THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS

AN Ordinary Meeting of the Society was held at the Institute of Chemistry, on Friday, March 4th, at 3 p.m., the President, Dr. G. Roche Lynch, being in the chair.

Certificates were read in favour of Ruth Bennett, B.Sc.,* Gordon Jack Budds, John Gaius Ashwell Griffiths, B.A., Ph.D., A.I.C., William John Chetwynd Horne, John Edwin Ritchie, M.A., B.Sc., F.I.C.† Marshall Jeffreys Robb, B.Sc., F.I.C.,† George Hugh Turner, B.Sc., F.I.C.*

The following were elected Members of the Society:—Elmer Bruce Ashcraft, B.S., M.S., Ph.D., Alfred Ernest Cross, Frederick Raine Ennos, B.A., B.Sc., F.I.C., Reginald Lee Lord, M.Sc., Maison Gabriel de Navarre, Ph.C., B.S., Frank Arnold Robinson, M.Sc.Tech., A.I.C., William Henry Smith, Stanley Robert Thompson, Charles Edward Waterhouse, A.I.C.

The Annual General Meeting then followed. The Honorary Treasurer presented the accounts for the year, and the Honorary Secretary presented the Annual Report of the Council. The President delivered his Presidential Address.

The following were elected as Officers and Council for the year 1938:

President.—Professor W. H. Roberts, M.Sc., F.I.C.

Past-Presidents serving on the Council.—F. W. F. Arnaud, E. Richards Bolton, J. T. Dunn, Bernard Dyer, John Evans, Edward Hinks, G. Roche Lynch, G. Rudd Thompson.

Vice-Presidents.—B. S. Evans, J. R. Nicholls, W. H. Simmons, T. P. Hilditch (*Chairman, North of England Section; ex-officio*), J. F. Tocher (*Chairman, Scottish Section; ex-officio*).

Honorary Treasurer.—E. B. Hughes.

Honorary Secretary.—Lewis Eynon.

Other Members of Council.—E. B. Anderson, Miss E. A. M. Bradford, W. G. Carey, R. C. Chirnside, S. Dixon, F. W. Edwards, S. Elliott, G. van B. Gilmour, H. E. Monk, J. G. Sherratt, F. G. H. Tate, E. Voelcker, J. B. McKean (*Honorary Secretary, Scottish Section; ex-officio*), J. R. Stubbs (*Honorary Secretary, North of England Section; ex-officio*).

* Application through the North of England Section.
† Application through the Scottish Section.

Anniversary Dinner

ON Friday, March 4th, 1938, the Society held a dinner at the Royal Palace Hotel, Kensington, to commemorate the sixty-fourth year of its foundation.

The members and guests, who numbered 152, were received by the President, Dr. Roche Lynch, and Miss Roche Lynch, and Dr. Roche Lynch afterwards took the chair at the dinner.

The guests of the Society and of the President included the Rt. Hon. the Lord Cornwallis, M.C., and Lady Cornwallis, the Hon. Mr. Justice Singleton, Mr. Norman Birkett, K.C., and Mrs. Birkett, Air Vice-Marshal Sir Philip Game, G.C.V.O., G.B.E., K.C.B., K.C.M.G., D.S.O. (Commissioner of Police of the Metropolis), Sir E. Tindal Atkinson, K.C.B., C.B.E. (Director of Public Prosecutions), Sir Robert H. Pickard, D.Sc., F.R.S. (Vice-Chancellor, University of London; President, Institute of Chemistry), and Lady Pickard, Professor F. G. Donnan, C.B.E., M.A., LL.D., D.Sc., F.R.S. (President, Chemical Society), and Miss Donnan, Dr. Wm. Cullen (President, Institution of Chemical Engineers), and Mrs. Cullen, Sir William Willcox, K.C.I.E., C.B., C.M.G., M.D., F.R.C.P., F.I.C. (Medical Adviser to the Home Office), and Lady Willcox, Mr. R. B. Pilcher, O.B.E. (Registrar, Institute of Chemistry), Mr. H. N. Linstead (Secretary, Pharmaceutical Society), and Mrs. Linstead, Dr. R. H. Slater, D.Sc., F.I.C., Mr. F. A. Greene, M.I.Chem.E., M.I.E.E., and Mrs. Greene, and Mr. and Mrs. R. L. Wilkinson.

After the loyal toasts had been proposed by the President and honoured, Mr. E. R. BOLTON proposed the health of His Majesty's Judges, coupled with the name of the Honourable Mr. Justice Singleton. He said that most analysts sooner or later found themselves in the position of expert witnesses, and that never in his long experience of the Courts had he known anything but the greatest courtesy to be accorded to members of the analytical profession. He had always been greatly impressed by the ability of judges and counsel to express in a few simple English words the scientific jargon of a chemist in the witness-box. He spoke of a learned K.C. who pretended to be unable to grasp the rudiments of a simple chemical problem, and yet the next day his cross-examination of Otto Hehner was like the clash of arms between two great chemists.

Mr. Bolton deplored the fact that not one of our Universities had instituted a Chair of Chemical Jurisprudence for the instruction of young chemists in the art of giving evidence and of writing an understandable report on a chemical subject; if it were possible, he added, for such a Chair to be occupied by Mr. Justice Singleton he would sit gladly at his feet. We in England were proud of His Majesty's Judges, who were the envy of every country in the world and no respecters of persons and whose motto was "Fair Play." Mr. Justice Singleton was not only a great Judge, but was also patron of the Manchester and District Medico-Legal Society.

THE HONOURABLE MR. JUSTICE SINGLETON, in responding to the toast, thanked Mr. Bolton for his kind reference to the Judges, and for his tribute to them and the administration of justice. The administration of justice, however, depended not so much upon the Judges and those who practised before them, as upon the courage and extraordinary fairness of juries. The trouble taken by jurors in serious cases to get the right result spoke well for the future of the country. It was the system of administration of justice in this country that tended to good government and settlement. We were able to go about our business in a normal way because of our confidence in the police, in jurors and in the administrative system.

He reminded Mr. Bolton that in Ireland, as in this country, there were Judges of all kinds and of all ages, and that when trouble occurred in some other countries a Judge was sent out from home to put things right. Referring to an incident

in Sierra Leone, where a native appreciated the way in which British justice had been administered, Mr. Justice Singleton said that he hoped that everyone in every class of life would realise that when he had been tried by a jury he had received fair play.

Mr. NORMAN BIRKETT, K.C., proposing the toast of the Society, said that his first duty was to acknowledge the sense of honour he experienced at being invited to the dinner and to propose this toast. He would like to add his word to what Mr. Justice Singleton had said about the administration of justice in this country. Notwithstanding the vagaries of juries, it was a very great satisfaction to feel that this country's reputation for rightness and fairness had the admiration of the civilised world.

His sense of honour in presenting this toast was only equalled by his sense of pleasure in coupling the name of Dr. Roche Lynch with that of the Society. There was one claim that all must accord to their President—his consummate fairness in presenting all matters to the Court. Very frequently it happened that there was nobody in Court who could contradict him, but the reliance that everyone felt could be placed in his fairness made one forgive him all the difficulties that he placed in the way of defending counsel.

Before the dinner he (Mr. Birkett) had received a copy of a book which gave reminiscences of fifty years of the Society. He was horrified to find described on the opening pages the most extraordinary adulteration of food. There was no single article of food that was not a source of most terrible danger! Continuing his researches into this book he thought that it would be wisest to refrain from commenting about anything in particular and merely to comment on the Society. The public advantages of a Society of this kind were tremendous. For instance, if one looked at the learning and scientific research involved in the day-to-day work of its members, it was an admirable thing that there should be research work of this nature, as it was of very great importance in every great field of activity. It was, to the ordinary man, a matter of profound satisfaction to think that in every field of life, so far as these matters were concerned, the learning and research of this Society were devoted to the public service and the public well-being. Millions of poor people must have benefited by the self-sacrificing work of members of a Society such as this. It was perhaps well that someone from outside should pay his tribute, not only to their work but to the great public advantages they conferred.

The PRESIDENT, responding, said that he would first like to thank Mr. Norman Birkett for his very kind words, not only about himself, but about the Society as a whole. He appreciated, too, the kindly remarks that Mr. Birkett had made on the past of the Society, culled from *Fifty Years of the Society of Public Analysts*.* He was very right in saying that the Public Analyst in those difficult days of gross food adulteration did yeoman service and did save poor people from receiving foul concoctions which were sold to them designated by the word "food." The Society they were honouring that evening had been in existence for sixty-four years, and it was true to claim it as the second oldest chemical society in London. It was founded originally by Public Analysts in order that they might meet together and discuss problems and so assist in the administration of the first Food and Drug Acts, and generally to contribute to the well-being of the great profession of chemistry. It was also true to say that analytical chemistry in general, and particularly food chemistry, owed a very great deal to the activities of those early chemists. As time went on, the study of analytical chemistry became more general, and the Public Analyst's work became very complicated. And so, in time, "Other Analytical Chemists" joined the ranks of the Society. To-day, of course, the Society's activities and interests were concerned with all forms of analytical chemistry.

He wished to make a personal reference to Sir William Willcox, whom he had

* By Dr. Bernard Dyer and Dr. Ainsworth Mitchell.

known for over thirty years, and to whom he owed his first introduction to medico-legal work. He was anxious to pay a personal tribute to him for his valuable help, advice and friendship, and a general tribute to a distinguished scientist.

The Society was fortunate in securing Professor Roberts to be its next President.

It had been a great pleasure to him (Dr. Roche Lynch) to have been able to serve the Society first as Secretary, and then as President, and it was a matter of much regret that that night must see the severing of his connection as an active officer, but it would be his constant endeavour to do his utmost for the Society as a Past-President. On this 64th Anniversary of its foundation, it was a great pleasure to have with them one of the original members—Dr. Bernard Dyer. He believed that at that time Dr. Dyer was too young to be a full member and had to be an Associate Member; as they could all see, he still preserved his youth.

(The company here drank the health of Dr. and Mrs. Dyer, and Dr. Dyer responded.)

Mr. A. L. BACHARACH, proposing the toast of "The Society's and the President's Guests," referred to the several distinguished members present from the two great professions, Law and Medicine. Scientific Societies were also represented by the Presidents of the Chemical Society, the Institute of Chemistry and the Institute of Chemical Engineers, and by the Secretary of the Pharmaceutical Society. Representatives of the Press, lay and medical, were also welcomed. The toast was coupled with the name of Lord Cornwallis, Chairman of the Agricultural Advisory Committee under the Fertilisers and Feeding Stuffs Act, and also representing the Houses of Parliament, as a member of the House of Lords; Sir Philip Game, the Commissioner of the Metropolitan Police; and Sir Robert Pickard, President of the Institute of Chemistry, Chairman of the Chemical Council, Vice-Chancellor of London University and Director of the Cotton Research Association.

THE RIGHT HONOURABLE THE LORD CORNWALLIS said that one possible reason why he had been given the privilege of responding first for the guests was that he had served for some time as Vice-Chairman and for a short time as Chairman of a County Council, and so could realise to the full the great service and great value of our County Analysts. He had a very sincere and personal feeling for the particular County Analyst of his own county, who was one of their Past-Presidents. He had had a perfect evening in the most charming company, and he hoped that in any activities he had in the future he would be able to work in the closest possible co-operation with the Society.

AIR VICE-MARSHAL Sir PHILIP GAME said that he regarded it as a very great honour to be allowed to take a minor part in expressing to the Society the gratitude of the guests for a very delightful evening. He would like to acknowledge the great help that the analytical chemist, for many years past and to an increasing extent every year, had been in assisting the police in what was a very difficult job. Members of the analytical profession had, indeed, already been "roped in" for full-time work in the police laboratories, and, like Oliver Twist, the police would soon be asking for more.

Sir ROBERT PICKARD said that he noted on the back of the menu a list of distinguished Past-Presidents and, as President of the Institute of Chemistry for the third time of asking, he could not help feeling a certain amount of pride in calling attention to the fact that all these Presidents had been members of the Institute. During the past year this Society had elected him an Honorary Member, and as the representative of the Institute of Chemistry he wished to thank them personally for the great honour they had done him. He could not forbear to mention that all the Public Analysts in Great Britain, Northern Ireland, and, he thought, also Eire, with, he believed, two exceptions, were members of the Institute of Chemistry. The Institute of Chemistry was proud of that fact. They had been told that Judges were badly paid; all agreed that Public Analysts were

badly paid. He was not quite sure that the calling of the Public Analyst was regarded quite as highly as it should be.

He wished to conclude by thanking the Society for the very pleasant evening given to the guests, and, as last speaker, asked to be allowed to offer the congratulations of all present to Professor Roberts on his accession to the chair and to wish him a very successful period of office.

Annual Report of Council

MARCH, 1938

THE Roll of the Society stands at 831, an increase of 43 over the membership of last year and of 250 in the past ten years.

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

W. J. Lesley
A. R. Ling
J. J. Morgan
F. R. O'Shaughnessy
J. H. Totton
J. A. Voelcker

Lesley, who died at the early age of 41, was elected a member of the Society in 1930. Apart from war service in the R.A.M.C., the whole of his professional life was spent in the sugar refining industry with Messrs. Tate & Lyle, Ltd. He obtained the London Ph.D. degree in 1930.

Ling, who died in his 76th year, had been a member of the Society for over 40 years, and had served on the Council as an Ordinary Member and as a Vice-President. He was engaged in practice in London as consulting chemist and analyst for many years and during the last 11 years of his professional life he was Professor of Biochemistry of Fermentation at Birmingham University (Obituary, ANALYST, 1937, **62**, 585).

Morgan, who died at the age of 74, had been engaged in the metallurgical industries during practically the whole of his career, in the latter part of which he was engaged in private practice as a metallurgist and analytical chemist. He was elected a member of the Society in 1910.

O'Shaughnessy, who died in his 64th year, had been a member of the Society since 1898, and served on the Council in 1910–11. After a short period as assistant to the late H. Droop Richmond, he was appointed, in 1899, chemist to the Birmingham, Tame and Rea District Drainage Board, and held that appointment until his death.

Totton, who died at the age of 57, was elected a member of the Society in 1930. He worked for two years with the late C. E. Cassal and became Public Analyst and Official Agricultural Analyst for the County of Armagh in 1907, and subsequently for the City of Belfast and for the Counties of Londonderry and Antrim (Obituary, ANALYST, 1937, **62**, 773).

Voelcker, who died in his 84th year, held a long and distinguished record of service to the Society. Elected a member of the Society in 1886, he subsequently served on the Council as Ordinary Member, Vice-President, President and Past President for many years, filling the office of President in 1901–2. His loss is keenly felt, both by his colleagues on the Council and by the numerous members of the Society to whom he was so well known (Obituary, ANALYST, 1938, **63**, 79).

During the year seven ordinary meetings of the Society have been held and the following papers have been communicated:

"Quantitative Microscopical Analysis of Feeding-stuffs: I, Determination of Rye, Wheat and Barley Starches in Mixtures; 'Ground Oats' Mixtures." By J. G. A. Griffiths, B.A., Ph.D., A.I.C.

"The Hartridge Reversion Spectroscope: Improvements in Design, Assembly and Technique." By R. C. Frederick, F.I.C.

"The Properties of Calciferol." By F. W. Anderson, B.Sc., A.I.C., A. L. Bacharach, M.A., F.I.C., and E. Lester Smith, D.Sc., F.I.C.

"The Determination of Bismuth as Phosphate." By W. R. Schoeller, Ph.D., F.I.C., and D. A. Lambie, B.Sc., A.I.C.

"The Distillation of Strong Acids." By H. I. Coombs, Ph.D., M.D.

"Resins and Pitches from Ancient Egyptian Tombs." By J. G. A. Griffiths, B.A., Ph.D., A.I.C.

"A New Apparatus for the more Rapid and Economical Determination of the Freezing Point of Milk." By P. L. Temple.

"Fluorine in Food Products." By H. C. Lockwood, B.Sc., F.I.C.

"Analysis of Coffee Infusions." By F. W. Edwards, F.I.C., and H. R. Nanji, Ph.D., D.I.C., F.I.C.

"The Determination of Tannins in Cacao Kernel." By D. W. Duthie, M.A., Ph.D., A.I.C.

"The Ascorbic Acid Content of Fruit and Vegetables." By Mamie Olliver, M.Sc., A.I.C.

"World's Dairy Congress." Report by W. L. Davies, Ph.D., M.Sc., F.I.C.

"A Method for the Routine Determination of Glycogen in Oysters." By J. P. Tully.

"The Detection and Determination of *p*-Hydroxybenzoic Acid in the Presence of Salicylic Acid." By S. G. Stevenson, M.Sc., B.Pharm., F.I.C., and J. Resuggan.

"The Contamination of Whale Oil with Fuel Oil." Parts I and II. By E. R. Bolton, M.I.Chem.E., F.I.C., and K. A. Williams, B.Sc., F.I.C.

"The Analysis of Glucose-Fructose Mixtures, with Special Reference to Honey." By C. R. Marshall, Ph.D., A.I.C., and A. G. Norman, M.Sc., D.Sc., Ph.D., F.I.C.

"The Influence of Feeding Stuffs on Meat." By V. C. Fishwick.

"Fat Absorption and Metabolism." By A. C. Frazer, M.B., B.S., M.R.C.S., L.R.C.P.

"The Concentration of Fruit Juices by Freezing." By P. L. Bilham, B.Sc., F.I.C.

The February meeting was a joint meeting with the Food Group of the Society of Chemical Industry.

THE ANALYST.—Once more THE ANALYST has created a record for size with 914 pages as compared with 882 in 1936. During the year 50 papers and 29 notes were published, and the increasing use that is being made by inorganic chemists of this journal as the medium for the publication of analytical papers is shown by the welcome fact that no fewer than eighteen of the papers and six of the notes related to inorganic subjects. Food and drugs now take the second place, accounting for sixteen of the papers and seven of the notes. One of the outstanding contributions was the printed version of Professor Barger's lecture on "The Alkaloids of Ergot." The remaining papers included four on physical methods and apparatus, seven on organic analyses, two on water analysis, two on biochemical subjects and one on toxicological analysis.

The abstracts, which numbered 534 in all, covered every branch of analytical chemistry and relative subjects. It will have been noticed that increased space

has been given to bacteriological abstracts relating to food, air and water, but not including the more strictly medical side of bacteriology.

As usual, notes from the Reports of Government Analysts in the Dominions and Colonies have been published, and the Editor continues to receive letters from chemists abroad expressing their appreciation of the interest shown by THE ANALYST in their scientific work.

TREASURER'S REPORT.—The Hon. Treasurer reports that the Society's accounts show that a satisfactory balance of income over expenditure is being maintained.

HONORARY MEMBERSHIP OF THE SOCIETY.—The Council is happy to record that Miss M. B. Elliott, M.B.E., has been elected an Honorary Member in recognition of her valuable assistance, during many years, in the secretarial work of the Society. A presentation of a wrist watch, set in diamonds, to Miss Elliott was made by the President on behalf of the past and present Members of the Council at the termination of the Council meeting on December 1st, 1937.

PRESIDENTIAL BADGE.—A badge in enamel and gold, bearing the effigy of Theophilus Redwood, the first President of the Society, has been obtained for the use of the President, on official occasions. The reverse side of the badge will be engraved with the following inscription:

"The cost of this Badge was met from a Bequest to the Society by Dr. J. A. Voelcker, January, 1938."

The design of the badge was exhibited at the Ordinary Meeting on November 3rd, 1937.*

THE KING'S BIRTHDAY HONOURS LIST.—The Council has congratulated Sir Robert Pickard, Honorary Member, on the Honour of Knighthood, Colonel Clive Newcomb on the honour of Commander of the Indian Empire, and Mr. Andrew More, Member of Council, on the award of the Imperial Service Order.

SECRETARYSHIP OF THE SOCIETY.—Dr. C. A. Mitchell, Secretary and Editor, tendered his resignation of the Secretaryship, to take effect on October 31st, 1937. The Council accepted Dr. Mitchell's resignation with great regret, and expressed high appreciation of his services during his tenure of office. Mr. J. H. Lane, Assistant Editor of THE ANALYST, has been appointed Secretary in succession to Dr. Mitchell, and the Registered Address of the Society is now 7–8, Idol Lane, London, E.C.3.

FOOD AND DRUGS BILL.—The Minister of Health, having invited the Society to make observations on a Draft of a Food and Drugs Bill shortly to be presented to Parliament, the Draft of the Bill was considered, in the first instance by the Public Analysts and Official Agricultural Analysts Committee, and then by a special meeting of the Public Analyst members of the Society. A Report on the Draft Bill as finally approved by the Council was sent to the Minister.

ANALYTICAL METHODS COMMITTEE.—Four reports from the Committee have been published during the past year, *viz.*:

"Determination of Dirt in Milk," ANALYST, 1937, **62**, 287.
"Determination of Esters in Essential Oils," ANALYST, 1937, **62**, 541.
"Determination of Free Alkali and Silica in Silicated Soaps," ANALYST, 1937, **62**, 865.
"Determination of Rosin in Soaps," ANALYST, 1937, **62**, 868.

In addition, notes dealing with "Allowable Limit of Dirt in Milk" (ANALYST, 1937, **62**, 301) and "Determination of Unsaponifiable Matter in Oils and Fats" (ANALYST, 1937, **62**, 863) have been published with the authority of the Council.

The Dirt in Milk Sub-Committee, having completed its work, has now been dissolved, and no new Sub-Committees have been appointed.

* A reproduction, from a photograph by Mr. E. R. Bolton, of the Badge is shown in the plate in the current issue of THE ANALYST.—EDITOR.

The Committee has accepted with much regret the resignation of Dr. Callan as Chairman of the Metallic Impurities in Food Colours Sub-Committee. Mr. T. Macara has been appointed to succeed him.

NORTH OF ENGLAND SECTION.—Five meetings have been held during the year. Twelve papers have been read and one discussion held. The attendances have been fully up to the level of any previous year. The President, Honorary Treasurer and Honorary Secretary of the Parent Society have attended some of the meetings.

In June the Summer Meeting was held at Scarborough; the attendance was equal to that of any previous year and the meeting was well supported by officers and members of the Parent Society.

Eleven candidates applied for membership of the Parent Society through the Section.

Ninety-seven subscriptions have been received, an increase of 8 on last year, previously the highest.

The Honorary Secretary wishes to express his thanks to the Chairman (Mr. A. R. Tankard) for his help and advice through another successful Session, and to the Committee for its loyal support, which has made the working of the Section a pleasure to all concerned.

List of papers and discussions held at meetings of the North of England Section during the Session 1937–38:

"Municipal Chemistry." Chairman's Address, by Arnold R. Tankard, F.I.C.
"The Acidity of Paper." By D. Burton, M.B.E., D.Sc., F.I.C.
"Flour Spoilage." By D. W. Kent-Jones, Ph.D., B.Sc., F.I.C.
"The Functions and Usefulness of Analytical Chemists." By J. J. Fox, D.Sc., F.I.C.
"The Evolution of the Public Analyst." By E. Gabriel Jones, M.Sc., F.I.C.
"The Practical Treatment of Dairy Effluents." By C. A. Scarlett, B.Sc., A.K.C., F.I.C.
"Non-alcoholic Wines." By C. J. H. Stock, B.Sc., F.I.C.
"A Note on Solution of Hydrogen Peroxide." By C. J. H. Stock, B.Sc., F.I.C.
"The Detection of Glucose Syrup in Jams and Honey." By G. D. Elsdon, D.Sc., F.I.C.
"Ginger with Low Water-soluble Ash." By G. D. Elsdon, D.Sc., F.I.C.
"The Standards of the British Pharmacopoeia, and the Relative Importance to be attached to Different Requirements." By A. D. Powell, F.I.C.
"Sidelights on Analytical Practice in the Near East." By E. S. Hawkins, B.Sc., F.I.C.
A discussion was introduced by Arnold R. Tankard, F.I.C., on "Misdescription of Food and Drugs."

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

"Boron; its Occurrence and Estimation in Plants." By Professor D. N. McArthur and K. L. Robinson, B.Sc.
"Trichlorethylene Vapour in Air: A Method of Determination." By Andrew Dargie, B.Sc., A.I.C.
"The Use of Selenium in the Determination of Nitrogen in Potato Tubers." By A. M. Smith, Ph.D., D.Sc., and W. Y. Paterson.
"Some Reactions of Sodium Metasilicate." By R. T. Thomson, F.I.C.
"Note on the Composition of Boiler Water." By J. B. McKean, F.I.C.
"A Method for the Estimation of Bismuth in Biological Materials." By S. L. Tompsett, Ph.D., B.Sc., F.I.C.
"Variations in the Composition of Blackcurrants." By John F. Brown, B.Sc., A.I.C.

Thirty-four subscriptions were received, an increase of three over the previous year. Seven members joined the Parent Society through the Section and one member ceased to be a member of the Section on taking up residence in England.

The Council of the Parent Society have altered the Rules of Local Sections in respect that the Annual General Meeting of Local Sections must now be held in January of each year, instead of February.

CONGRESSES.

International Dairy Congress, Berlin, 1937.—The Society was represented by Dr. W. L. Davies, whose report on the Congress has been published in THE ANALYST.

Société de Chimie Industrielle.—Dr. L. H. Lampitt represented the Society and presented a Congratulatory Address on the occasion of the Commemoration of the Twentieth Anniversary of the foundation of the Société de Chimie Industrielle. A bronze plaque, struck in commemoration of the event, was subsequently received by the President on behalf of the Society from the Société de Chimie Industrielle and exhibited at the Ordinary Meeting on November 3rd, 1937.

In conclusion, the Council desires to express its thanks to the members who served on the Committees and Sub-Committees and to those who represented the Society on other bodies.

(*Signed*) G. ROCHE LYNCH, *President*
LEWIS EYNON, *Hon. Secretary*

Address of the Retiring President

(G. ROCHE LYNCH, O.B.E., M.B., B.S., D.P.H., F.I.C.)

LADIES AND GENTLEMEN,

It is my first and very pleasant duty to record that this year, at the end of my term of office, the affairs of the Society are in a flourishing condition, and the Roll of the Society stands at over 830, for the first time in its history. As my predecessor has recorded, the Council have decreed that its President shall deliver only one address, which shall be given at the end of his term of office. Such a proceeding enabled the Council to invite Professor Barger to address us in March, 1937, and as his address has been printed, I make no comment, save that the principle of inviting distinguished visitors to address us on subjects which are of interest to analysts should prove of the greatest value to the members of the Society. Professor Barger set a standard which, if maintained in the future, will make these lectures an important feature of our Society. In modern times, analytical chemistry and the sciences allied to it have become so vast that I often find my knowledge of those subjects with which I have no immediate connection soon grows rusty, and I do feel that lectures which from time to time survey any branch of our interests, lectures which, as it were, report progress, are of immense service.

In considering the affairs of the Society, I cannot but express admiration of all the officers and committees who toil so generously in its interests; this voluntary service, coupled with a careful handling of the finances, has enabled the Society to continue its useful career without any rise in subscription, which, at its

present rate, is a remarkable achievement. In fact, as you know, a few years ago it was actually able to increase its benefits to members of over 40 years' standing.

The present position of THE ANALYST and its finances is highly gratifying, and reflects the greatest credit on Dr. Mitchell, under whose editorship the reputation of the journal has grown to such an extent that the sales to non-members now greatly exceed in number the membership of the Society itself. The figures are a testimony to Dr. Mitchell's brilliant achievement, and at the same time a warning that the very high standard of quality that has been set in the past must be maintained if we are to continue our success.

Our two healthy children, the North of England and Scottish Sections, still progress, as will be seen from their Reports, and we welcome Professor Hilditch and Dr. Tocher as Chairmen of the Sections, whilst Mr. Stubbs and Mr. McKean still give valuable service in a secretarial capacity.

During the last two years it is sad to have to report thirteen deaths, and among these, with the deepest regret, that two Past-Presidents are no longer with us—Ellis Richards and Voelcker. The former had not been amongst us for some time, owing to ill-health, but the latter was an active member until within a few weeks of his death. You will all have read obituary notices in THE ANALYST, and I will only say that their deaths have deprived the Society of wisdom which will be greatly missed. Dr. Voelcker's affection for our Society is shown in his legacy to us of £100, and the Council, with the cordial consent of his widow, has decided to spend part of this sum upon the Presidential badge which I am now wearing. It is to be suitably inscribed to this effect, so that his name will be remembered by future Presidents.

During the past year changes have taken place in our secretariat. Miss Elliott has reluctantly been compelled to give up her work for the Society, and Dr. Mitchell, too, has taken the opportunity to limit his work to the Editorship of THE ANALYST. We have been very fortunate in securing the services of Mr. Lane, who, in addition to his assistant editorship, has now undertaken the general work of Secretary.

The present and past members of the Council of the Society felt that they could not let Miss Elliott's resignation pass without expressing their appreciation of her services, and they accordingly presented her with a wrist watch as a token of their esteem. This presentation, at her request, was made privately.

For the first time a book has been published under the auspices of the Society —Dr. Schoeller's work on tantalum and niobium—and it is to be hoped that this will be the forerunner of a series. His researches in this field, over a period of 17 years, have now been collected together, and we are proud to think that this important contribution to analytical chemistry has throughout been associated with us.

I could allude to many other matters connected with the activities of the Society during my term of office, but time forbids, limiting me to but one or two.

I would mention the proposed new Food and Drugs Bill which the Minister of Health is to introduce into Parliament. Although this is a subject on which I am no authority, it would appear to me from conversations I have had with Public Analysts and with chemists in industry, that, whilst it contains some matters of controversy, it is a valuable piece of legislation and one which will

commend itself to all those who desire to see that only pure and wholesome foods reach the public.

There is one other matter I am anxious to submit to the younger members of the Society. We welcome papers both for THE ANALYST and for reading before the Society prior to their publication, and we encourage papers from the younger men, particularly those who are making their first effort. May I, to the younger men, offer these words of advice? First, the writing of a paper, I would remind you, is not the easiest job in the world. When you are familiar with a particular subject, you are apt to leave out details you assume are known by all; therefore, give too many details rather than too few. Choice of words, setting out of your material and style are all matters of great moment. But, above all, show the paper to some experienced person who will be able to guide your hand and advise you, so that your paper will at once appeal.

Secondly, leap at the chance of reading the paper in person. A well-read paper will often impress in such a way that you are at once accepted as having a competent knowledge of your subject, and may help you in your career. A badly-read paper may do you much harm. I appreciate that the man who is accustomed to lecturing has a great advantage, but the less practised may also make his mark if he will but take advice. I cannot here give all the advice which is necessary, but if the author works in a laboratory with a senior colleague he should ask to be allowed to read the paper to him, just as he proposes to do it on the evening, and hearken to his comments. If he is working independently, he should find a senior member to help him, or, if he does not know any, approach one of the officers of the Society for assistance. I am sure that all of us will help whenever we can.

I have used the words "reading the paper," and here is my last piece of advice. Never read from a manuscript, learn it by heart or get your subject matter into your mind, so that it becomes a speech. It is far better for portions to be accidentally omitted than to keep your eyes glued on the manuscript all the time. It is also no good giving every detail of your process; few of your listeners can follow all the details. Give the outline and show how your process is an improvement on the previous work. Lastly, never read tables; give them in summary form or on the blackboard, or on lantern slides.

During the last few months I have been musing over a choice of subject that I should take for my address to you to-day, and have been flirting with many ideas. At one time I re-read those presidential addresses which I have heard delivered during the period of my membership, in the hope that I might gain inspiration. When I turned to the old stalwarts who gave two addresses, I found the first that I heard were those of Bolton, and my memory of his addresses was his concern for the ablutions of the chemist. Then followed Hinks, who, whilst taking his duties very seriously, at the same time gave us little sparkling touches of humour which helped us to assimilate and appreciate the value of his contributions. Dunn was the next, and my memory of his was the delivery, aided by his charming, and one would almost say musical, voice, so that one fancied one could hear the harpsichord playing old English melodies. His addresses, too, were monumental in their value and usefulness, and they were none the worse

for the fact that they were written out in his fair handwriting on the backs of old galley proofs of THE ANALYST. The next President, Arnaud, went away into the fields and gave us a dissertation on the work of the Agricultural Chemist of to-day, from which I may say I learnt more on this subject in one short hour than I can ever remember learning on similar occasions. The last in my list is one from Wales, whose name you will remember is Evans, and with that gift, so often seen in his countrymen, of poetical interpretation mixed with a belief in fairies, an awe of the supernatural and a profound respect for Mother Earth and all that comes therefrom, he was able to show that the Cinderella of Chemistry—Analytical Chemistry—, was rapidly producing magical coaches from pumpkins.

Ladies and Gentlemen, I am sure you will appreciate that I turned away from these addresses with a feeling that, after the glamorous nights of vintage orations of which you had all partaken so freely in the past, my non-vintage effort would leave you nothing but a terrible headache in the morning.

My choice has finally fallen upon the toxicology of the narcotic drugs. I cannot pretend that, in the short time at my disposal, my survey will be profound or complete, but it will be in essence an account of a number of these drugs, based upon my personal experience.

THE TOXICOLOGY OF THE NARCOTIC DRUGS

THE BARBITURATES.—These drugs are cyclic ureides, prepared by combination of malonic acid with urea, the hydrogen atoms of the CH_2 group of the former substance being replaced by a variety of alkyl, and phenyl groups. Further, a hydrogen atom of an alkyl group may be replaced by a bromine atom, as occurs in Noctal, and an alkyl group may replace the hydrogen atom of one of the CONH-groups. It will thus be seen that the number of possible ureides that may be prepared is legion; in actual fact, there are only about seventeen of these substances on the market.

Recently, a new series, namely, the thiobarbiturates, in which the oxygen of the terminal CO-group is replaced by sulphur, has been placed on the market, but only one or two members are in use. One of these is called Pentothal. I have had an opportunity of examining one or two cases when a full medicinal dose of Pentothal has been given, and find that the drug is excreted in the urine, and can be extracted therefrom by the methods in use for the ordinary barbiturates. I have not had much experience of the thiobarbiturates yet, but it is probable that sufficient can be recovered to determine the melting-points and mixed melting-points.

All the barbiturates are definitely hypnotic and fall into two main groups: (1) where the narcosis is relatively light but is long-lasting, and (2) where the narcosis is deep with great relaxation, but the duration is relatively short. An example of the former is Veronal (diethyl barbituric acid), and of the latter, Evipan (*n*-methyl-*c*-*c*-cyclohexenyl-methyl barbituric acid).

From these properties it will be realised that members of the former group are used for insomnia, and members of the latter group for anaesthetic purposes. The barbiturates cannot be called drugs of addiction, although from time to time their

continued use will bring about dependence on them, inability to do without them, and in a few cases a true addiction, which may result in mental degeneration, etc.

The barbiturates are not cumulative in their effect, but when they are in regular use every day it may happen that complete excretion of a previous dose has not been effected by the time the next dose is taken. In such a case abstinence for a few days from the drug will result in complete elimination.

My observations on poisoning by barbituric drugs are based on the study of considerably over 100 cases. Unfortunately, the records of many cases are not available; consequently, I propose to discuss only those 100 cases of which there is some record. Of these, a few were investigated by my late colleague, John Webster, and with some of the others I have been helped by assistants, in particular by my present assistant, Dr. Slater, who has been invaluable to me.

In this series of 100 cases, all have been uncomplicated cases of barbituric acid poisoning, except a few in which drugs such as amidopyrine have also been taken, as, for example, with Allonal, a proprietary remedy, consisting of a barbituric acid mixed with amidopyrine. I have had cases of mixtures of, for example, morphine or cocaine with a barbituric acid, but they are not included in this series. Cases also have passed through my hands in which mixtures of these drugs have been taken, and in one case no less than four were taken, namely, Allonal, Veronal, Quadronox and Ipral.

Investigations of cases of poisoning in which this class of drug is suspected fall into two categories: (1) those in which the patient is found comatose and a rapid diagnosis is required by the physician for treatment purposes, and (2) those in which proof of the nature of the drug taken is required to be given as evidence.

(1) In the first group the analyst has to sacrifice everything to speed, even at the risk of a possible mistake being made occasionally, although so far our analyses have been correct. Life and death are at issue, and unless the proper treatment is applied at once, the consequences may be fatal. A competent physician should, in the majority of cases, be able to diagnose barbituric acid poisoning, but he is always glad of laboratory confirmation. The analyst should be able to give this in about 15 minutes. A direct extraction of the acidified urine with ether will remove the barbituric acid present, and the residue after evaporation should have three properties:

(*a*) It should be crystalline. This is not always the case, as certain barbiturates, *e.g.* Soneryl, Hebaral, and sometimes Luminal and Dial, etc., will not always crystallise readily from the crude ethereal extract. Veronal will always crystallise.

(*b*) A positive reaction should be obtained with Millon's reagent.

(*c*) A positive reaction should be obtained with the cobalt test.

I believe that if (*b*) and (*c*) are positive, and, better still, if all three are positive, I am justified in telling the clinician that he is dealing with a case of barbituric acid poisoning. One of the recognised parts of the treatment of barbituric acid poisoning is to perform a lumbar puncture, and if the fluid from this is available, its ethereal extract will contain the barbiturate. When these tests are applied to cerebro-spinal fluid and positive results are obtained, the results are, in my opinion,

much more conclusive. Although the quantity of the cerebro-spinal fluid generally available is such that it contains only a fraction of a milligram of the barbiturate, this quantity is ample for tests (*b*) and (*c*), and the microscope will reveal (*a*).

(2) Proof of the presence of a barbiturate for the purposes of evidence is, however, on a different footing. Here the problem resolves itself into the purification of the barbiturate sufficiently to be able to rely on the weight found and on the melting-point and mixed melting-point of the particular barbiturate present. Although with Veronal and usually with Luminal, this should be easy, many of the other members present difficulties because they are not readily obtained in crystalline form, and for other reasons with which I shall deal later. Generally speaking, the analyst will find that the stomach contents and stomach washings are likely to yield the barbiturate in the purest form for this purpose, the viscera next, and the urine last. It is very rare that sufficient barbiturate is obtained from the cerebro-spinal fluid for a melting-point determination. In connection with the melting-point it must be realised that one never gets the correct melting-point, but always a value several degrees lower; if a value roughly intermediate between this and the correct melting-point is obtained with the mixture, I consider it safe to state the nature of the barbiturate present. The mixed melting-point is especially important, as no less than four members of this group on the market melt between 168° and 172° C.

The fate of the barbiturate in the body varies considerably with the particular member of the group. Thus, in cases of Veronal poisoning, after a dose of, say, 50 to 80 grains, quantities of the order of 260 mg. may be found in the urine, and in one case I found 1·2 g. in the urine, which was equivalent to 397 mg. per 100 ml. of urine. Veronal, therefore, is not easily broken down in the body, and sufficient is excreted to enable it to be identified with ease.

In my Luminal series I have never found more than 120 mg. in the urine, and quantities of the order of 30 mg. are common. With Dial, however, except in one case, in which 25 mg. were found, I have never found more than traces, with which the determination of a melting-point was impossible.

This difference, of course, is in part due to the fact that the average fatal dose of the two latter is only about two-thirds that of Veronal, but it is mainly due to the fact that the rate of decomposition of the drugs in the body is considerably higher for Luminal and Dial than for Veronal.

So, too, with the stomach contents and the viscera. The former will contain little, if any, of the drug if the patient is found in a state of coma which has lasted several hours before discovery, and in the case of the viscera those drugs which are easily found in the urine will readily be found in the liver and kidneys, whereas those which are only found in traces in the urine will only be found in traces in the viscera, owing to rapid decomposition. Thus it will often happen that the analyst can only say in the witness box, in the case of some barbiturates, that he has found what are probably traces of a barbiturate. The cause of death then resolves itself into (1) the presence of the characteristic clinical picture and (2) a knowledge of the particular barbiturate present from, for example, the finding of a bottle beside the bed. Nevertheless, the finding of a trace of a barbiturate only, coupled with the clinical picture and the knowledge, *e.g.* that Dial was probably

taken, enables the analyst to say that his findings are consistent, in that he would only expect to find traces of this particular drug.

I do not propose to draw any conclusions from drugs such as Hebaral, Soneryl and Sandoptyl and Rutonal, and so on, as my cases are too few for any generalisations, save to say that moderate quantities appear in the urine; although extracts from this source do not crystallise easily, sufficient should be obtained for the determination of the melting-point.

It is fashionable for certain firms to compound a barbiturate with amidopyrine, *e.g.* Allonal, Veramon, Cibalgin, and so forth. Poisoning in these cases presents an additional picture. The stomach contents may show, if absorption is not complete, the presence of a barbiturate (extractable in acid ether solution) and amidopyrine (extractable in alkaline ether solution). The viscera, in my experience, may show the barbiturate (according to its nature), but will not show the presence of the amidopyrine. The urine, however, is so characteristic that a glance demonstrates that an excessive quantity of amidopyrine has been taken. The urine, if acid, is deep red in colour, but it changes colour to purple if alkalised, especially if ammonia is used. This colour is due to a pigment, rubazonic acid, which is formed, together with antipyryl-urea, from amidopyrine in the tissues. Both these bodies, if present in sufficient quantity, can be isolated from the urine by appropriate treatment. This we have done.

In my series of barbiturate cases, two of the hundred have been murders, a few have been accidents, where persons in bad health have taken doses which have been within medicinal limits, and in the remainder the cause might have been put down to suicide. Undoubtedly many of the cases were suicidal, the evidence at the inquest clearly pointing in that direction, but a number of cases have passed through my hands in which the alleged motive has appeared to be insufficient, and indeed there appears in some cases to have been but little motive at all.

Medical experience shows that a very large number of persons, more especially women, never take a night's rest without a tablet of a barbiturate, and occasionally, when this fails to produce the desired effect, a second is taken. This is not likely to do any harm, but recently medical men have been calling attention to a somewhat indefinable stuporous state, in which an individual, after taking one or two tablets, might take a whole packet, and in consequence be found unconscious in the morning. I can remember definitely three cases of this type in the last two or three years, and had my attention been drawn to this possibility earlier, I could, no doubt, add others. It is well known that women take these drugs regularly more than men, so that it is not surprising that my series of poisoning cases show 63 per cent. of female and 33 per cent. of male cases (records of the remaining 4 per cent. being incomplete), which, again, to some extent supports the accident theory. In my series 56 per cent. died and 29 per cent. recovered, leaving 15 per cent. in which I do not know the issue.

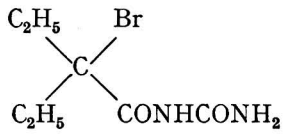
If, however, I were to analyse further this series, I could show that the percentage of recovery was far higher in the last 6 to 7 years than in the years previous to this period. This is due to improved methods of treatment, worked out mainly by Sir William Willcox and Sir James Purves Stewart. This is no place to discuss treatment, but I will say that one of the most important factors is the

frequent performance of lumbar or cistern puncture, coupled with saline injections and washing out of the stomach (with neutral or acid, not alkaline, liquid) and the colon, and powerful stimulants, *e.g.* strychnine, in full doses. As I have examined material from all these sources, I can, from the presence of a drug in the washings, speak of the importance of the treatment. Nor is this the place to discuss the clinical state, but I would mention that the most serious complication is development of bronchial pneumonia accompanied by a rise of temperature, which may supervene at any time after about the first 24 hours, and every case dying after this period will show this condition. This complication renders the prognosis more grave. Without my discussing the clinical features in greater detail, you will appreciate that, for this and other reasons, the clinical picture of poisoning by the barbituric acid group is not merely that of unconsciousness, but unconsciousness associated with certain features which will generally enable a diagnosis to be made.

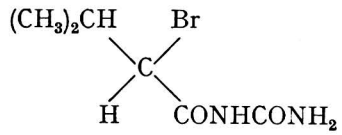
STRAIGHT-CHAIN UREIDES.—The next group of narcotic drugs, sometimes called the straight-chain ureides, are derived from mono-carboxylic acids and urea. They contain an atom of bromine.

Although there may be several of these drugs on the market, two only are known to me and need be considered. These are Adaline and Bromural.

Adaline



Bromural



These drugs are much less powerful than the barbiturates and are consequently safe hypnotics. When taken in large doses they do not produce the broncho-pneumonia which is so characteristic of the barbiturates. They are only slightly soluble in water, and of course, not being acids, do not form salts. They are soluble in ether and in benzene, so that if a mixture of a barbiturate and one of these drugs be encountered, a fairly good separation may be achieved by extracting the material with benzene to remove Adaline, etc., and subsequently extracting with ether in acid solution, to separate the barbiturate.

Leschke, in his *Clinical Toxicology*, is unable to record any fatal cases of poisoning by these drugs, and is able to cite a recovery after 150 grains of Bromural. I have had six cases through my hands, and, although my records are very incomplete, I am able to cite three fatal cases and two recoveries. The other was a recovery, so far as the drug was concerned.

Before referring briefly to these cases I would add a word about the analysis. In none of these cases where it has been possible to analyse the organs, or the urine, or both, have these drugs been found. They are readily broken down in the body, and I have never detected the drug in the tissues or the urine. In one case the drug was found in the stomach and contents, *i.e.* some of the drug which had escaped absorption. One finds bromide in large quantities in the tissues and in the urine, so that in the majority of cases one is able to say that the analysis is consistent with this class of drug having been taken.

Case 1. Urine only examined, bromine only found. Patient believed to have recovered. Probably a mixture of Adaline and Bromural taken.

Case 2. Known to have taken a mixture of Adaline and Bromural. Three-fifths of a grain of the mixed drugs found in the stomach and contents, or rather the mixture was assumed. Large quantities of bromine in liver, kidneys and urine. Patient recovered but killed himself by opening the radial artery in a hot bath.

Case 3. Died in coma, bromine only found in viscera and urine. History of having taken Adaline, quantity unknown.

Case 4. Known to have taken up to 300 grains of Bromural and Adaline in proportions of 15 of Bromural to 5 of Adaline. Urine only examined. Large quantities of bromine only found. Died.

Case 5. Believed to have taken up to 500 grains of Adaline. Bromine only in urine. Believed to have recovered.

Case 6. Large quantities of Adaline taken. Liver only examined. Bromine only found. Died.

Of these cases, two were females and four were males.

It will thus be seen that one has to be very cautious in these cases, and in not one of this series was the nature of the drug established by analysis. Nevertheless, if there is a history of the taking of the drug, coupled with the presence of bromide and failure to find by analysis any other hypnotic drug, and the post-mortem fails to reveal a cause of death, one is entitled by inference to draw the conclusion that one or both of these drugs have been taken, or, putting it at its lowest, that the findings are consistent with such a drug having been taken.

These drugs, on extraction from stomach contents, will yield a crystalline residue, but the Millon and cobalt tests are negative.* A quick diagnosis, for clinical purposes, may be made by a soda fusion and test for bromine in the urine. The broncho-pneumonia features so consistently seen with the barbiturates are not seen in these cases, and death is probably a primary cardiac failure, as a very low blood pressure is found towards the end.

MORPHINE.—The series of cases on which I shall base my observations is of course very much smaller in number than the barbiturate series, and numbers only 30, apart from four cases of morphine and barbiturate. With the exception of one case, they are not of special interest. The one case is of interest in view of the amount taken by the suicide. In this case I found 4 grains of Veronal in the urine, 10 grains in the stomach and contents (post mortem), and during life 212 grains in the stomach washings, *i.e.* 226 grains. In addition, 10 mg. of morphine were found in the urine. The infrequency of this poison, when compared with barbituric acid, is, of course, due to the operation of the Dangerous Drugs Act, and, although this Act was in operation during the period under consideration, there is no doubt that in the immediate post-war period many non-medical persons had possession of stocks of morphine. It is probable that during the next few

* With pure Adaline or Bromural, or both, in concentrated solution a faint positive cobalt reaction may be obtained.

years we shall see a decline in the number of barbituric acid cases, as this group of drugs is now in Schedule 4 of the Poisons and Pharmacy Act, 1932, which means that the drugs can only be obtained with a medical prescription.

Of my 30 cases, 20 were men and 10 women, and 26 of them died, leaving 4 whose fate is unknown. Four were cases of homicide, of which 2 were the subject of a prosecution. It will be seen that this drug is in more common use by males than by females, and it is much more fatal than those already considered.

There are some points in the analysis and the deductions therefrom which may be worthy of note.

First, I would say that I have never experienced any difficulty in isolating morphine in quantity from viscera by the ordinary Stas–Otto process. In our laboratory we have traditionally (dating, I believe, from Sir Thomas Stevenson) used ethyl acetate as the extracting solvent. Although many workers use amyl alcohol, and others favour the alcohol chloroform of Nicholls, our experience has been that both these solvents extract so much other organic material present in tissue extract that purification of the morphine is troublesome. Ethyl acetate may not be such an efficient solvent, but it is more selective.

Secondly, as a broad generalisation, we have found that the Stas–Otto process works more smoothly on liver that is rather decomposed than when practically no decomposition has taken place, and in general this applies to most alkaloids.

Thirdly, it has always been the practice in our laboratory when evaporating alcoholic extracts not to heat them above 40–50° C. Although the process is slow, we believe this to be a golden rule to success in alkaloidal work, and at the same time we take the greatest care to have the liquids only faintly acid. Again, this has been traditional since Stevenson's time, but it has worked well now for four generations, and I should be disinclined to change the practice until I had the most convincing proof of a superior one.

Finally, any process used must be capable of finding any alkaloid that may be present. The analyst may start with a knowledge that he may expect morphine, but unless his process is capable of detecting other alkaloids, he may find himself in grave difficulties at a later stage.

In dealing with cases of morphine poisoning, some of which have given me more anxiety than those with any other alkaloid, it is necessary to realise that three results may be obtained, and the medical deductions from the results of the analysis will lead to these three possibilities. Although perhaps I ought not to say it here, it is my firm conviction that the toxicologist should be a medical man with chemical training. This combination is essential in order that the whole picture from the details of the illness right to the analysis and the deductions therefrom may be in his mind for the final opinion. I say this, not from any personal point of view, but from a long experience of cases which have come my way, and of cases in which I have not been engaged, but about which I have had full knowledge.

To return to the three types of case.

First Group.—These are simple cases of an overdose of the drug for suicidal or homicidal purposes, where there is no question of addiction to the drug. In these cases the illness usually lasts from 6 to 12 hours, as contrasted with barbiturate cases, in which I have known the coma to last as long as 6 days. In this short time

much of the morphine will escape destruction and consequently plenty will be found on analysis. We have found as much as 10·5 grains in the stomach and contents, with 2 grains in the liver, and 0·5 grain in the kidney. Quantities from 0·5 to 2·5 grains in the stomach and contents, and from 0·2 to 1 grain in the liver, have several times been found.

In morphine cases, as indeed in all cases where a drug has been administered by hypodermic methods, and where the puncture mark is visible, the skin area, together with some of the muscle tissue underlying, should be fairly widely excised. When this is submitted to an analysis, morphine will, as a rule, be readily found. The urine, too, should always be saved in all cases of narcotic examination, as in practically every case the presence here, too, of the drug will be established.

The cause of death in these cases will rarely, if ever, be in doubt, and the matter is a simple one.

Second Group.—From time to time cases come into the analyst's hands where it is known that the deceased was a morphine addict, and that death appears to be from unnatural causes. The deceased may have taken a quantity of morphine far in excess of the usual daily dose, or, as appears to be probable in some cases, death may have been due to a sudden intolerance to the drug, and an analysis will be required. In these cases, although there may be evidence that a very large quantity of morphine has been taken, a quantity far in excess of what is regarded as an average fatal dose, the analyst will find only traces of morphine in the viscera, and in some cases he will have the greatest difficulty in establishing the presence of morphine. In fact, I have on more than one occasion suggested to the Coroner that, in view of the belief that the deceased, from the clinical and post-mortem picture, had taken morphine, while my analysis has shown only traces of the drug, the deceased was addicted to the drug, and subsequent enquiry has established the correctness of the supposition.

The whole question of addiction would appear to be bound up with an increased power to destroy the drug in the tissues, and despite the sudden loss of tolerance to the drug which sometimes occurs, and which results in grave mental change, it would appear that this power to destroy the drug is a reaction of the tissues to the ingestion of an abnormal substance, which once acquired is not lost.

Third group.—(Morphine in the exhumation case.)—From time to time it has been my duty to examine the viscera in cases where bodies have been exhumed after various periods, the longest being 19 months after burial. Naturally, the finding of morphine in such cases will depend on many factors, *e.g.* the amount of morphine taken, conditions of burial, state of the body, and so on. Nevertheless, in this case I found definite evidence of the presence of morphine, so that in some cases it is possible to demonstrate this poison in viscera after periods as long as 19 months. In the case in question the body was that of an old man with but little fat, well coffined in an oak coffin and buried in a clay soil, where very little change of temperature, moisture or air would be likely to occur, with the result that the body was well preserved. Although I have stated that morphine was detected, this is strictly incorrect, as I actually found oxydimorphine or pseudomorphine. This change occurred in the three exhumation cases in which I have been concerned, and where this drug was in

question and was found; and in a fourth case, not an exhumation, where the stomach contents only were available, and where these contents had been kept by the Coroner for nearly 14 days, with the result that gross decomposition had occurred, the change was also observed. The last case illustrates three points: (1) the change from morphine to pseudomorphine does occur in decomposed viscera; (2) morphine given hypodermically will be found in the stomach and contents; (3) even after a dose of 1/4 grain, given three hours before death, morphine may be detected, and in this case I would add that the administration of the morphine was not known to me until after my analysis was complete and as the result of enquiries made by me.

The important test which serves to differentiate between morphine and pseudomorphine is, of course, the Marquis test, and in this connection I looked up one of the *causes célèbres*, in which a conviction for murder was obtained after this drug had been found in an exhumation case after 8 weeks' burial. I refer to the case of *The People of the State of New York* against *Carlyle W. Harris*. This case was brought to a successful conclusion through the brilliant work of the late Prof. Witthaus, whose textbook on toxicology, although perhaps somewhat out of date (the last edition being 1911), still, in my opinion, remains the classical work on this subject. I was interested to see whether Witthaus had, in fact, found morphine or pseudomorphine. Although it would appear that some of the colour reaction tests made by him and described in his evidence would point to the presence of morphine rather than pseudomorphine, it is interesting to note that the Marquis test was not published by its discoverer until 1895, whilst the trial to which I have referred took place in 1892. The difficulty of describing some of the colour reactions of morphine in visceral extracts is well known, and, although it would appear that Witthaus had some evidence to justify the finding of morphine, one cannot help wondering whether he would have reported the presence of pseudomorphine had the Marquis test been available at the time of his analysis.

While on the subject of textbooks, one cannot but deplore some of the statements which are copied from book to book for generations. One, for example, is the statement, often quoted in the Coroner's Court, that the smallest fatal dose of Tinct. Opii. recorded in an adult is 2 drachms. This case is recorded in the *Edin. Med. and Surg. Journal*, July, 1840, p. 151. Expressed as morphine in terms of the tincture of to-day, this is equivalent to 1·3 grains of morphine. What was the amount in 1840? First, opium was not standardised; secondly, according to Henry (*Plant Alkaloids*) the strength of opium may vary from 3·2 to 12·3 per cent. of morphine; thirdly, in those days chemists made their own tinctures with variable skill, had no means of standardisation, and weights and measures were not of the highest accuracy. The Pharmacopoeia of that date only says: "Take hard opium and proof spirit, and when made about 19 minims should contain one grain of opium." Therefore, two drachms would contain about 0·6 grain of morphine, if opium contains 10 per cent. of morphine. But, having regard to the possible variants in the making of such a tincture in 1840, I think it is quite impossible to calculate how much morphine the deceased actually took, so that it would seem futile to continue recording such a worthless observation. This is an example of many unsound observations which abound in textbooks on

this subject, and it is to be hoped that one day a book will be written which has eliminated such misleading and unreliable statements.

In my series of morphine cases a few were opium cases, which necessitate the search for meconic acid, but the majority were morphine cases. Two were chlorodyne, and two probably heroin cases.

I have not dealt in detail with the symptomatology of morphine poisoning, as there is but little to say. The patient passes from sleep to coma and from coma to death, generally within 12 hours. Cyanosis is marked, owing to the action of the drug on the respiratory centre, and towards the end the respiratory rate may slow down to about 4 per minute. At the time of death the heart may beat for a while after respiration has ceased. Everyone knows that the pupils are contracted in poisoning by this drug, but this requires a word of warning. This effect is central in origin, *i.e.* originating in the brain, but when the final paralytic stage is reached the pupil often relaxes and becomes dilated. On several occasions I have known the failure to appreciate this fact lead to a faulty diagnosis, and it has only been the subsequent analysis which has demonstrated that morphine and not atropine was the poison used.

The following list gives the melting-points of the barbituric acid compounds now on the market and also the melting-points of certain of the other common hypnotic drugs. These values have actually been found in our laboratory, and the determinations have been made on the substances extracted and purified from tablets and so forth. The list also gives most of the names under which these substances appear on the market. So far as the barbiturates are concerned, it will be appreciated that the majority of them are marketed as the corresponding sodium (and in one case calcium) salt.

There are a large number of compounded proprietary drugs on the market which contain barbiturates mixed with other substances, but it is impossible to give a complete list of these.

	m.p. of acid °C.
Soneryl, Neonal, Butyl-ethyl barbiturate	123–4
Hebaral, Ortal, Hexyl-ethyl barbiturate	126
Nembutal, Pentobarbital. Ethyl-isomethyl-butyl barbiturate ..	130
Pernocton, Butyl-bromo-allyl barbiturate	132
Sandoptyl, Isobutyl-allyl barbiturate	138–9
Allonal, Allyl-isopropyl barbiturate (mixed with amidopyrine) Numal	141
Evipan, N-methyl-*c*-*c*-cyclohexenyl-methyl barbiturate	144–5
Amytal, Isoamyl-ethyl barbiturate	155
Phanodorm, Cyclohexenyl-ethyl barbiturate	168–9
Dial, Currial, Allobarbitonum, Diallyl barbiturate	168–9
Prominal, N-methyl-ethyl-phenyl barbiturate	170–1
Luminal, Phenobarbitonum, Gardenal, Phenyl-ethyl barbiturate ..	171–2
Noctal, Bromopropanyl-isopropyl barbiturate	176–7
Veronal, Barbitone, Malonal, Hypnogen, Di-ethyl barbiturate ..	191
Ipral, Ethyl-isopropyl barbiturate	198
Rutonal, Phenyl-methyl barbiturate	221
Proponal (not now on the market)	145
Seconal, Propyl-methyl-carbinyl allyl barbiturate (5)α-Methyl-*n*-butyl-(5)allyl barbituric acid	—
Pentothal, Ethyl-methyl-butyl-thio-barbiturate	156–7

	m.p. of acid °C.
Adaline, Carbromalum, Uradal, Nyctal, Planadalin, etc., Bromo-ethylbutyryl carbamide	116
Bromural, Bromoisovalum, Bromo-isovaleryl carbamide	145
Sedormid, Allyl-isopropyl-acetylurea	194
Sulphonal	126
Trional, Methyl sulphonal	76

For the purpose of illustration the histories of a few selected cases are given in the accompanying table.

In conclusion, I desire, once again, to pay a tribute to my officers, and without making distinctions I would specially desire to mention Dr. Hughes and Dr. Mitchell, who have both for so many years given us devoted service, and to whom we owe the present prosperous state of the Society. Mr. Eynon, I can say from personal experience, has given his best to us, and I can assure you it is a very good "best," and I am convinced from my experience of Mr. Lane that our choice of him as Secretary could not have been bettered. Lastly, I would pay a tribute to the Members of Council, who devote so much time to the meetings and who, in many instances, are so willing to come at their own expense from long distances.

I now leave the President's chair and I do so with mixed feelings. Increasing work has made it difficult for me to carry out my duties with the efficiency that I should have liked, but, on the other hand, it has been a real pleasure to me to play a part in the direction of the Society's affairs, and I can assure you that, as a Past President on the Council, it is my intention in the future to do everything in my power to further the interests of the Society.

The Determination of Bismuth in Biological Materials

By SIDNEY LIONEL TOMPSETT, Ph.D., B.Sc., F.I.C.

Bismuth is not a normal constituent of human tissues and excreta. In syphilis, bismuth in either the combined or the metallic form is frequently administered as an intra-muscular injection. Under these conditions bismuth will be present in the tissues and excreta. The efficiency of a particular bismuth preparation in the treatment appears to depend, to a certain extent, on its rate of absorption from the site of injection. This may be evaluated by a study of the rate of excretion of bismuth.

The following is a description of a method for the determination of minute amounts of bismuth in such materials as urine, faeces and animal tissues.

Senzi and Seghezzo[1] have shown that thiourea produces an intense yellow colour with bismuth in acid solution. The reaction is specific, but it does not

SOME SELECTED CASES

	Patient	Sex	Date	Amounts taken	Amount found in urine	Amount per 100 ml. of urine	Amount found in stomach contents	Stomach washings	Amount per 100 ml. of C.S.F.	Liver	Kidneys	Melting-point determined	Time of illness	Result	Remarks
ADALINE + BROMURAL	N	M	9/34	——	Br found	——	0·6 gr. Adaline + Bromural	——	——	Br found	Br found	——	——	Death	Glass found containing Adaline and Bromural.
	C	M	10/35	——	Br found	——	——	——	——	Br found	Br found	——	——	Death	History of taking of Adaline.
ALLONAL	L	F	11/37	——	3·2 mg.	2·5 mg.	——	6 gr. + 5·4 gr. of amidopyrine	14 mg.	——	——	Yes	——	Death	Rubazonic acid and antipyl urea found in urine.
	B	F	8/33	——	Trace. Rubazonic acid found	——	——	Trace, BA. No Amidopyrine	——	——	——	No	——	Recovery	
DIAL	W	F	1/34	——	Trace	——	Trace	——	——	——	——	No	20 hours at least	Death	
	R	F	12/33	18 gr.	25 mg.	——	——	14 mg.	——	——	——	No	——	Recovery	Known to have taken Dial.
	F	F	7/35	Possibly 54 gr.	Trace	——	——	0·5 gr.	——	——	——	No	About 60 hours	Death	Known to have purchased Dial.
HEBARAL	D	F	1/34	75 gr.	6 gr. (1) 3·3 gr. (2)	110 mg. 88 mg.	——	Trace	6.25 mg. (1) C.P. 4·3 mg. (2) C.P.	——	——	Non-crystal-lisable	——	Recovery	Known to have taken Hebaral.
LUMINAL	R	F	7/36	——	0·75 gr.	17 mg.	——	0·26 gr.	18 mg. (1) 10 mg. (2)	——	——	168–169° C. (mixed, 171° C.)	——	Death	
	A	F	3/37	——	25·1 mg.	19·8 mg.	——	——	6·5 mg.	——	——	No	——	Death	Known to have taken Luminal.
	W	F	5/35	——	0·5 gr.	18 mg.	——	2·3 gr.	24 mg.	——	——	172° C.	——	Death	
NEMBUTAL	D	F	1/38	37 gr. alleged, probably less	21·5 mg.	3·3 mg.	——	29·7 mg.	1·8 mg.	——	——	Yes	——	Recovery	
RUTONAL	—	M	3/35	50 gr. (?)	35 mg.	——	——	——	——	——	——	198° C.	——	Recovery	
SANDOPTYL	W	F	2/34	150 gr.	2·25 gr.	11 mg.	——	——	Trace	——	——	——	——	Recovery	Known to have taken drug.
SONERYL	K	F	11/36	?	Trace (1) 0·3 gr. (2)	—— 10 mg.	Trace	——	Trace (1) 2·0 mg. (2)	Trace	Trace	No	——	Death	Known to have taken drug.
VERAMON	D	F	5/32	14 tablets	Trace, BA	——	Trace, BA	——	——	Trace, BA	Trace, BA	No	——	——	1 Tablet = 2 gr. Veronal, 4 gr. amidopyrine.
	C	M	11/33	——	Trace, BA Rubazonic acid	——	Trace, BA	——	Trace, BA ,, Amidopyrine	Trace, BA	Trace, BA				
VERONAL	S	M	2/27	——	0·77 gr.	——	1·84 gr.	——	——	5·0 gr.	1·27 gr.	185° C.	59 hours	Death	
	B	M	3/30	——	5·3 gr.	——	37·9 gr.	——	——	6·4 gr.	——	Yes	——	Death	
	W	F	10/35	160 gr. presumed	4·3 gr.	100 mg.	0·2 gr.	——	25 mg.	1·0 gr.	——	187° C.	7 days	Death	
	B	F	12/37	——	6·0 gr.	——	0·5 gr.	——	——	1·87 gr.	——	Yes	——	Death	Found dead in a wood.
MIXTURES OF BARBITURATES	D	F	5/33	65. gr. Allonal 125 gr. Veronal 90 gr. Quadronox 40 gr. Ipral	——	——	——	BA, 12 mg. Py. 5 mg.	C.P.(1) BA, 5 mg. Py. nil C.P. (2) BA, 2 mg. Py. nil L.P. (1) BA, 3 mg. Py. trace	——	——	No	——	Recovery	
	V.H.	F	1/34	48 gr. Luminal 30 gr. Soneryl	2 gr.	23·4 mg.	——	——	L.P. 2·3 mg. C.P.(1) 33 mg. C.P.(2) 2·0 mg.	——	——	No	——	Recovery	
TRACES OF UNKNOWN BARBITURATES	C	M	12/36	——	——	——	Trace, BA	——	——	Trace, BA	Trace, BA	No	——	Death	
	G	F	——	——	——	——	Trace, BA	——	——	Trace, BA	Trace, BA	No	——	Death	
MORPHINE	T	M	5/33	——	0·1 gr.	——	1·0 gr.	1·23 gr.	——	0·66 gr.	——	——	——	Death	Taken in tea; residue contained 0·25 gr.
	(Mrs.) B	F	1/36	——	——	——	Trace (oxy-dimorphine)	——	——	Trace (oxy-dimorphine)	Trace (oxy-dimorphine)	——	——	Death	Exhumation case; 5 months; Alkaloid as oxydimorphine. Murder.
	E	M	8/34	——	0·2 gr.	——	0·1 gr.	——	——	0·5 gr.	——	——	——	Death	Taken as chlorodyne.
	H.	M	6/21	——	Trace (morphine)	——	Trace (morphine)	——	——	0·05 gr. (morphine)	0·025 gr. (morphine)	——	——	Death	Taken hypodermically; skin from thigh, 0·1 gr. (Addict).
	L	M	7/28	——	0·07 gr. (morphine)	——	0·33 gr. (morphine)	——	——	0·15 gr. (morphine)	——	——	——	Death	Syringe and drug found. Not addicts.
	S	F	7/28	——	——	——	0·5 gr. (morphine	——	——	0·5 gr. (morphine)	——	——	——	Death	Suicide pact.

NOTE.—BA indicates barbiturate; L.P., lumbar puncture; C.P., cistern puncture; C.S.F., cerebrospinal fluid; Py amidopyrine; gr., grains.

appear to have been applied quantitatively. The present investigation shows that it can be so applied.

In the first place, the sensitivity and range of the reaction were studied. Solutions containing from 0·005 to 1 mg. of bismuth in 5 ml. of 20 per cent. sulphuric acid were prepared, and each was mixed with 5 ml. of a 10 per cent. solution of thiourea. The mixtures were compared colorimetrically. The colour was found to be proportional to the quantity of bismuth, within a range 0·005 to 0·8 mg., but larger quantities did not show proportionately greater depth of colour. It is necessary, therefore, to work within the range 0·005 to 0·8 mg. of bismuth. Concentrations of bismuth within the range 0·005–0·05 mg. of bismuth produced depths of colour, which, although distinct, were too weak to compare in a colorimeter. Concentrations of bismuth within this range were determined by comparison with a series of standards in standard tubes. Concentrations of bismuth within the range 0·05 to 0·8 mg. of bismuth were determined by comparison with a suitable standard in a colorimeter.

When applying such a method to biological material one is confronted with the difficulty that the amount of bismuth may be small, and thus a large quantity of material must be used. Since the ash-content of biological materials is usually high, the difficulty arises of making the solution in a volume of 5 ml. A method was therefore devised to separate the bismuth prior to its colorimetric estimation.

The separation was based upon the fact that bismuth combines with sodium diethyldithiocarbam. te to form a complex which is soluble in ether (Tompsett[2,3]). Certain other metals, *e.g.* copper and lead, are also extracted, but these do not interfere with the final colorimetric estimation. The separation was effected in alkaline solution (*p*H 8) in the presence of citrate; this prevents the precipitation of phosphates and also the extraction of iron.

The following is a detailed description of the method as applied to urine. *Solutions.*—(1) conc. sulphuric acid; (2) perchloric acid; (3) sodium citrate, 20 per cent. solution; (4) ammonia, sp.gr. 0·88; (5) sodium diethyldithio-carbamate, 2 per cent. solution; (6) thiourea, 10 per cent. solution; (7) standard solution of bismuth.

For the preparation of the standards 1 g. of pure metallic bismuth was heated with 20 ml. of conc. sulphuric and nitric acid until the bismuth dissolved; the nitric acid was then driven off. The digest was diluted to 100 ml. with water; 1 ml. of this solution contained 10 mg. of bismuth. Suitable standards were prepared by dilution of this solution with 20 per cent. sulphuric acid.

Chemicals of analytical reagent quality were used; these were found to be free from bismuth.

METHOD.—The urine (100 to 500 ml.) was digested with sulphuric and perchloric acids in a Kjeldahl flask until all the organic material had been destroyed and excess of perchloric acid had been driven off. The digest was diluted to about 150 ml. with water, 100 ml. of 20 per cent. sodium citrate solution were added, and the mixture was made alkaline (*p*H 8) by addition of ammonia. Ten ml. of 2 per cent. sodium diethyldithiocarbamate solution were added, and the mixture was extracted three times with ether. The combined ethereal extracts were evaporated to dryness in a Kjeldahl flask, and organic material was destroyed by

heating with 1 ml. of concentrated H_2SO_4 and 1 ml. of perchloric acid. After cooling, the digest was diluted to 5 ml. with water, and 5 ml. of 10 per cent. thiourea solution were added. The colour was compared with a standard prepared by mixing 5 ml. of a standard solution of bismuth in 20 per cent. sulphuric acid with 5 ml. of 10 per cent. thiourea solution.

A similar method was used in the determination of bismuth in faeces and tissues.

Known amounts of bismuth were added to samples of urine, faeces and liver. Accurate recoveries were obtained, as is shown by the following typical results:

		Bismuth added Mg.	Bismuth found Mg.
(1)	Urine, 200 ml.	0·01	0·01
(2)	Urine, 200 ml.	0·02	0·02
(3)	Urine, 200 ml.	0·08	0·07
(4)	Urine, 200 ml.	0·20	0·19
(5)	Urine, 500 ml.	0·40	0·39
(6)	Urine, 500 ml.	0·50	0·48
(7)	Faeces, 10 g., dried	0·10	0·10
(8)	Faeces, 10 g., dried	0·20	0·19
(9)	Liver, 50 g.	0·10	0·09
(10)	Liver, 50 g.	0·20	0·19

REFERENCES

1. G. Senzi and S. Seghezzo, *Annali Chim. Appl.*, 1929, **19**, 392.
2. S. L. Tompsett and A. B. Anderson, *Biochem. J.*, 1935, **29**, 1851.
3. S. L. Tompsett, ANALYST, 1936, **61**, 591.

BIOCHEMICAL LABORATORY
ROYAL INFIRMARY
GLASGOW

November, 1937

A Test for Traces of Oxidising Agents in Milk

BY R. C. WRIGHT, B.Sc., DIP.BACT., AND E. B. ANDERSON, M.Sc., F.I.C.

THE work to be described in this communication arose from an attempt to discover a test for the detection of traces of hypochlorite in milk.

The value of hypochlorites as sterilising agents is well known. They are particularly useful in open vessels where steam cannot be applied effectively. Their cost is low, and their use does not involve any capital outlay for plant. Despite this, their use for disinfecting dairy plant and utensils is forbidden by law in England, except for mechanical milkers. The reason usually given is that the milk producer or distributer might use them as preservatives, and that, as they are converted into chlorine ions in the milk, the addition could not be detected by chemical analysis. This argument is not really sound, because the work carried out by Lochhead[1] in Canada has shown that 80 p.p.m. of chlorine must be added before its effect on the milk can be detected organoleptically, and that this concentration has no preservative action.

It appeared reasonable to suppose that if a satisfactory test for milk contaminated with chlorine could be discovered, the major objections to its use would be nullified.*

THE DETECTION OF CHLORINE IN MILK AND CREAM.—This problem has received little or no attention in recent years and the only published test is that devised by Rupp.[1] Rupp states that his test depends upon the fact that when chlorine is added to milk, a chloro-protein compound is formed from which chlorine is liberated by hydrochloric acid. In the presence of potassium iodide the free iodine liberated by the chlorine then combines with the protein to form an iodo-protein compound, which reacts with starch to give a blue or purple colour.

In all our experiments carried out on this test the experimental details given by Rupp were strictly adhered to. The results obtained suggest that the test is more sensitive than is claimed by Rupp, as it was found possible to detect the addition of 6 p.p.m. of chlorine in milk and 50 per cent. cream, and 12 p.p.m. of chlorine in 25 per cent. cream. Various modifications of the method were tried in an attempt to increase its sensitivity, but without result.

In the course of these experiments two difficulties were encountered, namely:—(*a*) The results obtained with any one sample were very irregular, *e.g.* in duplicate tests on any one sample of milk the limit of detection might vary between 1·5 and 25 p.p.m. (*b*) Positive controls were occasionally obtained.

During the course of the work on the Rupp test, acids other than hydrochloric were tried, and it was observed that if conc. sulphuric acid was diluted slightly, well cooled and mixed with cold milk, normal milk remained white, provided that it was kept well cooled during the mixing, whereas milk to which hypochlorites had been added developed a greenish-yellow colour. Further, the greater the addition of hypochlorite the greater was the intensity of this colour, and the stronger the acid the more intense was the colour up to the point when the controls no longer remained white but began to char, with the formation of a yellowish-brown colour which masked, to a certain extent, the presence of the greenish-yellow colour.

This phenomenon was then developed into a practical test, with the result that it appeared possible to detect an addition of 12 p.p.m. of chlorine in milk and coffee cream and 17 p.p.m. of chlorine in full cream.

Briefly, the method adopted for milk consisted in adding 3 ml. of 78·4 per cent. sulphuric acid (4 vols of conc. acid and 1 vol. of distilled water), cooled to 0°–5° C., to 3 ml. of milk similarly cooled to 0°–5° C., and mixing the contents of each tube in an ice-bath. The tubes were left in the ice-bath for 10 minutes and then taken out and examined for the presence of any greenish-yellow colour. With full cream it was found necessary to first dilute it to a fat-content of 35 per cent. and to use 8 ml. of 71·3 per cent. sulphuric acid (8 vols. of conc. acid and 3 vols. of distilled water) and 2 ml. of the diluted cream. The use of 78·4 per cent. sulphuric acid

* In our own tests it was not possible to detect by its odour 200 p.p.m. of chlorine half an hour after its addition. In experiments on the preservative action we found that apparently the effect varied from milk to milk: to some, 100 p.p.m. of chlorine could be added without affecting its keeping quality, whilst with others, 50 p.p.m. had a preservative effect. From our own experiments we should feel justified in stating that amounts less than 50 p.p.m. have no preservative action.

resulted in control creams possessing a brownish-yellow colour which masked the presence of any greenish-yellow colour.

It was found that tubes showing this greenish-yellow colour emitted a strong yellow fluorescence under ultra-violet light. This property resulted in an increased sensitivity, and a further improvement was made by centrifuging the tubes after mixing in the ice-bath, in order to obtain a clear serum.

In all the experiments carried out on this new test the chlorine was added in the form of hypochlorite, and it was originally thought that the test was an index of the amount of available chlorine added. However, while using an old hypochlorite solution it was found that milks containing a definite volume of this old hypochlorite solution gave a much more intense fluorescence than milk containing an equal volume of a freshly manufactured hypochlorite solution, despite the fact that the percentage of available chlorine was very much higher in the fresh hypochlorite solution. Further investigation of these two hypochlorite solutions showed that the amount of fluorescence developed was proportional to the chlorate content. Subsequent tests on various hypochlorite solutions and on potassium chlorate solutions proved that this yellow fluorescence was due to the presence of chlorate in the hypochlorite solutions and was proportional to the amount present.

Table I shows that the amount of hypochlorite detectable is inversely proportional to the chlorate present in the hypochlorite solution.

TABLE I

Hypochlorite solution	Available chlorine Per Cent.	Potassium chlorate Per Cent.	Sulphuric acid test (a) Amount of chlorate per ml. at minimum fluorescence mg.	Sulphuric acid test (b) Equivalent to available chlorine p.p.m.
B	11·28	4·25	0·0008	2·2
C	13·65	3·65	0·0007	2·6
D	14·95	2·60	0·001	5·6
E	16·70	1·54	0·0007	13·0
F	18·04	0·87	0·0007	28·0

The chlorate-content of the hypochlorite solutions was determined by reduction in the presence of acid ferrous sulphate and potassium iodide.[3] The figure so determined represents the chlorate-content and the available chlorine-content of the solution, and from this was subtracted the available chlorine-content obtained by titration of the iodine liberated in the presence of potassium iodide at room temperature.

With pure potassium chlorate solutions it was found possible to detect 1 part in $2{\cdot}5 \times 10^6$ parts of milk $\equiv$ 0·0004 mg. per ml. of milk.

Since this test depends upon the presence of chlorate in hypochlorite solutions, it was thought that it would be of interest to determine the rate of formation of chlorate in such solutions. Therefore a freshly manufactured hypochlorite solution was obtained and divided into two portions, one being stored in the dark at 0° C. and the other in the dark at atmospheric temperature. At intervals the available chlorine-content and chlorate-content of each solution were determined.

The results of these determinations are given in Table II.

TABLE II

RATE OF FORMATION OF CHLORATE AND RATE OF DESTRUCTION OF AVAILABLE CHLORINE IN HYPOCHLORITE SOLUTIONS

Date of testing	At 0° C.		At atmospheric temperature	
	Available chlorine Per Cent.	Chlorate Per Cent.	Available chlorine Per Cent.	Chlorate Per Cent.
21–5–37	19·77	0·43	19·77	0·43
28–5–37	19·41	0·50	17·43	1·27
7–6–37	18·90	0·57	15·39	2·28
17–6–37	18·43	0·79	13·99	3·15

From these figures it will be seen that, except in a perfectly fresh solution, there is sufficient chlorate present to enable one to detect an addition of hypochlorite equivalent to 13 p.p.m. of chlorine.

During the course of this work it was found that, whereas control tubes showed the absence of any visible greenish-yellow colour in daylight, such tubes showed a slight yellow fluorescence when examined in ultra-violet light. Accordingly the concentration of acid used was altered slightly in order to obtain consistently negative controls. As a further safeguard against the production of any yellow fluorescence in the control tubes it was decided to introduce a small quantity of a reducing agent into the sulphuric acid. A reducing agent was chosen because the production of this fluorescence is due to oxidation, as proved by the fact that hydrogen peroxide and potassium nitrate are both capable of bringing about this reaction, but the concentration of these substances necessary is much higher than is likely to be found in milk or cream. The reducing agent chosen was stannous chloride and a concentration of 0·025 per cent. was used, as this amount was found to be sufficient to eliminate any possibility of positive controls occurring, whilst at the same time it did not seriously impair the sensitivity of the test.

The presence of added water in the milk did not affect the result, but it was found that if the test is to be carried out on milks which contain more than 20 per cent. of added water the control milk should be correspondingly diluted.

A few experiments were carried out to determine what constituent of the milk was responsible for the yellow fluorescence. These experiments showed that the production of the fluorescence was connected with the protein constituents and was independent of the presence of the fat, lactose or mineral salts.

The final method adopted of applying the test was as follows:—

MILK.—To 3 ml. of milk in a $\frac{3}{4}$-in. test-tube, cooled to 0°–5° C., are added 3 ml. of 73·5 per cent. sulphuric acid containing 0·025 per cent. of stannous chloride, also cooled to 0°–5° C. The contents of the tube are well shaken in a freezing-mixture of ice and salt, and allowed to stand in this mixture for 3 minutes. The contents are then transferred to a 12·5-ml. centrifuge tube and spun for 3 minutes at 2500 r.p.m. The tubes are immediately examined in ultra-violet light for the presence of any yellow fluorescence.

CREAM.—To 1·3 ml. of distilled water in a $\frac{3}{4}$-in. test-tube is added 1 ml. of cream. The tube is well shaken and cooled to 0°–5° C., and 3 ml. of 73·5 per cent. sulphuric acid containing 0·025 per cent. of stannous chloride, also cooled to 0°–5° C., are added. The contents of the tube are well shaken in a freezing-mixture of ice and salt and allowed to stand in this mixture for 3 minutes. The contents are then transferred to a 12·5-ml. centrifuge tube and spun for 3 minutes at 4000 r.p.m. The tubes are immediately examined in ultra-violet light for the presence of any yellow fluorescence.

Provided that these methods are strictly followed, no difficulty should be experienced in applying the test. If the tubes cannot be examined immediately after centrifuging they should be examined within half-an-hour, as a small amount of yellow fluorescence may appear, especially with cream, if they are allowed to stand for more than half-an-hour.

With this new test the results obtained are very consistent, *i.e.* they can be repeated on the same sample, with very little, if any, variation and no positive controls were ever obtained in the final method adopted.

The test was found to act equally well with milk and cream, whether raw or pasteurised, and it made no difference to the result whether the hypochlorite was added to pasteurised milk or to raw milk and the milk subsequently pasteurised. In addition, the sensitivity of the test remains constant up to 48 hours after the addition of hypochlorite, after which time it sometimes appears to decrease slowly, but, even then, the loss in sensitivity during the next 48 hours is only slight. Usually there was no loss in sensitivity over a 4-day period, the samples being kept at 38° F.

Our own tests show that by taste it is possible to detect between 50 and 100 p.p.m. of available chlorine in milk and cream. The test described, therefore, is much more sensitive.

SUMMARY AND CONCLUSIONS.—A simple method of detecting the presence of small quantities of hypochlorite solutions in milk and cream is described. The test depends upon the constant presence of chlorate in the hypochlorite solutions.

We are indebted to Dr. R. J. Macwalter for the suggestion that the colour produced might be more readily observed under ultra-violet light, and to United Dairies, Limited, for permission to publish the paper.

REFERENCES

1. A. G. Lochhead, *Report of the Dominion Agricultural Bacteriologist, Canada*, 1929–30.
2. P. Rupp, *U.S.A. Dept. Agric. Bull.*, 1114, 1922.
3. C. O. Harvey, ANALYST, 1925, **50**, 538.

UNITED DAIRIES, LTD.
CENTRAL LABORATORY
WOOD LANE, LONDON, W.12

The Determination of Parachlorometaxylenol in Antiseptic Solutions

By R. P. MERRITT, Ph.C., and T. F. WEST, M.Sc., A.I.C.

Antiseptic solutions described as non-toxic germicides are being introduced and are largely replacing lysol. Their Rideal–Walker coefficients range between 3 and 7 and, according to recent publications,[1,2] *p*-chloro-*m*-xylenol is often used as the high-powered antiseptic agent included in these solutions. Rapps[3] has shown that the Rideal–Walker coefficient of this phenol is 35·7 in aqueous, and 62·0 in saponaceous, solution. We have evolved a method by which the *p*-chloro-*m*-xylenol in antiseptic solutions containing soap and essential oils can be determined and identified. In addition, the method can be used by the analyst, confronted with an unknown sample, to obtain an approximate analysis involving percentage of oil, soap and phenol. The process has been applied successfully to a number of solutions containing different soaps and essential oils. The method could not be applied to a solution in which the essential oils employed contained phenolic bodies but, in view of the oils available, the presence of these would be improbable. The final method was based on observations which may be summarised as follows:—*p*-Chloro-*m*-xylenol was obtained in quantitative yield from an alkaline solution by passing carbon dioxide through it for half-an-hour and extracting the suspension with ether. Although the phenol is exceedingly soluble in ether, its solubility in petroleum spirit (b.p. 40 to 60° C.) is only 3·27 g. per litre at 21° C.; it could therefore be readily extracted from an alkaline solution with ether, but the amount extracted by petroleum spirit was negligible. When the phenol was dissolved in excess of sodium hydroxide solution, calcium chloride solution added, and the precipitated calcium hydroxide filtered off and washed with water, the *p*-chloro-*m*-xylenol could be recovered in quantitative yield from the filtrate after precipitation with hydrochloric acid or carbon dioxide. Experiments showed that it was not possible to free the phenol from fatty acids by the use of carbon dioxide, and this limited the applicability of this reagent. The following method, involving variations based on the results obtained in a number of experiments, was finally adopted:

Method.—Sodium hydroxide (2 ml. of a 50 per cent. solution) was added to 20 ml. of the solution (see Table I) under investigation in a 50-ml. distillation flask, and the alcohol was distilled. The solution was transferred to a separator by means of 10 ml. of water, and the essential oil was removed by shaking the solution gently twice with 10 ml. of petroleum spirit (b.p. 40°–60° C.). The petroleum spirit extract was washed twice with 10 ml. of water, and the soap in the combined aqueous liquor was precipitated with 20 per cent. calcium chloride solution. The calcium soap was filtered off on a Buchner funnel and washed three times with 10 ml. of hot water. The solution was acidified with conc. hydrochloric acid and extracted three times with 25 ml. of ether (A).

It was found that the calcium soap adsorbed a small quantity of the *p*-chloro-*m*-xylenol, which was not removed by three further washings with water. The

following method of recovering the adsorbed phenol was found effective. The calcium soap was extracted three times with 15 ml. of boiling ethyl alcohol, the solution was diluted with twice the volume of water, and filtered, and the alcohol was removed by distillation under reduced pressure. The aqueous solution (filtered if necessary) was acidified and extracted three times with 25 ml. of ether (B).

The combined ethereal extracts (A) and (B) were evaporated, and the residue was dissolved in the smallest possible quantity of 10 per cent. sodium hydroxide solution and made up to 60 ml. with water. The phenol was precipitated by passing carbon dioxide through the liquid for half-an-hour, and the suspension was extracted three times with 25 ml. of ether. The ethereal solution was dried over sodium sulphate and filtered into a tared alkaloid-flask, and the ether was distilled. The flask was then left in a vacuum desiccator over calcium chloride and paraffin wax for two hours and weighed. The melting-point of the *p*-chloro-*m*-xylenol obtained was usually about 110° C., but its identity could be confirmed after one re-crystallisation from benzene.

The results obtained with the various solutions containing *p*-chloro-*m*-xylenol, as given in Table I, are shown in Table II.

TABLE I

SOLUTIONS PREPARED CONTAINING *p*-CHLORO-*m*-XYLENOL

	I	II	III	IV	V	VI
p-Chloro-*m*-xylenol (g.) ..	2·491	3·500	3·318	3·078	3·507	3·339
Pine oil (*Palustris*) (ml.) ..	—	2	1	—	1	1
Sassafrassy camphor oil (ml.) ..	—	1	—	—	2	1
Phellandrene (ml.)	—	—	1	1	—	2
Ti-tree oil (ml.)	3	—	0·5	2	1	1
Terpineol (ml.)	—	—	7·5	6	3	4
Castor oil fatty acids* (ml.) ..	10	6	9	—	—	—
Potassium hydroxide (10 per cent.) (ml.)..	—	10	15	—	—	—
Triethanolamine (ml.)	10	—	—	—	—	—
Potassium soap of olive oil (g.)..	—	—	—	—	15	—
Potassium soap of palm kernel oil (g.)	—	—	—	—	—	15
Potassium soap of coconut oil (g.)	—	—	—	15	—	—
Ethyl alcohol (ml.)	—	10	15	—	15	15
Isopropyl alcohol (ml.)	10	—	—	10	—	—
Water	to 100 ml.	to 100 ml.	to 100 ml.	to 100 ml.	to 100 ml.	to 100 ml.

* Prepared by saponifying castor oil with excess of potassium hydroxide, dissolving the soap in water and acidifying with hydrochloric acid.

It was found that with solutions containing a high proportion of essential oils the phenolic residue, as prepared for weighing, sometimes contained a small amount of the oil. This was removed by washing the crude phenol twice with

10 ml. of petroleum spirit. The petroleum spirit solution was evaporated to dryness, and the residue was treated with 30 ml. of 1·7 per cent. sodium hydroxide solution and washed into a separator with 10 ml. of water. The solution was extracted twice with 10 ml. of petroleum spirit, and the extract was washed twice with 10 ml. of water. The small amount of phenol in the combined aqueous liquor was liberated with hydrochloric acid and extracted with ether, and the ethereal solution was dried in the usual way. This was added to the washed phenol residue, the ether was removed, and the *p*-chloro-*m*-xylenol was dried before being weighed, as described in the general method.

TABLE II

ANALYSIS OF SOLUTIONS IN TABLE I

Solution No.	*p*-Chloro-*m*-xylenol: Added g. in 100 ml.	*p*-Chloro-*m*-xylenol: Determined g. in 100 ml.	m.p. °C.	m.p. after re-crystallisation from benzene, °C.
I	2·491	2·465	107–109	114–115
II	3·500	3·442	109–110	114–115
III	3·318	3·192	108–109	114–115
IV	3·078	2·805	107–108	114–115
V	3·507	3·501	110–111	114–115
VI	3·339	3·343	108–110	114–115

We wish to record our indebtedness to the Directors of Stafford Allen & Sons, Ltd., for permission to publish this account.

REFERENCES

1. *Pharm. J.*, 1936, **137**, 273.
2. W. R. Heading, *Pharm. J.*, 1937, **138**, 321.
3. N. F. Rapps, *J. Soc. Chem. Ind.*, 1933, **52**, 175T.

THE RESEARCH LABORATORY
STAFFORD ALLEN & SONS, LTD.
WHARF ROAD, LONDON, N.1

November 20th, 1937

The Determination of Ethylene Glycol

BY R. CUTHILL, M.SC., PH.D., A.I.C., AND C. ATKINS, PH.D., A.I.C.

I. INTRODUCTION.—Whilst the determination of glycerol has been the object of extensive investigation, and various more or less trustworthy methods of determination have been described, the literature contains few references to the determination of ethylene glycol. It is the purpose of the present note to describe a new and convenient method of determination.

According to Müller,[1] glycol in concentrated solution may be determined by the acetin method of Benedikt and Cantor.[2] Oxidation by boiling with potassium dichromate, as in the Hehner–Steinfels method for the determination of glycerol, also gives satisfactory results. Modifications of this procedure consist in heating with a mixture of sulphuric acid and chromium trioxide and weighing the carbon

dioxide formed. Shutt and Mack[3] also describe a method for the determination of glycol by heating with dichromate solution. Recently Kedrinskii and Skornyakova[4] have stated that the potassium dichromate process is superior to determination of density as a means of determining glycol.

Various other oxidimetric methods of determination have been proposed in recent years. Malaprade[5] found that glycol could be determined volumetrically by means of periodic acid. Fleury and Marque[6] have suggested the use of an alkaline solution of potassium iodomercurate for a similar purpose. Criegee[7] observed that various glycols could be quantitatively oxidised with a solution of lead tetra-acetate in glacial acetic acid, but it proved impracticable to utilise this reaction as a means of determining ethylene glycol.

II. Determination with Permanganate.—Müller[1] states that the oxidation of glycol with permanganate in alkaline solution is incomplete, even after a long time and in presence of considerable excess of permanganate. We, however, were successful in working out a method for the determination of glycol with permanganate solution. The procedure ultimately adopted was as follows:—A mixture of 50 ml. of 0·1 *N* potassium permanganate solution, 10 ml. of approximately 0·025 *M* glycol solution, and 30 ml. of 4 *N* sodium hydroxide solution is allowed to stand for 1½ hours, after which 50 ml. of 4 *N* sulphuric acid are added, and the mixture is allowed to stand for a further hour. Finally, 10 ml. of 10 per cent. potassium iodide solution are added, and the iodine liberated is titrated with thiosulphate. Under these conditions glycol reacts with 5 atoms of oxygen, corresponding with complete oxidation to carbon dioxide and water. It is necessary also to make a blank experiment, omitting the glycol. The results described below were obtained with a sample of glycol prepared by re-distilling a good commercial sample three times under reduced pressure, the head and tail fractions in each distillation being rejected.

Variation of Time of Keeping in Alkaline Solution.—Mixtures of 50 ml. of 0·1 *N* potassium permanganate solution, 10 ml. of a glycol solution containing 1·647 g. per litre, and 30 ml. of 4 *N* sodium hydroxide solution were kept for varying periods; 10 ml. of 10 per cent. potassium iodide solution and 50 ml. of 4 *N* sulphuric acid were then added, and the iodine liberated was titrated.

Time of keeping Minutes	Atoms of oxygen used per mol. of glycol
15	2·639
30	3·082
60	3·579
90	3·982
120	4·010
180	4·023

It may be inferred from these results that in 1½ hours glycol reacts with 4 atoms of oxygen, corresponding with oxidation to oxalic acid. As, however, the extent of reaction varies appreciably with the time, attempts were made to discover whether by acidifying the permanganate and keeping the mixture for a further period before titration the glycol might be oxidised completely to carbon dioxide and water.

VARIATION OF TIME OF KEEPING IN ACID SOLUTION.—Mixtures of 50 ml. of 0·1 *N* potassium permanganate solution, 10 ml. of the above-mentioned glycol solution, and 30 ml. of 4 *N* sodium hydroxide solution were left for 1½ hours. They were then acidified with 50 ml. of 4 *N* sulphuric acid, and kept for further varying periods. Finally, 10 ml. of 10 per cent. potassium iodide solution were added and the iodine set free was titrated.

Time of keeping, Minutes	Atoms of oxygen used per mol. of glycol
7½	4·923
15	4·938
30	5·000
60	5·000
90	5·000
120	5·000

From these results it would appear that keeping for 30 minutes after acidification is sufficient for complete oxidation. Experiments with more dilute solutions of glycol, however, showed that it was desirable to extend this period to 60 minutes.

VARIATION OF GLYCOL CONCENTRATION.—Mixtures of 50 ml. of 0·1 *N* potassium permanganate solution, 10 ml. of glycol solution, and 30 ml. of 4 *N* sodium hydroxide solution were kept for 1½ hours, then acidified with 50 ml. of 4 *N* sulphuric acid, and kept for a further hour. Potassium iodide was then added, and the iodine liberated was titrated.

Amount of glycol present g. per litre	Amount of glycol found g. per litre	Percentage
2·108	2·104	99·82
1·647	1·647	100·00
1·318	1·320	100·15
0·8235	0·8235	100·00
0·4118	0·4118	100·00
0·1647	0·1645	99·89

These figures show that the method gives good results over a wide range of glycol concentrations.

Our thanks are due to the Bradford City Council for a grant which has made possible the carrying out of this work.

REFERENCES

1. B. Müller, *Chem.-Ztg.*, 1920, **44**, 513.
2. R. Benedikt and M. Cantor, *Monatsh.*, 1888, **9**, 522.
3. R. S. Shutt and E. Mack, *Ind. Eng. Chem.*, 1933, **25**, 687.
4. V. V. Kedrinskii and V. F. Skornyakova, *Reports on Cracking and Chemical Treatment of Products* (Leningrad), 1935, No. 2, 201.
5. M. L. Malaprade, *Bull. Soc. Chim.*, 1928, [iv], **43**, 683.
6. P. Fleury and J. Marque, *J. Pharm. Chim.*, 1929, [viii], **10**, 241.
7. R. Criegee, *Ber.*, 1931, **64**, 265.

PHYSICAL CHEMISTRY LABORATORY
THE TECHNICAL COLLEGE
BRADFORD

February 15th, 1938

Notes

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

VARIATIONS IN THE COMPOSITION OF BLACKCURRANTS

THE published literature on the variations in the constituents of blackcurrants is scanty, being mainly confined to publications by Mansfield (ANALYST, 1927, **52**, 351), by Macara (ANALYST, 1931, **56**, 35), by Hughes and Maunsell (ANALYST, 1934, **59**, 231), and by Hinton (ANALYST, 1934, **59**, 248), all of whom record values obtained over a very limited period of time.

In view of the suggestions that the composition of blackcurrants varies from season to season, and that the country of origin has some effect, at least on the major components, I have made a series of determinations of insoluble matter, total solids, sugars and soluble solids in fruit (not pulp) from Holland, France and England, and also on a few samples of blackcurrant pulp from Canada. These analyses have been made from 1932 until 1937, and show that during the period of observation there has been a general tendency for the insoluble solids content of blackcurrants, from the above-mentioned sources, to decrease until 1936. During the season 1937 a slight increase in this constituent was shown.

The results also showed that, speaking generally, blackcurrants containing a high percentage of soluble solids contain a proportionately low percentage of insoluble solids.

DUTCH BLACKCURRANTS

	SEASON 1932 Insoluble solids Per Cent.	SEASON 1933 Insoluble solids Per Cent.
Maximum	4·82	5·92
Minimum	4·5	4·12
Average	4·62	4·83
Number of samples	5	24

	SEASON 1934 Total solids Per Cent.	Insoluble solids Per Cent.	Soluble solids Per Cent.	Sugars Per Cent.	Decrease of insoluble solids from 1933 figures Per Cent.*
Maximum	16·7	5·67	12·22	7·39	—
Minimum	13·34	3·77	8·48	5·34	8·5
Average	15·53	4·69	10·68	6·64	3
Number of samples ..	18	—	—	—	—

	SEASON 1935 Total solids Per Cent.	Insoluble solids Per Cent.	Soluble solids Per Cent.	Sugars Per Cent.	Decrease of insoluble solids from 1933 figures Per Cent.*
Maximum	18·85	5·26	13·91	8·37	—
Minimum	13·78	3·71	10·07	4·08	9·95
Average	16·13	4·28	11·85	6·3	11·4
Number of samples ..	24	—	—	23	—

DUTCH BLACKCURRANTS—*continued*

SEASON 1936

	Total solids Per Cent.	Insoluble solids Per Cent.	Soluble solids Per Cent.	Sugars Per Cent.	Decrease of insoluble solids from 1933 figures Per Cent.*
Maximum	17·34	4·42	13·45	7·01	—
Minimum	11·46	3·5	8·16	4·22	15·05
Average	13·73	4·1	9·76	5·13	15·1
Number of samples..	27	25	25	22	—

SEASON 1937

	Total solids Per Cent.	Insoluble solids Per Cent.	Soluble solids Per Cent.	Sugars Per Cent.	Free acid (as anhydrous citric acid) Per Cent.	Combined acid (as anhydrous citric acid) Per Cent.
Maximum	17·74	5·36	12·85	6·77	3·24	0·532
Minimum	14·17	3·99	9·76	3·41	2·42	0·276
Average	16·03	4·75	11·3	4·84	2·88	0·435
Number of samples..	28	—	—	—	—	—

* Per cent. of the 1933 figures for insoluble solids.

FRENCH BLACKCURRANTS

	SEASON 1933 Insoluble solids Per Cent.	SEASON 1934 Total solids Per Cent.	Insoluble solids Per Cent.	Soluble solids Per Cent.	Sugars Per Cent.
Maximum	5·0	17·81	5·56	12·25	—
Minimum	4·5	14·58	4·64	9·31	—
Average	4·76	16·44	5·23	11·21	6·51
Number of samples..	5	5	—	—	3

SEASON 1935

	Total solids Per Cent.	Insoluble solids Per Cent.	Soluble solids Per Cent.	Sugars Per Cent.	Decrease of insoluble solids from 1934 figures Per Cent.*
Maximum	18·1	5·38	13·88	7·23	—
Minimum	14·32	4·22	9·21	4·27	9
Average	16·78	4·91	11·15	5·25	6·1
Number of samples..	7	—	—	—	—

SEASON 1936

	Total solids Per Cent.	Insoluble solids Per Cent.	Soluble solids Per Cent.	Sugars Per Cent.	Decrease of insoluble solids from 1934 figures Per Cent.*
Maximum	17·92	5·25	12·59	6·48	—
Minimum	13·2	3·75	9·32	3·72	19·2
Average	15·21	4·29	10·95	5·31	18·0
Number of samples..	13	—	—	—	—

SEASON 1937

	Total solids Per Cent.	Insoluble solids Per Cent.	Soluble solids Per Cent.	Sugars Per Cent.	Free acid (as anhydrous citric acid) Per Cent.	Combined acid (as anhydrous citric acid) Per Cent.
Maximum	19·22	6·08	13·73	7·01	3·29	0·55
Minimum	13·1	4·09	9·01	3·44	2·29	0·335
Average	16·74	5·09	11·32	5·45	2·95	0·436
Number of samples..	9	—	—	7	9	9

* Per cent. of the 1934 figures for insoluble solids

English Blackcurrants

Season 1934

	Total solids Per Cent.	Insoluble solids Per Cent.	Soluble solids Per Cent.	Sugars Per Cent.	Free acid (as anhydrous citric acid) Per Cent.
Maximum	21·75	7·67	15·42	8·69	2·97
Minimum	17·95	6·02	10·28	7·54	1·95
Average	20·26	6·71	13·54	8·16	2·56
Number of samples..	5	—	—	—	—

Season 1936

	Total solids Per Cent.	Insoluble solids Per Cent.	Soluble solids Per Cent.	Sugars Per Cent.
Maximum	18·64	6·26	13·36	5·26
Minimum	15·79	4·72	10·68	3·6
Average	17·12	5·4	11·61	4·11
Number of samples ..	14	—	—	—

Canadian Blackcurrant Pulp

Season 1936

(This pulp is reputed to contain 90 per cent. of fruit)

	Total solids Per Cent.	Insoluble solids Per Cent.	Soluble solids Per Cent.	Sugars Per Cent.
Maximum	14·96	4·22	8·95	5·09
Minimum	11·54	3·99	7·48	2·15
Average	12·8	4·09	8·28	4·16
Number of samples ..	7	—	—	—

Thus, during the years 1933 to 1936 Dutch blackcurrants showed a progressive decrease in insoluble solids content, amounting to approximately 15 per cent. over the period.

In the period 1934 to 1936 French blackcurrants showed a decrease of approximately 19·5 per cent. in insoluble solids content, and with English blackcurrants the decrease amounted to 19·5 per cent. in 1936, as compared with 1934.

It would greatly assist the analyst in his interpretation of analytical results if a scheme were established whereby the collective data relating to the major constituents of all types of fruit used for the production of jam under the agreed standards could be published annually.

J. F. Brown

Scottish Co-operative Wholesale Society, Ltd., Shieldhall Glasgow, S.W.1

THE DETERMINATION OF MECHANICAL WOOD PULP IN PAPER

The criticism of the Halse method for the determination of mechanical wood pulp in paper put forward by Aiyar and Krishnan (Analyst, 1937, **62**, 713) is somewhat misleading as it is not strictly correct in two respects:—(1) The Halse method is intended for newsprint consisting of mechanical and unbleached sulphite wood pulp, with or without filler (china clay). (2) The correction from "ash" (residue on ignition) to "filler" has not been applied by Aiyar and Krishnan to obtain the true pure lignin figure.

Under the first clause, samples numbered 1, 11 and 12 are unsuitable for the Halse treatment, being art papers, whilst Nos. 13 and 14 do not necessarily contain

unbleached sulphite and mechanical wood pulp alone. Only the percentage of the latter constituent appears to have been standardised.

The second point, the omission of the ash correction, has led to the entirely erroneous result given for sample No. 1, and it is also responsible for larger apparent differences between the Halse results and those obtained by the official microscope method than a corrected calculation indicates.

The results given in Aiyar and Krishnan's Table III have been re-calculated on the bone-dry ash-free basis, using factors for the two kinds of pulp only slightly different from those adopted by the authors, and making the necessary allowance for the moisture associated with the ash in its original state as filler.

The assumptions in this re-calculation were:—(1) The lignin-content of the mechanical wood pulp was 29·6 per cent., and that of the sulphite pulp 3·35 per cent. These figures are based on a large number of determinations, and agree very closely with those of Halse when the latter are calculated on the bone-dry basis. (2) The filler was china clay, so that the factor used for converting the weight of ash to that of filler was 1·13.

The effect of the first assumption was negligible, but the conversion of ash to filler made quite an appreciable difference in the results. It should be mentioned that the ash figures used for the calculation were those given by Aiyar and Krishnan for the original papers. This had to be done because the weights of acid-treated ash were not available in the published account. The weight of the residue after ignition is almost invariably lower than that of the ash in the paper, owing to the acid attack. This would mean a slightly higher "pure lignin" figure, and therefore a slight rise in the mechanical wood percentage. The differences between the revised and the microscope results in Table III are nearly all negative. Hence the use of the acid-treated ash figures for correction would lead to a smaller difference in nearly every instance.

TABLE III (REVISED)

THE PERCENTAGE OF MECHANICAL WOOD PULP IN PAPER

No. of paper	Average microscopic count (A)	Halse formula (B)	Halse formula after correcting ash to filler (C)	Difference between A and B	Difference between A and C
1	0·5	20·3	— 3·2	+19·8	— 3·7
2	71·3	79·3	72·9	+ 8·0	+ 1·6
3	75·0	79·9	72·0	+ 4·9	— 3·0
4	73·6	78·5	71·9	+ 4·9	— 1·7
5	75·8	76·5	74·4	+ 0·7	— 1·4
6	89·3	92·5	91·5	+ 3·2	+ 2·2
7	67·0	59·8	58·7	— 7·2	— 8·3
8	76·3	78·8	71·2	+ 2·5	— 5·1
9	74·9	82·9	69·4	+ 8·0	— 5·5
10	72·1	75·6	68·1	+ 3·5	— 4·0
11	76·6	86·4	61·3	+ 9·8	—15·3
12	58·8	78·4	52·1	+19·6	— 6·7
13	61·1	53·6	51·8	— 7·5	— 9·3
14	50·7	42·5	41·3	— 8·2	— 9·4
	Average difference (Nos. 1–14) . .			7·7	5·5
	Average difference (Nos. 2–10) . .			4·8	3·6
	Average difference (Nos. 2–10, omitting No. 7)			4·5	3·1

The revised table shows that the average difference between the results obtained microscopically and those by lignin determination is smaller with the

revised calculations, especially when papers which are obviously not newsprint are omitted. As stated above, the comparison would be even more favourable if the acid-treated ash figures were available.

It is interesting to note that if the standard papers Nos. 13 and 14, which are stated to contain 60 per cent. and 50 per cent. of mechanical wood pulp respectively, are assumed to have bleached sulphite pulp as the other constituent, the calculated percentages of groundwood become 56·4 and 46·9. These values are much nearer the correct figures and are based on a lignin-content of 0·57 per cent. of the bleached sulphite pulp.

S. V. SERGEANT

BOWATER'S PAPER MILLS
NORTHFLEET, GRAVESEND
KENT

SOME NEW FLUORESCENCE TESTS

TEST TO DISTINGUISH VISCOSE FROM BEMBERG SILK.—Viscose and Bemberg silk may be readily distinguished in ultra-violet light by the use of the following reagent:—Acronol yellow TS (0·2 g.) is dissolved in 50 ml. of alcohol and the solution is mixed with a solution of 0·5 g. of anhydrous sodium carbonate in 50 ml. of water, heated to boiling and allowed to cool. The material under examination is immersed for a few seconds in the reagent, then removed, washed with cold or warm water, and examined in filtered ultra-violet light. Bemberg silk fluoresces with a brilliant bluish colour, whilst viscose appears a very dull blue. Cellulose acetate treated in this way shows a luminous violet-blue fluorescence.

Another means of distinguishing between the two materials is to dye both for 5 minutes at 85° C., in a 1 per cent. solution of the dyestuff Primuline AS in 5 per cent. sulphuric acid. The ratio of the weight of dye liquor to that of the cloth should be approximately 30 to 1 and, after dyeing, the materials are rinsed in cold water, dried and examined under the ultra-violet lamp. Viscose appears a brilliant bluish-white, whilst Bemberg silk appears a vivid canary-yellow.

ALUMINIUM IN SIZING MATERIAL AND ADHESIVES.—A test for aluminium, sensitive to 1 part in 10^5, in sizing materials, adhesives, and so on, has been based on the vivid orange-red fluorescences obtained with aqueous alcoholic solutions of Solochrome Red ERS and Solochrome Violet RS. A portion of the material, weighing 2–3 g., is treated with 2 to 3 ml. of water and, if alkaline in reaction, is neutralised with conc. hydrochloric acid; a slight excess of acid does not interfere with the test. The contents of the tube are heated to 100° C. on the water-bath for 5 minutes and cooled, 1 ml. of a 0·02 per cent. solution of one of the above-mentioned Solochrome colours is added, and the contents of the tube are again heated for 5 minutes on the water-bath. After cooling, 10 ml. of alcohol are added, the tube is shaken and its contents are filtered, and the filtrate is examined under the ultra-violet lamp; a brilliant reddish-orange fluorescence indicates the presence of aluminium. Magnesium, calcium, sodium, potassium or ammonium ions do not interfere with the reaction. Zinc ions give a dull red fluorescence with both dyestuffs, but the brilliant orange-red fluorescence given by aluminium salts is not masked by the zinc fluorescence, even when zinc ions are present in great preponderance. These tests are therefore suitable for the detection of aluminium salts in sized materials or of aluminium soaps precipitated on fabrics.

I should like to thank the Directors of Imperial Chemical Industries, Ltd. (Dyestuffs Group), in whose laboratory this work was carried out, for their permission to publish this note.

J. A. RADLEY

11, WILTON ROAD, CRUMSALL
MANCHESTER, 8

Official Appointments

THE Minister of Health has approved the following appointments:

ERIC VOELCKER as Public Analyst for the County of Oxford, in place of J. Augustus Voelcker (deceased) (March 2nd, 1938).

REVERSAL OF OFFICES

HUGH CHARLES LOUDON BLOXAM as Public Analyst for the County Borough of Newcastle upon Tyne, in place of J. T. Dunn, who becomes Additional Public Analyst, the reversal of offices being due to the advancing age of Dr. Dunn.

The Minister of Agriculture and Fisheries has approved the following appointments as Agricultural Analyst:

ERIC VOELCKER	for	the	County	of		Buckingham (Dec., 1937).
,,	,,	,,	,,	,,	,,	East Riding of York (Jan. 26th, 1938).
,,	,,	,,	,,	,,	,,	Isle of Ely (Dec., 1937).
,,	,,	,,	,,	,,	,,	Middlesex (Jan. 12th, 1938).
,,	,,	,,	,,	,,	,,	Northampton (Jan. 25th, 1938).
,,	,,	,,	,,	,,	,,	Oxford (Feb., 1938).

Notes from the Reports of Public Analysts

The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of special interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

ROYAL BOROUGH OF KENSINGTON

REPORT OF THE PUBLIC ANALYST FOR THE FOURTH QUARTER, 1937

TABLE CREAM.—A sample sold as "table cream" was reported adulterated. It consisted, not of cream, but of a vanilla-flavoured custard powder. The name "table cream" is applied to cream of rather low fat-content, just as is the term "coffee cream," and the dairy trade may be said to have established a right to the use of such a name. Its application to a custard powder is, in my opinion, wholly unwarrantable; it is a particularly glaring example of deceptive labelling.

LEAD IN CURRY POWDER.—Samples of two curry powders were reported adulterated because of the presence of lead; the amounts were 24 and 30 parts per million respectively. A third specimen also contained lead, but the amount found was considerably below twenty parts per million. From an official source it was learnt that the presence of lead in this article of food is due to the treatment of the turmeric root, which in the ground state is one of the ingredients, with chromate of lead, so as to give it a fictitious appearance of high quality. The presence of chromium in these samples could not be demonstrated; this may have been due to a compound of lead other than the chromate having been employed as adulterant.

COFFEE EXTRACT.—Four samples, reported adulterated, were sold as "Pure Coffee Extract," but contained only about 4 per cent. of dry coffee extractives and about 14 per cent. of extraneous matter (other than sugars) not derived from coffee. In my experience pure coffee extracts (liquid) contain over 15 per cent. of dry coffee extractives, and therefore a very low standard of 10 per cent. was

suggested in my report. It seems most unfair that such an article, the major constituent of which is presumably burnt sugar, and which contains only about 4 per cent. of the extractives of coffee, should be allowed to compete on the market with genuine extracts and should be allowed to be described as *pure* coffee extract. The labels made anything but modest pretentions to excellence, but no notice was given of the presence of burnt sugar or of anything else than coffee.

F. W. EDWARDS

COUNTY OF KENT

REPORTS OF THE COUNTY ANALYST FOR THE FOURTH QUARTER, 1937

CHEWING GUM.—A sample of chewing gum that was thought to have caused illness amongst children was examined. Although the sales of chewing gum in this country in no way approach those in the United States, the amount of gum retailed is probably higher than is generally thought, and consumption is steadily growing. At one time a purified paraffin wax was used in this product, but now a wide variety of substances finds its way into the gum. Most chewing gum is prepared from the chicle or sapotilla tree, native to Mexico; in fact, the use of chewing gum is said to have spread through its use by Mexicans. As the tree grows only in very restricted areas, the amount of gum produced is very limited and is almost entirely absorbed by America. The difficulty of obtaining chicle has led to a variety of substitutes being either suggested or used, and among these is raw rubber which has been treated and mixed with resins, waxes, etc. It could not be certified that the chewing gum submitted had been the cause of illness.

FERTILISER WASTES.—*Feather Wastes.*—The two samples examined were both of poor quality, owing to their grit-content. It does not seem easy to account for the amount of earthy matter which is often found in feather wastes, but so frequently do large quantities occur that it becomes important to buy these wastes only with a warranty or on sample.

Rabbit Fur Wastes were almost all of rather poor quality, owing generally to the quantity of paws they contained, although sand occurred in one or two cases. Three fur wastes below warranty were inferior, owing to the presence of mineral matter in one instance, to paws and ears in another, and in the third to excessive water, which exceeded 28 per cent.

Hair Mixture.—A sample consisted of hair with large pieces of bone and hoof. Some of the bone and hoof was of no manurial value at all, and although the nitrogen-content of the mixture amounted to less than 9 per cent. and phosphoric acid to 10 per cent., the farmer was quite willing to pay £8 10s. 0d. per ton for it. Another manure which changed hands at rather more than £1 0s. 0d. per unit of nitrogen was that of slaughterhouse refuse. This refuse was certainly in good condition, but it contained only 7 per cent. of nitrogen and 54 per cent. of organic matter.

Ground Town Refuse has again been freely offered. Town refuse seldom contains any material proportion of a constituent of manurial value, sometimes, in fact, it is chiefly valuable on account of its lime-content, though this is low. The sample in question contained 0·7 per cent. of nitrogen, 0·5 per cent. of phosphoric acid, 0·4 per cent. of potash and only 14·2 per cent. of organic matter. Often the use of town refuse is advocated as an "organic manure," and it has been so described in literature, but much of the so-called organic matter consists only of the carbon contained in cinders.

F. W. F. ARNAUD

South Africa

DIVISION OF CHEMICAL SERVICES

ANNUAL REPORT OF THE CHEMICAL SERVICES OF THE STATE FOR 1936

THE Division of Chemical Services (which is under the direction of Dr. J. P. van Zyl) is intended, as its name implies, to render chemical services to all Government Departments that require them. The most important departments from this point of view are Agriculture and Forestry (with its various divisions), Public Health, Justice, Irrigation, Customs and Excise, Mines and Interior. The work on behalf of private persons is restricted to (*a*) minerals on behalf of prospectors, (*b*) agricultural materials for *bone-fide* farmers, and (*c*) water for domestic purposes, for the general public.

The samples examined included 30,000 in connection with soil surveys, 15,000 of butter, wine and fruit for the Department of Agriculture and Forestry, 5000 of foods, drugs, disinfectants, etc., for the Department of Public Health, and 1100 for the Ministry of Justice.

FOOD ADULTERATION.—Of the 3653 samples of milk examined, 397 (10·9 per cent.) were adulterated or deficient in fat or non-fatty solids; of 318 samples of minced meat, sausages, etc., 101 were adulterated, mainly in that they contained a prohibited preservative; 39 of the 124 samples of ice-cream were reported as adulterated, usually because of deficiency in fat.

MICRO-BIOLOGICAL PROBLEMS.—A comprehensive field experiment is being conducted on exhausted established pasture land in the Natal mist belt, in order to discover how such land can best be handled with a view to restoring its fertility. It is hoped that by ploughing up the land and applying treatments to promote the oxidation processes, large quantities of organic matter, which at present appear to be inert, will be oxidised, thus setting free available plant nutrients when the land is re-sown for grass.

Nodule Bacteria in Clover.—Considerable success has attended attempts to isolate more vigorous strains of nodule bacteria from indigenous clovers. Inoculation of imported clovers with these strains under different field conditions may point the way to the more successful use of legumes in the best grassland areas. Up to the present, the need for introducing suitable clovers has been keenly felt, but the problem of establishing them has proved difficult. It is hoped that by studying the influence of lime, phosphatic and other fertilisers on the growth of legumes under various field treatments, this problem will be brought nearer solution.

FOOD AND NUTRITION.—Work was continued with a view to defining standards for the most important constituents of cereals and cereal meals, particular attention being paid to maize and the products prepared therefrom. Owing to the fact that certain of the constituents of meals are separated by the millers and sold as by-products, there has always been a doubt as to the variability of the meal left, as well as of the by-products, and no reliable information was available as to their food-value.

Cereals, cereal products and by-products have been collected from nearly all the mills throughout the Transvaal and Orange Free State. The samples included maize of all the principal grades and mixtures, maize meals, straight-run—No. 1, refined, granulated—maize flour, samp, mealie rice, grits, germ meal, hominy chop, maize seconds, bran and cow meal; wheat of different types, wheat products, unsifted boer meal, flour, bran, pollard, and wheat germ; kaffir corn, kaffircorn meal and flour; oats, oatmeal, rolled oats and oat husks; rye, rye meal and flour. The work aims at determining to what extent the names actually represent the

materials supplied, and how far the milling process in South Africa is effective. The work entailed the analysis of some 150 samples of various cereals.

SULPHUR IN TOBACCO.—Special attention was again given to the use of sulphur in combating mildew in tobacco. Since the tobacco trade has been rejecting large parcels of tobacco because of excess sulphur-content, these investigations were mainly directed towards determining the lowest sulphur dosages that can be used in controlling mildew disease. The effect of certain variations in flue-curing technique is also being investigated as a means of reducing sulphur in tobacco leaves.

FLUORINE IN INSECTICIDES.—Large-scale experiments are being carried out in co-operation with the Division of Plant Industry with a view to determining to what extent fluorine is absorbed by citrus fruit as a result of such substances as cryolite and sodium fluosilicate being used as insecticides.

Report on the Preservative Principles of Hops

QUANTITATIVE STUDIES OF THE CHANGES IN PRESERVATIVE VALUE DURING THE BOILING AND FERMENTATION OF HOPPED WORTS*

HUMULON, the principal antiseptic constituent of hops, was boiled in wort under standard conditions simulating those in a brewery, and the following general conclusions were drawn from the experiments:—(1) The rate of loss of preservative value (P.V.) of humulon is greatest in the early stages of boiling. (2) Increase in the rate of boiling (*i.e.* rate of evaporation) is accompanied by increase in the rate of destruction of P.V. (3) At both high and low boiling rates the rate of destruction of P.V. gradually decreases as boiling proceeds, until the loss of P.V. may be more than compensated by concentration (by boiling) of the wort, and the P.V. per unit volume increases. (4) Over the normal range of *p*H in brewery worts the *p*H has not a great influence on the P.V., but the initial *p*H affects the solubility of humulon and participates in controlling the change to humulon transformation products. (5) Under normal brewery conditions there is a loss of 50 per cent. in the P.V. (due to humulon) in the first two hours of boiling, and, since there is an immediate loss of 40 to 60 per cent. of P.V. on adding humulon to wort, owing to retention by wort colloids, it follows that after 2 hours' boiling there remains only about 25 per cent. of the original P.V. (6) When humulon is boiled in water adsorption and other retention effects are absent, but the rate of destruction of P.V. is appreciably greater than in wort, and there is a greater mechanical loss owing to the adhesion of humulon aggregates on the sides of the vessel.

The humulon used in these experiments was obtained from the lead salt by the Ford and Tait process. The preservative value was determined by the log-phase method, using *L. bulgaricus*, titrating the acid produced, and calculating from the expression:—Percentage restriction of acid formation + percentage concentration of antiseptic = a constant.

The Report includes an appendix in which modern work on the antiseptic constituents of hops is reviewed. The inter-relationship of the hop resin constituents and the nomenclature of these substances are stated as follows:—An ethereal extract of hops contains (*a*) humulon (sometimes called α-bitter acid); (*b*) lupulon (sometimes called β-bitter acid); (*c*) soft resins, consisting partly of the oxidation products of (*a*) and (*b*); (*d*) hard resins, which represent a more advanced stage of oxidation than the soft resins. Petroleum spirit dissolves (*a*), (*b*) and (*c*).

* Sections I and II of Part XIX. By T. K. Walker, *J. Inst. Brewing*, 1938, **44**, 11–28.

When humulon is boiled for 2 hours in borate buffer solution at pH = 9·6, and also at 8·6, the whole is converted into α-soft resins; at pH = 7·0, 23 per cent., and at pH = 6·5, 18 per cent. is converted; as the solution becomes more acid more is then converted. The relative solubilities of humulon and lupulon in phosphate buffered solution of varying pH, as determined by Wöllmer and Kolbach, are given, from which it appears that only 14 mg. of humulon can dissolve to form a clear solution in 1 litre of beer of average pH (*viz.* pH = 4·3) and no lupulon; it follows, therefore, that the preservative material in beer consists principally of resinous transformation products of humulon and, to a less extent, of resinous transformation products of lupulon formed during the boiling of the hopped wort. D. R. W.

Imperial Institute

ANNUAL REPORT FOR THE YEAR 1937

In the past year the Institute has celebrated its jubilee, and in a foreword to the Report, Sir Harry Lindsay, the Director, surveys many developments. The Advisory Council on Minerals, under the chairmanship of Sir William Larke, has reorganised and re-named the old Advisory Technical Committees. These, formerly fifteen in number, are now only seven, *viz.* The Mining Law Technical Committee and the Consultative Committees on Precious Metals, Base Metals, Coal and Petroleum, Iron and Ferro-Alloy Metals, Chemical Industries and Miscellaneous Minerals.

The Advisory Council on Plant and Animal Products, whose Chairman is Sir Frank Stockdale, has also revised what were formerly known as Advisory Technical Committees, and are now Consultative Committees eight in number; these deal with Timbers, Hides and Skins, Tanning Materials, Vegetable Fibres, Oils and Oil-seeds, Essential Oils, Gums and Resins, and Insecticide materials of vegetable origin, in addition to two sub-committees, one on Grading Rules and Standard Sizes for Empire Hardwoods, and one on Tung Oil.

The number of technical enquiries dealt with continued to rise, the total of 1101 representing an increase of nearly 19 per cent. over that of 1936, and more than 38 per cent. above the 1935 total. As usual, the enquiries were of an extremely varied nature and came from almost every country of the Empire.

Laboratory investigations involved the examination of 317 samples from 30 countries, including such varied materials as Nyasaland waters, South African quartzite, East African soils, Nigerian tin and columbite concentrates, Tanganyika vermiculite, and numerous samples from the Gold Coast, including one very interesting mineral which has hitherto only been recorded from Brazil.

Plant and Animal Products.—The Committee on Timbers issued towards the close of the year a second edition of the "Grading Rules and Standard Sizes for Empire Hardwoods." It also dealt with the possibility of marketing Newfoundland birch in this country, and with certain fire-resisting timbers. Subjects considered by the Committee on Vegetable Fibres included comparative tests of tarred and untarred sisal cordage and developments in the phormium industry of New Zealand. The Sub-Committee on Tung Oil gave attention to the preparation of tung oil from the fruits, to samples of tung fruits grown experimentally in East and South Africa, and to the practicability of increasing the production of perilla oil in the Empire.

The Committee on Hides and Skins approved for publication a monograph on "The Preparation of Empire Hides and Skins," and also gave consideration to the certification of hides and skins by stamping, to denote the method of preparation, and to the improvement of goat and sheep skins in Gambia, Nigeria and East Africa.

The Committee on Essential Oils, which was reconstituted during the year, dealt, among other subjects, with supplies of *Eucalyptus dives* oil, the standardisation of Seychelles essential oils, and with various oils from East Africa and elsewhere.

The Committee on Insecticide Materials of Vegetable Origin decided that a monograph surveying the production of insecticide materials and the possibility of extension within the Empire should be prepared, and a Sub-Committee for the purpose was appointed. Arrangements were also made for the compilation of a quarterly bibliography of recent literature on insecticide materials of vegetable origin.

Details are given of the numerous raw materials investigated. These included the following:

COMBRETUM GUM FROM TANGANYIKA.—The sample examined was an ungraded gum obtained from trees of a species of *Combretum* growing in the Tabora District, and consisted largely of small tears, nearly colourless to dark reddish-brown in colour. The gum was mainly of the insoluble type, but a 10 per cent. mixture with water gave a jelly much less stiff in consistence than that produced by tragacanth.

DANIELLA OLEO-RESIN FROM NIGERIA.—This material, obtained by tapping *Daniella oliveri* trees, was a somewhat viscous product, yielding about the same amount of volatile oil as commercial varieties of copaiba. The material, however, did not conform to the requirements of the B.P. for copaiba oil, which specifies that the product must be derived from *Copaifera* spp., and also that it must yield a laevorotatory oil, whereas the Daniella oil was strongly dextrorotatory.

SUGAR-CANE WAX.—South African samples of two grades of sugar-cane wax at present being marketed were examined. It was suggested that producers should endeavour to prepare a hard and light-coloured material more likely to compete with beeswax and other waxes.

TEPHROSIA LEAVES AS AN INSECTICIDE.—Three samples of *Tephrosia* leaves from Uganda were subjected to insecticidal tests at Rothamsted. The results showed that, although one of the samples was rather more toxic than the other two, none of the materials would be likely to compete with derris or cube root of good quality as a commercial source of insecticides.

GRANADILLA SEED FROM KENYA.—With the increasing production of granadilla (passion fruit) juice in Kenya, there is likely to be an increasing accumulation of seed, and the present sample was forwarded in order to ascertain the possible value of the seed as a source of oil. The sample contained 22·4 per cent. of a pale yellow semi-drying oil, and would probably be suitable for edible purposes and as a low-quality paint oil (*cf.* ANALYST, **1937**, **62**, 471).

GROUND MICA FOR WALL-PAPER MANUFACTURE.—For many years the particular quality of ground mica required in printing wall-paper, especially ceiling-paper, had to be imported from U.S.A., despite the fact that the raw material used is largely of Empire origin. The problem is to reduce the mica to powder without destroying its sheen. Ultimately a promising sample was submitted, and co-operation between the inventor of a process and a firm using the material resulted in the successful production of ground mica on a commercial scale.

VERMICULITE.—A sample from Tanganyika Territory was examined. The use of this mineral is increasing rapidly. It is a variety of hydrous mica which has the property of exfoliating considerably when heated, expanding to many times its original bulk. Its principal uses are in connection with heat- and sound-insulation.

British Standards Institution

THE following Standard Specifications have been issued*:

NO. 769—1938. BRITISH STANDARD METHODS FOR THE CHEMICAL ANALYSIS OF BUTTER.

The work of the Empire Marketing Board Dairy Research Committee and its sub-committees was taken over by the British Standards Institution in November, 1933, and the continuance of the work of the *Sub-Committee on Chemical Analysis of Dairy Products* was confirmed on 1st November, 1934, at a meeting of the Technical Committee on Standards for Use in the Dairy Industry, C/11, with the addition of two new members representing the Society of Public Analysts and other Analytical Chemists.

The methods were again carefully reviewed and widely re-circulated both at home and in the Empire for further criticism. As a result of the comments and suggestions received it has been decided to confine this specification to the recognised methods used by chemists in the routine examination of all butters, and to include in an Appendix such supplementary special tests as have been found of value in the tracing back of defects in the condition and quality of defective butter. These special methods have not in all cases been the subject of co-operative investigation by the full Committee, and it is intended to review the methods at intervals of not less than two years, in order that modifications found to be necessary in their practical application can be incorporated.

During the work of the Committee it was felt that the technique for determining the volatile acids in butter should be investigated with a view to the elimination of variations which give rise to inconsistencies in the test results obtained. In accordance with the arrangement agreed between the Society of Public Analysts and other Analytical Chemists and the British Standards Institution, this problem was referred to them for consideration, and, after investigation, a report was forwarded to the Institution, which has been incorporated in this document. (*Cf.* ANALYST, 1936, **61**, 408.)

The Supplementary Special Tests given in the Appendix include the following:—A. Curd-protein in butter. B. Lactose in butter. C. Acidity. D. Common salt. E. Copper and iron. F. Iodine value of fat. G. Saponification value of fat. H. Refractive index of fat. I. Peroxide oxygen in fat (method of C. H. Lea, *Proc. Roy. Soc.*, 1931, B **108**, 188). K. Hydrogen-ion concentration of curd serum (reference is made to Arup and van Gilmour, *J. Dept. Agr. Irish Free State*, 1932, **31**, 180; 1933, **32**, 273; 1935, **33**, 23). The quinhydrone electrode is used (*cf.* Moir, ANALYST, 1931, **56**, 445; Davis, *id.*, 1931, **56**, 449).

NO. 770—1938. BRITISH STANDARD METHODS FOR THE CHEMICAL ANALYSIS OF CHEESE.

The account of the origin of Specification No. 669 is repeated in the foreword to this Specification.

The methods taken over from the Empire Marketing Board Dairy Research Committee and its Sub-committees were again carefully reviewed and widely re-circulated both at home and in the Empire for further criticism. As a result of the comments and suggestions received it has been decided to confine this specification to the recognised methods used by chemists in the routine examination of all cheeses and to include in an Appendix such supplementary special tests as have been found of value in the tracing back of defects in the condition and quality of defective cheese. These special methods have not in all cases been the subject of co-operative investigation by the full Committee, and it is intended to review the methods at intervals of not less than two years in order that modifications found to be necessary in their practical application can be incorporated.

The method for the determination of fat is based on the Schmid–Bondzynski–Ratzlaff method, as submitted by Mr. A. More to the International Convention at Rome. The method was adopted in 1930 and approved by the Convention in 1934 as an alternative method. The method adopted by the Convention is given in Appendix B.

The Supplementary Special Tests, given in Appendix A, include the following:—A. Hydrogen-ion concentration. B. Titratable acidity.

NO. 771—1938. BRITISH STANDARD SPECIFICATION FOR SYNTHETIC RESINS (PHENOLIC) MOULDING MATERIALS AND MOULDING.

This Specification provides the methods of test and technical particulars necessary in purchasing specifications for synthetic resin (phenolic) mouldings and for the moulding material from which the mouldings are made.

Several types of commercial moulding material have been selected which are in general use for the manufacture of moulded articles or component parts, and they are classified, according to their properties, into five types.

---

* These Specifications can be obtained from the Publications Department, British Standards Institution, 28, Victoria Street, London, S.W.1. Price 2s. each net, post free 2s. 2d.

No. 773—1938. British Standard Specification for Ostwald–Folin Pipettes.

The British Standard series of Ostwald–Folin pipettes comprise pipettes calibrated for delivery and pipettes calibrated for content. Each series includes the following seven sizes:—0·2 ml., 0·5 ml., 1 ml., 2 ml., 3 ml., 5 ml., and 10 ml.

No. 783—1938. British Standard Specification for Japanese and/or Korean Sardine Oil (Pale).

The requirements specified for this oil are as follows:—(1) It shall be the product of the Japanese sardine (*Clupanodon melanosticta*) and be free from admixture with other oils and fats. (2) It shall not contain more than 0·7 per cent. of total volatile matter, as determined by the specified method. (3) It shall contain not more than 0·3 per cent. of dirt, as determined by the specified method. (4) When the completely liquid, filtered oil is matched through a 1-in. cell with Lovibond colour glasses at 25° C. to 30° C. the red component of the matching glasses shall not exceed 10 units. (5) The sp.gr. of the filtered oil at 15·5°/15·5° C. shall not be lower than 0·928 nor higher than 0·933. (6) The iodine value (Wijs) shall not be lower than 170. (7) The saponification value shall not be lower than 189. (8) The acidity of the filtered oil shall not exceed the equivalent of 5·0 per cent. of free fatty acids as oleic. (9) Unsaponifiable matter shall not exceed 2·0 per cent., when determined by the specified method.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS

Food and Drugs

Adulteration of Port Wine. J. C. Botelho. (*Ann. Chim. anal.*, 1938, **20**, 33–40.)—It has been shown (Botelho, *Ann. Chim. anal.*, 1935) that the total acidity and the fixed acidity, expressed as tartaric acid, are generally very low in port wines of good quality. Esterification takes place rapidly when the alcohol-content is high and the acidity is low. The volatile acidity, expressed as acetic acid, is always less than 0·09 per cent. and diminishes with the age of the wine, thus accounting for the minute amounts found in very old port wines. Souring due to bacterial action increases the amount of volatile acid and reduces the amount of fixed acid and, to conceal the increase of volatile acid, alkaline bases are sometimes added to the wine. Since the acids of port wine vary only within narrow limits, the ratio of the fixed acid to the volatile acid (F/V), both being expressed as tartaric acid, is suggested as a useful criterion of the quality of the wine. A diminution in the value of the ratio to below 3 indicates that the volatile acid is relatively high, or that propionic fermentation of the tartaric acid has set in, or that the wine has been adulterated by the addition of alkalis or alkaline earths. Ammonium oxalate produces only a slight precipitate with genuine port wine, but, if the wine has been adulterated with calcium compounds an abundant precipitate is formed. The values for the fixed and volatile acidities and for the ratio F/V are given for 100 white port wines and 200 red port wines. The value of the ratio in these samples never falls below 3 and, for the majority, its value lies between 5 and 18. The following are typical examples, the acidities being expressed as tartaric acid per cent.:—volatile acidity 0·0850, fixed acidity 0·3425, ratio F/V 4·0; volatile acidity 0·0375, fixed acidity 0·3900, ratio F/V 10·4; volatile acidity 0·0330, fixed acidity 0·4395, ratio F/V 13·3. A port wine to which an alkaline earth base had been added gave the following figures:—volatile acidity 0·1590, fixed acidity 0·1410, ratio F/V 0·8. In all the analyses published previously (Botelho, *loc. cit.*)

and in the present work, only two samples were found to have values of the ratio F/V less than 3, these having an abnormally low fixed acidity and a normal volatile acidity. Wines adulterated by the addition of alkalis or alkaline earths give high figures for ash and alkalinity of the ash as well as low figures for the ratio F/V.

A. O. J.

Further Determination and Characterisation of the Component Acids of Butter-fat. T. P. Hilditch and H. E. Longenecker. (*J. Biol. Chem.*, 1938, **122**, 497–506.)—The present work concerns the identification, in butter-fat, of the unsaturated minor component acids of lower molecular weight than oleic acid. By using an electrically heated and packed column for the distillation of mixtures of volatile acids and esters, fractions have been obtained with very high proportions of unsaturated esters of low molecular weight. Oxidation of the fractions with permanganate in acetone showed that decenoic, dodecenoic, tetradecenoic and hexadecenoic acids were present. In each unsaturated acid the ethenoid linkage was found to occupy the $\Delta^{9,10}$ position, and this is suggestive in connection with the hypothesis that the lower fatty acids in milk-fat are produced by combined oxidation and reduction of oleic groups already existing as glycerides (ANALYST, 1937, **62**, 250; *Biochem. J.*, 1931, **25**, 507). The minor unsaturated components of milk-fat may represent fragments of transformed oleoglycerides which have escaped complete saturation to lower saturated glycerides. Bromination of the C_{18}-unsaturated acids and mild oxidation confirmed the conclusion that only oleic acid and an octadecadienoic acid were present; the difficulties of identifying the latter acid are discussed. Tetrabromostearic acid, characteristic of seed-fat linolic acid, could not be identified. A study of the ether-insoluble bromides indicated that arachidonic acid might be present. The total component fatty acids of the sample of cow's milk-fat examined were calculated to be as follows:—Butyric, 3·03; *n*-hexanoic, 1·39; *n*-octanoic, 1·51; *n*-decanoic, 2·68; lauric, 3·71; myristic, 12·01; palmitic, 25·17; stearic, 9·17; as arachidic, 1·32; $\Delta^{9,10}$-decenoic, 0·32; $\Delta^{9,10}$-dodecenoic, 0·42; $\Delta^{9,10}$-tetradecenoic, 1·58; $\Delta^{9,10}$-hexadecenoic, 3·96; oleic, 29·54; $\Delta^{9,10,12,13}$-octadecadienoic, 3·57; as arachidonic, 0·28; unsaponifiable matter, 0·34 per cent. The results of analyses with the electrically heated and packed column agreed well with those obtained in previous analyses with simpler equipment.

D. G. H.

Detection of Vegetable Gums in Dairy Products. P. A. Racicot and C. S. Ferguson. (*J. Assoc. Off. Agric. Chem.*, 1938, **21**, 110–112.)—The following method is recommended for the detection of vegetable gums used as adulterants, in place of gelatin, in cream cheese, cottage cheese, and sour cream.

Preliminary Test.—Ten g. of the sample are thoroughly mixed with 10 ml. of water and treated with 5 ml. of 20 per cent. trichloroacetic acid solution. The mixture is shaken for one minute and filtered, and 1 volume of the filtrate is mixed in a test-tube with 2 volumes of 95 per cent. alcohol. If the resulting mixture fails to show a distinct precipitate or turbidity after standing for 30 minutes, the test is negative. The appearance of a stringy or flocculent precipitate indicates the presence of locust bean gum or gum tragacanth. A turbidity settling out as a fine granular precipitate indicates gum arabic. A turbidity which persists,

even after standing overnight, is due to decomposition or fermentation products present in the sample and not to the presence of gum. If the preliminary test indicates the presence of vegetable gum, the following test is made:

Confirmatory Test.—One hundred g. of the sample are mixed well with 100 ml. of water and then treated with 50 ml. of 20 per cent. trichloroacetic acid, followed by shaking for one minute and filtering. Two volumes of 95 per cent. alcohol are mixed thoroughly with the entire filtrate, and the liquid is allowed to stand overnight, preferably in a tall cylinder or graduated vessel. The supernatant liquid is decanted as completely as possible without loss of precipitate, so that not more than 50 ml. remain. The precipitate is mixed with the remainder of the filtrate, poured into a 50-ml. centrifuge tube, and centrifuged for 5 to 10 minutes or until the supernatant liquid can be poured off without disturbing the precipitate; this is washed six times with 50 ml. of 75 per cent. alcohol, shaken well, and centrifuged, the supernatant liquor being drained off as completely as possible each time to remove all traces of lactose. The entire precipitate is mixed with 15 ml. of water. If the precipitate dissolves, the presence of gum arabic is indicated, and may be confirmed by U.S.P. tests on this solution. To prove the absence of lactose, Benedict's qualitative test is applied to 8 drops of the mixture, 5 ml. of Benedict's solution being used. The biuret test is carried out on 1 ml. of the mixture to prove the absence of protein, and Molisch's carbohydrate test is applied to 1 ml. of the mixture to prove that the precipitate is a carbohydrate. Ten ml. of the mixture are hydrolysed by boiling gently for 2 to 3 minutes with 10 ml. of hydrochloric acid (1 volume of conc. hydrochloric acid and 2 volumes of water). The liquid is cooled and neutralised exactly to phenolphthalein with sodium hydroxide, 50 per cent. sodium hydroxide solution being used at first and the final adjustment made with 0·1 *N* sodium hydroxide solution or 0·1 *N* hydrochloric acid, after which it is cooled, treated with 1 or 2 g. of decolorising carbon, shaken and filtered. Benedict's qualitative test is carried out on 8 drops of this filtrate, the mixture being allowed to stand overnight if there seems to be no immediate reduction. Comparison should be made with the result obtained by Benedict's test before hydrolysis. A negative biuret test for protein, plus a positive Molisch carbohydrate test and a negative Benedict test before hydrolysis, followed by a positive Benedict test after hydrolysis, afford proof of the presence of a vegetable gum. One g. of phenylhydrazine hydrochloride and 1·5 g. of sodium acetate are added to 10 ml. of the neutralised solution, and the liquid is mixed thoroughly and placed in a boiling water-bath for two hours. After cooling, it is noted if any osazones are present, and these are examined microscopically, being compared, if necessary, with osazones of known identity. Glucosazone is indicative of locust bean gum; tragacanth gives flat pale yellow osazones resembling maltosazone; gum arabic yields very small burr-like osazones.

Irish moss is precipitated by proteins in an acid solution, so that this method cannot be used for its detection; nor can Karaya gum be detected in this way, as it is not precipitated by the addition of alcohol to the trichloroacetic acid filtrate. The method is particularly valuable for the detection of locust bean gum. The confirmatory test is useful for prosecution purposes, but is not necessary in ordinary routine work. E. M. P.

Volatile Oil in Marjoram. J. F. Clevenger. (*J. Assoc. Off. Agric. Chem.*, 1938, **21**, 109–110.)—The following table shows the variations in yield and properties of volatile oils obtained from commercial samples of marjoram imported into New York during the past six years. The results were obtained by the methods outlined in *Methods of Analysis A.O.A.C.* (1935, 4th Ed., 447–449).

Yield v/w*	Sp.gr. at 25°/25° C.	Op. rot. 25° C.	Ref. ind. n_D^{20}	Acid value	Ester value
2·0	0·939	+ 9·0	1·501	3·7	13·1
1·7	0·937	+ 6·9	1·496	4·6	21·7
1·2	0·896	+13·1	1·475	3·9	33·0
1·7	0·897	+12·8	1·476	1·7	22·9
1·5	0·913	+ 9·1	1·486	3·2	25·8
1·9	0·910	+10·4	1·486	1·7	17·2
1·0	0·936	+ 9·3	1·491	4·1	20·9
2·9†	0·931	+ 6·2	1·497	1·9	2·0
1·6	0·935	+ 8·3	1·494	4·2	15·1
2·6†	0·935	+10·3	1·489	2·6	5·0
2·4†	0·926	+ 9·7	1·489	1·8	14·0
1·6	0·900	+16·3	1·473	2·3	23·6
1·8	0·913	+16·8	1·475	1·1	39·0
1·5	0·945	+ 8·6	1·495	3·5	12·0
1·0	0·909	+14·0	1·478	2·0	33·7
1·3	0·950	+ 6·05	1·502	2·6	9·3
1·6	0·961	+ 4·5	1·508	2·7	6·8
1·7	0·927	+ 5·9	1·493	5·3	23·0
0·3‡	0·912	+15·2	1·478	4·6	28·6

* ml. per 100 g. of marjoram. † Grown in Tunis. ‡ Contained exhausted leaves.

The positive optical rotation is valuable for the detection of spurious species of marjoram. The loss of volatile oil from marjoram leaves allowed to stand in an open pan in the laboratory for a year was very slight, as is shown by the following results:

Date	Yield v/w	Sp.gr. 25°/25° C.	Op. rot. 25° C.	Ref. ind. n_D^{20}	Acid value	Ester value
29/4/36	1·6	0·940	+5·2°	1·503	1·87	4·67
29/5/37	1·4	0·933	+5·2°	1·503	1·65	7·9

E. M. P.

"Bulgarian" Belladonna Root. R. H. Henriksen. (*Pharm. J.*, 1938, **140**, 240.)—"Bulgarian" belladonna root supplied by Raeff, with whom the treatment of post-encephalitic parkinsonism originated, was examined (*cf.* A. E. Bailey, *Pharm. J.*, 1938, **140**, 77; Abst., ANALYST, 1938, 199). Its properties and microscopical characteristics agree with those of the ordinary root. Two samples analysed for alkaloid by the B.P. (1932) process contained 0·345 and 0·351 per cent., respectively, calculated as hyoscyamine. By this method Bailey (*loc. cit.*) obtained 0·238 and 0·247 per cent., and Kuiper and van der Wielen found 0·58 per cent. by the Swiss Pharmacopoeia method. A decoction (1 in 20) for administration is prepared (*a*) by Neuwhal and Fenwick's method, in which the drug is macerated in 600 g. of white wine for 5 or 6 hours, boiled for 10 minutes, cooled and

filtered, or (*b*) by Raeff's method, according to which the drug is boiled with white wine for exactly 14 minutes, cooled and filtered. Analysis of decoctions similarly prepared, but with white wine replaced by 12 per cent. alcohol, gave the following results:

Raeff's method		Neuwhal and Fenwick's method			
A	B	C	D	E	
0·0072	0·0081	0·0126	0·0147	0·0116	} per cent.
0·0069	0·0075	0·0128	0·0150	0·0123	

Thus from 40 to 80 per cent. of the alkaloid in the root had been extracted. With the B.P. maceration process, and also by percolation with 80 per cent. alcohol, tinctures containing approximately 99 per cent. of the available alkaloid were obtained. These variations in the root and in the amounts extracted indicate the necessity for careful chemical control. Qualitative analysis of a solution of the alkaloid confirmed Bailey's identification of the root with ordinary belladonna.

E. B. D.

Detection of Ethylvanillin (Bourbonal). P. Stadler and K. Wagner. (*Z. anal. Chem.*, **1938**, **111**, 391–393.)—The reaction between vanillin, hydrochloric acid and hydrogen peroxide, resulting in the formation of a reddish-brown colour and a greyish-blue crystalline substance on subsequent dilution, as described by Griebel (*Mikrochem.*, 1931, **9**, 313; Abst., ANALYST, 1932, **57**, 200), is also given by ethylvanillin. Although the crystals formed from ethylvanillin are generally smaller and more compact than those formed from vanillin, the occurrence of transitional forms makes differentiation difficult. It was found that the precipitates behave differently when the liquids in which they are suspended are shaken with various solvents. The vanillin precipitate, for example, does not separate into two layers when shaken with carbon tetrachloride, chloroform or carbon disulphide although the precipitate from ethylvanillin does. The causes of these differences are probably physical, depending upon differences in density, surface action and solubility. The following is a useful method of distinguishing the two compounds. The substance (about 0·01 g.) is warmed on the water-bath to 50° C. with 1 ml. of 25 per cent. hydrochloric acid until complete solution is effected. To the cold solution 0·5 to 1 ml. of 3 per cent. hydrogen peroxide is added, and the mixture is allowed to stand, with frequent shaking, until the colour changes from yellow, through brown and red, to violet, and until the subsequent formation of a precipitate appears complete. This usually takes from 10 to 20 minutes. An equal volume of benzene is then added, and the mixture is frequently but moderately shaken. The blue precipitate from vanillin remains in the aqueous layer, and the benzene forms an upper colourless layer. The precipitate from ethylvanillin dissolves in the benzene to form a violet solution, the aqueous layer being almost colourless. With a mixture of the two substances both layers are coloured. For a further test, the test-tube is again placed in a water-bath at 50° to 60° C. and warmed, with frequent shaking. If vanillin is present, the crystals dissolve in the aqueous layer, forming a lemon-yellow solution; if ethylvanillin is present, the violet colour remains in the benzene layer for a longer time at this temperature.

With a mixture of both substances the aqueous layer is yellow and the benzene layer violet. In applying the test to sugar products the vanillin or ethylvanillin is first separated by extraction with ether. A. O. J.

Review of the Analytical Reactions of Ephedrine. New Methods of Identification. M. Pesez. (*J. Pharm. Chim.*, 1938, [8], **27**, 120–128.)—The identification reactions of ephedrine are briefly summarised, and the following new reactions are described:—(1) *Colour reaction with methanol.*—This is an adaptation of the Denigès reaction for benzene derivatives, and depends on the presence, in ephedrine, of a non-substituted benzene ring with only one side carbon chain. The alkaloid is dissolved in 2 ml. of conc. sulphuric acid, and 3 or 4 drops of a 40 per cent. officinal formaldehyde solution are added. A rose colour is formed in the cold, changing to blood-red and to wine-red on immersing the tube in boiling water. (2) *Acetone and dinitro-ephedrine.*—This is an application of the work of Janowski (*Ber.*, 1891, **24**, 971), who showed that certain *meta*nitro derivatives give characteristic colour reactions in alkaline solutions of acetone. From 1 to 2 cg. of ephedrine and 1 ml. of a mixture in equal vols. of conc. sulphuric and nitric acids are placed in a test-tube about 16 cm. long and of 16 mm. internal diameter. On warming the tube over a small flame, nitric fumes are evolved, and the liquid boils and becomes first yellow and then reddish-brown. After 2 or 3 seconds' heating, the tube is allowed to cool slightly and held vertically for 10 to 20 seconds. (The heating is not sufficient unless the brown colour persists.) Ten ml. of water are then added cautiously. On neutralising with sodium carbonate, a deep blood-red colour results. This change indicates that the medium is alkaline. Six to 8 ml. of ether are added and the tube is shaken. The lightly coloured ethereal layer is then pipetted off, the ether is evaporated, 2 ml. of acetone and 2 ml. of sodium carbonate solution (sp.gr. 1·3) are added, and the tube is shaken vigorously and allowed to stand. The lower layer of sodium carbonate solution is almost colourless, but the acetone layer shows an intense gooseberry-red or slightly violet-red colour. The reaction appears to be specific for ephedrine. (3) *Reaction with bromoform.*—Sodium hypobromite reacts with ephedrine, yielding bromoform; this reaction is analogous to that of Sanchez, in which iodoform is produced (*J. Pharm. Chim.*, 1935, **12**, 489; Abst., ANALYST, 1936, **61**, 126). It depends particularly on the presence of a secondary alcoholic chain in the side carbon chain. A few crystals of ephedrine (0·01 g.) are dissolved in 3 ml. of water, and 5 drops of sodium hypobromite solution, followed by 1 ml. of sodium carbonate solution (sp.gr. 1·33) and 1 to 2 ml. of pyridine, are added. When the mixture is vigorously shaken and warmed (with shaking), a rose colour, changing to gooseberry-red, is produced. On standing, the supernatant pyridine layer is seen to be bright red and the aqueous layer colourless. D. G. H.

Biochemical

Detection of Pyruvic Acid in Blood. T. Shindo. (*Z. physiol. Chem.*, 1938, **251**, 285–286.)—The blood to be examined is allowed to stand for one hour at room temperature with one-twentieth of its volume of 20 per cent. trichloroacetic acid and the solution is then filtered. The precipitate is twice triturated

with 5 per cent. trichloroacetic acid and filtered. The combined extracts are treated with a 5 per cent. solution of 2:4-dinitrophenylhydrazine (5 ml. to 20 ml. of the filtrate) and allowed to stand for a week at room temperature with occasional shaking. The mixture is then extracted several times in a separating funnel with ethyl acetate until the aqueous layer is colourless. The ethyl acetate solution containing the hydrazone and the excess of hydrazine is repeatedly extracted with 25 per cent. sodium carbonate solution until the aqueous layer is no longer coloured. This is then acidified with strong hydrochloric acid and extracted with ethyl acetate until colourless. These operations are repeated four or five times. The final ethyl acetate extract is washed two or three times with cold water and the solvent is distilled at room temperature. The brownish-yellow residue is dissolved in the smallest amount of half-saturated carbonate solution and precipitated with strong hydrochloric acid. By this method 1·5 mg. of pyruvic acid 2:4-dinitrophenylhydrazone were isolated from 48 g. of the blood of B_1-avitaminous pigeons and 2·0 mg. from 150 g. of the blood of a human being suffering from beri-beri. The m.-ps. were 213° C. and 212° C., respectively. F. A. R.

Form of Copper in Blood Plasma. R. Boyden and V. R. Potter. (*J. Biol. Chem.*, 1938, **122**, 285–290.)—To ascertain the type of compound in which copper occurs in the blood of red-blooded animals, blood was collected from one cow over a period of several months, 2 mg. of copper-free potassium oxalate per ml. being used to prevent clotting. The total copper was determined on a 10-ml. portion of the plasma by the Fischer and Leopoldi method (*Z. angew. Chem.*, 1934, **47**, 90). Aliquot parts of the plasma, with and without acid, were placed in cellophane holders and dialysed for 24 hours at 2 to 4° C. against 20 volumes of re-distilled water. The total copper in the plasma was between 10 and 15γ, so that extreme precautions had to be taken to obtain copper-free reagents, etc. When no acid was added the copper did not dialyse, but increasing amounts dialysed as the pH was progressively lowered. The amount of copper dialysed by hydrochloric acid at pH 1·67 was 46 per cent., and by sulphuric acid 84 per cent. The percentage of copper dialysable with these two acids and with phosphoric acid was determined over a pH range of 8·1 to 1·5, and it was found that the pH was quite constant for any given amount of added acid. The copper is regarded as existing in the plasma in an organic form dissociated by acid, and the possibility of the presence of more than one organic form is discussed. D. G. H.

Relation of Fat to the Utilisation of Lactose in Milk. E. J. Schantze, C. A. Elvehjem and E. B. Hart. (*J. Biol. Chem.*, 1938, **122**, 381–390.)—When rats, a pig and a calf were placed on a whole-milk mineralised diet all the milk sugar was effectively utilised, but when skim milk was substituted for whole milk, sugar was detected in the urine after a few days. To identify the sugar the urine was boiled with nitric acid and the insoluble compound which separated was identified as mucic acid. (The re-crystallised acid melted at 208° C. uncorrected, and had a neutralisation equivalent of 112). Galactosazone crystals were prepared from the urine sugar after clarification of the urine with mercuric acetate. No part of the reducing material disappeared from the sterilised urine inoculated with a pure *Saccharomyces cerevisiae* (this yeast fermented dextrose readily, but

not galactose), whilst all the reducing material disappeared when *Torula galactosa* was used. These results indicated that all the sugar in the urine was galactose. As much as 35 per cent. of the ingested galactose was recovered from the rats. The addition of fats, such as butter-fat, lard, maize oil, coconut and linseed oils, and palmitic and oleic acids, in quantities of 3 to 4 per cent. to the mineralised skim milk, prevented the loss of sugar in the urine. Glycerol or butyric, β-hydroxybutyric, caproic or lactic acids did not prevent the loss. The sugar in the blood rose to about 200 mg. per cent. with mineralised skim milk; with whole milk it seldom exceeded 140 mg. after feeding. D. G. H.

Isolation of some Crystalline Alcohols from the Unsaponifiable Matter of Rice and Wheat Germ Oils. A. R. Todd, F. Bergel, H. Waldmann and T. S. Work. (*Biochem. J.*, 1937, **31**, 2247–2256.)—The unsaponifiable matter of rice germ oil was freed from sterols and chromatographed on alumina. The oils obtained after purification by distillation in a high vacuum gave crystalline esters with *p*-nitrobenzoyl chloride or β-naphthoyl chloride. These esters were fractionated and gave apparently homogeneous compounds yielding on hydrolysis: α-orysterol (m.p. 121–122° C.; $[\alpha]_D$ in alcohol + 49°), β-orysterol (m.p. 113–114° C.; $[\alpha_D]$ + 51·3°) and γ-orysterol (m.p. 119–120° C.; $[\alpha]_D$ + 51·9°). These were unsaturated alcohols with a composition corresponding with the formula $C_{30}H_{50}O$ and were biologically inactive.

The unsaponifiable matter of wheat germ oil was similarly treated and yielded β-amyrin, α-tritisterol (m.p. 113–114° C.), a small amount of an unknown alcohol (m.p. 174–175° C.), and an oily alcohol giving a nitrobenzoate, m.p. 185° C. None of these was biologically active. F. A. R.

Isolation of β-Tocopherol from Wheat Germ Oil. A. R. Todd, F. Bergel and T. S. Work. (*Biochem. J.*, 1937, **31**, 2257–2263.)—The unsaponifiable matter of wheat germ oil was fractionated by partition, chromatographic analysis and digitonin treatment, and the fraction rich in vitamin E, which could be readily identified by its property of reducing in the cold an ammoniacal solution of silver nitrate in methyl alcohol with formation of a silver mirror, was dissolved in benzene and treated with cyanic acid. The mixed allophanates so obtained, when subjected to chromatographic analysis, yielded mainly β-tocopheryl allophanate (m.p. 143·5°–144·5° C.; $[\alpha]_{5461}^{21°}$ + 7·4°) with only a very small amount of α-tocopheryl allophanate, m.p. 158°–160° C. β-Tocopherol, obtained by hydrolysis of the β-tocopheryl allophanate, possessed full vitamin E activity in a dosage of 5 mg.; it showed an absorption maximum at 295$m\mu$. The oil remaining after separation of the tocopherol was biologically inactive. F. A. R.

Influence of Pimiento Pigments on the Colour of the Egg Yolk of Fowls. W. L. Brown. (*J. Biol. Chem.*, 1938, **122**, 655–659.)—Ground dehydrated pimiento shells were fed to hens which had been depleted of carotenoid pigments. The yolks from the eggs subsequently laid by these hens contained zeaxanthin as the main pigment, together with small amounts of cryptoxanthin, zeaxanthin ester and capsanthin. Pimiento was fed to other groups of hens, together with rations containing other carotenoids. Whereas at least 65 per cent. of the ingested

xanthophylls was deposited in the yolks, only about 1 per cent. of the ingested capsanthin was deposited. It is concluded that, for maximum deposition of a carotenoid in egg yolk, it is necessary that at least one ring of the molecule should contain one, and only one, hydroxyl group. F. A. R.

Carotene in Oranges. A. L. Taylor and P. J. Witte. (*Ind. Eng. Chem.*, 1938, **30**, 110–111.)—Estimations of carotene in the juice from various types of American oranges were made, each sample being obtained from 24 oranges. Fifty ml. of the strained juice were placed in an Erlenmeyer flask through which a gentle current of air circulated, concentrated to a syrup on the water-bath, and analysed by Guilbert's method (*Ind. Eng. Chem., Anal. Ed.*, 1934, **6**, 452), 100 ml. of alcoholic potassium hydroxide solution being used for the saponification. The final solutions in petroleum spirit were immediately compared with a dye standard in a Duboscq colorimeter. Instead of the dye solution prepared by Guilbert (*loc. cit.*), a 0·036 per cent. solution of potassium dichromate (*cf.* Russell, Taylor and Chichester, *N.J. Agr. Exp. Sta., Bull.* 560, 1934) was used for the comparison. This was colorimetrically equivalent to 2·66 mg. of carotene per litre. The average carotenoid pigment was much higher in the Californian than in the Florida varieties of oranges. The following results were obtained:

Variety	No. of samples	Carotene mg. per litre	
		Average	Range
California Valencias	14	1·65	0·65–2·74
California-Washington navels ..	68	1·07	0·24–2·97
Florida Valencias	34	0·57	0·18–1·05
Florida pineapple oranges ..	32	0·34	0·05–0·83
Florida assorted	16	0·32	0·15–0·70

Some of the varieties showed a possible seasonal trend in carotene value, but the variation between samples was too great to enable a decisive conclusion to be drawn. The results for the juice did not appear to depend on the colour of the skins. E. B. D.

Possible Discrepancy in the Estimation of Ascorbic Acid in Urine. G. W. T. H. Fleming and T. E. Burrows. (*Brit. Med. J.*, 1938, I, 333–334.)—The estimation of ascorbic acid in urine, for the purpose of detecting subclinical scurvy, must be carried out on a twenty-four-hours' sample of urine. Appreciable loss of ascorbic acid occurs during this time, and sulphuric acid has been suggested as a preservative. It is now shown that sulphuric acid, far from preserving the vitamin, actually destroys it, nor is acetic acid an effective preservative. Titration of each individual sample of urine immediately after voiding is recommended, tedious though this may be. F. A. R.

Note on the Vitamin D Content of Cow's Colostrum. K. M. Henry and S. K. Kon. (*Biochem. J.*, 1937, **31**, 2199–2201.)—Colostrum not only has a higher concentration of vitamin A and carotene than has later milk, but also contains more vitamin D. Fat from the colostrum of a Guernsey cow on pasture contained 1·2 I.U. per g. one day after calving, 0·56 I.U. per g. after 2–3 days

and 0·36 g. after 4–5 days. A normal control butter-fat contained 0·41 I.U. per g. The increase in the vitamin D content of colostrum as compared with later milk is smaller than that of vitamin A. F. A. R.

Toxicological and Forensic

Determination of Alcohol in Blood. B. Kratz and E. Plämbock. (*Chem.-Ztg.*, 1938, **62**, 148–149.)—During the past year, in applying the Widmark process (v. Brandt, *Chem.-Ztg.*, 1936, **60**, 485) to the determination of alcohol in blood for forensic purposes, the method of Liebesny (*Klin. Wochschr.*, 1928, No. 41), as modified by Heiduschka and Steulmann (*Pharm. Zentralh.*, 1936, **77**, 405; *Dissertation*, Steulmann, Dresden, 1935), and investigated by Wrede and Scriba (*Pharm. Zentralh.*, 1937, **78**, 267), was used as a control process. In addition to its advantage as a more rapid method, it was found to give a more accurate figure for the alcohol-content. Both methods depend upon the reduction of strongly acidified potassium dichromate solution, and there is little difference in the concentrations of sample and reagents. In the procedure of Widmark, the reaction takes place in a closed vessel, and all volatile compounds causing reduction of the dichromate solution, unless subjected to separate investigation, are included in the alcohol-content found. In Liebesny's procedure the blood is heated in an oil-bath at 100° C., and the alcohol vapour is brought into the reaction mixture by means of a stream of air. The transference of the alcohol from the blood into the reaction mixture takes a few minutes, during which time the temperature of the dichromate solution rises to about 85° C., and it is claimed that since, under these conditions, the reaction depends upon the intermediate formation of ethyl hydrogen sulphate, other volatile substances which might react are drawn through the mixture unchanged. By applying the two methods to solutions containing small amounts of such substances as ether, ethyl chloride, chloroform and acetone, any of which may occur in samples of blood, the effect of the presence of these bodies on the apparent alcohol-content was determined. It was found in every instance that Liebesny's procedure gave figures which for practical purposes might be neglected, but that the Widmark process gave high figures which would seriously affect the true alcohol-content. The following examples indicate the order of the discrepancy found:—an aqueous solution of ether (1 part per thousand) gave an apparent alcohol-content of 0·8 (Widmark) and 0·05 (Liebesny); ethyl chloride (3 parts per thousand) gave 1·0 (Widmark) and 0·05 (Liebesny); chloroform (1 part per thousand) gave 0·15 (Widmark) and 0·03 (Liebesny); acetone (5 parts per thousand) gave 0·6 (Widmark) and 0·15 (Liebesny). A sample of blood from a patient under ether anaesthesia gave an apparent alcohol-content of 0·62 (Widmark) and 0·04 (Liebesny). It is apparent that the Liebesny process gives results nearer the true alcohol-content and can usually be applied without correction. The following results are selected from a number obtained in forensic practice. With a sample of blood giving an alcohol-content of 1·14 parts per thousand by Widmark's method and 0·86 by Liebesny's process the discrepancy was found to be due to the presence of acetone. A sample of decomposed blood gave 0·73 (Widmark) and 0·48 (Liebesny), the difference being probably due to a decomposition

product. In connection with a sample giving the figures 2·45 (Widmark) and 2·51 (Liebesny), it was stated in defence that ethyl chloride had been administered as an anaesthetic before the taking of the blood sample and that the result found was partly due to the presence of this substance in the blood. The agreement between the figures found by the two processes indicates the undoubted presence of alcohol and the absence of interfering substances. If the alcohol had been determined by the Widmark process alone, it would not have been possible to rebut the defence. The conclusion is reached that, unless investigation of the possible presence of interfering substances is made, the Widmark process has not the accuracy desirable for forensic purposes. A. O. J.

Determination of the Quinine Content of Blood. F. J. Kaiser. (*Rec. Trav. Chim. Pays-Bas*, 1938, **57**, 117–132.)—Methods involving deproteinisation or direct extraction are unsatisfactory, owing to adsorption of quinine in the former case and to the formation of emulsions in the latter. In the author's method the quinine is evaluated in terms of the fluorescence of quinine sulphate in filtered ultra-violet light. Thus sodium citrate is added to the blood to prevent coagulation, and the gelatinous mixture produced when 5 ml. of sample are mixed with 2·5 ml. of 30 per cent. sodium hydroxide solution is heated for 10 minutes at 80° C. The resulting brown liquid is cooled and extracted for 3 hours with 10 ml. of chloroform. In the Schoorl extraction apparatus described the conical extraction flask is completely immersed in an electrically-heated air-oven, in such a way that the rest of the apparatus remains cool. The latter consists of a long vertical tube with two side-arms. The lower arm connects through a siphon tube with the base of the wide cylindrical glass vessel in which extraction takes place, and therefore serves to return the chloroform extract continuously to the flask; the upper arm leads into the top portion of the same vessel at a point above the level of the contents, and conveys the chloroform vapour from the flask to the extraction yessel, the top end of which is fitted with a vertical Liebig condenser. To the residue after evaporation of the extract are added 2 ml. of ether followed by 5 ml. of 0·1 *N* sulphuric acid, and after evaporation at a low temperature the fats remain as a film on the surface of the aqueous layer, whence they may be removed by shaking with petroleum spirit (b.p. 40° to 60° C.) and removing the extract by decantation and the dissolved solvent by warming gently. A solution of quinine sulphate which is clear and colourless should result; this is important. All the operations are carried out in the same flask, and only glass should be used in the apparatus, as cork or rubber may yield traces of substances which interfere with the subsequent determination of the quinine. Experiments carried out with different periods of extraction have shown that when these exceed 3 hours there is evidence of contamination of the final solution by colouring matters from the blood. Slight modifications in procedure are necessary according to the origin of the blood. Thus, a blank test on dogs' or cats' blood gave a solution, the fluorescence of which corresponded with a quinine-content of less than 0·05γ per ml.; with goats' blood, however, the fluorescence was considerably stronger. The fluorescence is evaluated by placing the solution in a cylindrical glass cell (4·5 cm. long, and 2 cm. in diameter) which is supported horizontally in a cylindrical copper holder in such a way as to

expose a circular opening (diameter 1·1 cm.) at the top to the ultra-violet light. This is the radiation from a mercury vapour lamp after filtration through a "Uviol" (UG 2) filter, 4 mm. thick, placed at a distance of 10 cm. from the lamp and 20 cm. from the cell; special provision must be made for ensuring a constant output from the lamp, the current being controlled by means of an adjustable resistance. The cell containing the sample is also illuminated from one side by means of an ordinary electric lamp (6 volts, 40 watts), the output of which is also controlled so as to eliminate fluctuations; this point is of particular importance when the lamp is first switched on. The lamp is mounted on an optical bench so that its distance from the cell may be varied and measured, and it is enclosed in a horizontal cylinder (diameter 5 cm.) which has a matt surface and is closed by a plate having an opening 4·5 mm. in diameter. The light emerging from this passes through a glass filter vessel (3 × 10 × 10 cm.) containing a solution which is intended to simulate the colour of the fluorescence of quinine sulphate, and which contains pyrrole blue (for the violet constituent) and patent green (for the green shade). The filtered light then falls on the side of the cell containing the sample, one end of which is blocked by a diaphragm having a small aperture (diameter 1·5 cm.), opposite which is placed a telescope. An observer at the latter, therefore, sees the fluorescent light from the sample (due to illumination by ultra-violet light from above) and the filtered visible light (produced by illumination from the side) in close juxtaposition, and by altering the distance of the lamp from the cell these may be matched in intensity; this distance should be between 40 and 80 cm. It is desirable to coat any reflecting surfaces with a paste consisting of 4 parts (by weight) of zinc oxide, 2 parts of alcohol, and 4 parts of a cellulose lacquer prepared by grinding 15 parts of camphor and 10 of colourless cellulose with 100 parts of alcohol; this enhances the intensity of the fluorescence. A series of 32 standard solutions, corresponding with the equivalent 8·0 to 0·5γ of quinine in 1 ml. of 0·1 *N* sulphuric acid is prepared, and a match is obtained first with one, the fluorescence of which is slightly more intense than that of the sample, and then with one having a slightly weaker fluorescence. If the corresponding respective concentrations are y and z per ml. and the distances of the light source from the cell, as measured on the optical bench, are b and c cm., then $c^2/a^2 = x/z$, and $b^2/a^2 = x/y$, where $x\gamma$ per ml. is the concentration of the sample, and a cm. is the scale-reading in this instance. It is assumed that the illumination may be regarded as being due to a point-source. Two values of x are thus obtained, from which a mean value is calculated. The method of ordinary linear interpolation may also be used, and this avoids the necessity of matching two standards, but it involves an error if the difference between the concentrations of the sample and standard exceeds 10 per cent. When 16 samples containing the equivalent of 16·80 to 1·10γ per ml. of quinine were tested, the error was +5·9 per cent. for the former sample and varied between 0 and −6·3 per cent. for the other 15 samples, the mean error being 4 per cent. Duplicate determinations on the same sample could be made with an error of 0·4γ in one instance, and a maximum error of 0·1γ in the others. The minimum quantity of quinine which may be determined in this way is 0·5γ per ml. (*cf.* Nicholls, ANALYST, 1934, **59**, 277). J. G.

Agricultural

The Insoluble Residue in the Potassium Chloroplatinate obtained in the Analysis of Certain Fertilisers for Potash. H. R. Allen. (*J. Assoc. Off. Agric. Chem.*, 1938, **21**, 134–140.)—When the official method for the determination of potassium in fertilisers is used (*Methods of Analysis, A.O.A.C.*, 1935, 30) a considerable quantity of insoluble residue is often found, this being determined by the difference between the weights of the crucible originally and after washing out the potassium chloroplatinate with hot water. In the present experiments attempts were made to reduce the quantity of this insoluble residue by ignition at higher temperatures; ignition over a Méker type (Fisher) burner, ignition in an electric muffle at 750° C., and double ignition with sulphuric acid over a Tirrill burner were tried. Platinum and silica dishes were used. The results, which are tabulated, indicate that the quantity of insoluble residue is smaller with higher temperature of ignition, except when silica dishes are used in the muffle, and that the quantity of potassium chloroplatinate is greater with higher ignition temperature. More potassium and less insoluble residue were obtained in platinum than in silica dishes, with higher temperature of ignition. It appears that ignition of the sulphates at about 750° C. is preferable to ignition at a lower temperature.

E. M. P.

"Sussex Ground" Oats. F. Robertson Dodd and A. Pattinson Telford. (*Fertiliser and Feeding Stuffs J.*, 1938, **23**, 3 and 4.)—The authors state that "Sussex Ground" oats were originally prepared from oats grown in Sussex which had been ground in "Sussex fashion" and in a Sussex mill, in a manner which eliminated a portion of the husks and resulted in a fine grist and a low proportion of fibre. Now that disintegrator or "beater" mills are in general use the fibre in what are now known as "Sussex Ground" is increased, since these mills disintegrate the whole oats without any removal of husks. The oats should be carefully selected and only those oats with thin husks and otherwise suitable for the purpose should be chosen. Adulteration is frequently practised by admixture with other farinaceous grains or meals and also with the ground oat shudes prepared from the offal obtained in the production of oatmeal for human consumption. The proportion of husk to kernel in genuine oats varies with the "plumpness" or "thinness" of the grain, and is shown by quotation from various authors to range from as little as 20 per cent. to over 40 per cent. It follows that the quantity of indigestible fibre in the whole oats shows a wide range of variation. The percentage of fibre in oat husks appears to vary from about 28·5 to 32·5 per cent. The authors themselves recorded, on hand-picked oat shudes, a figure of approximately 32 per cent. A recent sample of clean oat shudes obtained in the market by Dyer and ground in the laboratory to a degree of fineness comparable with that of "Sussex Ground" oats yielded 28·2 per cent. of fibre. The authors consider that for estimation of husks in a mixture from the percentage of fibre it would probably be safe to assume an average of 30 per cent. for the fibre in oat husks.

The authors give the following analyses of oats made by themselves:

Oats	Moisture Per Cent.	Oil Per Cent.	Albumin-oids Per Cent.	Digestible carbo-hydrates Per Cent.	Fibre Per Cent.	Ash Per Cent.	Silica Per Cent.
English A ..	8·25	6·72	12·16	56·27	12·80	3·80	1·71
English B ..	9·00	5·95	13·25	57·25	10·65	3·90	1·77
Plate	8·35	7·57	11·56	59·76	8·90	3·86	2·05
Canadian ..	10·95	5·07	12·94	58·23	9·40	3·41	1·15

They also quote the following analyses made by Dyer:

	Moisture Per Cent.	Oil Per Cent.	Albumin-oids Per Cent.	Digestible carbo-hydrates Per Cent.	Fibre Per Cent.	Ash Per Cent.	Silica Per Cent.
English	13·40	4·35	9·20	60·46	9·26	3·33	1·10
	12·40	4·50	10·00	57·54	11·86	3·70	1·30
	13·33	4·93	8·65	63·12	6·80	3·17	1·03
	12·67	4·30	14·30	56·81	8·19	3·73	1·00
	15·68	3·32	12·03	53·29	11·90	3·78	1·47
	13·30	4·27	11·23	58·75	9·12	3·33	1·41
	15·97	4·06	11·63	56·30	8·76	3·28	1·33
	13·80	5·30	11·90	57·53	8·90	2·57	0·70
	12·83	5·40	9·89	60·03	8·93	2·92	1·09
Scottish	16·07	7·80	9·94	—	7·63	—	—
	15·47	5·77	10·00	—	8·33	—	—
	16·37	6·90	10·31	—	7·57	—	—
	15·67	7·03	10·63	—	7·70	—	—
Canadian ..	10·73	5·25	12·70	61·27	7·02	3·03	1·20
	10·00	5·43	12·75	61·10	7·53	3·19	1·40
Danubian ..	10·72	5·95	12·50	58·33	8·58	3·92	1·68
Plate	11·67	6·10	11·44	56·75	9·97	4·07	—
	9·97	5·86	11·44	61·66	7·60	3·47	1·00
	11·53	6·50	8·90	57·97	10·90	4·20	2·50
Cape (average of 8 samples) ..	10·50	7·13	8·55	59·83	10·22	3·77	2·26

Analyses of 52 samples of oats grown in various parts of the Cape made by J. Lewis (*Cape Dept. of Agriculture*, 1908, *Bull.*, No. 35) gave the following average:

	Moisture Per Cent.	Oil Per Cent.	Albumin-oids Per Cent.	Digestible carbo-hydrates Per Cent.	Fibre Per Cent.	Ash Per Cent.	Silica Per Cent
Cape oats (average of 52 samples)	9·85	6·03	9·53	60·77	10·20	3·62	—

In these 52 samples the fibre ranged from a minimum of 8·46 to a maximum of 13·72 per cent.

Ground oats are described in the Fourth Schedule of the Fertilisers and

Feeding Stuffs Act as "the meal obtained by grinding commercially pure oats as grown"— a reasonable definition clearly prohibiting any admixture of extraneous oat husks. But as the prefix "Sussex" appears to be generally regarded by purchasers as an indication of good quality as well as of fine grist, the authors suggest that a separate description should be introduced into the Schedule for "Sussex Ground" Oats, indicating a maximum of 9 per cent. of fibre and possibly limits of not less than 5 per cent. and 9·5 per cent. respectively for oil and protein.

Determination of Nicotine on Apples Sprayed with Nicotine Bentonite. L. N. Markwood. (*J. Assoc. Off. Agric. Chem.*, 1938, **21**, 151–155.)—A dipping method, using a stripping solution containing brucine, was found to be the most efficient means of removing nicotine from films of nicotine bentonite on apples. The use of a wetting agent in the stripping solution gave no advantages. The following experiments were carried out:—Fifty Stayman Winesap apples (picked during the growing season) of approximately uniform size were sprayed with the greatest uniformity possible by laboratory technique and divided into lots of 10. Four lots were treated by immersion in boiling stripping solutions, each of which contained 180 ml. of water and 20 ml. of 1 : 4 hydrochloric acid, and in addition, for Lot (2), 0·25 g. of wetting agent, for Lot (3), 2 g. of brucine, and for Lot (4), 0·25 g. of wetting agent and 2 g. of brucine. Each apple was held in the solution for 4 minutes and then rinsed off in another beaker. The acid extracts were concentrated on the steam-bath to about 75 ml. before the alkaline distillation and the usual precipitation with silicotungstic acid (*Methods of Analysis, A.O.A.C.*, 1935, p. 60). The fifth lot was treated by a modified Ralston method, in which the apples were shaken in a closed container with a mixture of dilute sodium hydroxide solution and toluene, the nicotine being extracted from the organic solvent with hydrochloric acid, distilled from alkaline solution and precipitated with silicotungstic acid. The following results were obtained:

NICOTINE COVERAGE ON APPLES BY ACID-IMMERSION.

Lot	Method	Nicotine found mg.	Relative amounts found Per Cent.
	Hydrochloric acid solution with—		
1	no wetting agent or brucine ..	3·59	61
2	wetting agent	3·59	61
3	brucine	5·87	100
4	wetting agent and brucine ..	5·87	100
5	Modified Ralston	4·79	82

A second group of apples, also approximately uniform in size and sprayed with uniform care, was divided into six lots of 10 each, and the nicotine load of each lot was determined. The stripping solution for Lots (1) and (2) consisted of 180 ml. of water, 20 ml. of dilute hydrochloric acid, and 2 g. of brucine; for Lots (3) and (4) 0·25 g. of wetting agent was added. Lots (5) and (6) were treated by the original Ralston method, with ethylene dichloride and sodium hydroxide as the solvents. The following results were obtained:

Method	Lot	Weight of lot g.	Nicotine found per lot mg.	Nicotine found per kg. mg.	Relative amounts found per lot Per Cent.	Relative amounts found per kg. Per Cent.
Brucine without wetting agent	(1)	866	5·29	6·12		
	(2)	867	5·30	6·12		
		Average	5·30	6·12	100	100
Brucine with wetting agent	(3)	858	5·23	6·11		
	(4)	837	5·18	5·93		
		Average	5·21	6·02	98	98
Original Ralston	(5)	830	4·46	5·37		
	(6)	861	4·53	5·27		
		Average	4·50	5·32	85	87

A stripping solution containing 2 g. of brucine, 20 ml. of 1:4 hydrochloric acid, and 180 ml. of water is recommended; a larger volume of water may be used, if necessary, to obtain complete immersion. The apple is kept submerged by a pointed glass rod or a three-pronged device constructed of glass tubing.

E. M. P.

Losses of Chlorine in different Materials with various Ashing Temperatures. T. A. Pickett. (*J. Assoc. Off. Agric. Chem.*, 1938, **21**, 107–108.)—The following figures were obtained in experiments to determine the temperatures at which various materials can safely be ashed without loss of chlorine. The first and second ashings of the samples were started in a cold muffle and were each of one hour's duration, except with the group ashed at 800° C., which needed only one ashing to produce a white ash. The chlorine was determined by the Volhard method, 0·05 *N* silver nitrate and 0·05 *N* potassium thiocyanate solutions being used.

Material	Chlorine found, mg. Ashing temperature 500° C.	550° C.	600° C.	700° C.	800° C.
35·72 mg. of chlorine	35·59	35·23	34·92	30·21	13·72
35·72 mg. of chlorine*	35·70	—	35·65	35·30	33·73
35·72 mg. of chlorine* + 5 g. of sucrose	34·51	—	35·12	33·93	28·73
35·72 mg. of chlorine+2·5 g. of starch	31·69	—	31·60	28·68	—
35.72 mg. of chlorine + 5 g. of wheat	27·88	—	20·88	—	—
35.72 mg. of chlorine* + 5 g. of wheat	—	37·76	37·94	37·27	34·77
100 ml. of pineapple juice ..	—	—	11·82	11·65	10·50
35·46 mg. of chlorine* + 100 ml. of pineapple juice	—	—	46·32	45·53	40·11
4 g. of cotton leaves	22·77	—	22·04	—	—
4 g. of cotton leaves*	—	28·15	—	27·25	26·27
0·1 g. of potassium chloride* ..	48·50	—	48·49	48·55	46·00
4 g. of turnip tops*	75·95	—	75·60	75·05	71·15
5 g. of tankage*	21·45	—	21·00	21·30	19·25
5 g. of fish meal*	18·33	—	17·90	17·55	17·00
3 g. of mixed fertiliser*	66·05	—	66·11	64·10	63·95

* 20 ml. of 5 per cent. sodium carbonate solution added.

The results show that if an excess of sodium carbonate is present, the loss of chlorine is neglible at temperatures not greater than 600° C. Many of the materials analysed can safely be ashed at 650° C. in the presence of sodium carbonate, but none can be ashed at 800° C. without a considerable loss of chlorine. Some samples lost a large amount of chlorine, even at 500° C., when an excess of sodium carbonate was not present. E. M. P.

Organic

New Reaction for the Detection of Sulphonated Oils by means of Pyridine. H. Freytag. (*Z. anal. Chem.*, 1938, **111**, 385–391.)—Compounds formed by the addition of certain groups to the nitrogen of pyridine are easily decomposed by primary or secondary aromatic amines, or by alkalis, in such a manner as to break the pyridine ring, and the enol-glutaconic aldehyde so formed condenses with aromatic amines to form coloured compounds. The substances which form addition compounds with pyridine usually contain a halogen element, but Bayer (*Dissertation*, Dresden, 1912, p. 39) has shown that the halogen may be replaced by other acid-ester groups. This reaction may be used to detect the sulphonic group in sulphonated oils, such as Turkey red oil. The oil (0·1 to 0·5 g.) is mixed thoroughly with about 5 ml. of a 15 per cent. solution of sodium or potassium hydroxide, 5 ml. of pure pyridine are added, and the mixture is heated to boiling-point over a small flame. After two or three minutes the mixture is divided into two portions. The first portion is treated with a small amount of pure β-naphthylamine hydrochloride and is then acidified gradually with dilute hydrochloric or sulphuric acid. A red colour, deepening on standing, indicates the presence of the sulphonic group in the oil. The colour is usually adsorbed on the liberated fatty acids, and thus appears as a red ring on the surface of the liquid. The second portion of the hot liquid is treated with *p*-nitraniline which, after acidification, easily forms the corresponding dye having a light green fluorescence visible in sunlight. If the coloured compound is extracted with ether and drops of the extract are placed upon filter-paper, the fatty acids remain in the centre of the drop and the colour spreads outwards. Upon the addition of alkali, the colour changes to golden yellow and is regenerated by the addition of acid. The test was applied to a number of sulphonated compounds used in the textile industry. The majority answered to the test either with β-naphthylamine or with *p*-nitraniline, if not with both, but there were two exceptions, *viz.* Monopolspinnöl and Lanaclarin. With two substances (Tallosan K and Homogenit B conc.) the final colour appeared on cautious acidification, but disappeared when more acid was added. When the product contains an oil solvent with a halogen group (*e.g.* Tetrapol containing carbon tetrachloride) the solvent must be removed by distillation before applying the test to the residue, since the halogen group behaves in the same manner as the sulphonic group. One substance tested (Oleocarnit) contained pyridine bases, but did not answer to the test unless pure pyridine was added. Possibly a feeble reaction was masked by the dark colour of the substance, or the pyridine homologues or derivatives which were present prevented cleavage of the pyridine ring (this is known to occur with certain pyridine derivatives by steric

hindrance). With one substance (Fewa) the red colour with nitraniline was masked by the bright yellow colour of the solution, but could be made visible by vigorous shaking, when it appeared in the froth. The hypothesis that the sulphonic group of the sulphonated fatty acid is added on to the nitrogen of the pyridine is confirmed by the behaviour of cetyl-, octyl-, decyl-, tetradecyl-, and octodecyl-pyridinium chloride with alkalis. When aqueous solutions or emulsions of these substances are warmed with alkali and subsequently heated with an amine and acidified, the pyridine dye is formed. Ethylene bromide responds to the test in the same manner as chloroform and carbon tetrachloride. With chloramine-T the colour forms momentarily but, on shaking the liquid, the colour disappears and a yellow precipitate forms. Apparently this is due to the presence of free chlorine. Bayer (*loc. cit.*) has stated that *p*-toluene sulphochloride does not react with formation of the pyridine dye but, if strong alkalis are used instead of the primary and secondary amines he employed, an almost quantitative cleavage of the pyridine ring occurs. The sulphochloride dissolves in the pyridine, forming a yellow solution which, on the addition of alkali, becomes violet and, on dilution with water, changes suddenly to brown or yellowish-brown. Possibly the sodium salt of 2-pyranol is first formed and is changed by dilution to the sodium salt of enol-glutaconic aldehyde. A. O. J.

Evaluation of Ethyl Cellulose Solvents. T. A. Kauppi and S. L. Bass. (*Ind. Eng. Chem.*, 1938, **30**, 74–79.)—The solvent used in the preparation of ethyl cellulose lacquers, etc., should be one which yields a clear solution of low viscosity, from which a film of high tensile strength and elongation is deposited. Mixtures of ethyl alcohol with toluene, and mixtures of their homologues, are satisfactory when the hydrocarbon and alcohol are present in the proper proportion. Films of maximum tensile strength and elongation are obtained from some alcohol-hydrocarbon solvents, in which the alcohol evaporates more slowly than the hydrocarbon, if the last solvent to evaporate from the film is almost entirely hydrocarbon (*i.e.* the mixture is slightly richer in hydrocarbon than is that of minimum viscosity). If the evaporation rates of alcohol and hydrocarbon are approximately equal, the solvent mixture producing the lowest viscosity also produces the best films (*cf.* Doolittle, *Ind. Eng. Chem.*, 1935, **27**, 1169). The hydrocarbons used alone with alcohols are aromatic hydrocarbons, hydrogenated naphthas, and fractions from some asphalt-base crude oils. If naphthas from asphalt base crude oils are used, a small proportion of aromatic hydrocarbon must be added. For spraying-lacquers, mixtures of hydrocarbons and alcohols of various rates of evaporation must be used, to obtain the required flow and levelling characteristics. To keep the viscosity low enough throughout evaporation, each hydrocarbon must be matched with the correct proportion of the corresponding alcohol. Solvent mixtures of hydrocarbons with aliphatic ketones or esters have viscosities intermediate between those of the two components; they do not produce solutions of minimum viscosity, but large amounts of ketones or esters can be used without lowering the film properties. Addition of 10 to 20 per cent. of an alcohol to these mixtures yields a satisfactory solvent of low viscosity. No single solvent satisfied all requirements. For solutions of minimum viscosity, the proportion of

toluene to ethyl-, iso-butyl-, or *n*-propyl alcohol was 70 : 30 by volume. These are also the proportions for mixtures of benzene or xylene with ethyl alcohol. With benzene-alcohol solutions, Suida (*Cellulosechem.*, 1931, **12**, 310) obtained a similar minimum, but Danilov and Aleksandrova (*Plasticheskie Massy*, 1935, 100) found that 10 to 15 per cent. of alcohol gave the solution of minimum viscosity. The difference is considered to be due to differences in the ethyl cellulose samples used. Differences in ethoxyl content and in manufacturing conditions affect the properties of ethyl cellulose (*cf.* Aleksandrova, *Plasticheskie Massy*, 1937, 31; Traill, *J. Soc. Chem. Ind.*, 1924, **53**, 337T). In agreement with Danilov and Aleksandrova (*loc. cit.*), the authors find that the amount of alcohol required to produce the alcohol-hydrocarbon solution of minimum viscosity increases with the concentration of ethyl cellulose. E. B. D.

New Method for Distinguishing between the Heart Wood and Sap Wood of Pine. J. E. Koch and W. Krieg. (*Chem.-Ztg.*, 1938, **62**, 140–141.)—The distinction of the heart wood and sap wood of pine is important as an aid to the selection of timber for telegraph poles, railway sleepers, etc. The heart wood is usually darker in colour than the sap wood, although sometimes (especially when the wood is newly-cut) this difference is not easily visible. Impregnation with oils or salts used in impregnating processes, followed by exposure to sunlight or radiations from the mercury lamp, accentuates the difference, and if a section of the wood is exposed to the vapours of hydrochloric acid during the irradiation, the heart wood develops a green colour, or in some instances a violet colour; chlorine gas has also been used in this test. Staining with dyestuffs, such as eosin or nigrosine (*cf. Amer. Wood Preserv. Assoc.*, 1934, p. 139) is stated to produce a dark colour in the sap wood, but has been found to be unreliable. A more reliable method is to prepare a solution of 5 g. of benzidine in a mixture of 25 g. of 25 per cent. hydrochloric acid and 970 g. of water, and to add to it an equal volume of a 10 per cent. solution of sodium nitrite. If the sample of wood (*e.g.* a cross-section or a boring) is immersed in the solution of the diazo-benzidine compound so formed, the heart wood is coloured dark red and the sap wood yellow, the distinction being very sharp, especially if the excess of reagent is removed by rinsing. Staining is very rapid with cross-sections, but requires a few minutes with longitudinal sections; the yellow colour of the sap wood may darken after 1 hour and even become brown, and the extent to which this occurs depends on the age of the tree. Owing to the instability of the reagent the two constituent solutions should not be mixed until the test is to be made. The reaction is not given by woods used as substitutes for pine (*e.g.* beech, oak, larch, fir and spruce), and thus the reagent also enables pine wood to be distinguished from these without microscopical examination.

J. G.

Effect of Various Carbohydrate Materials on the Determination of Lignin by the Fuming Hydrochloric Acid Method. M. Phillips and M. J. Goss. (*J. Assoc. Off. Agric. Chem.*, 1938, **21**, 140–145.)—The tentative method adopted by the A.O.A.C. for the determination of lignin uses fuming hydrochloric acid for the hydrolysis of the cellulose and the associated carbohydrates under definite conditions of temperature and duration of reaction, it being assumed

that the insoluble residue left is lignin (Goss and Phillips, *J. Assoc. Off. Agric. Chem.*, **1936, 19, 341**). The present paper reports the results of experiments to ascertain whether the carbohydrates present may yield insoluble residues on treatment with fuming hydrochloric acid. The weighed sample was placed in one of the tubes of the apparatus of Goss and Phillips (cooled with ice), 50 ml. of fuming hydrochloric acid (sp.gr. 1·212–1·223 at 15° C.) were added, and the mixture was agitated for 2 hours with a current of dry hydrogen chloride. The reaction mixture was allowed to remain for 24 hours at +8° C. to +10° C., diluted with water to 500 ml., refluxed for 1 hour, cooled to room temperature, and filtered on a weighed Gooch crucible which was dried at 105° C. and re-weighed. In the first experiments the carbohydrate was used alone, and in the second series there was added 0·6 g. of wheat straw previously extracted successively with a 1 : 2 alcohol-benzene solution, hot water and 1 per cent. hydrochloric acid, ground to pass an 80-mesh sieve, and dried at 105° C.; 0·6 g. of the straw contained 0·1370 g. of lignin, determined by the method of Goss and Phillips. The results obtained were as follows:

CARBOHYDRATE ALONE

Substance	Weight of carbohydrate g.	Colour of solution after 24 hours' standing with 42–43 per cent. hydrochloric acid	Precipitate obtained	
			g.	Weight of substance Per Cent.
Arabinose	0·2000	Faint yellow	0	0
	0·5000	Yellow	0	0
Xylose	0·2000	Light yellow	0	0
	0·5000	Light brown	0	0
Dextrose	0·2000	Colourless	0	0
	0·5000	Colourless	0	0
Mannose	0·2000	Faint yellow	0	0
	0·5000	Yellow	0	0
Galactose	0·2000	Colourless	0	0
	0·5000	Light yellow	0	0
Fructose	0·2000	Brown	0·0018	0·90
	0·5000	Dark brown	0·0246	4·92
Sucrose	0·2000	Light brown	0·0006	0·30
	0·5000	Brown	0·0078	1·56
Maltose	0·2000	Faint yellow	0	0
	0·5000	Light yellow	0	0
Starch	0·2000	Faint yellow	0	0
	0·5000	Light yellow	0	0
Inulin	0·2000	Brown	0·0025	1·25
	0·5000	Dark brown	0·0209	4·18
Pectin	0·2000	Light brown	0	0
	0·5000	Brown	0·0046	0·92
Cellulose (filter-paper) ..	0·2000	Colourless	0	0
	0·5000	Colourless	0·0020	0·20

CARBOHYDRATE PLUS 0·6 g. OF WHEAT STRAW

Substance	Weight of carbohydrate g.	Yield of lignin g.	Increase of weight of lignin g.	Increase of weight of lignin on weight of sample Per Cent.
Arabinose ..	0·2000	0·1365	0	0
	0·5000	0·1361	0	0
Xylose.. ..	0·2000	0·1365	0	0
	0·5000	0·1375	0·0005	0·04
Glucose ..	0·2000	0·1358	0	0
	0·5000	0·1366	0	0
Mannose ..	0·2000	0·1350	0	0
	0·5000	0·1369	0	0
Galactose ..	0·2000	0·1367	0	0
	0·5000	0·1353	0	0
Fructose ..	0·2000	0·1521	0·0151	1·88
	0·5000	0·1748	0·0378	3·43
Sucrose ..	0·2000	0·1441	0·0071	0·88
	0·5000	0·1587	0·0217	1·97
Maltose ..	0·2000	0·1361	0	0
	0·5000	0·1357	0	0
Starch ..	0·2000	0·1361	0	0
	0·5000	0·1359	0	0
Inulin	0·2000	0·1516	0·0146	1·82
	0·5000	0·1687	0·0317	2·88
Pectin	0·2000	0·1357	0	0
	0·5000	0·1405	0·0035	0·31

The only carbohydrates which might interfere with the determination are fructose, sucrose, inulin and pectin; these would never be present in large quantities in lignified plant material, and would probably be removed by the preliminary extractions of the material with 1:2 alcohol-benzene solution, hot water and 1 per cent. hydrochloric acid, as specified in the method. E. M. P.

Inorganic

Determination of Copper and its Separation from Lead. J. Dick and A. Radulescu. (*Z. anal. Chem.*, 1938, **111**, 394–396.)—The method is based on the precipitation of copper pyridine benzoate $[C_6H_5CO_2]$ $[Cu(C_5H_5N)_2]$. The copper solution (50 ml.) is treated, drop by drop, with a small excess of pyridine; the azure-blue solution is treated in the cold with a solution of 3 g. of ammonium benzoate in 25 ml. of water during agitation. After 2 minutes' stirring, the precipitate is collected and washed with water containing 1 ml. of pyridine and 1·5 g. of ammonium benzoate per 100 ml. The precipitate is dried and ignited gently at first, then strongly for 15 to 20 minutes, and weighed as CuO. Ammonium salts, alkalis, alkaline earths, magnesium, manganese, cobalt, and nickel do not interfere. For the separation of copper from lead, the solution is treated with 2 to 3 g. of ammonium acetate prior to the precipitation of the copper. The lead in the filtrate may be determined by precipitation as chromate. W. R. S.

Determination of Small Quantities of Arsenic in White Metals. C. W. Anderson. (*Ind. Eng. Chem., Anal. Ed.*, 1937, **9**, 569–570.)—After precipitation by reduction with hypophosphite, the elemental arsenic is dissolved in conc. sulphuric acid, the solution is reduced with sodium sulphite, and the arsenic is ultimately titrated with potassium bromate solution. *Tin and Bearing Metals.*—A 1-g. sample is dissolved, by heating, in a mixture of 5 ml. of 1:1 hydrochloric acid and 12 to 15 ml. of a solution of bromine in conc. hydrochloric acid (12 ml. in 100 ml.), and 0·3 to 0·5 g. of cuprous chloride and 5 to 10 ml. of calcium hypophosphite solution (15 g. in 100 ml. of 10 per cent. hydrochloric acid) are added. The liquid is boiled for 10 to 15 minutes. The black precipitate of arsenic is filtered off with suction on a small Gooch crucible with a mat prepared from asbestos which has been kept in dilute hydrochloric acid containing some bromine, the mat being washed with water before use to remove bromine; high results are obtained if ordinary asbestos is used without this treatment. The precipitate is washed with a little conc. hydrochloric acid followed by two or three washings with water. The crucible with precipitate is placed with 3 ml. of conc. sulphuric acid in a test-tube, and shaken to loosen the mat; the tube is heated in a flame until the arsenic has dissolved. After cooling, a few ml. of water are added, the contents of the tube are transferred to a small beaker, and 10 mg. of anhydrous sodium sulphite are added. The liquid is then heated to boiling, and a stream of air (about 3 or 4 bubbles per second) is passed through it for 1 minute; 1 drop of methyl orange indicator (0·1 per cent.) is added, and the solution is titrated slowly with 0·015 *N* potassium bromate solution (standardised against arsenious oxide) to the disappearance of the pink colour; 0·1 ml. of potassium bromate solution is deducted as a blank; 1 ml. = 0·5622 mg. of arsenic. The titration is presumably done hot, although no temperature is stated. *Lead.*—A 2-g. sample of filings is taken and the bromine-hydrochloric acid alone is used as solvent, a slight excess of bromine being maintained during the solution process. When attack is complete the process is continued as for tin. Good results were obtained in test experiments, the quantities of arsenic dealt with being generally less than 1 mg.; antimony in quantities up to 0·5 g. did not interfere. S. G. C.

Separation of Magnesium as Oxalate. P. J. Elving and E. R. Caley. (*Ind. Eng. Chem., Anal. Ed.*, 1937, **9**, 558–562.)—The method depends on the quantitative precipitation of magnesium oxalate from a solution containing 85 per cent. of acetic acid, and has the advantage that alkali metals may be readily determined in the filtrate. The neutral solution containing the magnesium is concentrated by evaporation to 5 ml. When less than about 40 mg. of magnesium are present, 85 ml. of glacial acetic acid are added, followed by 10 ml. of saturated ammonium oxalate solution added slowly with constant stirring. If a larger amount of magnesium is present (up to 100 mg. maximum), only 70 ml. of glacial acetic acid should be added, and the magnesium is precipitated with 25 ml. of a solution containing 1 g. of ammonium oxalate in a mixture of 15 ml. of acetic acid and 10 ml. of water. When sulphate is present with the larger amounts of magnesium, the 25 ml. of the last-mentioned reagent should be added first, after being warmed, and then the 70 ml. of acetic acid; this avoids the danger of

precipitating magnesium sulphate by the acetic acid addition. The liquid is heated for 30 to 60 minutes on a steam-bath until the magnesium oxalate settles out in a flocculent mass, when it is filtered off and washed with hot 85 per cent. acetic acid. The filter is ashed in a platinum crucible, and the precipitate is ignited at the highest temperature obtainable with a Méker burner or blast-lamp and weighed as magnesium oxide. If not ignited at a sufficiently high temperature, the magnesia tends to be hygroscopic, and should be converted into magnesium sulphate for weighing. When alkali metal salts are present in large quantities, a double precipitation of the magnesium oxalate is required for accurate results: it is dissolved in hot 3 *N* hydrochloric acid, the solution is evaporated to dryness, and the oxalic acid is destroyed by heating with a mixture of 2 ml. of conc. nitric acid and 5 ml. of perchloric acid; the acids are removed by evaporation, the residue is dissolved in 5 ml. of water, and the precipitation is carried out as described above. For the determination of alkalis, the filtrate from the magnesium oxalate precipitation is acidified with 25 ml. of conc. nitric acid and evaporated to dryness; the oxalic acid is destroyed by heating with nitric and perchloric acids, which are removed by evaporation, and the alkali metals are determined by the usual processes. In test experiments results agreeing very closely with the theoretical were obtained. Sodium and lithium were completely removed by two precipitations. Large amounts of sodium, such as might be present after a sodium carbonate fusion, cannot, however, be separated conveniently, owing to the low solubility of sodium oxalate. Alkaline earths and manganese require prior separation. The magnesium oxalate may be determined volumetrically by permanganate titration: it is recommended that, after being heated on a steam-bath for an hour, the precipitation liquid should be centrifuged; the liquid is decanted, the precipitate is washed by decantation several times, with intervening centrifuging, and is finally dissolved in 5 per cent. sulphuric acid, and the oxalic acid is titrated with standard potassium permanganate solution in the usual way; 1 ml. of 0·1 *N* permanganate solution is equivalent to 0·001216 g. of magnesium. The centrifuging procedure is not recommended for use with more than 25 mg. of magnesium. S. G. C.

Determination of Chlorides and Bromides. L. A. Reber and W. M. McNabb. (*Ind. Eng. Chem., Anal. Ed.*, 1937, **9**, 529.)—The procedures described by Kolthoff and Sandell (*Quantitative Inorganic Analysis*, 1936, p. 544) were followed, but Volhard's method of titration was replaced by one using stable standard solutions. I. To 25 ml. of 0·03658 *N* potassium chloride solution were added 5 ml. of 6 *N* nitric acid followed by 25 ml. of 0·1 *N* silver nitrate solution. The precipitated silver chloride was coagulated by stirring, filtered off and washed with 60 ml. of *N* nitric acid. To the combined filtrate and washings 3 ml. of 0·5 per cent. starch solution and 0·1 ml. of 0·1 *N* ceric ammonium sulphate were added, and the silver was titrated with 0·1 *N* potassium iodide solution until a permanent blue-green colour appeared, 0·1 ml. being subtracted from the reading as indicator blank. II. To the same amount of potassium chloride solution 15 ml. of 6 *N* nitric acid were added, followed by 25 ml. of 0·1 *N* silver nitrate solution. The mixture was made up to 100 ml., shaken and filtered, the first portion of filtrate was rejected, and 50 ml. were titrated with 0·1 *N* potassium iodide solution as in I. The results agreed very closely with those obtained by the Volhard methods, in which the

filtrate is titrated with standard potassium thiocyanate solution, with the use of ferric alum as indicator. The methods were also checked by gravimetric determinations. Iodides may be determined similarly, and it is unnecessary to remove the precipitated silver iodide before titration. In the reverse titration (silver nitrate into the iodide solution) the end-point appears too soon, introducing an error of about 1 per cent., owing perhaps to adsorption of iodide ions by the silver iodide. D. G. H.

Determination of Cerium in Welded Copper Wire. E. Pache. (*Chem.-Ztg.*, 1938, **62**, 102.)—The metal (10 g.) is dissolved in *aqua regia*, the red fumes are boiled off, and the diluted solution is boiled and treated with 1 ml. of 10 per cent. ferric chloride solution and an excess of ammonia. The precipitate is collected after half-an-hour's settling and dissolved in dilute hydrochloric acid, and the solution is saturated with hydrogen sulphide. The sulphides are filtered off, the filtrate is boiled until free from the gas, oxidised with hydrogen peroxide, concentrated to 100 ml., and filtered from sulphur. The filtrate is rendered slightly ammoniacal, boiled, and treated with solid oxalic acid (5 g.). The precipitate dissolves, and, after some boiling, the cerium is precipitated as oxalate. This is left to settle, collected, washed with 1 per cent. oxalic acid solution until free from iron, and ignited to CeO_2. W. R. S.

Titration of Fluorine in Aqueous Solution. R. J. Rowley and H. V. Churchill. (*Ind. Eng. Chem., Anal. Ed.*, 1937, **9**, 551–552.)—The necessity for the use of a 48 per cent. alcoholic solution in Willard and Winter's method (*id.*, 1933, **5**, 7; Abstr., ANALYST, 1933, **58**, 242) can be avoided if the titration-liquid is buffered at *p*H 3. To the aqueous solution (100 ml.) containing 1 to 50 mg. of fluorine are added 8 drops of sodium alizarine sulphonate indicator (0·05 per cent. in water). The acidity is adjusted by means of dilute sodium hydroxide solution or dilute hydrochloric acid until the pink colour of the indicator is just discharged; 1 ml. of buffer solution (9·448 g. of monochloroacetic acid and 2·000 g. of sodium hydroxide in 100 ml., which furnishes a half-neutralised solution of monochloroacetic acid) is added, and the solution is titrated with 0·1 *N* thorium nitrate solution to the appearance of a permanent pink colour of the thorium-alizarine complex, which indicates the end-point. The thorium nitrate solution is standardised by titration of the distillate obtained from pure sodium fluoride or fluorspar according to Willard and Winter's method. S. G. C.

Determination of Silicon in Aluminium. Method for Referee Analysis. Callendar (*Ind. Eng. Chem., Anal. Ed.*, 1937, **9**, 533) refers to a paper by Churchill, Bridges and Lee (*Met. Absts.*, 1937, 348) containing the results of a comparison, by the American Society for Testing Materials, of the soda and the mixed acid methods for determining silicon. He gives results from his papers (ANALYST, 1932, **57**, 500; 1933, **58**, 81, 580) to show that when the silicon is entirely in solid solution the results by the mixed acid process are about 20 per cent. too low.

In a reply by Churchill, Bridges and Lee (*id.*, p. 534), Callendar's contention is accepted in view of further results, which they give, for metal annealed at 550–570° C. and quenched. They concur in the proposal that, for referee determinations of silicon in aluminium and aluminium alloys, the sodium hydroxide method (ANALYST, 1932, **57**, 506) should be used. L. H. C.

Microchemical

Micro-determination of Arsenic and Antimony. F. Hecht and M. v. Mack. (*Mikrochimica Acta*, 1937, **2**, 218–226.)—Arsenic is determined as magnesium ammonium arsenate by a micro method adapted from Dick's macro method (*Z. anal. Chem.*, 1933, **93**, 429), using Jena glass micro filter-beakers. The precipitate is not dried *in vacuo* but in a current of air. Antimony is determined as the tersulphide by a method adapted from that of Henz (*Treadwell's Analytical Chemistry*, 1930, 11th Ed., p. 179). Excellent results are obtained in the determination of the individual elements. The two methods can be combined to separate and determine both arsenic and antimony. J. W. M.

Micro-gravimetric Separation of Tin and Antimony. M. v. Mack and F. Hecht. (*Mikrochimica Acta*, 1937, **2**, 227–241.)—Determination of tin in the presence of other metals or considerable quantities of alkalis by precipitation with phenylarsinic acid and ignition is found to give unreliable results. In the absence of other metals, precipitation with ammonia and ammonium nitrate, followed by ignition of the precipitate to stannic oxide, gives accurate results. The method of precipitation with cupferron and ignition to stannic oxide is also reliable, and has been applied to the separation of tin from antimony, in the absence of large amounts of alkali salts. The cupferron precipitation is carried out in tartaric acid solution, the cupferron in the filtrate is oxidised by perhydrol in ammoniacal solution, and the antimony is then determined as antimony tersulphide. J. W. M.

Direct Acidimetric Micro-titration Method for Calcium. A. E. Sobel and S. Sklersky. (*J. Biol. Chem.*, 1938, **122**, 665–671.)—To 2·0 ml. of solution, containing 0·1 to 0·4 mg. of calcium ions, in a 10- or 15-ml. Pyrex tube, 1 ml. of saturated ammonium oxalate solution and 1 drop of bromocresol purple indicator (0·04 per cent.) are added. The sides of the tube are washed down with 2 ml. of water, and the whole is mixed. The *p*H is adjusted so that the solution is of a grey colour, and, after standing for one hour, the liquid is centrifuged. The supernatant layer is decanted, and the precipitate is suspended in 3 ml. of 0·5 per cent. ammonium oxalate solution and again centrifuged. The supernatant liquid is again decanted, and the washed precipitate is dried at 100–110° C. and heated in a muffle furnace or sand-bath at 475–525° C. for 20 to 30 minutes. The tube is then placed in a boiling water-bath, and 0·5 ml. of hot 10 per cent. boric acid solution is added. The precipitate of calcium carbonate dissolves completely in 1 to 2 minutes. The solution is diluted to 3 ml. with distilled water, and 1 to 2 drops of Patterson's indicator* are added. The solution is then titrated with 0·01 *N* sulphuric acid to the *p*H of a pure solution of boric acid of equal concentration, adding indicator to a control tube in which 0·5 ml. of boric acid solution is diluted to approximately 4·0 ml. The method is directly applicable to fresh serum except that at least 4 hours should be allowed for precipitation of

* Patterson's indicator is prepared by mixing 100 ml. of a 0·02 per cent. solution of methyl red with 30 ml. of a 0·1 per cent. solution of methylene blue and diluting to 500 ml.

the calcium oxalate. The average error of 71 determinations was −0·0006 mg.; the results obtained on blood serums agreed excellently with those given by the Kramer–Tisdall method. F. A. R.

Micro-determination of Potassium. B. Norberg. (*Mikrochimica Acta*, 1937, **1**, 212–219.)—Minute amounts of potassium, of the order of 10^{-4} mg.-equivalents, are determined by the platinic chloride method, with an error of the order of $1-2 \times 10^{-6}$ mg.-equivalents. The platinic chloride is then converted into the corresponding iodine compound and titrated with thiosulphate solution. *Reagents.*—(1) Approximately 0·02 *M* chloro-platinic acid: 0·1 g. of $H_2PtCl_6.4H_2O$ is dissolved in water and diluted to 10 ml. This solution will keep at 0° C. for at least a month. (2) Wash liquid: pure absolute alcohol. (3) Buffer solution of *p*H 6·98 to dissolve the precipitate: 4 ml. of 1/15 *M* potassium hydrogen phosphate solution and 6 ml. of 1/15 *M* disodium hydrogen phosphate are mixed with 90 ml. of water. (4) 2 *N* potassium iodide solution, free from iodine. (5) 1/50 *N* sodium thiosulphate solution. (6) Indicator colour: buffer solution of *p*H 6·36 (1/15 *M* potassium dihydrogen phosphate solution and 1/15 *M* disodium hydrogen phosphate solution mixed in the proportion of 3:1), of which 4 ml. are mixed with 6 ml. of a 0·04 per cent. aqueous solution of bromothymol blue. This solution will not keep longer than a week. *Detail.*—The precipitation is carried out in small centrifuge tubes, 2 cm. high, with either pointed or rounded tips. To the test solution 10 m.ml. of precipitating reagent are added and the water is evaporated by heating the tube in a metal block at 90° to 95° C. The excess of platinic chloride is removed by washing with 100 m.ml. of alcohol, the precipitate being stirred with a platinum needle. The tube is covered with a rubber cap and centrifuged. The supernatant liquid is carefully removed with a fine-tipped suction-pipette. It is advisable to note with the aid of a horizontal lens or microscope if any crystals are sucked up; in that event the liquid must be centrifuged again. When not more than 5 m.ml. of liquid remain in the centrifuge tube the washing procedure is repeated. Finally the tube is dried at 95° C., and 45 m.ml. of the boiling hot buffer solution (6·98 *p*H) are added carefully with a micro-pipette. The tube is then covered and left for 6 to 12 hours, after which 15 m.ml. of 2 *N* potassium iodide solution are added, and a Linderstrom–Lang magnetic stirrer is introduced. After 30 minutes the mixture may be titrated. The disappearance of the wine-red colour of the potassium iodo-platinate gives sufficient indication of the end-point in green light, obtained by using the colour solution (6) in a small cell, 3 mm. thick, with walls of clouded glass. The thiosulphate solution is added from a micro-burette of the Rehberg type, as adapted by Linderstrom–Lang. The method may also be applied to animal tissue, after ashing and removing the interfering ions by treatment with baryta. J. W. M.

Micro-silicate Analysis. F. Hecht. (*Mikrochimica Acta*, 1937, **2**, 188–208.)—Methods are given for the determination of fifteen of the most important constituents of a silicate mineral or a silicate rock, eight separate samples of the substance, weighing 10 to 20 mg. each, being used. The samples are allocated as follows: (1) SiO_2; (2) Al_2O_3, total iron, CaO, MgO, TiO_2, MnO; (3) H_2O (loss below 110° C.) and P_2O_5; (4) H_2O (above 110° C.); (5) total sulphur; (6) Na_2O and K_2O;

(7) TiO_2; (8) CO_2. "Loss on ignition" is considered useless, as it is derived from several components. (1) Silica is determined on a 10-mg. sample in a 20-ml. platinum crucible weighing 10 to 11 g. The sample is mixed with 0·2 g. of sodium carbonate and heated to 1000° C. in an electric furnace. After cooling and treatment with about 10 ml. of water, the alkali is neutralised with hydrochloric acid gas blown on to the surface of the liquid for about an hour, through a capillary tube passing through a cover glass. After being heated for half-an-hour, covered, on the water-bath, the solution is evaporated to dryness. Evaporation with hydrochloric acid is repeated twice. Finally, the residue is treated with 4 to 5 ml. of hydrochloric acid (1:3) and heated, and the liquid is filtered through a platinum filter stick, with spongy platinum filtering surface (Heraeus), into a second platinum crucible. The silica is washed first with 0·5 ml. of acid and then with 0·5-ml. portions of water until free from chlorides. The crucible containing the silica, together with the filter-stick, is dried, the silica is heated with 3 drops of sulphuric acid until fumes appear, and then for 3 hours in an electric furnace at 950° C. to volatilise any traces of alkalis. The heating is repeated until the residue attains constant weight. The silica is then volatilised with 0·5 ml. of nitric acid and 4 to 5 ml. of hydrofluoric acid, and the mixture is evaporated to dryness on the water-bath. The evaporation is repeated with 0·1 ml. of sulphuric acid and a little hydrofluoric acid, and the crucible is then heated to constant weight at 700° C. The difference in weight before and after volatilisation represents the silica, and to this is added the silica separated from the filtrate in the usual way.

(2) The mineral for the determination of Al_2O_3, Fe_2O_3, TiO_2, MnO, CaO, MgO, is decomposed as in the method of Berzelius, the hydrochloric acid solution is filtered through a platinum filter-stick into a porcelain crucible, and the R_2O_3 is precipitated with hydroxyquinoline reagent. The precipitate is dissolved and iron is separated as sulphide and finally determined as the oxine. Titanium and aluminium are determined together, and finally the titanium is precipitated as the cupferron compound, which is ignited and weighed. Calcium is determined as the oxalate, magnesium as the oxine compound, manganese having previously been separated as sulphide and weighed as Mn_3O_4. Complete analyses on five minerals gave percentages adding up to 100·11, 100·08, 101·59, 100·47, and 100·20.

J. W. M.

Physical Methods, Apparatus, etc.

Ultra-violet Light and Photomicrographic Studies of Animal Skin. E. R. Theis and E. J. Serfass. (*J. Amer. Leather Chem. Assoc.*, 1938, **33**, 67–79.)—The paper is divided into 4 parts, *viz.*: (1) A brief account of previous work on the subject is given. (2) *Transmitted Ultra-violet Light.*—Since ultra-violet light contains rays (notably those of 365$m\mu$) having wave-lengths shorter than those of visible light, it should give greater resolution of structure and detail, and comparative photographs of a section of calf-skin, 30μ thick, made after 24 hours' soaking, and stained with Weigert's stain, demonstrate that this is the fact; the magnifications used are 375× and 385×, respectively. A quartz lens system is required, but rather less expensive objectives which transmit ultra-violet light

have recently become available. Since these produce an image in the same plane with both ultra-violet and monochromatic green light, the techniques of focussing and photography are greatly simplified. (3) *Primary Fluorescence in Ultra-violet Light.*—Although several skin proteins and fats fluoresce in ultra-violet light, the fluorescence is weak, and structural differences are not, as a rule, readily observed. On the other hand, the primary fluorescence of oils may be used to study penetration phenomena associated with fat liquoring and "oiling-off." This is shown by a photograph of an unstained section, 50μ thick, of chrome-tanned calf skin, mounted in glycerin jelly (magnification 100×). Dark-field illumination with ordinary objectives may be used, and the method is free from the error due to the solvent action of the solution of sudan IV in alcohol required in the usual method. Foreign substances in leather, which may be missed by the usual staining methods, are also often detectable in this way. (4) *Secondary Fluorescence in Ultra-violet Light.*—This is illustrated by 8 further photomicrographs prepared by staining with geranine-GL (1:10,000), phosphine-G (1:5,000) or thioflavine-S (1:100,000). The colours of the respective fluorescence effects obtained by dark-field illumination are as follows:—elastin, white, white to yellow, white; collagen, faint pink, nil and nil; sebaceous glands, brilliant white, white, blue-white; epidermis, red, light orange, yellow to green; hair, green, blue, yellow to green. The specimens shown are oil glands and elastic fibres in calf-skin, the epidermal layer of calf-skin after soaking for 24 hours and also after 6 days in saturated lime solution, calf skin after dehairing in lime and bating for 1 hour, and calf-skin after dehairing for 4 hours in lime and sodium sulphide solutions. Photographs taken in visible light with the aid of three stains, namely, Weigert's stain with a counter-stain containing erythrosin and toluidine blue, are also shown for comparison. The presence of the hyaline layer and its subsequent decomposition during bating are demonstrated. Thus ordinary light shows that decomposition is actually taking place above the corium minor during liming, but it gives no idea where such decomposition begins. Ultra-violet light, however, not only shows the progress of the decomposition, but also indicates clearly that it occurs at the stratum germinatium and shows that neither the structure nor the content of the elastin is changed to any great extent during the soaking and liming periods; the gradual solution of the decomposed layer during bating is also visible. A thin fluorescent band just below the decomposed layer may be either a hyaline layer or a grain membrane. The former alternative is favoured because the grain membrane is known to consist largely of collagen fibres which are non-fluorescent. This is in accordance with the observation that the highly-hydrolysed cells in the stratum germinatium are the first to be attacked by the liming process. It is also known that the elastin-content is decreased by the bating process, but the present method indicates that the elastin is not completely removed, the individual fibres being foreshortened and the connecting links destroyed by strong bating. This may account for the difference in " feel " between well-bated and under-bated leathers.

J. G.

Reviews

THE THERMOCHEMISTRY OF THE CHEMICAL SUBSTANCES. By F. RUSSELL BICHOWSKY and FREDERICK D. ROSSINI. Pp. 460. New York: Reinhold Publishing Corporation; London: Chapman & Hall, Ltd. 1936. Price 35s. net.

This compilation is described on the title page as "the assembly of a self-consistent table of 'best' values for the heats of formation of the chemical substances (except carbon compounds containing more than two carbon atoms), including heats of transition, fusion and vaporisation." It will be seen, therefrom, that the title of the book is somewhat misleading, for it is not a text-book but consists merely of tables of thermochemical data.

The collection and critical selection of these data constitute not only an onerous task but one of exceedingly great difficulty. One of the compilers has already demonstrated his ability in carrying out such work, for he was responsible for the Section on Thermochemistry of the International Critical Tables. The present volume is a revision and extension of those tables. It must, therefore, be regarded as the standard work on the subject, and will be of considerable service to physical chemists.

The book falls into two main divisions. Approximately one-half is devoted to the tables themselves. In addition to the selected heats of formation, heats are recorded for the different physical states in which the elements may exist. The concluding half gives the actual heats of formation obtained, and discusses their probable accuracy. References to the original memoirs are included.

The compilation seems to have been carried out with considerable care, but here and there a few wrong spellings were noticed. To chemists on this side of the Atlantic it is somewhat irritating to find Sir William Ramsay's name misspelled.

The book is a welcome addition to the existing physico-chemical tables, and although its appeal is distinctly limited, it should, nevertheless, find a place on the shelves of university chemical libraries. H. T. S. BRITTON

POLYMERISATION, AND ITS APPLICATIONS IN THE FIELDS OF RUBBER, SYNTHETIC RESINS AND PETROLEUM. By R. E. BURK, H. E. THOMPSON, A. J. WEITH, and I. WILLIAMS. American Chemical Society Monograph. Pp. xviii + 524. New York: Reinhold Publishing Corporation; London: Chapman & Hall, Ltd. 1937. Price 37s. 6d.

There are some subjects which, ramifying like mycelia, penetrate into those regions of chemistry that for convenience are classed as "pure" and "applied." Polymerisation is a subject of this kind, and the preface to the volume under review points out that such subjects, not being "the special obligation of any one company to develop at its own expense and for the partial benefit of the others; do not receive systematic and enlightened attention. . . ." The editors of the A.C.S. monographs therefore are to be highly commended for the production of this book, which undoubtedly meets a wide need.

Apart from theoretical considerations, on matters such as the mechanism of polymerisation, "applied" subjects are well discussed; they include rubber, synthetic resins and petroleum. It is hardly necessary to say that all the authors are well-known experts. The scope of the "pure" side of the book is shown by the following chapter headings:—I, Introduction; II, The Relation between Molecular Structure and the Rate of Polymerisation; III, Catalysis and Polymerisation; IV, The Mechanisms of Polymerisation; V, The Liquid State and the Structure of Polymers. These constitute a notable review of the subject. For those in any way interested in the subject Chapter III, which reviews the whole of the existing literature on polymerisation catalysts in tabular form, is alone worth the price of the book.

The chapter on Polymerisation in the Rubber Industry, which is by Ira Williams, opens with a brief review on raw rubber, in which the author sums up the pros and cons for considering that polymerisation plays a part in the vulcanisation process. He concludes that "at present the process of vulcanisation is only partially understood, and it is probable that polymerisation plays an unimportant part" (see also ANALYST, 1938, 147).

The author then proceeds to discuss "Synthetic Rubber." Owing to the recent advent of various artificial rubbers (this term is to be preferred), the subject is of great interest and importance. The present account is both timely and valuable, and the concluding paragraph sets this much-discussed subject in a proper light, as follows:—NEW SYNTHETIC RUBBERS.—"Much improvement can be expected in the future in the art of producing rubber with special properties, and artificial rubber-like substances. It is unnecessary to attempt the duplication of natural rubber, but the most fruitful field for future development lies in the production of synthetic rubbers having properties which make them superior to natural rubber for special uses. Rubber having better resistance to abrasion, heat, low temperature, ozone, oxygen, oils and chemicals, would be desirable. Rubber with lower hysteresis loss, higher modulus of elasticity or greater extensibility would find wide application. Vulcanisable semi-liquid rubber, free from solvent, would also be extremely useful. New raw materials, which need not be conjugated dienes, and new methods of polymerisation should produce many new types of synthetic rubber in the future."

In Chapter VII, A. Weith discusses "Polymerisation and Synthetic Resins," and includes a brief account of drying oils. The importance of polymerisation in the production of plastics is well known, and this account may be strongly recommended to those interested in the subject.

The effects of polymerisation processes are not always desirable, and this applies particularly in the petroleum industry, where undesirable gum or sludge formation causes serious losses. This subject is fully discussed by R. E. Burk in Chapter VIII, which, too, is well worthy of recommendation.

Having regard to the numerous industries discussed, the book should have a wide sale. There are extensive lists of references (directly numbered in the text) at the end of each chapter, and very full author- and subject-indexes are provided. The book is well printed and bound, and should be widely welcomed at the reasonable price at which it is published.

W. H. STEVENS

LECTURE EXPERIMENTS IN CHEMISTRY. By G. FOWLES, M.Sc., A.I.C. Pp. xvi + 564. London: G. Bell & Sons, Ltd. 1937. Price 16s.

Books confined entirely to lecture experiments are comparatively few and very much out of date, the last being some thirty years old, and thus a new one is bound to be of, at least, passing interest.

The present volume "opens with a discussion of the relation of lecture experiments to the rest of teaching, and then deals with the technique of lecture demonstration." The main body of the work is arranged under definite subjects in an order expedient for teaching, and in this respect differs fundamentally from the old, but famous, works of Heumann, Benedict and Newth. The main chapter headings are:—Introductory: A Study of Water and Air; Acids, Bases and Salts; Sulphur and its Compounds; The Halogens, Phosphorus and Silicon; Quantitative Experiments; Physical Principles and Common Phenomena. Many of the experiments are prefaced by an historical note and references abound throughout the text. The book closes with three appendixes, one of which is an interesting thesis on teaching aims and methods as applied to chemistry.

On first reading through the book the experienced chemist may possibly become irritated by the minute details which are so numerous as to be wearisome, and apparently so trivial that scant compliment seems to be paid to the intelligence of the reader. He may wonder, too, why such experiments as numbers 149 and 532 (the former dealing with the absorption of bromine by acetylene, and the latter with the fire-extinguishing action of carbon dioxide) are recorded at all. The young teacher, on the other hand, will find such minutiae valuable in helping him to avoid failure and even disaster. The index, although copious, is not very complete, for many important items mentioned in the text are not easily traceable through the index.

Mr. Fowles's book is much more than its title claims; it is, in addition, a thesis on the teaching of chemistry. To the young and inexperienced the reviewer would recommend the whole book; to the old and disillusioned he would recommend the preface, the introduction and Appendix I. Throughout the book, and especially in these last-mentioned parts, there is much that is stimulating; also there is evidence of a fine enthusiasm for chemistry which, if caught by others, will do much to maintain chemistry as a leading subject in education. H. TOMS

VITAMIN D IN CACAO SHELL. REPORTS ON RECENT RESEARCHES, INCLUDING INVESTIGATIONS INTO ITS USE AS AN ACCESSORY FODDER. Edited by A. W. KNAPP. Pp. 64. Publication Dept., Bournville. Price 1s.

Most analysts must have read with interest the paper by Knapp and Coward (ANALYST, 1934, 474), in which the discovery of vitamin D in cacao shell was announced. Later work on the genesis of the vitamin and on the use of cacao shell as a cattle food may not be so familiar, but the whole story is here told in the form of reprints of the original papers, and is well worth reading. The claim made for cacao shell as a valuable accessory fodder appears to be substantiated, and it is to be hoped that its use for this purpose will not be neglected.

F. A. ROBINSON

THE MIND OF THE JUROR. By ALBERT S. OSBORN. Pp. xv + 239. The Boyd Printing Company, Albany, New York, U.S.A. 1937. Price $4.50.

Most Public Analysts are called upon from time to time to give evidence at the Assize Courts, and will therefore have some experience of the difficulty of presenting scientific evidence in such a way that it will be both intelligible and convincing to members of a jury who will rarely have had any scientific training. According to the author, however, the lot of the scientific witness is far happier in this country than in most of the American States. Mr. Osborn has had more than forty years' experience in the courts of practically all the United States of America and in Canada, and his book is largely based on his own observations of the reactions of the jury towards counsel and witnesses. Except in the State of New Jersey, where the trial system has much in common with that of Great Britain and Canada, the expert witness in America is handicapped by the restrictions governing the selection and functions of the jury. In England our haphazard method of selection may chance to result in the collection of an intelligent jury, but in most of the American courts a premium is set on lack of intelligence by allowing each household on the jury list to choose its representative, with the result that frequently the most unsuccessful members of a family become virtually the judges in a highly technical trial.

A still more serious drawback is that in most of the States the judge is precluded from helping the jury to sift the evidence, and the jurors have to find their own way through a mass of irrelevant detail and to assess the value of conflicting statements as interpreted by opposing counsel. It is small wonder, therefore, that the verdict may finally be given on some fact of minor importance.

Thus the impression left after reading this book is that it is primarily an indictment of the American jury system and a plea for its reform, but at the same time the work is informed with a shrewd wisdom which finds its expression in many terse aphorisms that crystallise the author's advice to those who have to plead or give evidence before a jury. For instance, "Technical accuracy alone is not sufficient, for it avails little to talk to a man in a language that he does not understand" (p. 136). "A little learning may not merely be dangerous; it may be fatal" (p. 140). "It is a common experience of expert witnesses that they give in some cases testimony that is twice as effective as in other cases, because of the proper and intelligent advance co-operation of attorney and witness" (p. 49). "Those who are most successful in convincing us are those who make us, or at least seem to make us, do some of the thinking" (p. 157).

The incompetent or dishonest specialist witness is not unknown in this country, but his systematic introduction into a trial to enable counsel to direct the attention of the jury to a conflict of testimony has fortunately not become the recognised practice, as it is in many of the American States. There it is open to anyone to put himself forward as an expert witness on any subject, provided that the judge does not object. In this respect a strong judge, notwithstanding the limitation of his powers, can still do much to protect litigants and the jury.

Referring to the dishonest witness, the author remarks: "The contention is made by some investigators that blood pressure, respiration and pulse are unconsciously affected by guilt and by perjury . . . some of us are inclined to think that if

this blood-pressure theory is true, there are others [in addition to the defendant] at a trial who should thus be tested."

This is a book that will repay careful re-reading, not only for what one can learn from it, but because it has a broad human outlook extending far beyond the narrow purview of the law courts. EDITOR

THE B.D.H. BOOK OF ORGANIC REAGENTS FOR DELICATE ANALYSIS AND "SPOT" TESTS. Sixth Edition. Pp. viii + 100. The British Drug Houses, Ltd. 1937. Price 2s. 6d.

The value of this little book to an analytical chemist lies (*a*) in the care taken by its compilers, the staff of the B.D.H. laboratories, to include only such reagents and methods as have been tried and found reliable, (*b*) its select list of references to original papers, and (*c*) its annual revision and extension.

This edition describes seventy-two organic reagents for the determination or detection of inorganic and organic substances. It is well written, is free from misprints, and contains a good index.

Since the last edition the following monographs have been amplified. That on alizarin-S now covers the determination of aluminium, and that on nitroso-R-salt, for cobalt, covers the indirect determination of potassium in biological material. The monograph on 2:4-xylen-1-ol, for the determination of nitrates, has been largely rewritten and extended. A new reagent, *p*-nitrobenzene-diazo-amino-azobenzene, for the detection of cadmium and magnesium, is included.

A few omissions were noted. There is no mention of the interference of bismuth with the diphenyl-thiocarbazone extraction method for the determination of lead.

As a reagent for the determination of tin, α-dinitro-diphenylamine-sulphoxide has been discredited (J. R. Nicholls, *Aids to Analysis of Foods and Drugs*, 1934).

The improvement effected by Clarke in the 4-methyl-1:2-dimercaptobenzene reagent for tin (ANALYST, 1937, **62**, 661) was unfortunately published too late for inclusion.

The full title of the book has been altered in order to place more emphasis on the use of organic reagents for quantitative work than on "spot" tests. If this is a first step to the removal of the word "spot" from the title, it is to be commended. Even when accompanied by apologetic inverted commas, its associations with blind chance, derived from its race-course usage in connection with a probable winner, is too close for scientific use. Is not that felicitous phrase "Delicate Analysis" a sufficient title in itself? F. L. OKELL