge said: "Then I shall tell the jury not to pay any attention to anything you say on the subject." This ruling was contrary to that given in *Rex* v. *Silverlock* (1894, 2 Q.B. 766), in which it was held that the solicitor for the prosecution might give evidence after comparing admitted handwriting of the prisoner with that on documents produced by the prosecution.

SCIENTIFIC INSTRUMENTS IN COURT.—Although counsel sometimes suggest that it should be done to impress a jury, it is not advisable to give a demonstration with actual scientific instruments in court. In the case of *Rex* v. *Podmore*[4,9] a dirty, oily fragment of paper was found on the scene of a murder, and, when cleaned with benzene, revealed writing and an address which led to the identification and conviction of the murderer. Although ultra-violet light showed more of the writing (in copying-ink pencil) than could be seen with the naked eye, it was considered safer to rely upon what the jury could see unaided. Even an outlined photograph was excluded by the Judge (Lord Hewart, C.J.).

In the case of *Shashoua* v. *Shashoua* (1935) a claim for £50,000 was made against Mrs. Shashoua by her husband on the strength of a contract made just prior to her marriage, the consideration being the marriage itself. The writing of the document was admittedly that of the plaintiff, but the defendant, while admitting the genuineness of her signature, said that she was in the habit of signing papers put before her but had not seen this marriage contract. The plaintiff asserted that, after writing the document, he had stamped it, and that then the defendant had signed across the stamp. This statement was proved to be false by means of a lens fitted with an electric bulb so that light could be transmitted from the underside of the paper. This showed clearly that the contract stamp was below two parts of lines in the writing, which cut across the edges, and that therefore the stamp had been affixed before and not after the body of the document had been written. The Judge (Mr. Justice Bennett) was anxious to examine this illuminating lens, but counsel for the defence objected, and so the Judge contented himself with trying the lens on his own notes and not on the actual exhibit. Nevertheless, the plaintiff lost his case.

In a case in 1936 (*Rex* v. *Hopson*), in which a sawdust merchant was charged with using "chemically washed" insurance stamps, ultra-violet photographs of cancelled stamps were made, and showed clearly where the ink marks had been removed. Since to the naked eye the "washed" stamps would pass unchallenged and there was no direct link between the defendant and the man who sold the stamps, the accused was acquitted.[4]

EVIDENCE RELATING TO AN INTERVAL BETWEEN TRIALS.—In *Rex* v. *Thurburn* (1926) a young girl was charged with writing anonymous letters to many people in Sheringham. The case was tried before Mr. Justice Horridge, at the Norwich Assizes, and, in addition to evidence of handwriting, chemical evidence was given that two adjacent capitals in an anonymous letter were in artists' black ink similar to that admittedly used by the defendant in a cartoon she had sent out; the rest of the letter was in ordinary writing ink. This suggested accidental dipping of the pen into a bottle of artist's black ink (a modern carbon ink). The jury disagreed and were discharged.

At the second trial, before Mr. Justice Sankey, evidence of anonymous letters having been received by the defendant, together with other points in her favour, were admitted, but the judge would not allow documentary evidence that told strongly against her to be given. When counsel for the Crown (Mr., now Sir, Gerald Dodgson, the present Recorder of London) protested, the Judge said that he was willing to allow to the defence a latitude that he would not allow to the prosecution. The jury again disagreed and, as the High Court would not allow the hearing to be transferred to London, the case was dropped.

In this connection the ruling of the same Judge (now Lord Justice Sankey) in *Maxwell* v. *Director of Public Prosecutions* (1935, A.C. 309, 323) is worth recording: "If in any case the evidence against a prisoner (other than that which is admissible) is very strong and is abundant to justify a jury in convicting, it may well seem unfortunate that a guilty man should go free because some rule of evidence has been infringed by the prosecution. . . . The sanction of the observance of the rules of evidence in criminal cases is that, if they are broken in any case, the conviction may be quashed. . . . It is often better that one guilty man escape than that the general rule evolved by the dictates of justice for the conduct of criminal prosecutions should be disregarded or discredited."

SELECTION OF EXPERT WITNESSES BY THE COURT.—In *Hawes* v. *Skelton* (1934)[4] a will had been found, 6 months after the death of the testator, in the pocket of an overall worn by his widow while bathing a dog. Evidence had been given before Mr. Justice Horridge to the effect that the signature was not genuine because it showed signs of retouching and the ink varied in colour. But the Judge was not satisfied, and told his clerk to telephone to Dr. Mitchell and ask him to come to the court. He then said that counsel on each side had agreed that Dr. Mitchell should be asked to examine the ink on the will and also that in a bottle in the testator's house and to report his finding direct to the court. Examination of the ink in the bottle, which was stated to contain a mixture of inks from three bottles bought at a sale, showed that it dried rapidly on paper, and gave writing of different colours according to the depth to which the pen was inserted into it. Thus, the peculiarities of the mixture fully accounted for the mixed colour and the retouching in the signature. Having heard this evidence, the Judge held that the will was valid.

Selection of expert witnesses by the court in this way has many advantages over the present system, in which each side selects its own expert witnesses, who are not always properly qualified. It would, of course, be necessary to have a panel of recognised specialists from whom the selection would be made.

SUMMARY OF CERTAIN CASES INVOLVING EVIDENCE ON INK.—The following is a list of selected cases, in many of which new methods of examining ink were applied.

Rex v. *Brinkley*, 1907. (Optical identification of blue-black ink.)
—— *Wood*, 1907. (Identification of copying-ink pencil pigment.)
—— *Pilcher*, 1910. (Chemical proof of age of ink.)
—— *Kuepferle*, 1915. (Identification of invisible ink.)[25]
—— *Müller and Hahn*, 1915. (do. do. .)

Rex v. *Brekoff*, 1915. (Proof of forgery of passport.)
—— *Cornwallis*, 1920. (Photographic differentiation of inks.)
Wakeford v. *Bishop of Lincoln*, 1921. (Continuity of flow of ink.)
Rex v. *Thurburn*, 1926. (Identification of artist's black ink.)
—— *Podmore*, 1930. (Deciphering copying ink pencil writing.)
—— *Rogers*, 1931. (Identification of silver ink used to imitate antique ink.)
Howes v. *Skelton*, 1934. (Effect of mixed inks on writing.)
Rex v. *Hopson*, 1934. (Ultra-violet detection of "chemically washed" stamps.)
Shashoua v. *Shashoua*, 1935. (Ink above, instead of below, contract stamp.)
Rex v. *Harris*, 1936. (Identification of typewriting and inks.)
—— *Taylor*, 1936. (Inks on false burial certificate and income tax returns.)

When Samuel Butler wilfully perverted the meaning of the Psalmist's "One day in Thy courts is better than a thousand," he had in mind the tribulations of a litigant; but to the expert witness, who can be present throughout the proceedings, unharassed by the outcome of a case, a court of law presents ever-changing opportunities of studying human nature, for in no other place can so many cross-sections of society be seen under such revealing conditions.

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9. —— *id.*, 1932, **57**, 144 (Scientific documentary evidence in criminal trials).

A New Colorimetric Method for the Determination of Boron

BY DENIS DICKINSON, M.Sc., F.I.C.

(*Read at the Meeting, February* 4, 1943)

OWING to the importance of boron as a plant nutrient, search has been made for analytical methods suitable for determining minute quantities. Robinson[1] and Naftel[2] have applied the turmeric test quantitatively, and Berger and Truog[3] and Maunsel[4] have expanded Smith's work on the quantitative application of quinalizarin[5] into a simple and rapid method of determining boron in plants and soils. For plant materials (here the primary concern) all that is needed is to ash the material, dissolve the ash in dil. sulphuric acid, mix a 1-ml aliquot portion of the soln. with 9 ml of conc. sulphuric acid containing quinalizarin, and match the resulting colour with boric acid standards freshly made each day. Alternatively, to save time, the standards may be stored in a desiccator to protect them from moisture, but it is then necessary to wait for 24 hrs. after mixing the sample with the quinalizarin before comparison with such matured standards.[4] The use of a photoelectric colorimeter would no doubt overcome these drawbacks, but, in my experience, and contrary to Maunsel,[4] the colour is not sufficiently intense for visual colorimetry.

Synthetic colour standards, prepared in this laboratory from alkaline solutions of phenolphthalein and thymolphthalein, have been used with fair success for several months, duplicate determinations usually agreeing within 3·5 p.p.m. of boron. The method has been used to follow changes in the boron content of plum leaves during the summer, but towards the end of the season the results began to vary widely. As the cause of this could not be traced, search was made for an alternative method, preferably one more suitable for visual colorimetry. As the turmeric methods appeared unsuitable for rapid routine determinations, experiments with other dyes of the anthraquinone group were made. Feigl[6] found that purpurin and alizarin-S were less sensitive than quinalizarin as reagents for boron. However, in our experience, alizarin-S, although giving a colour change less definite than that with quinalizarin, was not necessarily less sensitive. In fact, when boric acid and alizarin-S react in conc. sulphuric acid soln. the colour of the dye is only intensified, and remains substantially the same as that of methyl-orange in aqueous alcohol; this observation suggested the possibility of using artificial colour standards.

The modification of the quinalizarin method (using synthetic standards) and the new method (using alizarin-S) are described in the following sections.

1. MODIFICATION OF THE QUINALIZARIN METHOD.—Prepare solns. of phenolphthalein and of thymolphthalein containing, respectively, 0·333 g and 0·100 g in 100 ml of alcohol. Use matched test-tubes (6 × 5/8 in.) fitted with ground-glass stoppers to contain the sulphuric acid solns. Prepare a series of standard solns. of boric acid containing 0·001–0·005 mg of boron per ml. Put 1 ml of the appropriate standard soln. into a stoppered tube and run in 9 ml of conc. sulphuric acid. Add 0·5 ml of a soln. of quinalizarin (0·01 g in 100 ml of 94% w/w sulphuric acid) mix, and leave for at least 15 min. for the colour to develop. Meanwhile prepare alkaline solns. of the indicators by adding 0·5 ml of the phenolphthalein soln., and 1·0 ml of the thymolphthalein soln., each to a separate 100 ml of sodium carbonate soln. containing 3 g of the anhydrous salt per litre. Match the colour of each standard by running into a similar tube the alkaline indicator solns. from burettes and looking through the depths of the liquids with the tubes held over a white tile. In this way a calibration curve can be drawn showing the vols. of the indicator solns. required to match the colour produced by any quantity of boric acid from 0 to 0·005 mg when allowed to react with quinalizarin for a definite time (*e.g.*, 15 min.).

2. NEW COLORIMETRIC METHOD USING ALIZARIN-S.—*Solutions required.*—(*a*) Alizarin-S: dissolve 0·01 g of the dye in 250 ml of conc. sulphuric acid (not less than 98% H_2SO_4 w/w), (*b*) Standard boric acid soln. (*c*) Methyl-orange: dissolve 0·10 g in 100 ml of 95% alcohol and 100 ml of water, and make up to 500 ml with water.

Procedure.—To standardise the methyl-orange soln. prepare a series of dilutions of boric acid containing up to 0·005 mg of boron per ml. Measure 1 ml of the standard soln into a test-tube fitted with a ground-glass stopper and add from a burette 9 ml of the alizarin-S soln. Mix by inverting and allow 30 (±2) min. for the colour to develop. Then run the methyl orange soln. from a burette into a precisely similar tube until the colours match on looking *down* the tubes (they do not match if one looks across the tubes) and note the vol. of the methyl orange soln. required. Plot the mg of boron present against the ml of methyl orange soln. required; the graph is a straight line.

A simple tube holder, made especially for use in this determination, consists of a block of wood bored with two parallel holes, about ¼ in. apart, just wide enough to take two similar flat-bottomed test-tubes. The diameters of the holes at the lower ends are slightly narrower than the tubes, so that these cannot slip through.

To determine the boron content of a plant material, ash 0·5–1·0 g of the dry sample in a porcelain crucible at 480–520° C. until a white or grey ash remains. Porcelain crucibles used for this purpose should be boiled in dil. hydrochloric acid before and after use. Add to the ash exactly 5 ml of dil. sulphuric acid (approx. 0·4 *N*) and triturate with a rubber-tipped rod. When the residue has settled, pipette 1 ml of the clear supernatant soln. and run it into one of the special test-tubes. Add 9 ml of the alizarin-S soln., replace the stopper, invert several times, cool if necessary, leave for 30 min., and then match the colour with the methyl-orange soln., as described above. Make a blank determination on 1 ml of the dil. sulphuric acid soln. mixed with 9 ml of the alizarin-S soln.

The effect of the concn. of the sulphuric acid used is illustrated in Table I.

TABLE I. EFFECT OF CONCENTRATION OF SULPHURIC ACID

Boron present, mg	Concn. of H_2SO_4 by wt., %	Methyl orange soln.* to match, ml
0·01	94·3	7·5
0·01	88·1	3·5
0·01	81·2	0·6
0·01	73·4	0·2
0·01	64·9	too faint

* The concn. of this soln. was different from that recommended above.

Clearly the concn. of the acid used should be the highest practicable (*i.e.*, *ca.* 94·5% w/w); a variation of ±1% will introduce an error of ±6% in the determination.

In expts. to ascertain the rate of colour development the amounts of methyl orange soln. required to match after different reaction periods were as follows:—5 min., 10·8 ml; 15 min., 12·0 ml; 30 min., 12·5 ml; 60 min., 13·0 ml. These results show that a 30-min. reaction time is sufficient; although the reaction is not complete, the error introduced by variations of ±2 min. is negligible. The method adopted for dealing with large numbers

of samples is to mix the unknowns and the alizarin-S at 5-min. intervals. This means that the colours become ready for matching at 5-min. intervals, which allows adequate time for matching and for calculation of the result.

The standard graph was determined by 3 observers independently. (See Table II.)

TABLE II. RESULTS BY DIFFERENT WORKERS

Boron, mg	Methyl orange soln. required, ml: Observer: (A)	(B)	(C)	Mean	Graphically corr. mean
0·0	3·6	—	3·2	3·4	3·3
0·001	4·1	3·8	4·0	4·0	4·1
0·002	4·9	5·3	5·3	5·2	4·9
0·0033	5·9	5·9	6·0	5·9	5·95
0·004	6·3	—	—	—	6·45
0·005	7·0	—	—	—	7·25
0·006	8·2	—	—	—	8·05

Examples of results obtained are as follows:

For six samples, each analysed by two or more out of four workers, A, B, C, D, the following boron contents (p.p.m.) were found: (B) 34·0, (C) 32·0; (A) 50·1, (C) 49·5; (A) 46·3, (B) 48·7; (A) 40·5, (B) 43·7, (C) 37·6; (A) 42·0, (D) 38·7; (A) 21·5, (D) 19·8.

For a series of 44 expts. in duplicate, by B, covering the range 0–55 p.p.m., the mean diff. between duplicates was 1·5 p.p.m., with a standard deviation of 1·27.

In a "blind" test carried out by observer A, comprising 7 test expts. with amounts of boron ranging from 0·001 to 0·005 mg, the differences between the amounts found and those present ranged from nil to 0·0004 mg.

Finally, a comparison of results obtained by this method, by the modified quinalizarin method and by the mannitol method[7] on various samples is set out in Table III.

TABLE III

Material	Method used and p.p.m. boron found: Alizarin-S	Quinalizarin	Mannitol
Plum leaves, A	42	42	—
,, ,, B	30	26	30
,, ,, C	28	24	26
,, ,, D	32	36	35
,, ,, E	31	36	30
Carrots	23	19	—
Cabbage	38	36	—
Broccoli leaves	25	26	—

CONCLUSIONS.—The alizarin-S method, used in conjunction with the ashing procedure as described, gives results which are correct to within 2 p.p.m. when 0·5-g samples are used, *i.e.*, within 0·2μg of boron. Moreover, the colour can be matched much more easily than the violet quinalizarin colour, and this factor alone reduces considerably the time required for the completion of a series of determinations.

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DISCUSSION

Mr. N. L. ALLPORT welcomed any new method for the determination of traces of boron, as existing procedures were far from satisfactory. Other investigators, besides Mr. Dickinson, had experienced unaccountable failures with the quinalizarin procedure, and he thought that they might be due to the concentration of the sulphuric acid in the final test mixtures having considerable influence upon the intensity of the colour produced. He wished to supplement a question from the Chair about the possible influence of aluminium by asking whether there was any interference with the alizarin-S test by nitrates, chromates or fluorides, all of which inhibited the reaction with quinalizarin. The author had said very

little about the turmeric method; although it had been greatly improved by K. L. Robinson (ANALYST, 1939, **64**, 324), the procedure was still lengthy and had the disadvantage that the final red colour was mixed with a considerable preponderance of yellow from the excess of reagent. Robinson's paper included a graph correlating the quantity of boron with the red colour produced, as measured in a Lovibond tintometer, but the size of the cell used was not stated; trial had shown that the figures correspond with a depth of liquid equal to 1 cm. Boron had been recommended as a hardener for steel, and so analysts engaged in the iron and steel industry had attempted to apply both the quinalizarin and the turmeric methods to their particular purposes (H. A. Kar, "*Metals and Alloys*," 1938, **9**, 175). All the colorimetric methods so far proposed for the determination were subject to the limitation imposed by the fact that the reagents themselves were highly coloured.

Mr. S. G. E. STEVENS asked whether the author was satisfied that the ashing method used ensured complete recovery of the boron, as he had found, when dealing with fuel oil which had been "doped" with boron, that the presence of aluminium and silica in relatively larger amounts resulted in a loss of boron as determined by the normal mannitol method.

Mr. H. AMPHLETT WILLIAMS asked if fluorine interfered, as apparently it could also be titrated in the same way as the boron.

Mr. DICKINSON, replying, said that the reaction between boric acid and the anthraquinone derivatives which was employed in both the quinalizarin and the alizarin-S methods was thought to be almost distinctive, only one other element—germanium—being known to react in a similar manner. Nitrates, if not decomposed by the ignition of the sample, interfered with both methods by decolorising the solns. Fluorides interfered in the same way if present in relatively large quantities. Aluminium did not interfere under the conditions prescribed. Smith had suggested the use of two strengths of sulphuric acid, the lower concentration being used when the amount of boric acid in 1 ml of the final soln. exceeded 0·040 mg. When, as in the present investigation, the amount of boric acid was much less than this, the higher acid concentration should be used (*i.e.*, 9 vols. of conc. sulphuric acid to 1 vol. of the soln. under test). Probably the alizarin-S method could be adapted for greater amounts of boron by using a lower concentration of acid, but for the present purpose the maximum practicable concentration of acid, to which the maximum sensitivity of the reaction corresponds, should be employed. The referees of the Association of Official Agricultural Chemists, J. S. McHargue and W. S. Hodgkiss, had compared the quinalizarin, turmeric, and mannitol methods and had found that the turmeric method gave results rather lower than the other two. (*J.A.O.A.C.*, 1942, **25**, 311.) They had also investigated the efficiency of the recovery of boron when the simplified ashing technique—as quoted in the above paper—was used and had found it satisfactory. The author could confirm this, subject to the proviso that the final ash must be alkaline.

The Estimation of Vitamin A in Vitaminised Cocoa and Chocolate by Means of the Carr-Price Test

BY E. G. RAYNES, B.SC., A.I.C., AND B. G. MCLELLAN, F.I.C.

THE increasing use made of chocolate as a vitamin-carrying foodstuff makes it important that accurate and simple methods of estimation should be available. As a result of prolonged investigation the following method is recommended for an accurate assessment of the Lovibond units per g of sample. To translate this into International Units reference must be made to the potency of the original concentrate which is supplied to a definite potency, usually determined spectrophotometrically. The factor given here was obtained by the blue unit procedure on a series of actual concentrates used, of specified potency. The procedure consists in : (1) extraction of the fat, (2) saponification, (3) extraction and isolation of the unsap. matter, (4) determination of the Lovibond units per g of sample on the unsap. matter in chloroform soln. and conversion into International Units by means of the conversion factor 1·41.

Early expts. showed that ordinary methylated ether, when used as the fat solvent and for extracting the unsap. matter, gave unsatisfactory results, on account of impurities present—particularly peroxides. This major difficulty was overcome by allowing the ether to stand over metallic sodium for 24 hrs. and redistilling. A rapid extraction of the fat with the use of a kieselguhr filter-aid was adopted to minimise loss of vitamin A through exposure to light and heat. Saponification of the fat and extraction of the unsap. matter follow the usual procedure, except that a Röse–Gottlieb syphon-tube in conjunction with a blow-ball is used to blow off the ethereal layer into the second separator for washing. The final isolation of the unsap. matter by heating under reduced pressure requires special care, to ensure that, as far as possible, all traces of moisture and solvent are removed from the sides and neck of the flask. If this is done carefully, there is no need to add abs. alcohol to get rid of the last traces of moisture, nor is it necessary to add anhydrous sodium sulphate to the final chloroform soln. to clear it.

The matching of the blue value needs a little practice owing to the fading of the blue component. As a rule it is necessary to determine the order of the colour first, and

then to repeat the operation until a satisfactory match has been obtained. It is usual to take the mean of at least 4 closely agreeing results. A clear blue soln. should always be obtained. Any turbidity is usually due to moisture, or to working in a cold atmosphere. In our experience matching is most accurate in the region of 4 Lovibond blue units.

Low results may be due to the chloroform (which contains 1–2% of abs. ethyl alcohol to preserve it) if the Winchester has been opened for some time and only a small vol. remains at the bottom. If there is any doubt, it is wise to run a control test on a chocolate or concentrate of known potency. On no account should recovered chloroform be used. We have found no advantage in using alcohol-free chloroform, but rather the reverse. It is quite satisfactory when freshly made, but deteriorates rapidly on standing, with a very adverse effect on the proper development of the blue colour.

The empirical conversion factor 1·41 was arrived at by determining the Lovibond blue units per g of concentrates of specified potency and taking the mean. Its use has been found satisfactory over a long period covering the analysis of very many samples. It is advisable not to conduct any of the operations in direct sunlight, and to complete the estimation in the min. time, or at any rate the same day. When a number of estimations are being made at the same time it is advisable to leave in the dark between consecutive states. We have not found it necessary to work in any special light.

METHOD.—*Reagents.—Purified Ether.*—Prepared by standing methylated ether over metallic sodium (*ca.* 20 g for 3 litres) under a reflux condenser for 24 hrs. and redistilling; store in a brown Winchester in the dark. *Chloroform.*—AnalaR, fresh and stored in the dark. *N/2 Alcoholic Potash.*—3 g of AnalaR potassium hydroxide dissolved in 100 ml of ethyl alcohol. Made up in 100-ml quantities to avoid deterioration. *Antimony Trichloride Soln.*—8·85% w/w Sb_2O_3 in chloroform (B.D.H.). *Kieselguhr Filter-aid.*—Filter-Guhr or Dicalite Speedex. *Asbestos.*—Gooch filter-fibre shaken up with purified ether.

Special Apparatus.—(i) A 150-ml "fat" flask fitted with a 2-holed rubber bung carrying a Gooch adapter and a piece of glass tubing bent at right angles. (2) Sintered glass Gooch crucible, coarse grade (1 G.). (3) Automatic 2-ml pipette and bottle for the antimony trichloride (B.D.H.). (4) Lovibond Tintometer provided with sliding colour glasses. (5) All-glass cell, 1-cm internal measurement.

Procedure.—Weigh 5 g of the cocoa or chopped chocolate in a 60-ml beaker, and add 30 ml of purified ether. Break up the lumps with a glass rod and warm for a few min. with stirring, in a warm water-bath until completely disintegrated. Add about 2 g of filter-aid, stir and filter through a sintered glass Gooch crucible previously prepared with a pad of asbestos, about 1/8 in. thick, covered with about 1/8 in. of tightly packed filter-aid. Filter by means of gentle suction into a "fat" flask. Wash the beaker and crucible twice with about 10 ml of purified ether. Remove the crucible and rinse the bottom with purified ether into the adapter. Transfer the contents back to the beaker by means of a spatula, leaving the pad intact. Add 30 ml of purified ether, stir and filter as before. Wash twice with 10 ml of purified ether. Remove the Gooch crucible and again rinse the bottom and the adapter with purified ether. Introduce a glass bead, and boil off and recover the ether on the Soxhlet water-bath. Add 15 ml of *N/2* alcoholic potash, and saponify with gentle boiling on the steam-bath for about 20 min., using an air-condenser. Add about 15 ml of water and pour into a separating funnel, rinsing the flask with about 20 ml of water. Leave to cool, rinse the flask with about 50 ml of purified ether and pour into the separator. Shake well, and, after separation, blow off the ether layer into another separating funnel containing about 15 ml of water. A Röse–Gottlieb syphon-tube arrangement in conjunction with a blow-ball is used for this purpose. Repeat the extraction twice with about 40 ml of purified ether. Wash the united ethereal extracts with 10–20 ml of water until the ether is clear and the aqueous layer neutral to phenolphthalein. Dry the stem of the separating funnel with filter-paper, and filter the ethereal layer through a dry 7-cm No. 41 Whatman filter-paper into the original "fat" flask, which has in the meantime been washed and dried. Wash the separating funnel and filter-paper with purified ether, introduce the glass bead and evaporate almost to dryness on the Soxhlet water-bath, recovering the ether. Transfer the flask to a boiling water-bath and remove as much as possible of the remaining liquid. Meanwhile remove the adapter from the rubber-bung, replace it by a cork and fit the bung in the flask. Continue the heating under reduced pressure until the walls and neck of the flask are dry, and only the yellow unsap. matter remains. (It has been found advantageous to remove momentarily the rubber-bung, to

wipe it and to dry the tube by means of suction before completing the final heating under reduced pressure.) Leave to cool under reduced pressure, and for a chocolate with a potency of about 3000 I.U. per oz., add immediately 20 ml of chloroform from a pipette. Allow the unsap. matter to dissolve and accurately pipette 0·2 ml into a 1-cm all-glass cell. Place the cell in the Lovibond Tintometer, add 2 ml of antimony trichloride soln. and match the blue colour at its max. intensity, using blue, yellow and neutral glasses. Repeat the "blue" determination several times and take the mean of at least four closely agreeing results. The yellow and neutral figures are recorded, but are not used in the calculation.

In working with samples of different potency, the weight of sample taken, and the vol. of chloroform used to dissolve the unsap. matter should be adjusted so as to match in the region of 4 Lovibond blue units.

CALCULATION.—Let B = mean Lovibond blue in 1-cm cell; V = ml of chloroform used to dissolve the unsaponifiable matter; W = weight in g of sample taken. Then

$$\frac{B \times 5 \times V}{W} = \text{Lovibond blue units per g.}$$

Lovibond units per g × 1·41 (conversion factor) = International Units per g.

LABORATORIES OF ROWNTREE & CO., LTD., YORK

November, 1942

Notes

A NEW METHOD OF TITRATING PRECIPITATED ARSENIC

IN determining arsenic in copper, etc., by Evans's method (ANALYST 1929, **54**, 523) the reduced arsenic may be dissolved in potassium dichromate soln. containing sulphuric acid, the excess of dichromate titrated with ferrous ammonium sulphate, and the arsenic, oxidised to the As_2O_5 condition, calculated from the result. In the following expts. different amounts of arsenious oxide were boiled with sodium hypophosphite soln. for *ca.* 15 min. in 25 ml of conc. hydrochloric acid and 25 ml of water and cooled, and the ppt. of reduced arsenic was collected on asbestos which had been ignited, treated with nitric acid (sp.gr. 1·2) at b.p., and washed free from acid. The ppt. was washed with 30% hydrochloric acid and finally with 5% ammonium sulphate soln. to remove all acid, and then (together with the asbestos) transferred to a small beaker, the sides of the funnel being wiped with a little wad of asbestos which was then added to the main filter.

The arsenic was oxidised to the As_2O_5 condition by adding excess of $N/100$ potassium dichromate soln. and 20 ml of dil. (1 : 3) sulphuric acid and stirring for a few min. in the cold; the soln. was then filtered through fresh asbestos, the filter washed, and the filtrate made up to 150–200 ml. The excess dichromate was titrated with excess of ferrous ammonium sulphate soln., 5 drops of 0·1% disulphine blue soln. were added, and standard permanganate soln. was run in until the colour became faint pink. The reaction is:

$$3As_2 + 5K_2Cr_2O_7 + 20H_2SO_4 \rightarrow 3As_2O_5 + 5Cr_2(SO_4)_3 + 5K_2SO_4 + 20H_2O.$$

The following results were obtained. The strength of each of the solns. was $N/100$.

As_2O_3, ml	$K_2Cr_2O_7$ added, ml	$KMnO_4$ ml	Ferrous amm. sulphate, ml	$K_2Cr_2O_7$ used, ml	As added g	As found g
2·0	10·0	3·1	8·0	5·1	0·00075	0·00076
5·0	20·0	2·2	10·1	12·1	0·00187	0·00182
7·0	20·0	4·65	8·0	16·65	0·00262	0·00250
5·0	20·0	2·0	9·6	12·4	0·00187	0·00186
15·0	40·0	4·2	8·1	36·1	0·00562	0·00540

(1 ml $N/100$ $K_2Cr_2O_7 \equiv 0{\cdot}00015$ g of arsenic.)

In other expts., in which the arsenic ppt. was washed with 15% nitric acid instead of 30% hydrochloric acid, the results were also good; this proved that, contrary to expectation, the dil. nitric acid did not oxidise any arsenic. The advantage of this method, for small amounts of pptd. arsenic, over that used by Evans is that the end-point is more sensitive.

Permission to publish this note has been given by A. D. Tech.A. (Tech. A [4A]).

ARMAMENT RESEARCH DEPT. W. J. AGNEW

January, 1943

ADDED PREPARED CHALK IN FLOUR

THE Ministry of Food requires each sack (280 lb.) of National flour to contain 7 oz. of Prepared Chalk. The published methods[1] for estimating the chalk take too much time when many determinations are required for control purposes. On the other hand, the short method here described has been found useful as a general sorting test and satisfactory as a control test in mills where unsupplemented flour samples can be drawn as "blanks." The method depends on the fact that the alkalinity of the flour ash varies directly with the chalk content of the flour. The determination of the ash is a routine procedure in milling control, and the general availability of the ash shortens this Prepared Chalk test. The ash content of a flour increases with the addition of chalk, but the increment is relatively smaller than that shown by the alkalinity. This is illustrated by plotting the data given below. Furthermore, the difference in ash content is the less reliable guide, owing to the possibility of variation in the degree of decomposition of the calcium carbonate, with loss of carbon dioxide.

Any of the accepted procedures for determining the ash content of flour may be used, but in the examples cited below 10 g of flour were ignited in a muffle furnace at 600° C. for 17 hrs. The ash (still in

its silica capsule) was dissolved by gentle heating in sufficient 0·1 *N* sulphuric acid to cover the capsule; generally 25 ml were used. After cooling, the excess acid was back-titrated with 0·05 *N* sodium hydroxide, methyl orange being used. In the table below are given figures for both National and white flours which had been made up with known supplements of Prepared Chalk.

Prepared chalk added	Ash %	Total calcium present %	Alkalinity of ash in ml of 0·1 *N* H_2SO_4 per 100 g of flour
National Flour			
Nil	0·770	0·027	53·0
4-oz per sack	0·835	0·063	69·5
6-oz. ,,	0·877	0·076	77·5
7-oz. ,,	0·895	0·086	82·5
8-oz. ,,	0·907	0·094	85·5
10-oz. ,,	0·940	0·107	92·5
12-oz. ,,	0·982	0·131	100·5
White Flour			
Nil	0·442	0·015	30·5
4-oz. per sack	0·511	0·058	44·5
6-oz. ,,	0·542	0·066	52·5
7-oz. ,,	0·555	0·075	57·5
8-oz. ,,	0·570	0·080	62·5
10-oz. ,,	0·600	0·104	68·5

Much of the experimental work was carried out by Miss J. Sanders.

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SPILLERS CENTRAL LABORATORIES
BIRKENHEAD
ALBERT GREEN
February, 1943

ESTIMATION OF COCOA SHELL

THE estimation of cocoa shell in a nib-shell mixture is most conveniently carried out by determining the crude fibre on the dry fat-free material, and referring the result to standard fibre figures for the two components. These standard figures must necessarily be averages, and, so long as it can be fairly assumed that the amount of shell or nib present is unlikely to differ considerably from the average, a sufficiently accurate estimate of a sample's composition can be obtained. Recent investigation, however, has shown that with certain by-products of cocoa manufacture, this assumption is not always justified. Cocoa shell is not homogeneous, but consists of a number of botanically differentiated layers, and winnowing and sieving treatment tends to produce "fractions" with widely varying fibre content. Thus, although freed from all nib and germ, the lighter portions, which tend to accumulate in cocoa siftings, have been found to give fibre figures as low as 9·5%, on the fat-free dry material, as against 20·5% in heavier fractions.

It is thus obvious that when dealing with cocoa products in which nib is mixed with a disproportionate amount of the lighter shell fractions, the use of the usual fibre standards may lead to very erroneous results, giving a nib content far in excess of that actually present. For such samples other methods of examination instead of, or in addition to, fibre determination, must be employed. Microscopical methods, based on the direct recognition and quantitative estimation of the various shell tissues by means of "counts," have so far been more promising than indirect methods based on the estimation of cocoa starch, but both lines of approach are under investigation in the Laboratory.

It is interesting to note that the percentage and constants of the fat from cocoa shell, determined during the course of this work, were found to differ considerably from those usually quoted. The fat in shell was found to average 1·8%, and never exceeded 2% in any sample. The iodine val. of this fat ranged from 78–84, and the refractive index (Z.B.) was 63–64. The fact that the fat content of shell has been found to be so low and the fat to have such a high iodine val. and refractometer reading suggests that, in the past, expts. have often been made on shell imperfectly freed from adhering nib. The extracted fat has, in consequence, been contaminated with nib butter.

We wish to express our thanks to the Government Chemist, Dr. J. J. Fox, C.B., O.B.E., F.R.S., for permission to publish this preliminary note, and hope later to give a full account of our investigations.

GOVERNMENT LABORATORY
LONDON, W.C.2
A. H. RHEINLANDER
V. H. FIELD
March, 1943

ESTIMATION OF LINT IN COTTONSEED MEALS

PURE cotton wool yields 99% of fibre in the usual official process. When treated in the cold with 80% v/v sulphuric acid under standard conditions the fibre of cotton wool (lint) dissolves completely, whilst that from cottonseed meal loses a third of its weight. To estimate the lint in cottonseed meal, determine the fibre in 3 g of the sample in the usual way, but do not burn off. Dry the separated fibre until constant in weight, detach it from the filter as completely as possible, drop it into 30 ml of 80% v/v sulphuric acid at 15° C., and stir vigorously for exactly 5 min., breaking up the mass as thoroughly as possible. Then quickly transfer the contents of the beaker into 400 ml of cold water, filter off the residue, wash it free from acid, and dry until the weight is constant. The lint $= \frac{T - (T_1 + T_1/2)}{0{\cdot}9}$, where T represents total fibre and T_1 fibre after treatment. The true lint thus found represents about a quarter of that obtained by mechanical separation.

LABORATORY, 59, VICTORIA STREET, LIVERPOOL, 1
R. ROBERTSON DODD
March, 1943

Ministry of Food

STATUTORY RULES AND ORDERS*

1943—No. 11. The Flour Order, 1943. Dated January 4, 1943. Price 6d.

This Order deals, in 7 Parts, with the control of production and packing, control of sales, registration, control of use and prices, and miscellaneous regulations relating inter alia to records, entry upon premises, construction of references to national wheatmeal and manufacturers' flour. Two Schedules deal with prices and one with revocations of licences.

In Part I "flour" includes flour containing licensed non-wheaten substances; also semolina and flour unfit for human consumption, but does not include (i) *flour which prior to milling has been subjected to any process other than cleaning, blending, dressing or conditioning,* (ii) *any product licensed by the Minister under the Manufactured and Pre-packed Foods Control Order,* 1942, *as amended.* (*S.R. & O.*, 1942, *Nos.* 1863 *and* 2073.)

"'D' flour" means flour so designated by the Ministry.

"Imported flour" does not include flour to which any substance has been added after importation.

"'M' flour" means flour conforming to the definition of national flour (infra) except that it does not contain the max. quantity of wheat germ which could have been included.

"Millers' offals" means the by-product obtained from milling wheat and other authorised ingredient of flour in accordance with an approved specification. Such by-product must not contain more than 4% *of other vegetable substances, and these must have been extracted from the wheat and other ingredients in the process of cleaning.*

"National flour" means flour (other than Canadian Springs flour) produced in the United Kingdom and complying with the following conditions:—(i) *it must contain the max. quantity of wheat germ compatible with the type of milling plant;* (ii) *it shall not include any coarse bran,* (iii) *it shall consist wholly of wheat flour of* 85% *extraction and such other ingredients as the Minister may authorise.*

"Non-millable wheat" means wheat so certified by a Local Wheat Committee under the Wheat Bye-laws, 1932 (*S.R. & O.*, 1932, *No.* 588).

"Per cent. extraction" means, in relation to flour obtained from any description of cereal, the percentage which the weight of flour of that description separated in the milling bears to the total weight of all the products obtained in the milling from that cereal together with all screenings, seeds and dust extracted from the cereal by the miller prior to the milling thereof.

"Self-raising flour" means national flour, "M" flour, or imported flour, to which has been added sodium bicarbonate and one or more of the following substances only:—(i) common salt; (ii) any acid or acidic substance capable of liberating the carbon dioxide in the sodium bicarbonate.

"Speciality flour" means any flour other than national flour, "M" flour, imported flour or "D" flour.

"Wheat offals" is defined like millers' offals (supra), but does not include other authorised ingredients of flour.

"White flour" means flour consisting wholly or partly of wheat flour of less than 85% extraction, but shall be deemed not to include (i) semolina, (ii) national flour or *"M"* flour, (iii) any flour by reason only that it contains national flour or "M" flour.

The moisture content of any flour shall be determined in accordance with the method prescribed in the Second Schedule to the Wheat (Examinations and Analyses) Bye-laws, 1939 (*S.R. & O.*, 1939, *No.* 850).

PART II (Control of Production).—*Except under licence, no person shall produce any flour other than national flour. A person shall be regarded as producing flour if* (*a*) *he is the miller thereof,* (*b*) *if he abstracts from or adds any substance or applies any treatment to flour, not being "D" flour, with the intention of selling the product as flour.* (*This does not apply to the drying of semolina.*)

The only wheat by-products that may be produced are millers' offals, wheat germ and the following descriptions of wheat offals:—fine wheatfeed, straight-run bran, coarse bran and fine bran.

PART III (Control of Sales).—*Only national flour as defined in this Order may be sold as "national wheatmeal" or "national flour." No description of the quality or composition of national flour for sale may be used on any notice or on the label or container in which the flour is sold, other than those appropriate in the following list, viz.,* "Winters," "Springs," "Strong," "Stone-ground," "Stone-milled," "All-English," "Milled from home-ground wheat," "Biscuit" and "Scaling."

"M" flour may not be sold by retail, or to any person, other than a flour miller, a licensed factor or an importer, without a written declaration that it will not be re-sold but will be used only (*a*) *in the preparation of self-raising flour, or* (*b*) *as an ingredient in the manufacture of an article of food other than bread.*

Flour other than "D" flour or speciality flour may not be sold or supplied to a licensed factor except under a "Factor's Licence" granted under this Order. The sales and delivery of white flour are restricted to those holding a licence to produce bread for sale under the Bread (Control and Maximum Prices) Order, 1943 (*S.R. & O.*, 1943, *No.* 42), *but at the same time such quantity of national flour as the Minister may direct must also be sold or delivered.*

PART V (Control of Use).—(*a*) *Wheat products (other than flour sweepings, sack shakings and any product of non-millable wheat) may be used only in the production of an article of food; when a wheat product has become unfit for use as food, it may not be sold to, or bought by, any person other than the Minister.* (*b*) *Flour sweepings or sack shakings may not be used otherwise than*

* Obtainable from H.M. Stationery Office. Italics signify changed wording.

in the production of feeding stuffs for livestock. (*c*) *No product of non-millable wheat or wheat by-product may be bought or sold except for the manufacture of a feeding stuff for animals, other than a compound or mixture for feeding cats or dogs. Any wheat product may be used or bought or sold for use in the preparation of a bait for destroying rats or mice, or for destroying leather-jackets, caterpillars, slugs or snails. "D" flour, bought under the authority of the Minister, may be used in the preparation of feeding stuffs for livestock.*

For the purpose of this Article. (*a*) "Flour sweepings" means the product known as such, consisting mainly of flour which, having become accidentally spilt in the ordinary course of trade, has become unfit for use in the manufacture of an article of food.

(*c*) "Sack shakings" means the product known as such, consisting mainly of flour extracted from flour bags which have previously been emptied so far as is practicable in the ordinary course of emptying flour bags.

(*d*) "Wheat products" means flour, flour sweepings, sack shakings and any other product obtained from wheat, except wheat by-products.

White flour may not be used by way of trade or business, in the production of any article of food, other than flour or bread. For bread produced in Scotland and known as batch bread the max. permissible amount of white flour is 25% and for any other bread, 12·5%. Semolina may not be used as an ingredient in any manufactured product containing meat or any meat or fish paste as defined in S.R. & O., 1942, No. 1381. It may be used as a dusting material for any such product, but not as a dusting material in the production of bread.

No. 42. The Bread (Control and Maximum Prices) Order, 1943. Dated January 13, 1943. Price 4d.

In this Order "bread" includes rolls but not fruit loaves or bun loaves. "Cake" includes fruit loaves, bun loaves and flour confectionery, but does not include (i) *bread,* (ii) *biscuits within the meaning of S.R. & O., 1942, Nos. 678, 1636 and 2341 ;* (iii) *any product with a filling containing meat or fish as an ingredient.*

"National bread" means bread in the production of which—(i) *for batch bread in Scotland at least 75% and for any other bread at least 87·5% of the flour used is national flour, and* (ii) *no ingredient besides national flour has been used except one or more of the substances specified in the First Schedule.*

The production of bread for sale except under the terms of a licence is prohibited. No person shall produce for sale any bread from dough containing more than 2 lb. of oils and fats per 280 lb. of flour. Yeast food in excess of the quantity normally used in the trade may not be used in the production of bread. The types of loaves that may be sold and their weights are specified.

In any prosecution for deviations from the prescribed weights the Court shall disregard any inconsiderable variation in the weight of a single loaf and shall take the average weight of a reasonable number of loaves or rolls of the same kind sold, or for sale by the defendant. It shall be a satisfactory defence for the defendant to prove that a deficiency was due to a bona fide mistake or accident, notwithstanding all reasonable precautions, or was due to the fault of another person over whom the defendant had no control.

The Miscellaneous regulations provide that records shall be kept, and deal inter alia with licences and descriptions of bread to the sale of which most of the provisions of the Order do not apply.

THE FIRST SCHEDULE.—The following substances, other than national flour, may be included in national bread:—White flour, oils and fats, water, salt, yeast, improvers of the nature of yeast food, any acid or acidic substance suitable for regulating the acidity of the dough, potato and potato flour, barm.

Notes from the Reports of Public Analysts

The Editor would be glad to receive Reports containing matter of special interest

CITY OF SALFORD: ANNUAL REPORT FOR 1942

EGG SUBSTITUTES.—A sample sold as "Liquid Egg Substitute" consisted of 2·4% of gummy matter derived from cellulose, 1·4% of sodium bicarbonate and 95·7% of water. The directions on the label stated "Use one good teaspoonful where you would use one egg." It was ascertained on enquiry that the Ministry of Food had refused to license this product for retail trade. The wholesaler stated that his salesman had been instructed not to sell it to retailers. Caution issued.

Formal and informal samples of Egg Substitute Powder contained only 1·3% of available carbon dioxide, whereas, in my view, they should have contained at least 6%. As the product bore a Ministry of Food licence, enquiry was made of the Food Substitutes Control of that Ministry, and it was learned that the formula for which the licence was granted would yield a much higher proportion of carbon dioxide than that found in the sample. Proceedings were instituted and the summons was dismissed under the Probation of Offenders Act on payment of £5 5s. costs.

As the result of a consultation between the Ministry of Food and the Food Manufacturers Federation towards the end of 1942, egg substitutes will in future be known as "golden raising powders" and so avoid the confusion in the public mind between "egg substitute powder" and dried egg.

RICE PUDDING POWDER.—A sample labelled "—— vanilla flavoured rice pudding powder" contained rice and wheat starches. The packers agreed to alter the label.

"EMPRESS" COCKTAIL.—A formal sample consisted of: water, 99·8; phosphoric acid, 0·15; saccharin 0·05% with flavour and colour. It was sold at 3s. 6d. per bottle. The manufacturer could not be traced.

READY-MEAL—STEWED STEAK.—Formal and informal samples contained 35 and 36% respectively of meat, the remainder consisting of vegetables, including carrots, beans and potatoes. The labels declared

"This product is made from choice meats and complies with the statutory requirements of the Ministry of Food." No indication of the presence of vegetables was given. Legal proceedings were instituted and the packers were fined £5 with £4 4s. costs.

EXCESS OF MEAT IN SAUSAGES.—Formal and informal samples contained 54·7 and 54·3% of meat; also 680 and 620 p.p.m. of sulphite as SO_2. Two summonses were issued: (1) under the Food and Drugs Act in respect of undeclared preservative, and (2) under the Meat Products and Cooked Meat (Control and Maximum Prices) Order. Proceedings were instituted and the defendant was fined £1 under the first, and £2 under the second summons with £3 3s. costs.

PROSECUTION UNDER THE PHARMACY AND MEDICINES ACT, 1941.—Under Sec. 11 of this Act the vendor of an article recommended as a medicine must declare on the label the appropriate designation of the substance, or of each of the active constituents or of each of its ingredients; in the latter two cases the appropriate quantitative particulars of the constituents or ingredients must also be stated. Briefly, the expression "appropriate designation" means either (1) the name given in the Poisons List of the Pharmacy and Poisons Act, 1933, or (2) the name given in the B.P. or the B.P. Codex, or (3), if the name is not given in these publications, its accepted scientific name. (*Cf.* ANALYST, 1942, **67**, 392.)

Cream Emulsion of Cod-liver Oil with improved Chemical Food.—Informal and formal samples contained 32·3% of cod-liver oil and 1·1% of mineral matter consisting of phosphates of iron, calcium, etc. No declaration of the amounts was given. The four cod-liver Oil Emulsions of the B.P. and B.P. Codex contain 50% by vol. of cod-liver oil, except the Emulsion of Cod-liver Oil and Creosote, which contains 33%. The sample was not an official preparation and the statements as to tonic value and dosage on the label left no doubt that the substance was intended to be used as a medicine, and that the requirements of Sec. 11 of the Act should have been complied with. Proceedings were instituted against the vendor, and at the hearing of the case on December 14, 1942, the Stipendiary Magistrate said he was satisfied there must be a conviction. In view of the fact, however, that this section of the Act only came into force on July 1, 1942, the summonses were dismissed under the Probation of Offenders Act on payment of £3 3s. 0d. costs. G. H. WALKER

Legal Notes

The Editor would be glad to receive particulars of cases of legal or scientific interest

SUB-DIVISION OF RETAINED SAMPLE

BRITISH FERMENTATION PRODUCTS, LTD. *v.* TEAL

ON Jan. 28, 1943, the King's Bench Divisional Court heard an appeal against a conviction by the Staple Hill (Glos.) magistrates for the sale of an article (an egg substitute powder) not of the nature, substance and quality demanded. The article had been supplied by the appellants to a wholesale firm, who had sold it to a grocer at Mangotsfield (Glos.), from whom the respondent in the appeal (an inspector under the Food and Drugs Act) had bought it. The inspector, when making the purchase, had complied with the formalities of the Act, dividing the sample and retaining a third part for production at the hearing of the case. Later, having decided to take proceedings against the manufacturers, he took the seal off the retained part, divided it into two and sent one of the sub-divided parts to the defendants. In these circumstances it was contended by counsel for the defence that a condition precedent to proceedings under the Act had not been complied with, since the part ultimately retained by the inspector was only part of the part which should have been retained in accordance with Sec. 80 (4). The magistrates who tried the case on June 8, 1942, took the view that the manufacturers had not been prejudiced by what had happened and convicted them.

Mr. JUSTICE TUCKER, giving the judgment of the Court, said that the sample had been procured in circumstances requiring its division into parts pursuant to the provision of the Act. Its production in Court at the hearing was essential and a condition precedent. In doing what he did, the inspector thought that he was doing what was right, proper and fair to the appellants, but the result was to render it impossible for him properly to comply with the Act. The sample produced was not the retained sample but something different, and it had become different by the inspector's own intentional act. The statute, a penal one, laid down conditions of sampling and analysis in the interest of prospective defendants. The requirements of the section must be strictly obeyed; non-compliance must vitiate the conviction. The Court allowed the appeal.

The Lord Chief Justice (Viscount Caldecote) concurred.

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(Supplementing the series published in the ANALYST, 1933, **58**, 340, and bringing the Bibliography up to date)

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(To be continued)

Portugal: Official Methods for the Analysis of Edible Oils and Fats*

IN July, 1942, the Dept. of Agriculture of the Ministry of Economics, Portugal, officially adopted methods of analysis of alimentary oils and fats recommended by a Technical Sub-Committee. These methods are classified as general and special. The former include the collection and preparation of samples, organoleptic examination, determination of water and other foreign matter, including mineral oils, unsap. matter, (*a*) insol. in ether (international method), (*b*) insol. in light petroleum, b.p. 40–60° C. (Hönig and Spitz), acidity, ketonic rancidity, autoxidative rancidity, (*a*) aldehydes (von Fellenberg), (*b*) epihydrin aldehyde (Kreis), mineral preservatives (salt, boric acid, fluorides, sulphites, thiosulphates), organic preservatives.

Physical Methods.—Sp.gr., m.p., Shukoff's and Dalican's titre, freezing pt., n_D, fluorescence and spectrogram, viscosity.

Chemical tests.—Acid val., neutralisation val. of fatty acids, sap.val., Reichert–Meissl and Polenske vals., butyric acid val. (Kuhlmann and Grössfeld), Bertram's val., iodine val. (*a*) Hanus (international), (*b*) Wijs (international), thiocyanogen val. (Wizöff or Kaufmann), polybromide val. (bromides insol. in ether), acetyl val. (international method), special tests for erucic acid (Wizöff method), sesame oil (Baudouin), arachis oil (modification of Bellier's method), cottonseed oil (modification of Halphen's test) and animal oils (Tortelli–Jaffe's bromine in chloroform reagent).

Methods are also given in detail for the separation of solid and liquid fatty acids (Baughman and Jamieson's lead-alcohol method), iodine val. of the liquid fatty acids (separated by Tortelli and Ruggeri's lead-ether method, and calculation of saturated and unsaturated acids (from iodine and thiocyanogen vals).

ANALYSIS OF OLIVE OIL FOR FISCAL PURPOSES.—The colour is measured in a Lovibond Tintometer using a 100-watt lamp. *Alkalinity of ash* from 50 g of filtered oil determined with $N/600$ sulphuric acid (rosolic acid as indicator) and expressed as ml of $N/10$ per 100 g of oil. *Sesame oil.*—The following modification of the Baudouin test is applied: Add to 5 ml of the oil 0·1 ml of a 2% soln. of furfural and 5 ml of conc. hydrochloric acid. Shake and leave for 10 min. Add 5 ml of water, shake and note colour of separated acid layer. *Arachis oil.*—The following modification of Bellier's test must be used. Put 1 ml of the sample in a 100-ml Erlenmeyer flask, add 5 ml of an 8% alcoholic soln. of potassium hydroxide (80 g of KOH dissolved in 80 ml of water and made up to a litre with 90% alcohol), and heat on the water-bath under reflux, with constant shaking. After saponification cool to *ca.* 25° C., add exactly 1·5 ml of dil. acetic acid (1 : 4 vols.), 3 drops (not more) of glacial acetic acid and 50 ml of 70% alcohol and shake until any turbidity disappears. Replace the flask in the water-bath (a thermometer, with its bulb in the liquid, replacing the reflux tube) and shake until the temp. falls to 16° C. Maintain at that temp. for 5 min. and, if there is no turbidity, cool to 15·5° C. for 5 min. If there is still no turbidity the sample contains no arachis oil or less than 5%. *Separation of Arachidic and Lignoceric Acids.*—Renard's method is used. *Detection of Resin Oils.*—Shake 20 ml of the sample with 200 ml of 90% alcohol, decant the alcohol and evaporate it in a porcelain capsule with flat bottom. Add 4–5 drops of sulphuric acid (sp.gr. 1·63) without shaking. A pink colour indicates resin oils. *Extracted Bagasse Oil.*—To detect oil extracted with carbon disulphide, heat 5 g of the sample (free from water) with 20 mg of recently prepared dry silver benzoate at 150° C. A chestnut-brown colour indicates oil extracted with carbon disulphide. To prepare the reagent, treat a fresh soln. of sodium benzoate with silver nitrate, filter off the ppt., wash with water and dry with alcohol and ether. *Semi-drying Oils* (*Soya bean oil, etc.*).—Vizern and Guyot's test with a soln. of bromine in light petroleum is used.

STANDARDS FOR OLIVE OIL.—The sample must be free from abnormal odour or taste and contain no water, inpurities insol. in light petroleum, inorganic acids or mineral oil. *Alkalinity of ash.*—Max. 0·1 ml of $N/10$ soln. per 100 ml of filtered oil. *Unsap. matter:* Max., 1·5% by Hönig and Spitz's method. *Acidity:* max., 4% (as oleic acid). *Ketonic and autoxidative rancidity* must be absent.

Physical and Chemical Constants.—Sp.gr. at 20° C., 0·910–0·913; titre of fatty acids, 18°–29° C.; n_D^{20}, 1·4699–1·4677; sap. val., 186–196; Reichert–Meissl val., 0·2–0·5; Polenske val., 0·9–2·1; iodine val. (Hanus), 75–88; iodine val. of total fatty acids, max. 92; of liquid fatty acids, max. 112; Bellier val., max., 16° C.

ANALYSIS OF LARD FOR FISCAL PURPOSES.—The temperature at which samples are blended should not exceed 60° C. The bulk sample should weigh not less than 250 g.

Colour.—This is measured by comparison of the sample, melted at 50° C., with a series of standards (1 to 10) consisting of 0·001 to 0·010 g of iodine in 100 ml of 10% potassium iodide. The colour should not exceed 4 (≡0·004 g of iodine). *Water* (determined by Polenske's turbidity method), 0·5–2%. *Impurities insol. in chloroform:* above 2%, unsuitable for food; above 5% adulterated. *Unsap. matter*, max. 0·6% (Hönig and Spitz). *Salt*, max. 1%. *Bömer val.*, min. 71° C.

Physical and chemical constants.—Sp.gr. at 20° C., 0·920–0·936; m.p., 30°–50° C.; titre of fatty acids, 32°–42° C.; n_D^{50}, 1·4538–1·4577; acid val., max. 2; sap. val., 192–200; Reichert–Meissl val., max. 1; Polenske val., max. 2; iodine val. (Hanus or Wijs), 53–72; iodine val. of fatty acids, max. 75; m.p. of phytosteryl acetate, max. 117° C. Tests for polybromides, sesame oil and hydrogenated fats must be negative.

ANALYSIS OF ARACHIS OIL FOR FISCAL PURPOSES.—*Colour:* max. 2 on the iodine scale. *Water* and *impurities insol. in light petroleum, and mineral oils* must not be present. *Unsap. matter*, max. 1·5%.

Physical and Chemical Constants.—Acidity (as oleic acid), max. 0·3%; sp.gr. at 20° C., 0·912–0·918; titre of fatty acids, 22°–32·5° C., n_D^{60}, 1·4680–1·4727; sap. val., 185–202; iodine val. (Hanus), 82–105; Bellier test, min. 32° C.; Baudouin test, negative.

Errata (Analysis of Beverages containing Citrus Juices).—Feb. issue, p. 47, 5th item, Table VII, *for* "4·5" *read* "4·6." Date at end: *for* "1941" *read* "1942."

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS

Food and Drugs

Contamination of Cuprous Oxide in Reducing-sugar Analysis. R. F. Jackson and E. J. McDonald. (*J. Assoc. Off. Agr. Chem.*, 1942, **25**, 988.)—In previous work (Jackson and McDonald, *J. Assoc. Off. Agr. Chem.*, 1941, **24**, 767) it was shown that cuprous oxide pptd. in Munson and Walker's method of reducing-sugar analysis (*J. Amer. Chem. Soc.*, 1906, **28**, 663; ANALYST, 1906, **31**, 337) was contaminated with organic decomposition products even when pure sugars were being analysed, and thus the values for copper, which were estimated from the weights of cuprous oxide, were slightly too high. This confirms work by Zerban and Naquin (*J. Amer. Chem. Soc.*, 1908, **30**, 1456; ANALYST, 1908, **33**, 436), which had been overlooked at the time. Zerban and Naquin, confining their attention to invert sugar from the acid inversion of sucrose, found, for concns. of sugar yielding 80 to 430 mg of copper, a mean contamination of 1·7 mg. Jackson and McDonald's analyses of invert sugar (*loc. cit.*) show exactly the same mean difference from Munson and Walker's tables. Hammond in his revision of the Munson and Walker tables (*J. Res. Nat. Bur. Stds.*, 1940, **24**, 579) found a mean difference of 1·9 mg for pure invert sugar. If Munson and Walker's table is corrected by deducting the 1·7 mg excess, there will be available four independent series of analyses yielding the same average results almost to within 0·1 mg. This applies to the average of the whole invert sugar column; individual values fluctuate somewhat. Zerban and Naquin found an almost constant difference between copper calc. from cupric oxide and that calc. from cuprous oxide. Both Hammond's and Jackson and McDonald's difference from the Munson and Walker table pass through a max. of 2·4 mg in the median concns. of sugar. When the cuprous oxide is dissolved in nitric acid, the asbestos in the Gooch crucible becomes progressively blacker owing to carbonisation of organic substances, thereby suggesting that the contaminants are organic decomposition products. Zerban and Naquin believed the apparent excess of copper calculated from cuprous oxide to be due to water retained by the ppt. The contamination is very variable, being practically negligible with dextrose and rising to more than 5 mg with some mixtures of sucrose and invert sugar. A. O. J.

Volatile Oils in Fennel Seed. J. F. Clevenger. (*J. Assoc. Off. Agr. Chem.*, 1942, **25**, 962–963.)—Fennel seeds entering New York during the past 8 years have been grouped into 6 classes: Roumanian, Argentinian, German, French, Indian and Italian. The dark brown Roumanian and Argentinian seeds were similar in size and appearance, *ca.* 5 mm long and 1·5 mm wide. The dark brown German seeds were *ca.* 9 mm long and 2·3 mm wide. The French and Italian seeds were usually light yellow and similar in size and appearance, *ca.* 6·5 mm long and 1·6 mm wide. The bitter fennel, usually of Italian origin, varied considerably in size and apparent maturity. The mature seeds were somewhat lighter in colour but otherwise resembled the Argentinian seeds. The yield of volatile oil varied considerably, being highest in the German and Roumanian seed (3·6–6·0 ml per 100 g) and lowest in the Indian seed (0·8–1·6 ml per 100 g). Over the whole series of oils, α_D ranged from +3·5° to +22·1° and, as a rule, the oils from Roumanian, German and Italian seeds gave uniformly high values and those from French and Argentinian seeds distinctly lower values. Although n_D^{25} was fairly uniform for all the oils (1·507–1·554), the oils from bitter fennel had definitely lower values (1·507–1·511) than the others (1·524–1·554). The congealing temp. for most of the oils lay between 1° and 12·5° C., but oil from one shipment of Argentinian seed congealed at −3·0° C., and the oils from bitter fennel would not congeal even at −15° C. The sp.gr. for the whole series ranged from 0·951 to 0·991. The congealing temp. was determined by the method of U.S.P., XI, 442; the other figures were obtained by the methods of the A.O.A.C. ("*Methods of Analysis*," 1940, 469–472.) A. O. J.

Separation of Non-carbohydrate Substances from Cereal Starches. T. J. Schoch. (*J. Amer. Chem. Soc.*, 1942, **64**, 2954–2956.)—The fatty acids of maize starch can be removed without hydrolytic degradation by extraction, in a Soxhlet apparatus or by refluxing, with hydrophilic fat solvents, *e.g.*, methanol, 80% dioxane or methyl "Cellosolve." Similarly, fatty material may be separated from rice starch as soap, and from wheat starch as a phospholipid plus substantially all the phosphorus of the original raw starch. The lipids, therefore, are not characteristic of any amylose fraction, but, rather, are natural adsorbed impurities. By means of these hydrophilic fat solvents, maize or potato starch can be impregnated with fatty acid, which can be removed only by solvent of the same type as that used to introduce it. When required for fundamental studies, the common cereal starches should first be purified by suspending 1 part of the starch in 3 parts of 85% (by vol.) methanol, refluxing for several hrs. on the steam-bath with adequate stirring, filtering hot, resuspending in fresh 85% methanol, and repeating the extraction 4 times. Five such extractions remove practically all the fat from maize, wheat and rice starches. E. M. P.

Fractionation of Starch by Selective Precipitation with Butanol. T. J. Schoch. (*J. Amer. Chem. Soc.*, 1942, **64**, 2957–2961.)—Fractional pptn. of starch sols by means of *n*-butyl alcohol (Schoch, *Cereal Chem.*, 1941, **18**, 121) yields products with different physical and chemical properties. Procedures are as follows: *Maize Starch.*—Remove the fatty material (see preceding abstract). The *p*H of the defatted starch becomes stabilised at 5·9–6·0 during the separation and prevents glucosidic hydrolysis. Heat to b.p. a mixture of 14 litres of water and 2 litres of butanol in a 5-gall. Pyrex stock bottle, on a steam-bath, and add slowly, with vigorous stirring, a suspension of 150–450 g (preferably 300 g) of defatted starch in 1 litre of water. Autoclave for 2–3 hrs. at 18–20 lb. pressure. For max. purity, pass the hot starch soln. through a continuous supercentrifuge. Alternatively, heat the butanol-water-starch paste under reflux for 5–6 hrs., with vigorous agitation, and then pass it through the supercentrifuge. Cool the hot starch sol. slowly and without agitation to room temp. during 24–36 hrs. Usually a "crystalline" floc forms at *ca.* 50° C. as six-segmented spherulites, 15–50μ in diam. Separate in a Sharples continuous centrifuge, fitted with a clarifier bowl (50,000 r.p.m.)

and capable of passing 20 litre of liquid per hr. Purify by suspending in water saturated with butanol, stirring and centrifuging. For more thorough purification, add the moist product slowly, with vigorous stirring, to a boiling mixture of 10 litres of water and 1 litre of butanol on slow cooling, the butanol pptd. fraction fluocculates as minute particles. Dehydrate by suspending in butanol and filtering on a Buchner funnel. The yield of butanol pptd. fraction averages 22% (on dry starch basis). To obtain the non-pptd. fraction flocculate with excess of methanol and triturate with fresh portions of methanol until dehydrated. The non-pptd. fraction dissolves in cold water (4–5%) and in hot water (10–12%); on cooling, 10% solns. give pasty gels readily liquefied by heating. The solns. do not undergo retrogradation on long standing, even in a refrigerator; the average alkali number is 5·6. The pptd. fraction gives relatively clear solns., in boiling water, up to 10–15%; these have exaggerated retrogradation properties; the average alkali number is 25.

Potato Starch.—Defatting is unnecessary, and the concn. of the autoclaved paste should not exceed 2%. Centrifugal clarification of the hot autoclaved soln. may be omitted; aver. yield of butanol pptd. fraction, 22%. It forms either large 6-petalled rosettes, 50–80μ in diam., or clumps of hair-like needles. When quite dry, this fraction remains relatively water-soluble, with little tendency to gel or retrograde; its aver. alkali number is 11.

Waxy Maize Starch.—Butanol treatment of autoclaved pastes of defatted waxy maize starch gave no trace of crystalline flocculate; less than 3% of slime and impurities separated, but most of this could be dispersed by further autoclaving and was not subsequently pptd. by butanol.

E. M. P.

Separation of Alkaloids in Certain Species of Nicotiana. H. H. Smith and C. R. Smith. (*J. Agric. Res.*, 1942, **65**, 347–359.)—The alkaloids were determined and separated as follows:—Digest 50 g of the air-dried, ground sample with water in a 1-litre Erlenmeyer flask on the water-bath for about 2 hrs. Filter and wash the residue several times with boiling water. Combine the extracts, concentrate on the steam-bath, make strongly alkaline with sodium hydroxide, and extract several times with ether. Combine the extracts and wash out the alkaloid with several portions of dil. hydrochloric acid. Use the washed ether repeatedly to extract more alkaloid from the alkaline aqueous extract, taking care to prevent emulsification. Separate the acid soln. from the ether, make strongly alkaline and extract with several small portions of ether. Combine the ethereal extracts and dry for several hrs. with sodium hydroxide sticks or, preferably, potassium hydroxide pellets. Evaporate the dry soln. in a weighed 50–ml Erlenmeyer flask and heat on the steam-bath for the min. time required to remove all traces of ether. Dry over sulphuric acid for 18 hrs. and weigh. About 95% of the alkaloid is thus extracted.

To separate and identify the alkaloids, rinse the total alkaloids into a distilling flask with 75–100 ml of water, and distil through an efficient fractionating column (*e.g.*, Widmer's) down to 1/5 vol. Add 60–80 ml of water to the residue in the flask and distil again. The nicotine is thus removed as an azeotropic mixture with water, 100 ml of the mixture containing 2·5 g of nicotine. Neither anabasine nor *nor*nicotine forms such a mixture. The distillate contains all the nicotine, with some "non-volatile" alkaloid. Titrate with standard acid to obtain the approx. quantity of nicotine. Make the titrated distillate alkaline with slightly more than the calculated quantity of standard alkali and repeat the distillations. Titration of this distillate gives a close check on the quantity of nicotine, the result being somewhat less than that in the first titration if some non-volatile alkaloid has distilled over with the nicotine. Make the final distillate appreciably acid to methyl red, evaporate to low vol. if necessary, and add picric acid soln. to form nicotine picrate. Ppt. any residual non-volatile alkaloid as picrate and recrystallise at least once. The presence of anabasine or *nor*nicotine is indicated by the crystalline form and m.p. of the picrate (*nor*nicotine, 188°–190° C.; racemic anabasine, 208°–209° C.; optically inactive anabasine, 213° C. The main and secondary alkaloids found in 29 wild species of *Nicotiana* are tabulated. In no species did anabasine and *nor*nicotine occur together; 4 apparently contained only nicotine, 5 only *nor*nicotine, 2 had mixtures of anabasine and nicotine and the rest mixtures of nicotine and *nor*nicotine. Nicotine was always present in the *l*-form, anabasine principally in the optically inactive form, and *nor*nicotine largely in the *l*-form mixed with different proportions of the optically inactive form. The amounts of total alkaloids ranged from 0·02% (*N. plumbaginifolia*) to 1·80% (*nor*nicotine in *N. glutinosa*). The lowest nicotine content was 0·10% (*N. nudicaules*) and the highest 1·02% (*N. wigandioides*).

E. M. P.

Rapid Method for Determination of Small Quantities of Copper on Apples when Lead Arsenate is also present. H. W. Rusk. (*J. Assoc. Off. Agr. Chem.*, 1942, **25**, 980–987.)—Two methods of preparing a soln. of the spray residue on the fruit have been successfully employed, *viz.*, wet digestion of the peel with nitric and sulphuric acids and the removal of the residue from the surface of the fruit by means of an aqueous soln. containing 5% of ammonium nitrate and 5% of nitric acid. The second method has the advantage of requiring no reagent blank and of providing a soln. available for the determination of copper, arsenic (by subsequent digestion and distillation) and lead (by electrolysis). The method for isolation and determination of copper is as follows:—Neutralise an aliquot portion of the soln. (≡0·1–2·0 mg of copper), diluted to *ca.* 200 ml, with ammonia (28%), to the neutral point of alizarin red S. Add 2 ml of conc. nitric acid to bring the *p*H to *ca.* 2·0, and extract the soln. with 0·1% soln. of dithizone in carbon tetrachloride, using *ca.* 25 ml of the reagent for each mg of copper. If the solvent layer is red to bluish-purple, repeat the extraction until the solvent remains dark blue-green. Finally, extract the aqueous layer with 10 ml of carbon tetrachloride. Evaporate the combined extracts just to dryness, treat the residue with 1 g of potassium chlorate and 20 ml of dil. nitric acid (1 + 1), boil vigorously for a few min., evaporate just to dryness, add 5 ml of water and again evaporate to dryness, avoiding decomposition of the nitrate by overheating. Dissolve the residue in 5–10 drops of conc. nitric acid and 50 ml of water and dilute the soln. so that an aliquot portion of *ca.* 10 ml will contain 10–100μg of copper. Dilute 5–10 ml of the soln. to 30 ml with water, add 5 ml of dil. ammonia (1 + 5) and 5 ml of aqueous sodium diethyldithiocarbamate soln. (1 g per litre). After

15 min. measure the colour in a photoelectric photometer (a Brice-type photometer with a Corning blue-green No. 430 filter is recommended). Prepare a range of standards by measuring 0·5, 1, 3, 5, 8 and 10 ml of a dil. standard copper soln. made by acidifying 25 ml of a soln. of 0·393 g of crystalline copper sulphate in a litre of water with 10 drops of conc. nitric acid and diluting to 250 ml. Treat each standard soln. in the same way as the aliquot portion of the unknown soln. Use the proper aliquot portion of a soln. prepared by extraction of the reagents used in the preparation of the sample and treated in the same manner as the unknown and standard solns. to determine the zero point of the instrument. Plot a curve to show the relation between the copper concn. and the photometer readings, and from this curve determine the amount of copper in the unknown soln. The curve deviates from linearity as the concn. of copper increases, and its slope is affected by the reaction temp. and by the age of the sodium diethyldithiocarbamate soln. Expts. showed that the recovery of copper is practically complete.

A. O. J.

Alternative Method for Decomposition and Determination of Acid-insoluble Constituents of Face Powders. G. McClellan. (*J. Assoc. Off. Agr. Chem.*, 1942, **25**, 964–965.)—A modification of the Berzelius procedure has been applied to the determination of acid-insol. substances in face powders (McClellan, *J. Assoc. Off. Agr. Chem.*, 1942, **25**, 909–918). The decomposition residue is dissolved in dil. sulphuric acid, the soln. is diluted to 500 ml, and the oxides of magnesium, aluminium, iron and titanium are determined in aliquot portions. Silica is determined in a separate sample by a rapid method. It is possible to determine all these constituents on the same 2-g sample, but the acid-insol. oxides will be contaminated with *ca.* 0·4% of the total silica. Fuse the ignited acid-insol. residue from 2 g with 12 g of sodium sulphite at 950°–1050° C. for 15 min. Dissolve the melt in 40 ml of dil. nitric acid (3 + 1) in a *porcelain* casserole, add 10 g of ammonium chloride and treat the soln. slowly and cautiously with 40 ml of conc. sulphuric acid. Boil the liquid in the covered casserole for 15 min. after nitrous oxides have been expelled, cool, add 150 ml of hot water, and collect the pptd. silica on filter-paper, washing with hot water and reserving the filtrate and washings. Ignite the residue of silica to constant wt., treat it on the steam-bath with dil. sulphuric acid (1 + 9) and excess of hydrofluoric acid, expel the sulphuric acid and again ignite at red heat. Weigh the residue to determine the silica by the loss in wt., fuse with 1 g of potassium pyrosulphate, and dissolve the fused mass in the filtrate already reserved, boiling until soln. is complete. A turbidity indicates barium sulphate, which should be collected and weighed. The filtrate is then diluted to 500 ml for the continuation of the original method (*loc. cit.*).

A. O. J.

Biochemical

Method of Physiological Assay of Pyrethrum Extracts. O. Lowenstein. (*Nature*, 1942, **150**, 760–762.)—Parkin's method (ANALYST, 1942, **67**, 306; *cf.* Busvine, *id.*, 401) gives satisfactory results, but involves the possible difficulty of maintaining an adequate supply of insects from controlled stocks; the following method is therefore proposed as a rapid preliminary test having a fairly high degree of accuracy:—It is based on the recording of the action-potentials in the exposed abdominal nerve cord of *Blatta orientalis* (cockroach) by means of a standard 3- to 4-valve resistance-capacity coupled amplifier, in conjunction with a 1-valve power stage, and an oscillograph and loudspeaker. The abdomen of an adult female cockroach is severed from the thorax and pinned down, ventral side upwards, on a piece of cork; a medium strip of the body wall is then cut out, deflected backwards and pinned down, so that the ventral nerve cord is fully exposed for the insertion of the recording electrodes, and for the application of the pyrethrum extract to be tested. The grid electrode is placed on the fifth abdominal ganglion, and the neutral electrode on a pad of cotton-wool which is soaked with saline and is in contact with the abdominal tissues. The electrodes are mounted in an earthed metal screen box as a protection against outside electrical interference. If the hair sensillae on the cerci are now stimulated by a puff of air, a high amplitude action potential, which produces a characteristic sound in the loudspeaker, arises in the so-called giant fibre neurons which are situated in the sixth abdominal ganglion, and which make synaptic connection with a different sensory fibre from the hairs on the cerci. The acoustical recognition of this effect (crackling reports) is so easy that the use of an oscillograph and camera is unnecessary, unless a permanent record is required. The effect of pyrethrin is to produce an initial excitory phase, followed by abnormalities in the response of the giant-fibre neurons to stimulation, and eventually the complete failure of this effect. The time taken from the moment of application of the insecticide to the disappearance of the response (and therefore, of the sound in the loudspeaker) is dependent on the concn. of pyrethrum extract used. The sample is applied with a No. 1 water-colour brush, care being taken that no part of the ventral nerve cord is left protected by fat remnants. When the mean survival-time of giant-fibre response at 19°–20° C. (in min.) was plotted against the % of total pyrethrins in the sample (10 expts. with each of 7 solns. in heavy oil; concns., 0·07–1·3%; survival times, over 45·5–0·6 ± 0·2 min., respectively) a hyperbola resulted. There was considerable "scatter" of individual readings within each group, but this was expected in view of the variability in the experimental animals available and the difficulty of applying standard amounts of insecticide, especially where the amount of body fluid present is variable. The discriminating power of the method is poor for concns. exceeding 0·33% of total pyrethrins, and this appears to be its upper limit. Over the range 0·23–0·13%, however, the sensitivity is a max. and the results are statistically satisfactory; thus the difference between the effects of 2 solns. differing in concn. by 0·1% pyrethrins is comparable with that obtained by Parkin's method for solns. differing in concn. by 0·2%. In comparing these methods, however, consideration should be given to the fact that the insecticide comes into contact with exposed nervous tissues, and with an intact animal, respectively. There appears to be an approx. correspondence in toxic effect between a 0·3 and 1·6% soln. applied according to the respective methods. Advantages of the method are that the 10 cockroaches required for each test are easily obtainable; the preparatory experimental procedure requires no specialised knowledge and is performed rapidly; the 10 tests can be completed in 5–8 hrs.

J. G.

Quantitative Estimation of Haemoglobin and Related Haeme Pigments in Faeces, Urine and Blood Plasma. E. B. Flink and C. J. Watson. (*J. Biol. Chem.*, 1942, **146**, 171–178.)—Haeme pigments can be determined in faeces, urine and blood plasma by conversion into pyridine ferro-haemochromogen, and measurement of the absorption under standard conditions by means of an Evelyn photoelectric colorimeter. Prepare a range of blood dilutions containing from 0·06 to 3·0 mg of haemoglobin in Evelyn colorimeter tubes. In each tube put 2 ml of pyridine, 2 ml of freshly prepared 2% sodium hydrosulphite soln. and enough 10% ammonia to make up the vol. to 10 ml. Measure the reading after 5 min., using a 550$m\mu$ filter and a centre setting from a blank tube. In estimating the haemoglobin content of plasma, blood is collected in 3% sodium citrate soln. and immediately centrifuged. One ml of the plasma is then diluted in an Evelyn colorimeter tube with 10% ammonia to 7·5 ml, and finally to 10 ml with 0·5 ml of pyridine and 2·0 ml of freshly prepared 2% hydrosulphite soln. Faeces are treated as follows:—Extract a weighed portion with ether or acetone to remove fat, acidify the residue with 2 to 4 ml of 5% hydrochloric acid and 1 ml of glacial acetic acid, and extract 4 times by thorough grinding with 25 ml of a mixture of 1 part of 95% alcohol and 4 parts of peroxide-free ether. Decant the extract, filter, make alkaline with 10% ammonia and extract 2 or 3 times with water. Dilute the aqueous soln., which contains the haeme pigments, to a known vol., and treat 1 or 2 ml of the extract in a colorimeter tube with pyridine and hydrosulphite soln. as before. With urine, acidify a suitable vol. with 5 ml of 5% hydrochloric acid and extract with four 25-ml portions of alcohol-ether mixture. Wash the extract once with water, and then proceed as for faeces. With amounts of haemoglobin ranging from 18·6 to 266 mg per 100 g, the error was ±10%. From 75 to 95% recovery of haemoglobin added to faeces was obtained.

F. A. R.

Microbiological Method for the Estimation of *p*-Aminobenzoic Acid. M. Landy and D. M. Dicken. (*J. Biol. Chem.*, 1942, **146**, 109–114.)—In view of the difficulties of measuring *p*-aminobenzoic acid by chemical or animal assay, a microbiological method was devised, using *Acetobacter suboxydans*. Cultures are carried on yeast-extract—glycerol-agar, and are transferred at monthly intervals and stored in the refrigerator in the meantime. Inoculum for assays is prepared by transfer to the basal medium, to which has been added 0·05μg of *p*-aminobenzoic acid. The culture is incubated at 30° C. for 24 hr. prior to use. The basal medium consists of casein ("vitamin-free") hydrolysate 0·6 g, glycerol 10·0 g, tryptophan 20 mg, cystine 15 mg, K_2HPO_4 100 mg, KH_2PO_4 100 mg, $MgSO_4 7H_2O$ 40 mg, NaCl 2 mg, $FeSO_4 7H_2O$ 2 mg, $MnSO_4 2H_2O$ 2 mg, calcium pantothenate 200μg, nicotinic acid 200μg, with distilled water to 100 ml (*p*H 6·0). The medium should be filtered through a hardened filter-paper before use. Introduce 5 ml of basal medium and 5 ml of the solns. under test into 50-ml conical flasks, and prepare standards containing 0·01 to 0·1μg of *p*-aminobenzoic acid and "blanks" containing no *p*-aminobenzoic acid. Plug the flasks with cotton wool, autoclave at 15 lb. for 15 min., and, after cooling, inoculate with the test organism as follows:—Centrifuge a 24-hr. culture and wash the cells twice with 10 ml of sterile saline. Re-suspend in 15 ml of saline and transfer 1 drop of the saline suspension to each flask. Incubate the flasks at 30° C. for 48 hr., add 10 ml of water and measure the turbidity with a photoelectric colorimeter. Prepare a standard curve and calculate from it the *p*-aminobenzoic acid contents of the samples under test. Materials that are sol. in water are assayed directly, but insol. materials, *e.g.*, foodstuffs, are minced and extracted with 10 to 20 vols. of water at 15 lb. for 30 min. and centrifuged or filtered. Body-fluids contain inhibitory substances which can be destroyed by autoclaving. For example, blood is laked with an equal vol. of water and autoclaved, 3 vols. of water are added to the autoclaved blood while hot, and the sample is shaken for several min. to disperse and extract the protein ppt. The supernatant liquid is then centrifuged and filtered. The following values were obtained for the *p*-aminobenzoic acid contents (μg per g) of various materials:—brewer's yeast 102, fresh calf-liver 0·2, peptone 0·4, wheat germ 1·0, alfalfa meal 2·0, maize meal 0·3, rolled oats 0·33, wheat middlings 0·52. Cow's milk contained 0·15 and molasses 0·01μg per ml. Several substances related to *p*-aminobenzoic acid were tested for growth-promoting properties, but in no instance was the activity comparable with that of *p*-aminobenzoic acid. Recoveries of added *p*-aminobenzoic acid ranged from 90 to 105%, and the accuracy of the method was similar to that of other microbiological methods, *viz.*, ±15%.

F. A. R.

***p*H Change as a Measure of Growth of *Lactobacillus Casei* in Vitamin Assays. R. H. Silber and C. W. Mushett.** (*J. Biol. Chem.*, 1942, **146**, 271–272.)—Direct determination of the *p*H is recommended in place of titration or turbidity measurements as an indication of the growth of the organism in pantothenic acid assays. With a range of 0·02 to 0·10μg of pantothenic acid, a variation of 2 *p*H units was observed. The method is extremely rapid, and assays of blood, tissue, etc., can be made after only 24 hrs.' incubation, since assays after 24 and 72 hr. give no significant difference by the *p*H method. Quantitative recovery of added pantothenic acid was observed in every expt. It is unnecessary to prepare fresh medium for each assay. Undiluted stock medium (prepared without glucose), is run into a series of suitable bottles, which are plugged, autoclaved and stored in the cold until needed. The medium is filtered before use and adjusted to *p*H 6·9 to 7·0. This is reduced to 6·6–6·7 on autoclaving. The procedure is also recommended for riboflavin assays.

F. A. R.

Stability of Ascorbic Acid in Dehydrated Vegetables. W. R. Aykroyd. (*Nature*, 1943, **151**, 22–23.)—Commercially dehydrated (steam-blanched) vegetables were kept for several months in an incubator in sealed (not exhausted) tins (to simulate tropical conditions), which were opened at intervals. Dehydrated cabbage, cauliflower and khol-khol lost 50% of the ascorbic acid originally present after 12 weeks at 37° C.; other samples of these vegetables, in closed, but not sealed, tins lost 70–75% in 6 weeks at 18°–23° C. Absorption of moisture was noted, and may have accelerated the loss, since a sample of crisp dehydrated potato stored at room temp, in an unsealed closed tin lost only 10% in 12 weeks. Dehydrated amla fruit (*Phyllanthus emblica*), powdered, in tablets and vacuum-packed, lost ascorbic acid steadily, but

only slowly, but the tannins present may have had a protective effect, as has the exclusion of oxygen.
J. G.

Vitamin C Saturation Test of Harris and Abbasy. W. R. G. Atkins. (*Nature*, 1943, **151**, 21.)—In this test (*Lancet*, 1935, i, 71; 1937, ii, 1429; 1942, i, 642) much time is saved by retaining the urine during the fourth and fifth hrs. after dosing with 0·75 g of the vitamin, and by diluting the sample to 0·5 or 1 litre instead of measuring the vol. of each sample. Calculation is simplified by adjusting the 2 : 6-dichlorophenol indophenol reagent so that 1·0 ml ≡ 0·1 mg of vitamin. The method gave consistent results and distinguished between groups of 100 men living under slightly different conditions of vitamin C intake. These differences are shown clearly by plotting the number of daily doses required to approach saturation (*i.e.*, an excretion of 35 mg of vitamin in a 2-hr. period) against the total % of men who have reacted as saturated before and on that day. Cases of scurvy required *ca.* 10 doses to become saturated. The curves indicate the existence of an appreciable personal variation in a population on a fairly uniform diet, and therefore explain why (*e.g.*) a portion only of a ship's company may be affected by scurvy; such differences may rise from differences in muscular exercise undergone or in individual tastes and cooking. Two sections of men fed from the same cookhouse responded very differently to the doses, their saturation-peaks being separated by *ca.* 2 days. It was later found that one section had breakfasted before and the other after being dosed, and this indicates that the vitamin suffers less destruction when taken after food. Unlike vitamin D, vitamin C is not stored in quantity for long. Some of the men in a unit which had not previously been dosed were found in May to have much higher reserves than men who had been saturated in Jan., although they had all since lived on the same diet.
J. G.

Colorimetric Determination of Vitamin C. M. L. Isaacs. (*Ind. Eng. Chem., Anal. Ed.*, 1942, **14**, 948–949.)—Grind a weighed quantity of the sample in a mortar with sand and 5% acetic acid, filter, wash the residue with 5% acetic acid, and dilute the filtrate to a suitable vol. This ext. is stable for *ca.* 3–4 hr.; if it is kept overnight, even in a refrigerator, the loss of vitamin is *ca.* 10%. To a vol. of this soln. ≡ up to 1 mg of vitamin C, in a 50–ml flask, add *ca.* 25 ml of water and 5 ml of reagent (*vide infra*), and dilute to the mark with water; use 1 ml of reagent for 0·01–0·1 mg of vitamin. Mix thoroughly, filter if necessary, and match the blue colour of the reduction product in Nessler cylinders or, preferably, in a photoelectric colorimeter. A Duboscq colorimeter is unsuitable because of the varying excess of the yellow reagent. Suitable standards contain 0·01–1·0 mg of pure ascorbic acids per 50 ml, in steps of 0·01–0·20 mg; with the higher ranges, match the colours by viewing the Nessler cylinders horizontally. The standards tend to increase in colour after 2 days, but if they are then re-adjusted with dil. reagent, they remain stable for several weeks before fading. *Reagent.*—Dissolve 2 g of ammonium molybdate in 50 ml of water at *ca.* 55° C., and add 10 ml of a fresh 1% sodium silicate ($9H_2O$) soln. and 5 ml of glacial acetic acid; dilute to 100 ml, and leave overnight. The reagent keeps indefinitely, and its activity is not impaired by the white ppt. of molybdic acid which forms after several weeks; sensitivity, 0·01 mg of vitamin per 50 ml of soln. With a photoelectric colorimeter use a Pyrex No. 241 pyrometer red glass or a Klett-Summerson No. 66 filter; this eliminates the effect of the colour of the reagent, of any turbidity which cannot be removed by filtration (*e.g.*, in fresh lemon juice), and of any natural red or yellow colouring matters from the sample. The instrument should, however, be calibrated in terms of readings obtained with solns. of known concns. The method is suitable for use with fruits and vegetables, especially when the concn. of other reducing substances is low. With other materials the results are comparable with those obtained by iodine titration, and therefore tend to be rather higher than with the indophenol method. The reagent is readily reduced by ferrous and stannous ions and, more slowly, by cysteine, sulphites and thiosulphates.
J. G.

Vitamin C Saturation: Standardisation Measurements at Graded Levels of Intake. L. J. Harris. (*Nature*, 1943, **151**, 21–22.)—Groups of boys (12–36 per group) were kept on a basal diet of known vitamin C content, and were given in addition various graded supplements of natural and synthetic vitamin C over periods of 3–4 months or more; each boy in a group received the same intake. About twice yearly (*i.e.*, after an interval sufficient to allow the effect on their reserves of any previous saturation test to have become negligible), the boys were tested by the author's method; 700 mg per 10 stone were given daily, and the number of days were counted until the approach of saturation. This was taken as an excretion of 50 mg or more per 10 stone in the 2·25-hr. specimen of urine collected in the fourth and fifth hrs. after each test dose. The results were graded as follows:—Daily intakes of 75, 60, 50 and 45 mg gave first-day responses of decreasing magnitude; 40 mg, first- to second-day responses; 25 mg, second- to third-day responses. The "scatter" in the responses of all subjects on the same intake-level was small. In earlier tests children on still lower intakes had third- to fifth-day responses. Similarly-graded results were recorded for adults on varied intakes; scorbutic subjects required 7–10 days for saturation. An intake corresponding with, or slightly in excess of, the League of Nations requirement (30 mg) sufficed to give responses in nearly all subjects on the first or second day of dosing; this therefore may be used as a reference standard in assessing surveys, and the number of days after the second may be taken as an index of the relative deficit in the past intake. With intakes of the order of 50–75 mg per day (the optimum recommended by some American workers) saturation is attained on the first day of dosing; whilst with intakes appreciably below 30 mg, 3 or more days are required, with a limit of 7–10 days in cases of developed scurvy.
J. G.

Chemical Determination of Tocopherols in Muscle Tissue. H. B. Devlin and H. A. Mattill. (*J. Biol. Chem.*, 1942, **146**, 123–130.)—The method of Emmerie and Engel (*Rev. trav. chim. Pays-Bas*, 1938, **57**,1351; ANALYST, 1939, **64**, 216) was recently applied to the determination of tocopherol in blood serum (*J. Biol. Chem.*, 1942, **143**, 695). By further refinements the method has been made applicable to muscle tissue, as follows:—(1) The procedure of Parker and McFarlane (*Canad. J. Res., Sec. B*, 1940, **18**, 405), in which double bonds capable of reducing ferric iron are destroyed by treatment with 85% sulphuric acid; (2) the use of

a Waring blender for shredding and extracting the tissue; (3) the use of the foaming solvent recommended by Moore and Ely (*Ind. Eng. Chem., Anal. Ed.*, 1941, **13**, 600), which enabled complete extraction of the unsaponified material to be made; (4) cholesterol, which was found to interfere with the estimation, was removed by adsorption on purified Super-sorb (Florisil); (5) an all-glass apparatus was used to eliminate impurities introduced from rubber tubing. *Method.*—Mince in the blender 10 to 25 g of muscle in presence of 200 ml of a mixture of purified Skellysolve B and abs. alcohol (8·5 : 10). After 5 min., centrifuge, and extract the residue 3 times more. Add water to the combined supernatant liquids so that the mixture has an alcohol concn. of *ca.* 40%, and break the emulsion by addition of a little alcohol. Evaporate the light petroleum layer to dryness under reduced pressure in presence of nitrogen, and dissolve the residue in 15 ml of benzene. Run 2·5-ml portions through adsorption columns (30 × 12 mm) of purified Supersorb. Wash each column with four 5-ml portions of benzene, and evaporate the filtrates on a boiling water-bath in a stream of nitrogen. Treat an aliquot portion as in the Parker–McFarlane technique, thus: Add 10 ml of Skellysolve E and 2 ml of 85% sulphuric acid, and centrifuge for 2 min. Decant the supernatant liquid into another tube, add 5 ml of 2% potassium hydroxide soln., and centrifuge for 10 min. To 5 ml of the supernatant liquid, which contains the tocopherols, add 20 ml of glacial acetic acid reagent (250 mg of ferric chloride and 500 mg of $\alpha\alpha'$-dipyridyl in 1 litre of glacial acetic acid). Measure the colour in a Klett–Summerson photoelectric colorimeter after 30 min. The recovery of added tocopherol was 91% of the theoretical by the method recommended above, but was only 74% when the treatment with sulphuric acid was omitted.

F. A. R.

Bacteriological

Growth of Coliform Bacilli in Distilled Water. J. W. Bigger and J. H. Nelson. (*Proc. and J. Inst. Chem.*, 1943, 33.)—The remarkable growth of *B. coli* in boiled or autoclaved tap water and in distilled water previously passed through a Pasteur–Chamberland filter is recorded. In absence of rubber connections growth occurred in the filtered water. It was noted, however, that after rubber tubing had been boiled in distilled water there was a deposit of talc in which coliform organisms increased from several hundred to one million per ml, and that even after the talc had been roasted in a muffle-furnace and repeatedly washed with distilled water growth still occurred, but that in absence of carbon dioxide or ammonia in the atmosphere there was no growth. In long-period investigations at 37° C and at 22° C. growth occurred at the former temp. for 220 days and at the latter for 335 days. Other bacteria, including *B. pneumoniae* and *B. alkaligenes* behaved in a similar way, and substances other than talc were found to render distilled water growth-supporting.

D. R. W.

Susceptibility of Wood to Decay [Decay Tests]. F. H. Kaufert and E. A. Behr. (*Ind. Eng. Chem.*, 1942, **34**, 1510–1515.)—Blocks of treated and untreated southern pinewood and the heartwoods of cypress, Douglas fir and red oak, 2 × 1 × 3/8 in. (1/4 in. for one series) cut across the grain, were used in the tests, the blocks being either air-dried or dried at 70° C. and their oven-dry weights computed from data on control blocks dried at 100° C. The action of the most important wood-rotting fungi (*Lenzites trabea, Lentinus lepideus, Trametes serialis* and, for red oak, *Daedalea quercina*) was tested by the following method.—Grow the fungi on malt agar in square quart jars with metal caps. Lay two sterilised birch applicator sticks, 0·1 in. in diam., on the fungus mat in each jar, and on these place the test blocks previously dipped for 1 sec. in boiling water. After 60 (or 90) days remove the blocks from the jars, dry them at 100° C. and weigh. Compute loss in weight as follows:

$$\% \text{ loss in wt.} = \frac{\begin{pmatrix}\text{original oven-dry}\\ \text{wt. + wt. of}\\ \text{absorbed chemical}\end{pmatrix} - \begin{pmatrix}\text{oven-dry}\\ \text{wt. after}\\ \text{decay}\end{pmatrix}}{\begin{pmatrix}\text{original oven-dry wt. +}\\ \text{wt. of absorbed chemical}\end{pmatrix}}$$

Express the retention of chemicals used for treating the wood in lb. per cb. ft. and as % of the oven-dry weight. The aver. sp.gr. of the woods tested were: red oak 0·62, Douglas fir 0·44, cypress 0·44, southern pine 0·46. At least 6 blocks of each were used in the tests described. Concentrations of urea or ammonium sulphate above 0·30% reduced the rate of decay, and, if above 1%, prevented all growth of the fungi on the surface of the blocks. The addition of soil bacteria to urea-treated wood had no apparent effect upon its susceptibility to decay by wood-rotting fungi.

Organic

Identification of *o*- and *p*-Sulphobenzoic Acids as their S-Benzylthiuronium Salts. E. Campaigne and C. M. Suter. (*J. Amer. Chem. Soc.*, 1942, **64**, 3040–3041.)—The three sulphobenzoic acids can be distinguished by treating a soln. of the acid, or an acidified soln. of a salt, at b.p., with the calculated quantity of a 10% soln. of S-benzylthiuronium chloride. *o*-Sulphobenzoic acid gives an immediate ppt. of di-(S-benzylthiuronium) *o*-sulphobenzoate, recrystallisable from 70% alcohol in fine white needles, m.p. 205·5–206·5° C. (corr.). *p*-Sulphobenzoic acid yields, on cooling, flat plates of S-benzylthiuronium hydrogen *p*-sulphobenzoate, recrystallisable with difficulty from ethyl alcohol, m.p. 212·6–214·4° C. (corr.). *m*-Sulphobenzoic acid gives either no ppt. or an oil.

E. M. P.

Detection and Determination of 4-Amino-2-Methyl-1-Naphthol. A. R. Menotti. (*Ind. Eng. Chem., Anal. Ed.*, 1942, **14**, 601–602.)—After preliminary expts. with similar reagents sodium pentacyanoammineferroate was chosen as the most suitable for the photometric estimation of 4-amino-2-methyl-1-naphthol. The formation of a blue colour is attributed to the replacement of the co-ordinately bound mol. of ammonia by a mol. of the aminonaphthol. The max. absorption of the coloured soln. occurs at 650$m\mu$ and the soln. obeys Beer's law within the limits of experimental error. To prepare a standard soln. of the drug, dissolve 50·53 mg of the pure hydrochloride and 50 mg of sodium bisulphite in 50 ml of water in a dark glass-stoppered bottle. The soln. is stable for 4 to 6 hr., but may decrease in strength by 10 to 15% when allowed to stand overnight. To prepare the reagent, dissolve 250 mg of sodium pentacyanoammineferroate and 500 mg of anhydrous sodium carbonate in 25 ml of water. This soln. is

stable for about a week. For the assay of ampouled solns. of the drug, dilute the soln. until the concn. of the drug is about 1 mg per ml. Extract powdered material with water containing 0·1% of sodium bisulphite, filtering the extract if necessary. Place the unknown soln. (1 ml) in a 50-ml flask and an approx. equiv. amount of the standard soln. in a similar flask and, if necessary, make the vols. equal. Treat each soln. with 1 ml of the reagent soln. and leave the flasks in the dark for 15 min. Dilute the solns. to 50 ml and compare the colours in a colorimeter, repeating the estimation with a more appropriate amount of the standard soln. if the results differ by more than 10%. The reagent may be used for the identification of the drug by adding a drop to a little of the unknown soln. in the depression of a spot-plate, when a green or blue colour appearing in a few min. will denote the presence of 0·5μg of the drug. The reagent gives blue colours in alkaline soln. with other primary aromatic amines, green colours with aliphatic and aromatic nitroso compounds, and red or yellow colours with hydrazine or its derivatives, but these are unlikely to occur in preparations of the drug intended for oral or parenteral administration. Aliphatic amines and secondary and tertiary aromatic amines do not interfere with the method. To prepare crystalline sodium pentacyanoammineferroate,

$$Na_3[Fe(CN)_5NH_3],$$

dissolve 10 g of finely powdered sodium nitroprusside in 30 ml of conc. ammonia soln., allow the soln. to stand overnight at 0°–10° C., collect the crystals by filtration and wash them several times with ethanol (95%) and once with abs. methanol. A further crop of small crystals of lower but satisfactory purity may be obtained by treating the mother liquor with ethanol. A. O. J.

Nigerian Cassava Starch. J. R. Furlong. (*Bull. Imp. Inst.*, 1942, **40**, 257–271.)—Samples of 6 varieties of cassava starch (*Manihot utilissima*) from the Nigerian Dept. of Agriculture, gave the following results:—Moisture, 12·2–12·4; protein, 0·11–0·33; crude fibre, 0·05–0·13; ash, 0·15–0·24%. Nine further samples prepared from 10 varieties (including 5 of the 6 above) and washed 6 times, gave: moisture, 14·7–18·8% and (on dry basis) ash, 0·035–0·107; protein, 0·025–0·11%. By commercial evaluation all the first 6 samples were reported to be saleable, but, owing to lumpiness, the price would be lower than that of finely powdered starch. All the second batch were also approved, apart from lumpiness, and all had satisfactory dextrinising properties. Other tests of good quality are acidity: low; colour: a good white; cleanliness: freedom from coloured specks, which, if present, cannot be removed by dressing. "Squeak": A well-washed finely-divided sample (which can be pulverised easily) "squeaks" under knife-pressure; an impure starch (containing non-starchy matter) does not squeak. A table indicates correlation between these tests of quality and the results of analytical determinations. On this basis the Imperial Institute suggested max. limits for analytical figures for the highest grade as follows:—moisture 12·5, ash 0·25, protein 0·15, and fibre 0·05%. The dextrinisation test used by the Imperial Institute is as follows:—Rub 10 g of cassava starch in a mortar with 0·2 ml of dil. hydrochloric acid (3·4% w/w) and 0·2 ml of dil. nitric acid (3·0% w/w) until thoroughly mixed. Leave the mixture uncovered in a beaker overnight; then spread in thin layer on filter paper and dry in oven at 35° ± 3° C. for 3 hr. Roast the paper and mixture in an oven at 155° ± 5° C. for 1 hr. If the dextrinisation test is successful, the product should be cream or pale buff to pale yellow in colour, and water-sol. either in the cold on shaking or on warming slightly. For the iodine test, treat a soln. (*ca.* 10%) of the dextrinised product with 2 or 3 drops of 1% iodine soln. and note the colour produced by the falling drops. A reddish-brown colour indicates a well-dextrinised product; blue in variable amounts indicate lower degrees of dextrinisation. An appendix by K. T. Hartley (pp. 268–271) gives particulars of production of this starch as a Nigerian village industry; so far, equipment for fine grinding has not been provided. E. B. D.

Determination of Free Hydroxyl Content of Cellulose Derivatives. C. J. Malm, L. B. Genung and R. F. Williams, Jr. (*Ind. Eng. Chem., Anal. Ed.*, 1942, **14**, 935–940.)—The free or unsubstituted hydroxyl groups of cellulose derivatives may be measured by acetylation with acetic anhydride in pyridine soln., under controlled conditions (*cf.* Schaefer, *id.*, 1937, **9**, 449; Marks and Morrell, ANALYST, 1931, **56**, 428). The effects of time, reaction-temp., concn., and amount of anhydride used were studied, and the following optimum conditions were established:—Dry the sample for 2 hr. at 100°–110° C., cool in a desiccator, and weigh out 1-g samples into 250-ml conical flasks. Add 40 ml (pipetted accurately) of a mixture of 50 ml of acetic anhydride (concn., at least 97%) with 950 ml of pyridine (*vide infra*) to each, and to a blank flask, and heat under reflux condensers, using ground-glass joints, for *ca.* 24 hr. at 75°–80° C. With cellulose acetate and other esters having only a moderate water-resistance, decompose the excess of reagent and ppt. the ester by carefully adding water in a fine stream, with rapid stirring. With acetate butyrates of high butyryl content, and similar esters of high water-resistance, add *ca.* 5 ml of water through the condenser, with shaking, heat for a few min., cool, remove the flask, and rinse the condenser with 100–150 ml of water which are collected separately. Thin the reaction mixture with pyridine if necessary, and pour it into the rinsings, with vigorous stirring so as to produce a soft, fine ppt. of ester; if this is lumpy or hard, the determination must be repeated. Titrate the mixture of liquid and ppt. with standard (*e.g.*, 0·5 *N*) sodium hydroxide soln. (phenolphthalein as indicator) or preferably, electrometrically to *p*H 9·0. The result is conveniently expressed as % of free hydroxyl = (vol. required by blank − vol. required by sample) × normality of alkali × 1·7/wt. of sample taken. Triangular diagrams are given showing the relationship between this figure and the compositions of cellulose acetate propionates and butyrates, and between the number of free hydroxyls per glucose unit and the acetyl, butyryl and propionyl contents of the above esters. Equations corresponding with these calculations are given. The pyridine used for the reagent should be purified by fractional distillation, and the fraction of b.p. 115·5°–115·5° C. taken. If the water-content exceeds 0·1% as determined by the method of Smith, Bryant and Mitchell (*J. Amer. Chem. Soc.*, 1935, **57**, 841; 1939, **61**, 240), add an additional 5 ml of the anhydride for each 0·1% present, and allow the mixed reagent to stand for 24–48 hr. in an all-glass container before it is used. Pyridine is preferred because it is a solvent for most cellulose esters over a wide range of concns., it produces a vigorous and practically irreversible

reaction, and it does not interfere with the titration. The tests of the method were confined to samples containing not more than 1·25 hydroxyl groups per glucose unit of cellulose. The max. deviation for 12 samples of normal viscosity corresponded with an error of 0·1% free hydroxyl or 0·25% acyl (as apparent acetyl). The average deviation was 0·006 hydroxyl group per glucose unit, corresponding with an error of 0·03% free hydroxyl or 0·08% acyl. The method has been found useful for checking the accuracy of other analytical methods; correlating the hydroxyl content with the physical properties of cellulose esters; analysing multi-component derivatives, one component of which may be calculated by difference. With cellulose derivatives of very low viscosity, the average degree of polymerisation may be calculated from the deviation from 3 hydroxyls per glucose unit, as found by analysis. J. G.

Nature of Fibre Staining by Iodine Stains. H. W. Rowe. (*Paper Ind.*, 1942, **24**, 873–877.)—An aqueous iodine stain gave the following colours:—Arrowroot starch, deep violet; dextrin, deep red-blue; waxy maize (corn) starch, red-brown; bleached soda pulp, faint orange; cotton cellulose, very faint yellow. These results indicate that with the carbohydrates there is both a progressive change from blue, through red, to yellow, and a decrease in colour-intensity, corresponding with the change to more strongly-oriented structures and with the decrease in the number of surface-active hydroxyl groups. Amorphous substances containing lignin, and methylated and acetylated cellulose were stained yellow. Both iodine vapour and alcoholic iodine soln. stained arrowroot starch yellow, but the colour changed to violet on addition of a drop of water. The addition of a small amount of potassium iodide increased the intensity of iodine stains; larger amounts tended to change blue and red reactions to violet, red or orange shades, but had little or no effect on the yellow reactions. This is probably the result partly of the dispersing effect of the iodide on the large aggregations of iodine, and partly of the formation of a potassium triiodide complex. Addition of zinc chloride had little effect on blue reactions (*e.g.*, that of arrowroot starch), or on those of lignified materials, but carbohydrates which gave a yellow shade with iodine (*e.g.*, cotton cellulose) acquired a definite blue shade. Calcium chloride behaved similarly, but its effect was less marked. Aluminium chloride had little or no effect on the shades of the stains, but it increased their intensities in some instances. The total amount of iodine absorbed by the fibres may be determined by adding a known vol. of the stain, and titrating the excess with sodium thiosulphate soln., with starch as indicator after a definite period (*e.g.*, 5 min.) of contact with the fibres; however, this procedure fails to distinguish between adsorbed and combined iodine. The latter is obtained by titrating the suspension of fibres in the iodine soln., so as to determine by difference the iodine which has reacted with the fibres and, therefore, is not titratable. The adsorbed iodine is given by the difference between the total iodine retained by the fibres and the combined iodine. In general, more iodine was adsorbed by unbleached pulps and groundwood than by bleached and purified fibres; the results obtained with groundwood may be due to the larger specific surface of the amorphous lignin. Bleached or unbleached alkaline pulps usually adsorbed more iodine than did acid (sulphite) pulps, particularly if they were prepared from hardwoods. Potassium iodide had relatively little effect on the adsorption of iodine, but adsorption was less in presence of aluminium chloride than if zinc or calcium chloride was present. Both zinc chloride and calcium chloride showed concn. regions over which there was max. adsorption; this may have been due to the presence of a concn. of salt conducive to max. swelling. There is evidence that the zinc chloride hydrolyses part of the cellulose, forming reducing groups which react with the iodine. The amount of combined iodine appeared to have no influence on the colour reaction, indicating that an oxidation-reduction action takes place between it and the groups and so did not form a coloured compound with either the cellulose or the lignin. Groundwood pulp which had been exposed to ultra-violet light or to direct sunlight gave a staining reaction similar to that of unbleached sulphite pulp. This indicated that the lignin had been so modified that it could be removed partly by water or sodium hydroxide soln., both of which are used in the preparation of the slides for staining. J. G.

Inorganic

Some Allyl Nitrophenyl Thiosemicarbazides and their Analytical Properties. A. W. Scott and J. T. Andrews. (*J. Amer. Chem. Soc.*, 1942, **64**, 2873–2874.)—Nitro substitution in the ortho, meta, or para positions does not affect the selectivity of 1-allyl-4-phenyl thiosemicarbazide in reaction with silver, mercuric, mercurous and copper ions, but the four reagents differ greatly in sensitivity; the *p*-nitro compound is much the most sensitive reagent for the four ions, the *o*-nitro compound comes next, and the *m*-nitro and unsubstituted compounds last. 1-Allyl-4-(*p*-nitrophenyl) thiosemicarbazide gives a red ppt. on standing with 1:1,000,000 mercuric mercury solns., and a slight colour at a concn. of 1:10,000,000; attempts to use this reagent for the gravimetric determination of mercury were unsuccessful. E. M. P.

Colour Reactions with 1,10-Phenanthroline Derivatives. M. L. Moss, M. G. Mellon and G. F. Smith. (*Ind. Eng. Chem., Anal. Ed.*, 1942, **14**, 931–933.)—Tests were made with the 5-bromo, 5-chloro, nitro, 5-methyl, and 5-nitro-5-methyl derivatives. A spectrophotometric study of the ferrous complexes proved that the substitution compounds can be used for the colorimetric determination of iron, but the parent compound is preferable. The cuprous and reduced molybdenum complexes are more or less unsatisfactory for quantitative purposes. W. R. S.

Volumetric Determination of Antimony with Dichromate. R. B. Neill. (*Ind. Eng. Chem., Anal. Ed.*, 1942, **14**, 955.)—*Tin Base Alloys.*—Boil 0·5 to 1 g with 10 ml of strong sulphuric acid in a 300-ml conical flask. Cool, add 40 ml of water, boil for 1 min. to expel any sulphur dioxide (arsenic may then be removed by adding 50 ml of strong hydrochloric acid and boiling down gently to 20–30 ml). Add 20 ml of hydrochloric acid, dilute the clear soln. to 150 ml with cold water, cool to 40° C., and titrate with dichromate soln. until the green liquid shows an orange tint. Add 2 to 3 drops of indicator and 5 ml of phosphoric acid, titrate with ferrous sulphate soln., adding 0·2 to 0·3 ml excess, and finish the titration with the dichromate soln. To

obtain the dichromate: ferrous sulphate ratio, add to the titrated liquid the same vol. of ferrous soln. as was used in the titration, and titrate with dichromate. Standardise against pure antimony. *Indicator.*—1 g of barium diphenylaminesulphonate in 100 ml of strong sulphuric acid. *Lead Base Alloys.*—Boil 0·5 to 1 g with 15 ml of nitric acid (1:1) and 0·5 g of tartaric acid in a 300-ml conical flask. When the sample has dissolved add 10 ml of strong sulphuric acid and 10 g of potassium sulphate, boil down to a melt, with partial oxidation by nitric acid if much frothing occurs. Expel the excess of sulphuric acid and cool. Boil with 50 ml of hydrochloric acid to remove arsenic if necessary, add 25 ml of water, 20 ml of strong hydrochloric acid and 10 g. of ammonium chloride, and warm to complete solution. Dilute as above to 150 ml and titrate. W. R. S.

Determination of Cobalt and Manganese by Photometric Methods. L. Waldbauer and N. M. Ward. (*Ind. Eng. Chem., Anal. Ed.*, 1942, **14**, 727.)—This method is suitable for the determination of small quantities of cobalt in presence of major amounts of manganese and has been extended to serve for the determination of small proportions of manganese in cobalt. Cobalt is determined by measuring the transmission factor of the chloroform extract of the cobaltinitroso-β-naphthol compound, using a standard form of photoelectric absorptiometer with a green filter. A suitable range of samples for the calibration curve contains amounts of cobalt from 0·01 to 0·60 mg. Manganese does not interfere. For the determination of manganese in large amounts of cobalt, the manganese ammonium phosphate is pptd. and then converted into the purple manganese formaldoxime compound, the transmission factor of which can be measured in the absorptiometer. Since some cobalt is also pptd., this must be determined by the cobaltinitroso-β-naphthol procedure and the manganese content then deduced from a calibration curve using various known manganese concns. in association with this particular cobalt concn. B. S. C.

Determination of Molybdenum in Steel. E. R. Vance. (*Steel*, 1942, **111**, 56, 58.)—Dissolve a 0·1 g. sample by heating in 6 ml of nitric-sulphuric acid (60 ml of water, 18 ml of conc. sulphuric acid, 28 ml of conc. nitric acid); add 5 ml of water and boil the liquid for 1 min.; then add 30 ml of 1:5 hydrochloric acid, cool, and add 5 ml of 5% potassium thiocyanate soln., 50 ml of ether and 10 ml of stannous chloride soln. (50 g of stannous chloride in 40 ml of conc. hydrochloric acid and 160 ml of water). When the iron thiocyanate colour is bleached by the stannous chloride, leaving orange-red molybdenum thiocyanate, shake the soln., separate the aqueous layer, and measure the extracted molybdenum colour in the ethereal layer in a photoelectric colorimeter previously standardised against the colours of known amounts of molybdenum. The sample weight is suitable for steels with up to 0·6% of molybdenum. S. G. C.

Separation of Ytterbium from Thulium and Lutecium. J. K. Marsh. (*J. Chem. Soc.*, 1943, 8–10.)—Ytterbium acetate readily forms an ytterbium amalgam when its soln. is treated with sodium amalgam. Under the same conditions the neighbouring elements thulium and lutecium do not react, and can thus be separated very satisfactorily from ytterbium. The reduction of the latter takes place in 2 stages: (1) to ytterbous salt, (2) to the metal. A little ytterbium (*ca.* 0·01%) remains with the non-reducible earths, but by addition of samarium acetate followed by renewed amalgamation, most of the ytterbium is removed with the samarium (ANALYST, 1942, **67**, 404). In this way lutecium containing only 0·001% of ytterbium has been prepared. The purification of ytterbium itself is simple, since with oxides free from early or middle members of the rare-earth group, nothing but ytterbium enters the amalgam and the resulting metal is spectroscopically free from its neighbours (*i.e.*, contains less than about 0·01%). Ytterbous ion, produced by sodium amalgam in concentrated soln., imparts an orange-yellow colour to the liquid. Cerous acetate cannot be used as a vehicle for the elimination of ytterbium from lutecium and thulium solns. W. R. S.

Determination of Selenious Acid. D. F. Adams and L. I. Gilbertson. (*Ind. Eng. Chem., Anal. Ed.*, 1942, **14**, 926–927.)—A method for the determination of selenious acid and selenites in presence of selenate and nitric acid is based on bromate oxidation to selenic acid and determination of excess bromate by arsenite. Treat about 35 ml of 0·1 *N* sodium arsenite soln. (neutralised to litmus with *N* sulphuric acid) with 10 ml of strong hydrochloric acid and 2 drops of Fast Red B indicator; titrate with 0·1 *N* potassium bromate until the colour fades. Add 1 more drop of indicator and finish the titration dropwise until the liquid is colourless: $HBrO_3 + 3H_3AsO_3 = HBr + 3H_3AsO_4$. This gives the ratio arsenite : bromate. Selenious acid (0·1 to 0·3 g) in 25 ml of water and 1 ml strong nitric acid is boiled with a measured excess of potassium bromate soln. until bromine-free;

$$5H_2SeO_3 + 2HBrO_3 = 5H_2SeO_4 + H_2O + Br_2.$$

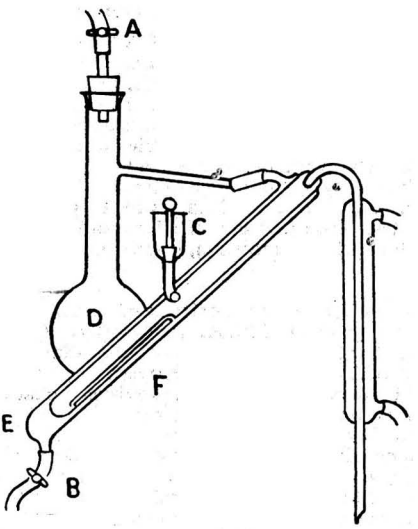
Add a known excess of sod. arsenite soln., then 10 ml of strong hydrochloric acid, and titrate with the bromate soln., using Fast Red B indicator. The vol. of soln. under treatment should be *ca.* 50 ml and the final titration carried out at or below 80° C. The vols. of bromate and arsenite solns. required may have to be determined in a pilot assay. Barium and lead, which yield insol. selenates, ferric and chromic ions and halide ions (which react with bromate), interfere. W. R. S.

Analysis and Composition of Crude Boron. E. H. Winslow and H. A. Liebhafsky. (*J. Amer. Chem. Soc.*, 1942, **64**, 2725–2726.)—Samples of commercial boron were analysed by methods admittedly of an approx. and provisional character. Total boron was determined by fusion with 12:1 sodium peroxide and sucrose mixture in a Parr bomb or with 2:1 sodium carbonate and potassium nitrate mixture in an open platinum crucible. The soln. of the melt was titrated for boron by the "usual method" [presumably mannitol acidimetric titration] the end-point being determined potentiometrically with the use of a glass electrode. Impurities were determined by analysis of the residue or sublimate from heating the boron in a glass tube in a stream of chlorine. Boron volatilised as boron trichloride was absorbed in water and the boron was determined. It was concluded that materials sold as pure boron may contain less than 80% of total boron, much of which is not present as the element. The most likely impurities are oxygen and reducing agent, *e.g.*, magnesium or aluminium used in the process of preparation. S. G. C.

Determination of Ammonia by a Diffusion Method. A. N. Prater, E. J. Cowles and R. P. Straka. (*Ind. Eng. Chem., Anal. Ed.*, 1942, **14**, 703.)—This method is based on the diffusion cell technique of Conway (*Biochem. J.*, 1933, **27**, 430), but makes use of apparatus and supplies generally available in the laboratory. The diffusion unit consists of a 50-mm or 100-mm diam. Petri dish with ground rims, and a plane circular cover plate, large in diam. than the dish by *ca.* 10 mm. The ammonia, liberated from the sample in the dish by treatment with saturated solns. of sodium metaborate and potassium chloride, is absorbed in a number of separate drops of boric acid in glycerin suspended from the glass cover-plate. After absorption of the ammonia is complete, the suspended drops are washed into a flask and the ammonia is determined by electrometric titration, using a glass electrode *p*H meter, or colorimetrically by titration, using the mixed indicator methyl red—bromocresol green. The method was tested on known solns. of ammonium salts, on a meat extract and on a meat extract *plus* ammonium chloride soln. With the size of Petri dish used, amounts of ammonia nitrogen from 0·1 to 9 mg can be determined. Check tests gave results within 1% of the known concn. B. S. C.

Microchemical

Steam Distillation Apparatus suitable for Micro-Kjeldahl Analysis. R. Markham. (*Biochem. J.*, 1942, **36**, 790–791.)—The apparatus shown has few rubber connections and can be



mounted on a single retort stand. It is convenient for the distillation of volatile fatty acids and for micro-Kjeldahl analyses. After the apparatus has been steamed out, the screw clips A and B are opened and the soln. to be analysed is rinsed in with 5–10 ml of water through the funnel C, which has a ground-in stopper. The stopper is replaced, and 2 ml of 40% sodium hydroxide soln. are put into the funnel. The flask D, which serves as steam-generator, is connected to the apparatus, clip A is closed, and steam is passed into the apparatus until the temp. reaches 100° C.; clip B is then closed. The soda in the funnel is added by opening the stopper, and distillation is continued at 8–10 ml per min. for 1–1½ min. The end of the condenser should not dip under the dil. acid in which the ammonia is collected unless more than 0·5 mg of nitrogen is present; there is practically no loss and the vol. of distillate is not increased by the necessity of rinsing the tip of the condenser. The burner is removed and the liquid in F runs automatically into E. Water is then passed into C, and passes through into E. Finally, the clips A and B are opened, and the apparatus is ready for the next run. F. A. R.

Physical Methods, Apparatus, etc.

Rapid Determination of Small Gas Flows. L. Silverman and R. M. Thomson. (*Ind. Eng. Chem., Anal. Ed.*, 1942, **14**, 928.)—The gas is led through a capillary tip previously dipped in soap soln. The moment a bubble begins to form, a stop-watch is released; this is stopped when the gas is turned off. The diameter of the bubble is measured by observation through a lens, an illuminated scale being placed behind the bubble. The scale consists of thin cross-section ruled paper illuminated from behind. The bubbles are quite stable (1·5 to 2 min.); repeated measurements of known vols. agreed within 4% when the horizontal diameter was measured. W. R. S.

Modified Calomel Cell for *p*H Measurements. A. D. E. Lauchlan and J. E. Page. (*Nature*, 1943, **151**, 84.)—In view of the wastage of potassium chloride that results from the frequent flushing of the liquid junction in calomel electrodes prepared with saturated potassium chloride soln., expts. were made with saturated sodium chloride soln. Well-defined potentials were obtained, and, although the potential of the new cell (245·8 millivolts at 20° C.) is slightly lower than that of the potassium chloride cell, inconvenience is unlikely to arise from this difference in routine *p*H determinations. J. G.

Electrical Resistance of Wood. W. W. Barkas, R. F. S. Hearmon and G. H. Pratt. (*Nature*, 1943, **151**, 83.)—The electrical resistance of wood increases very rapidly with a decrease in its moisture content, so that if two probes are driven into a board about 2 cm apart, the resistance between them may be used to measure the moisture content. If beech wood containing 30–40% of water is heated at atm. pressure to just over 100° C. in a confined space which is suddenly evacuated, the resistance suddenly increases by *ca.* 200%. The fact that on re-admission of air or maintaining the vacuum by further pumping the resistance falls to near its original value in about 200 min. is taken as indicating that the path of the current in moist wood is principally along the interior surfaces of the cells. The temperature coefficient of the resistance of wood is negative, but too small to account in terms of local cooling for the above big resistance change; however, it indicates that the mechanism of the conduction is ionic, as in a soln. J. G.

Fluorescence of the Extractives of Wood. W. W. Martey. (*Paper Ind.*, 1942, **24**, 877–879.) The fluorescence of unextracted black spruce (*Picea mariana*) heartwood (measured photo-electrically) was similar to but slightly more intense than that of the sapwood; extraction of

either with ether or of the sapwood with a mixture of alcohol and benzene, markedly increased the intensity of the fluorescence in the violet region, but extraction of the heartwood with alcohol increased the intensity only slightly, and removed the intense violet shade. Extraction with 1% acetic acid, 5% hydrochloric acid or 5% sodium hydroxide soln. reduced the fluorescence intensity at 450–700$m\mu$. The violet fluorescence of the sapwood was intensified after extraction with water or hydrochloric acid, whereas with heartwood only the latter had this effect. The sapwood and heartwood of western hemlock (*Tsuga heterophylla*) were less fluorescent and the changes in fluorescence at 450–700$m\mu$ were less marked than for the spruce. The heartwood could be distinguished from the sapwood by the intensification of the violet fluorescence of the former after extraction with water or hydrochloric acid. Loblolly pine (*Pinus taeda*, L.) sapwood, on the other hand, had an intense violet fluorescence which was reduced only at 400$m\mu$ by extraction with ether or alcohol, whilst water or acids produced a high intensity at 400$m\mu$, and if acetic acid was used, a second max. at 600$m\mu$. A final extraction with sodium hydroxide soln. left the residue practically non-fluorescent. All 3 woods showed a marked decrease in fluorescence intensity on ageing. The location and nature of the fluorescent extractives were determined. It was found that, since the fluorescence is a sensitive indication of the presence of impurities, it may be used to control the course of purification of the fractions; a const. fluorescence in successive fractions is an indication that purification is complete. In separating the highly fluorescent resenes from the almost non-fluorescent phytosterols it was observed that a small quantity of the latter considerably reduced the fluorescence of the former, whilst large quantities were without effect. The water-sol. fluorescent extractives come from the cell-walls of all 3 woods; alcohol-sol. extractives from the resin ducts, from the tracheid walls, and from the resin ducts and wood rays; ether-sol. extractives from the wood rays, resin ducts and pits, from the wood rays, and from the resin ducts for black spruce sapwood, western hemlock, and loblolly pine, respectively.

J. G.

Separation of Isotopes [and Other Similar Substances by Ionic Migration] and Thermal Diffusion. J. Kendall. (*Nature*, 1942, **150**, 136–140.)—The method depends on the more rapid migration of lighter ions under the influence of an electric current, and it may be illustrated by a description of the technique employed in a simple instance, *e.g.*, for the isotopes of chlorine. An agar-agar gel containing sodium chloride serves as a short middle portion in a long horizontal Pyrex tube (internal diam., 1·5 in.), and it is flanked by sections containing a sodium hydroxide and a sodium acetate gel, respectively. The ends of these latter sections are connected with right-angled Pyrex bends of similar diam. and containing the same gel as the respective horizontal portions; after the gels have set conc. solns. of sodium hydroxide and of sodium acetate in acetic acid are poured into the respective vertical tubes. A platinum electrode is inserted in each of these solns., and a current is passed through (the sodium hydroxide being the anode); the tube is kept cool by immersion in running water. The boundaries between the various sections of the gel move towards the anode, and remain distinct because there is a faster-moving ion in front of the chloride ion and a slower ion behind it. The rate of travel depends on the potential drop between the electrodes, the length of the tube and the concns. of the solutions; *e.g.*, with several 3-ft. sections and 100–500 volts, it is 12–18 in. per day. When the front chloride boundary has almost reached the end of the tube the 2 rear sections are discarded, 2 new sections filled with sodium hydroxide gel are inserted in front of the chloride, and new bends are fitted on. The chloride ions are now forced to migrate into these new sections, and the procedure is repeated until they have traversed about 100 ft. of tube. The chloride gel is then removed from the tube, and cut up into strips of suitable width; the front portions contain the faster and the rear the slower ions. With mixtures of isotopic ions (*e.g.*, those of chloride) no significant separation was detectable, and it is therefore concluded that the mobilities of such ions are equal to within 1%. On the other hand, very successful separations were obtained with mixtures of the rare earth elements (at Nos. 39 and 57–71 inclusive). Thus, complete separation of yttrium and erbium was effected in 4 days (distance of travel, 2 m), with potassium as the preceding faster ion and chromic as the slower following ion. The visibility of the rear boundary was improved by the colour of the chromic salt, and it was possible also to follow the progress of the separation in the rare earth section by means of a small, direct-vision hand spectroscope; the yttrium accumulated in the front of the section and the erbium in the rear. The method is much more convenient and requires less time than the usual method of fractional crystallisation. The separation of neodymium and praseodymium required about 10 days, and there is evidence that the method may also be applicable to separations of radium and barium, and of the hydrochlorides of closely-related alkaloids. The separation of hafnium and zirconium was incomplete. The possibilities of separations based on thermal diffusion and ionic discharge potentials are also discussed, but are of less analytical interest.

J. G.

Physiological Theory of Colour Perception. R. Granit. (*Nature*, 1943, **151**, 11–14.)—It is now possible to record the discharge of the retinal elements directly by means of electrodes connected with more or less isolated fibres of the optic nerve. The electrical impulses following illumination, which are the physiological means of communication between the retina and the higher centres, are amplified and recorded photographically with the aid of an oscillograph. The use of micro-electrodes enables a rapid survey to be made of the response of single or grouped fibre units to light of varying spectral characteristics. In order to analyse colour sensitivity the amount of energy necessary to produce a threshold response is measured for lights of different wave-lengths, and the reciprocal of this energy is plotted (as a percentage of the max. for the spectral range investigated) against the wavelength. The curve obtained for the dark-adapted eye having its rods fully charged with visual purple is an exact reproduction of the absorption curve for visual purple (corrected for quantum intensity); max. at *ca.* 0·500μ. After the eye has been light-adapted, the max. shifts to *ca.* 0·560μ (Purkinje shift), and the new curve determines the distribution of brightness in a spectrum strong enough to produce colour sensations, the retina is then supposed to utilise the cones as receptor elements. Such curves are of 2 types;

according as the absorption bands are broad or narrow. The former are obtained with cat, frog and snake eyes (the last being a pure cone eye, so that light adaptation is not necessary to produce this type of curve), but not with guinea pig or rat eyes. It is therefore concluded that the factors responsible for the production of curves with broad bands are responsible for the sensation of brightness. The modulation of the dominant impression of brightness to colour is brought about by very narrow bands in the 3 preferential regions, 0·580–0·600, 0·520–0·540 and 0·450–0·470μ. In addition, the eyes of some animals (*e.g.*, rat and guinea-pig) have a narrow band at *ca.* 0·500μ (*cf. supra*). The nature of the modulators present varies according to the animal, but of all those studied, the frog has the most complete set. This theory of the production of colour sensations by the modulation of the dominant impression of brightness can be used to explain (*a*) differences in the degrees of brilliance of different colours, (*b*) the great general reduction in brightness and lack of colour selectivity following selective adaptation of the human eye to any colour, (*c*) the general reduction in the colour strength of a spectrum with a diminution in intensity, (*d*) the reduction in colour without loss of brightness distribution which results when the area of a visual object is reduced, and (*e*) certain forms of colour-blindness, particularly in animals (which arise from the absence of certain modulators). The relative merits of this and the trichromatic theories are discussed. There is reason to believe that visual purple (which is described as the "mother substance" of these phenomena) consists of a molecule having a protein nucleus which serves as a carrier for about 10 chromophoric groups; the different colour-sensitive substances may arise from changes in the linkage between carrier and chromophore. J. G.

Reviews

A Laboratory Handbook of Pulp and Paper Manufacture. By Julius Grant, Ph.D., M.Sc., F.I.C. Pp. viii + 320. London: Edward Arnold & Co. 1942. Price 28s.

This is definitely a good book. Stevens' standard work, "*The Paper Mill Chemist*," has been admirably absorbed, extended, and modernised with no loss, and indeed some gain, in prestige.

Dr. Grant has made something of a speciality of compiling readable summaries of the work of his contemporaries and giving them his own added lustre, and this volume is no exception. It covers a wide field thoroughly, if not quite exhaustively; the treatment is rational and the result satisfactory. Chemists, whether their concern be primarily with paper manufacture or only in testing paper for some of its manifold uses, will find the book of great service.

After an introductory chapter on the scope and function of a mill laboratory, there are two chapters on the control of processes and raw materials for the production of "half-stuff," one on their evaluation, and two on papermaking processes proper. The last three chapters are devoted to paper testing by physical, chemical, and microscopical means. There is finally a short section on water and fuel, and some useful tables form an appendix. Both author and subject indexes are included. Cross-referencing is copious, and the use made of TAPPI standard methods admirable.

Although the book professedly is not concerned with papermaking practice, sufficient attention has been paid to the need for discussing the function of the various control processes in terms of operational requirements, and this tends both to clarity and to easy reading. Its readability is indeed one of its outstanding features. In the few instances where mill processes are explained, however (*e.g.*, the multiple effect evaporator and certain types of beater) illustrations would have been very useful, especially where new apparatus was being discussed.

The paper testing and analytical sections are comprehensive, but it is permissible for a friendly reviewer to draw attention to a few omissions and errors of fact. (1) Logwood and Vandyke Brown (p. 137) are still used in appreciable quantities; the presence of logwood in black papers can be detected by the red colour obtained on warming with dilute HCl. (2) Differentiation between the machine and cross directions of a sheet of paper, Method (*d*) (p. 182), will not be clear if one does not understand what is meant by "the chief line of rupture"; a sketch would therefore have been helpful. (3) The explanation of the effect of handle speed on the Mullen burst (p. 193) is unsatisfactory; the compressibility of glycerin is only 22 $\times 10^{-6}$ per atmos. between 1 and 10 atmos. at 15° C. (4) The section on Colour (p. 202) might well contain a description of a few of the simple colour blindness tests, although none is completely satisfactory; in papermaking a colour-blind person may be given important colour matches to carry out! (5) The *p*H of untreated papers (p. 236)

may range from 3·7 to 9·0 under certain conditions. (6) The presence of copper (p. 239) is sometimes an indication of the use of copper sulphate to "fix" such dyes as Sky Blue. (7) A test for chromium should be included as a means of establishing the presence of chrome pigments in yellow and green papers, and the use of potassium dichromate in blacks. (8) In the identification of fibres by staining (p. 249), the Schulze method of differentiating between chemical and mechanical pulps is too valuable to omit, and the description of permanent microscopic slide mounts (p. 260) should include mention of such media as polystyrene resins. (9) The list of dirt specks (p. 262) contains no reference to lime spots.

There are, as is almost inevitable in a first edition of a book of this nature, a few misprints, but the only ones of any consequence noted by the reviewer are 1·5 mg. for 1·5 g (p. 92, line 8), hydrogen for hydrogen sulphide (p. 241, line 10), 25 for 2·5% (p. 274, line 26), 10% for 10 per sq. ft. (p. 274, line 27), and the formula of smalt (p. 141, line 17). It is unfortunate that it takes as much space to remark on a few small blemishes as can be spared for the rest of a review, and the reader should not allow his sense of balance to be impaired by this fact.

It only remains to add that the volume, although produced in complete conformity with the authorised economy standards of to-day, is excellently printed; and perhaps to ask with some diffidence why treaties (p. 277) should nowadays require to be prepared on paper of the highest permanence! Yes, this is quite definitely a good book.

H. AINSWORTH HARRISON

GAS WARFARE. W. K. FITCH. A "P.J." Monograph for Instructors. Pp. 103. London: The Pharmaceutical Press. 1942. Price 2s. 6d.

This booklet is concerned with gas warfare in a very general way, the main chapters dealing with the properties, detection and identification of the war gases, respirators, first aid and treatment of gas casualties, and decontamination. In such a small volume only the main principles of this complex subject can be given, and although the author has managed to condense a considerable amount of information in a very handy form, the subtitle "a monograph for instructors" appears to be rather ambitious. From the instructor's point of view the Ministry of Home Security and other official publications, of which this booklet is a compressed form, deal much more adequately with the subject and a more suitable title for the monograph would be "An Introduction to Gas Warfare." With a little more care in reading the reference books the author would have avoided several errors and inaccurate statements; for example, the boiling point of diphosgene is given as 106·7° C. on page 5, instead of 127–128° C., the wrong formula and molecular weight have been ascribed to bromomethylethylketone, and one stage in the preparation of ethyl dichloroarsine is given as "the reduction of the ethyl arsenious oxide by sulphur dioxide." A further source of irritation is the use of drachms, grains and minims, in the part dealing with gas simulants (*i.e.*, non-toxic materials with the odour and appearance of particular war gases and having, perhaps, some similar chemical properties), in what are certainly not pharmaceutical preparations. In spite of these failings from the chemist's angle, this little book should prove very useful to those who require a bird's-eye view of the subject at a very low price. The appendixes dealing with the decontamination of weapons, a Home Guard gas exercise and 23 questions stated to have been taken from platoon gas instructors' examination papers, should appeal to the more military-minded. W. J. STAINSBY

AN INTRODUCTION TO INDUSTRIAL MYCOLOGY. By GEORGE SMITH, M.Sc., A.I.C. 2nd Ed. Pp. xii + 260. London: Edward Arnold & Co., Ltd. 1942. Price 20s. net.

Most mycological problems that arise in industry have to be investigated along individual lines, each according to the characteristics of the fungus concerned and the material involved. It is probable that the author had this in mind when writing this textbook on industrial mycology, the first edition of which appeared in 1938, as the work is primarily concerned with methods of culture of fungi, their examination and classification rather than with methods of control. It may even be considered that too close adherence has been given to this principle, and that the section on the control of mould growth, one of the most fundamental problems of the industrial mycologist, deserves a greater allocation than a dozen pages out of 260.

After a general introduction on the fungi with their terminology and classification, the *Zygomycetes, Ascomycetes,* yeasts and related fungi, *Fungi Imperfecti, Hyphomycetales, Aspergilli, Penicillia* and related genera are considered in separate chapters. Many fungi of economic importance are described and illustrated in each section by superb photomicrographs, and references are also given to standard works where the different fungi can be studied in greater detail. A condensed, but eminently practical, section on laboratory equipment and technique together with one dealing with the maintenance of cultures follow, and the physiology of mould fungi is also considered. Many industrial uses of the fungi are indicated in the short closing chapter.

The new edition contains nearly 50 pages less than the original, but this is the result of more compact presentation, and the actual quantity of the text has been increased. Most of the original subject matter remains unchanged but, from the applied mycology angle, considerable improvement has been made in some sections, notably in that dealing with yeasts, which has now been extended to form a separate chapter. The chapter on laboratory equipment and technique has been improved by the addition of, for example, the use of Cellophane technique for the examination of moulds, and of greater details for the preparation of permanent slide cultures. A new section dealing with the methods used for growing moulds on comparatively large volumes of liquid media is not only of general interest but should be of very real value to those engaged in the isolation of the products of mould metabolism. The control of that menace to the mycologist—the mite—is now also given the attention that, unfortunately, it deserves.

It will be a relief to all those familiar with the first edition, and should be a cause of pride to the publishers, that in spite of war-time difficulties the high standard of presentation, especially of the 136 really beautiful photomicrographs, has been maintained.

M. OLLIVER

Papers for Publication in THE ANALYST

THE Editor welcomes Papers and Notes for insertion in THE ANALYST, whether from members of the Society or non-members. They are submitted to the Publication Committee, who decide on their suitability for insertion or otherwise.

Authors and prospective authors are reminded that, owing to the paper shortage, all contributions to the journal must be condensed as far as possible.

The Publication Committee have recently issued a circular containing Advice to Authors on the writing of Papers for THE ANALYST. This can be obtained on application to the Secretary, Society of Public Analysts and Other Analytical Chemists, 7–8, Idol Lane, London, E.C.3. All Papers submitted will be expected to conform to the recommendations there laid down and any that do not may be returned for amendment or rejected.

Volume 68 April, 1943
No. 805, Pages 99—132

Contents

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS

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