THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS

THE Annual General Meeting of the Society was held at 3.30 p.m. on Friday, March 7th, 1947, in the meeting room of the Royal Society, Burlington House, London, W.1. The chair was taken by the President, Dr. G. W. Monier-Williams. The Financial Statement for 1946 was presented by the Hon. Treasurer and approved and the Auditors for 1947 were appointed. The Report of the Council for the year ending March, 1947 (see pp. 131–137) was presented by the Hon. Secretary and adopted. The following were elected Officers and Council for the coming year.

President—Lewis Eynon, B.Sc., F.R.I.C.

Past Presidents serving on the Council—F. W. F. Arnaud, Bernard Dyer, E. B. Hughes, G. Roche Lynch, S. Ernest Melling, and G. W. Monier-Williams.

Vice-Presidents—R. C. Chirnside, D. W. Kent-Jones, (Mrs.) J. W. Matthews and, *ex officio*, C. H. Manley (Chairman, North of England Section), and H. Dryerre (Chairman, Scottish Section).

Hon. Treasurer—G. Taylor.

Hon. Secretary—K. A. Williams.

Other Members of Council—C. A. Adams, R. Belcher, H. E. Cox, (Miss) I. H. Hadfield, J. H. Hamence, J. Haslam, A. D. Mitchell, J. R. Nicholls, Norman Strafford, R. W. Sutton, H. N. Wilson, E. C. Wood and, *ex officio*, Arnold Lees (Hon. Secretary, North of England Section), and R. S. Watson (Hon. Secretary, Scottish Section).

After the business outlined above had been completed the meeting was opened to visitors, and the retiring President, Dr. G. W. Monier-Williams, O.B.E., M.C., M.A., F.R.I.C., delivered his Presidential Address, in which he made reference to some outstanding features in the affairs of the Society during his tenure of the presidency, and then proceeded to a consideration of certain aspects of food law administration with particular reference to the control of the use of preservatives and the presence of injurious substances in foods (see pp. 137–142).

NEW MEMBERS

Richard Ralph Appleby, B.Sc. (Lond.); Geoffrey Browne, B.Sc.(Lond.), F.R.I.C.; Stanley James Bush, A.R.I.C.; Terence John Cahill, F.R.I.C.; (Miss) Eileen Mary Chatt, B.Sc. (Lond.), F.R.I.C.; Thomas Morton Clark, B.Sc. (Lond.); Harry Kenneth Dean, B.Sc., Ph.D. (Liv.), F.R.I.C.; John Arthur Eggleston, B.Sc. (Lond.), A.R.I.C.; Charles Arthur Hallas, B.Sc.(Lond.), F.R.I.C.; Wynford Price Jones, A.R.I.C., Ph.C.; Ralph William Latter, B.Sc. (Lond.), A.R.I.C.; George Frederick Longman, B.Sc. (Lond.), F.R.I.C.; Thomas Barton Mann; John Vernon Morris, B.Sc. (Lond.), A.R.I.C.; Geoffrey Padget, Assoc.Met. (Sheff.); Arthur Edward Pickford, A.R.I.C.; Joseph Brian Rickson, B.Sc. (Lond.), A.R.I.C.; Albert Arthur Smales, B.Sc. (Lond.), A.R.I.C.; William Stross, M.D. (Prague); Cyril Gordon Sumner, M.Sc., Ph.D. (Manc.), A.R.I.C.; Leslie Stuart Theobald, M.Sc. (Lond.), A.R.C.S., F.R.I.C.; Walter Frederick Wilkinson, M.P.S.

Anniversary Dinner

IN the evening of the day of the Annual General Meeting the Society held an Anniversary Dinner, the first since 1938, at the Dorchester Hotel, Park Lane, London, W.1, to celebrate its seventy-third anniversary.

The members and guests, who numbered 110, were received by the President, Dr. G. W. Monier-Williams and Mrs. Monier-Williams The President afterwards took the chair at the dinner.

The guests of the Society and of the President were: Professor Sir Robert Robinson, M.A., D.Sc., LL.D., F.R.I.C., President of the Royal Society; Sir Arthur S. MacNalty, K.C.B., M.D., F.R.C.P., K.H.P., formerly Chief Medical Officer, Ministry of Health; P. N. R. Butcher, Esq., Assistant Secretary, Ministry of Health; Dr. J. Alison Glover, O.B.E., F.R.C.P., formerly

Senior Medical Officer, Ministry of Education; Dr. G. M. Bennett, M.A., Sc.D., F.R.I.C., F.R.S., Government Chemist; Professor Alexander Findlay, M.A., Ph.D., D.Sc., LL.D., Past President of the Royal Institute of Chemistry; Dr. L. H. Lampitt, F.R.I.C., M.I.Chem.E., President of the Society of Chemical Industry; R. O. Hall, Esq., and Mrs. Hall.

After the loyal toasts, proposed by the President, had been duly honoured, Professor FINDLAY proposed the health of the Society. Beginning with an outline of the early history of the Society, founded by a small number of Public Analysts who were deeply interested in the health and welfare of the community and anxious that the administration of the then newly enacted Food and Drugs Adulteration Acts should not fall into the hands of incompetent or unscrupulous practitioners, Professor Findlay traced its development, during the past seventy-three years, to its present position as one of the leading Societies for the study and propagation of analytical chemistry. This progress had been made in close collaboration and co-operation with the Royal Institute of Chemistry and had been greatly accelerated and expanded by the inclusion, in the Society, of analytical chemists other than Public Analysts and, more recently, by the formation of groups within the Society, for the special study of microchemical, physical, and biological methods of analysis. These ever-growing activities, coupled with the increased circulation and prestige of THE ANALYST, which had now become the most important journal of analytical chemistry in this country and one of the most important in the world, had made it necessary for the members to realise that the Society had now become the guardian of analytical chemistry. He expressed regret that there should still be no Chair of analytical chemistry in Great Britain, although his own University, Aberdeen, had recently appointed a member of the Society as a lecturer in that subject. He concluded by recalling older, more intimate annual dinner parties which were often enlivened by musical contributions by members. He particularly regretted that Dr. Bernard Dyer, the sole remaining original member of the Society, was unable to be present, though he was quite well and with us in spirit.

The PRESIDENT, in replying to the toast, thanked Professor Findlay, on behalf of the Society, for the kind way in which he had referred to its activities. He said that although our Society was not the oldest of the publishing societies (that honour fell to the Chemical Society, which was holding its delayed centenary celebrations this year) it had undergone a more significant development than any of the others. Starting as a very small group of enthusiastic men, for the most part Public Analysts, it had now become the central society for all analytical chemists, but despite its great increase in membership and diversity of interests, it had retained throughout its existence a close association with Public Analysts, whose great services to our Society, throughout its past and at the present time, was recognised by all our members. The proposer of the toast had mentioned the remarkable diversity of analytical methods that had been evolved in recent years. In these activities the Chairmen and Committees of the new subject Groups were to be congratulated upon the valuable work that they were doing. The success of the Society as a whole was proved by the demand for THE ANALYST, particularly by increased sales to non-members. For many years THE ANALYST had been well served by Dr. Ainsworth Mitchell. It had now a strong Publication Committee and an augmented editorial staff, that looked like having some hard work ahead. The Publication Committee prided itself on producing a distinctive type of informative abstract. The difficulty of covering the whole of the published analytical work was almost insuperable; that used for abstracting was selected with care and discrimination from the original papers that appeared to be of the greatest practical value. In conclusion, he thanked Professor Findlay for the interest that he had always shown in analytical chemistry as an educational subject and for the able and effective way in which he had furthered the interests of our Society.

Mr. A. L. BACHARACH, in proposing the health of the kindred Societies, welcomed Sir Robert Robinson, President of the Royal Society, who in proposing a similar toast, some years ago, had recognised the Society of Public Analysts as one of the Learned Societies of this country. He wished Sir Robert a safe journey and success in his forthcoming visit to America. He regretted that the President of the Chemical Society had found it impossible to be present. On behalf of the Society of Public Analysts he wished the Chemical Society, and its President, a successful celebration of its centenary, which should have been held six years ago; for not only was that Society the oldest, it was also the most important Chemical Society in the world. He regretted also the absence of the President of the Royal Institute of Chemistry, Dr. G. Roche Lynch, who was on a visit to Ireland, but we had the pleasure of the presence of his

predecessor in that office, Professor Alexander Findlay. In Dr. Lampitt we had with us the President of the Society to which we were the most closely linked by our activities, the Society of Chemical Industry. Dr. Lampitt was concerned with food not only as the founder of that Society's Food Group, but also as its first Chairman and the instigator of the annual joint meeting held by the Group and our Society. For his part, he saw no reason why the practice of joint meetings should not be extended to other Groups of the Society of Chemical Industry.

Dr. LAMPITT, replying for the Kindred Societies, said that they were kindred in spirit—the spirit of science at its best and free from the baser uses to which science could be put. But he trusted that while retaining the spirit of kinship and co-operation, they would never amalgamate, for only through competition could they remain virile.

The health of the Guests, coupled with the name of Dr. G. M. Bennett, Government Chemist, was proposed by Dr. H. E. Cox, who likened his Toasts to a constellation of stars, and in a felicitous speech placed them in their several orders of magnitude and traced their various orbits.

Dr. G. M. BENNETT, replying for the guests, thanked the Society for an enjoyable evening as well as for the opportunity of acknowledging a debt owed to the Society by all chemists. He said that THE ANALYST had a very high standard and that he had been much impressed by its prompt publication of papers; in this respect it compared favourably with journals of other societies. He mentioned the Society's entry into the Chemical Council and said that this would be to their mutual advantage. He spoke of the cordial relationship that exists between his Department and the Society, also of the pleasure that he took in helping its members in any way possible.

Sir ROBERT ROBINSON made some noteworthy observations on the importance of analytical chemistry and said that a committee had recently been formed to discuss the teaching of that branch of science in universities. He urged the need for a University Chair in analytical chemistry and asked for the active assistance of all chemists in securing that object.

In proposing the health of the President Elect, Mr. Lewis Eynon, the PRESIDENT said that Mr. Eynon had served the Society as Honorary Secretary for the past eleven years, but that he, personally, had known him for many more years than that, having met him for the first time in a trench in Northern France. He assured the members that a better man could not have been chosen for the office of President.

Mr. EYNON replied in a short and appreciative speech.

Finally the PRESIDENT proposed that a message be sent to Dr. Bernard Dyer conveying the Society's good wishes and lively appreciation of his work in its interest. The proposal was received with acclamation.

Annual Report of Council: March, 1947

THE roll of the Society numbers 1345, an increase of 75 over the membership a year ago.

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

H. G. Battye	H. Firth	T. McGrath
J. J. Blackie	J. A. Foster	R. S. Morrell
G. E. Boizot	P. F. Frankland	M. Nierenstein
A. Bruce	C. D. Garbutt	F. E. Needs
J. L. Buchanan	A. D. Heywood	W. H. Roberts
W. J. A. Butterfield	E. Hinks	K. H. Vakil
W. R. Dracass	P. H. Jones	G. H. Warburton
G. W. Edwards	J. F. Liverseege	S. Watkinson

Battye, who died in his 58th year, was elected a member of the Society in 1940. He studied at the Central Technical College and Leeds University, and after being engaged in private practice for some years he went to Canada. In 1941 he became an Inspector of Explosives and was subsequently a chemist in the Department of Mines, Ottawa.

Boizot, who died at the age of 33, received his scientific training at Battersea Polytechnic and obtained the B.Sc. degree with first class honours. He was subsequently employed by May & Baker, Ltd., and in 1931 became Food Analyst to United Dairies, Ltd. In 1938 he

was appointed Assistant Government Analyst at Singapore. He died in 1943 whilst a prisoner of war.

Bruce, who died in his 69th year, was elected a member of the Society in 1925. He studied at Edinburgh University and obtained the B.Sc. degree in 1900. After a short period of service at the Royal Arsenal, Woolwich, he went to Ceylon and engaged in consulting and analytical practice. He published a number of papers and became a Fellow of the Royal Society of Edinburgh.

Buchanan, who died at the age of 73, became a member of the Society in 1923. He worked for some years in the laboratory of Tatlock, Readman and Thomson, City Analysts, Glasgow. In 1894 he joined Messrs. Lever Bros. at Port Sunlight, became a Managing Director in 1911 and, in 1922, Chairman of the Associated Company, John Knight, Ltd.

Butterfield, who died in his 80th year, had been a member of the Society for 43 years. He received his scientific training at Oxford, obtaining the B.A. degree with honours in chemistry and subsequently M.A. In 1891 he became chemist to the Gas Light and Coke Co., Beckton, and afterwards a Gas Referee under the Board of Trade, and in 1938 Advisor to the Board of Trade under the Gas Undertakings Act, 1934. He was also engaged in consulting practice. He served for two periods as an Ordinary Member of the Council of the Society and, in 1920–21, as Vice-President.

Dracass, who died in his 37th year, was elected a member of the Society in 1933. He studied at King's College, London, where he graduated B.Sc. in 1930, and at the Sir John Cass Technical Institute, obtaining the M.Sc. degree in 1935. He worked for some years in the laboratory of the late Mr. E. Hinks, then with Messrs. Burroughs, Wellcome & Co., and in 1944 he became Senior Analyst to Dr. H. E. Cox until, shortly before his death, he became employed by Allied Supplies, Ltd.

Edwards, who died in his 26th year, was elected a member of the Society in 1944. He was trained at the Manchester College of Technology and was for 8 years an assistant analyst to Messrs. Melling & Ardern. In 1943 he was appointed as a chemist to the War Department.

Firth, who died in his 46th year, became a member of the Society in 1926. He received his scientific training at Bradford Technical College. After some years of analytical experience he became Senior Analyst to Lipton, Ltd., and later, Chemist to C. & T. Harris (Calne), Ltd. In 1937 he became Manager of the Food Cannery of Messrs. J. Travers & Son.

Foster, who died in his 75th year, had been a member of the Society for 46 years. After a period of service as Second Assistant Admiralty Chemist he became Public Analyst for the East Riding of Yorkshire, Grimsby, Beverley and Louth, Official Agricultural Analyst for Grimsby and Gas Examiner for Lincolnshire.

Frankland, who died at the age of 88, was elected an Honorary Member in 1909. He occupied the Chairs of Chemistry at University College, Dundee, and Birmingham, successively. He was elected President of the Institute of Chemistry in 1906 and of the Chemical Society in 1911, and was awarded the Davy Medal of the Royal Society in 1919.

Heywood, who died in his 57th year, had been a member of the Society for 35 years. After studying at University College, London, and long experience in analytical work he was appointed head of the laboratory of Messrs. Courtaulds, Ltd., Castle Works, Flint, and subsequently Technical Manager at the Wolverhampton works of the same firm.

Hinks, who died in his 67th year, had been a member of the Society for 41 years. He received his scientific training at King's College, London, and obtained the B.Sc. degree. He became assistant to Sir Thomas Stevenson, Home Office Analyst and Public Analyst for Surrey, succeeding to the latter appointment on Stevenson's death. He subsequently held a number of other appointments and was also engaged in private practice. For his services as an officer in the Army Ordnance Corps during the 1914–18 war he was awarded the M.B.E. He served on the Council of the Society continuously from 1911 until his death, first as an Ordinary Member, then as Honorary Treasurer, then as President (in 1928–29), and finally as Past President. (Obituary, ANALYST, 1946, **71**, 347.)

Jones, who died in his 84th year, was elected a member of the Society in 1930. Having qualified as a pharmacist, he attended University College, Liverpool, and qualified as A.I.C. in 1894. He became asistant to Professor Campbell Brown and subsequently senior assistant analyst to Professor W. H. Roberts, Public Analyst to the City of Liverpool. He published numerous papers on the microscopy of starches and spices.

Liverseege, who died in his 83rd year, had been a member of the Society for 53 years. He was trained at University College, Nottingham, and Mason College, Birmingham. In

1885 he became assistant to the City Analyst for Birmingham and succeeded to that appointment in 1902, retiring in 1929.

Morrell, who died in his 80th year, became a member of the Society in 1920. From Bradford Grammar School he went to Gonville and Caius College, Cambridge, in 1886. After a period at Wurzburg he returned to his College as a Fellow in 1896 and retained his Fellowship until 1904; then he went to Mander Bros., the well-known paint and varnish manufacturers at Wolverhampton, and retained the position of Chief Chemist until 1930.

Needs, who died in his 63rd year, was elected a member of the Society in 1934. He was trained at the Merchant Venturers Technical College and Bristol University. For some years until 1914 he was assistant to the Public Analyst for Bristol and during the war of 1914–18 served in the R.E. and R.F.A. After that war he became Senior Assistant in the Bristol Corporation Laboratory and in 1930 was appointed Public Analyst for Bristol.

Roberts, who died in his 69th year, had been a member of the Society for 35 years. He graduated at Victoria University and subsequently took the M.Sc. degree. He became City Analyst for Liverpool at the age of 34 and held many other Public Analyst appointments and the Associate Professorship in Public Health Chemistry at Liverpool University. He served the Society as Ordinary Member of the Council, Vice-President in 1934–5 and President in 1938–9. (Obituary, ANALYST, 1947, **72**, 36.)

Watkinson, who died at the age of 60, was elected a member of the Society in 1943. He worked at first in the laboratory of the late Mr. A. H. Allen, and in 1916 went to the Great Central Laboratory, Gorton, Manchester. In 1925 he was moved to Doncaster as a result of the railway amalgamation and was District Chemist there until his retirement.

ORDINARY MEETINGS—Five meetings of the Society were held during the year and the following papers were communicated:

"The Determination of Boron." By E. C. Owen, B.A., M.Sc., Ph.D.

"Separation of the Cobalt Complex of Nitroso-naphthol from other Coloured Metallic Complexes." By E. Boyland, D.Sc.

"Exhibition and Description of Apparatus:

1. For the Control of Delivery from Burettes.
2. A Vacuum-operated Circulating Pump.
3. A Thermostatically-controlled Low-Temperature Bath.
4. For the Continuous Production of Doubly Distilled Water."

By J. T. Stock, M.Sc., F.R.I.C., and M. A. Fill.

"The Microbiological Assay of Amino Acids. I. The Assay of Tryptophan, Leucine, *Iso*leucine, Valine, Cystine, Methionine, Lysine, Phenylalanine, Histidine, Arginine and Threonine." By E. C. Barton-Wright, D.Sc., F.R.I.C.

"The Microbiological Assay of Amino Acids. II. The Distribution of Amino Acids in the Wheat Grain." By E. C. Barton-Wright, D.Sc., F.R.I.C., and T. Moran, D.Sc., Ph.D.

"The Determination of Some Products of Sugar and Molasses Fermentations." By G. G. Freeman and R. I. Morrison.

"The Estimation of the Volatile Matter Content of Propellant Explosives. Part I—The Estimation of Water by an Improved Fischer Method." By T. G. Bonner.

"The Analysis of Barium Carbide." By A. H. Edwards.

"The Fundamental Laws of Polarography." By J. Heyrovsky, D.Sc., Ph.D.

"Reductometric Determination of the Sulphoxide and Amine Oxide Groups." By Mrs. Erica Glynn.

"The Determination of the Composition and Constitution of Ammonium Phosphomolybdate and the Conditions affecting its Precipitation." By W. P. Thistlethwaite, B.Sc., A.R.I.C.

The December Meeting was a Joint Meeting with the Food Group of the Society of Chemical Industry. The subject was "The Application of Statistical Methods to Food Problems," and the following papers were read and discussed:

Introductory Address: "The Inevitability of Statistics." By D. J. Finney, M.A.

"The Use of Statistical Analysis in Research on Food Canning." By W. B. Adam, M.A., F.R.I.C.

"The Evaluation of the Nutritive Value of Animal Feeding Stuffs." By Dr. K. L. Blaxter.
"Application of Statistical Methods in Calculating Proportions of Ingredients in Certain Food Products." By E. H. Steiner, B.Sc., A.R.I.C.

RULES OF SECTIONS AND GROUPS—The Rules of Sections and Groups, prepared by the Special Committee appointed for that purpose, were approved by the Council and issued to all members of the Society with the December number of THE ANALYST.

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

"A Photoelectric Method of Assaying Vitamin A in Margarine." By J. L. Bowen, N. T. Gridgeman, B.Sc., and G. F. Longman, B.Sc., F.R.I.C.
"The Determination of Carotene and Vitamin A in Butter and Margarine." By T. W. Goodwin, M.Sc., A.R.I.C., and Prof. R. A. Morton, Ph.D., D.Sc., F.R.I.C.
"An Application of Photoelectric Spectrophotometry to the Analysis of Mixtures." By Prof. R. A. Morton, Ph.D., D.Sc., F.R.I.C., and Dr. L. A. Stubbs.
"The Determination of the Pyridine Content of Technical Pyridine." By A. Hamer, B.Sc., A.R.I.C., R. Pomfret, A.R.I.C., and W. V. Stubbings, B.Sc., A.R.I.C.
"The Rapid Determination of Sodium in 50 Per Cent. Potassium Hydroxide Liquor, 50 Per Cent. Potassium Carbonate Liquor and Solid Potassium Carbonate." By J. Haslam, M.Sc., F.R.I.C., and J. Beeley.
"The History of Chocolate and Sugar Confectionery." By Dr. L. E. Campbell, F.R.I.C.
"The Determination of Traces of Lead, Zinc and Tin in Phenol." By W. Hutchinson and H. N. Wilson, F.R.I.C.
"Soil Biochemistry." By Dr. H. Lees, B.Sc., A.R.I.C.

There have been good attendances at the meetings. The Hon. Secretary wishes to express his appreciation of the loyal support and assistance accorded to him by the Chairman, Vice-Chairman and members of the Committee during the year.

SCOTTISH SECTION—Three meetings were held during the year, one of them jointly with the Physical Methods Group, at which the following papers were presented and discussed:

"Determination of Vitamins A and A_2 by Photoelectric Spectrophotometry with some remarks on General Principles." By Professor Morton and Dr. A. L. Stubbs.
"Modern Aids to Spectroscopy." By B. S. Cooper, B.Sc., F.Inst.P.
"Application of Spectrographic Analysis to Soil Investigations." By Dr. R. L. Mitchell.
"Spectrographic Analysis of Rare and High Purity Materials." By D. M. Smith, A.R.C.S., B.Sc., F.Inst.P.
"Semimicro Method for the Determination of Magnesium." By James A. Hunter, B.Sc.
"Some Observations on the New Ice Cream Order." By C. W. Herd, Ph.D., B.Sc., F.R.I.C.

The total membership remains the same as for the previous year, *viz.*, sixty-four.

MICROCHEMISTRY GROUP—Two meetings were held during 1946, one in London and one in Cardiff. The latter was held jointly with the Cardiff and District Section of the Royal Institute of Chemistry and the South Wales Section of the Society of Chemical Industry. It was also intended to hold a meeting in Birmingham during September, but owing to circumstances beyond control the arrangements had to be cancelled.

The following papers have been read:

"Chemical Microscopy in Metallurgical Analysis." By Miss I. H. Hadfield.
"A Review of Methods for the Micro Analysis of Gases." By W. A. Kirby.
"Determination of Cyanide by the Picrate Method: A Water-bath for Heating Simultaneously many Tubes of Reactants." By J. G. A. Griffiths and J. K. Whitehead.
"Some Observations on the Kjeldahl Method for the Determination of Nitrogen." By A. E. Beet.
"Methods for the Construction of Microchemical Apparatus." By R. Belcher.

Three meetings will be held in 1947. These include the Annual General Meeting, held in London in January, a meeting at Sheffield in May, and it is hoped a meeting at Cambridge jointly with the Physical Methods Group in September.

The number of Group members is now 181, an increase of 21 since the last Report.

Activities of the Committee—The Committee has met three times during the year. Following the proposal made at the last Annual General Meeting, British Intelligence Objectives Service was asked if a team could be sent to Germany and Austria to report on advances in microchemical methods made during the war. The matter was referred to the Department of Scientific and Industrial Research who agreed to sponsor such a team, and the Committee was invited to make nominations for the team. It was hoped that the trip would take place in September, but it has been postponed several times. The delays are stated to have been due to other bodies demanding representation on the trip, and it is now expected that two teams will travel.

The report on educational institutions teaching microchemistry, prepared by Dr. Cecil L. Wilson, has been published in *Nature* and reprints have been circulated to all members.

A census has also been taken of industrial laboratories using microchemical methods; Dr. Wilson kindly undertook the task at the request of the Committee. A report is now being prepared.

PHYSICAL METHODS GROUP—During the past year the activities of the Physical Methods Group have expanded considerably. The policy announced last year has been continued and each meeting of the Group has taken the form of a symposium on a particular physical method of analysis. Two meetings have been held in London and one each in Edinburgh and Cardiff. The Edinburgh meeting was held jointly with the Scottish Section of the Parent Society and the Cardiff meeting jointly with the Cardiff and District Section of the Royal Institute of Chemistry and the South Wales Section of the Society of Chemical Industry. All the meetings were well attended. The following papers were read at meetings of the Group:

Barker Index Meeting in London on November 28th, 1945.
"The Barker Index." By R. C. Spiller.
"The Utility of the Barker Index in Analytical Chemistry." By M. W. Porter and A. E. J. Vickers.

Chromatographic Analysis Meeting in London on January 26th, 1946.
"General Principles." By F. A. Robinson.
"Partition Chromatography." By R. L. M. Synge.
"Chromatography in the Analysis of Fatty Oils." By K. A. Williams.
"Some Applications of Chromatographic Analysis in Industry." By F. R. Cropper.

Spectroscopic Analysis Meeting in Edinburgh on May 23rd, 1946.
"Photoelectric Spectrophotometry Applied to the Analysis of Mixtures and Vitamin A Oils." By R. A. Morton and A. L. Stubbs.
"Modern Aids to Spectroscopy." By B. S. Cooper.
"Applications of Spectrographic Analysis to Soil Investigations." By R. L. Mitchell.
"Spectrographic Analysis of Rare and High Purity Materials." By D. M. Smith.

Electrometric Analysis Meeting in Cardiff on October 11th, 1946.
"Recent Developments in Apparatus for *p*H Measurements and Electro-titrations." By A. D. E. Lauchlan.
"Some Applications of Electrometric Methods to Analysis." By R. J. Carter.
"Polarisation End Points." By D. P. Evans.

The Publication Committee of the Parent Society has decided to publish in booklet form the proceedings of some of the Group meetings. The booklets covering the Polarography, Chromatography and Spectroscopy meetings will be available shortly.

In May, Dr. F. Wokes resigned from the office of Honorary Secretary and the Committee appointed Dr. J. E. Page to succeed him. The sincere thanks of the Group are due to Dr. Wokes for all the work that he has done since the inception of the Group.

The Committee of the Group has formed a Polarographic Discussion Panel. The objects of the Panel will be to hold and sponsor informal discussions on polarographic analysis. The Committee of the Panel will be elected at the forthcoming Annual General Meeting of the Group.

The Group has been represented on the Vitamin Sub-Committee of the Analytical Methods Committee by Mr. G. F. Lothian and Dr. F. Wokes and on the Barker Index Committee by Dr. J. G. A. Griffiths, Dr. J. H. Hamence and the Honorary Secretary.

The number of the Group members is now 148, an increase of 20 since the last Annual General Meeting.

BIOLOGICAL METHODS GROUP—In the Report of the Provisional Committee which was presented at the first Annual General Meeting of the Group on February 25th last it was stated that the Committee was considering a Draft of Rules for the constitution and conduct of the Groups of the Society which had been put forward for consideration by the Council. A final version of these Rules has now been agreed upon and has been sent to every member of the Group. It is provided in these Rules that the Annual General Meeting of the Biological Methods Group shall be held in December of each year, and it will therefore be convenient for the business year to end on the 30th November. This Report, therefore, covers the period from the first Annual General Meeting up to the 30th November, 1946.

During this period two meetings have been held. The Annual General Meeting was followed by an Ordinary Meeting at which Messrs. N. T. Gridgeman and E. C. Feiller read papers entitled respectively "The Transformation of Metameters, with special reference to Vitamin D Assays," and "Some Remarks on the Statistical Background of Bio-Assays." At a subsequent meeting on the 27th May papers were read by Dr. K. Mather and Dr. G. Pontecorvo on the genetical background of bio-assays, Dr. Mather dealing with the larger animals, and Dr. Pontecorvo with micro-organisms. Both of these meetings were well attended, not only by members, but also by a number of visitors, some of whom have since joined the Society. The membership of the Group is now 87.

PUBLIC ANALYSTS AND OFFICIAL AGRICULTURAL ANALYSTS COMMITTEE—The Committee met during the past year on three occasions. Recommendations have been made in respect of financial matters directly concerning Public Analysts, also in relation to definitions of cocktails and other liquors; some of the latter have since been adopted in part by the Ministry of Food. Two Bulletins have been issued to Public Analysts and a quantity of routine business transacted.

ANALYTICAL METHODS COMMITTEE—Reports of good progress have been received from sub-committees. The appeal published in THE ANALYST (1946, p. 300) to assist the work of the Standard Methods Committee on compilation of a bibliography met with a very satisfactory response and much progress has been made. Considerable work has been done by the Sub-Committees on Vitamin Estimations, Gum Tragacanth and Determination of Metallic Impurities in Foodstuffs.

Two reports from the Committee have been published during the year, *viz.*

"The Determination of Phenols in Soaps." ANALYST, 1946, p. 301.

"Report on the Microbiological Assay of Riboflavine and Nicotinic Acid." ANALYST, 1946, p. 397.

During the year the Committee lost the services by resignation of W. H. Simmons as Hon. Secretary, a position he had held since the inception of the Committee, in 1924, but he remains an active member. Mr. Simmons, whose valuable work for the Committee is greatly appreciated, has been succeeded by Dr. D. C. Garratt.

HON. TREASURER'S REPORT—The financial position for 1946 is satisfactory inasmuch as the income balances the expenditure. But it is the view of the Hon. Treasurer that, having regard to the probability of increased expenditure, due mainly to the increasing activities of the Society, and to the increasing cost of publication of THE ANALYST, it is desirable that the future commitments of the Society should now be critically examined.

THE ANALYST—At the beginning of 1946 the restricted consumption of paper permitted to THE ANALYST by the Paper Control made it difficult to accommodate the increasing number of original papers offered for publication, and some restriction of space given to the other sections of the journal became necessary. Later the paper position was greatly eased by a considerable increase in the permitted consumption, granted on the ground that the journal was "primarily concerned with the first publication of original research." The 1946 volume has 600 pages, compared with 486 in the 1945 volume and 388 in 1944. The number of original papers published in 1946 was 80, compared with 56 in 1945 and 40 in 1944. On the other hand there were only 31 Notes in 1946, compared with 45 in 1945 and 39 in 1944; but these are not a very important factor in the distribution of space in the journal. The circulation of the journal was greater by several hundreds in 1946 than in 1945.

A new development this year is the issue, in the form of stiff-covered reprints, of certain important papers or sets of papers from THE ANALYST. Three are on the point of publication,

viz., the Symposia of Polarography, Chromatography and Spectroscopic Analysis, respectively, held at the meetings of the Physical Methods Group.

In consequence of the increasing quantity of matter in THE ANALYST and the need for more efficient organisation of the Abstract section of the journal, it has been found necessary to augment the editorial provisions hitherto made. The Publication Committee have been fortunate in obtaining for this purpose the part-time services of Mr. F.L. Okell, F.R.I.C., as general Assistant Editor, and Mr. L. S. Theobald, M.Sc., A.R.C.S., F.R.I.C., as Associate Editor responsible particularly for the Abstract section of the journal.

EMERGENCY COMMITTEE—This Committee was appointed in September, 1938, with full power to act on behalf of the Council should a state of emergency warrant it. The Committee has met on several occasions since its appointment. The state of emergency having passed, the Committee was not re-appointed for 1946–47.

CHEMICAL COUNCIL—The Society has been co-opted on the Chemical Council and the President and Honorary Treasurer have been appointed as representatives of the Society on the Council.

ROYAL INSTITUTE OF CHEMISTRY—Dr. G. Roche Lynch, Past President of the Society, was elected President of the Royal Institute of Chemistry in March, 1946. The Council has offered its congratulations to Dr. Roche Lynch on this high distinction.

BRITISH STANDARDS INSTITUTION—Dr. W. F. Elvidge was appointed representative of the Society on Committee C/8 and Sub-Committee C/8/1B of the British Standards Institution.

BUREAU OF ABSTRACTS—Mr. A. L. Bacharach was appointed a representative of the Society on a Committee of the Bureau to report on the suggestion to issue separate sets of abstracts covering both the scientific and technical aspects of foods and nutrition.

INSTITUTION OF WATER ENGINEERS—Dr. J. H. Hamence and Mr. S. E. Melling were appointed representatives of the Society on a Joint Committee on the standardisation of methods of chemical analysis of potable waters.

The Office of the Society is available for meetings of small committees; for the accommodation of larger committees the Council again desires to record its thanks to organisations and to members of the Society.

G. W. MONIER-WILLIAMS, *President*
LEWIS EYNON, *Hon. Secretary*

Address of the Retiring President

G. W. MONIER-WILLIAMS, O.B.E., M.C., M.A., PH.D., F.R.I.C.

IT is my privilege on retiring from the chair to address the members of this Society and I propose, with your permission, to follow the usual custom of referring shortly to some of the Society's activities during my term of office and then discussing at greater length a subject of particular interest to many of our members.

The chief events of the past two years are fully recorded in the Council's annual reports, but there are a few matters to which special reference should be made. We have been most unfortunate during this period in losing three of our Past Presidents, Evans, Roberts, and Hinks, to all of whom the Society owes a great debt of gratitude for the work they did for us. Edward Hinks was for many years honorary treasurer before succeeding to the chair, and the value of his work for the Society can hardly be over-estimated. I personally used often to consult him on difficult or controversial matters concerning our profession and could always count on getting thoroughly sound and practical advice. He was ready at all times to take the utmost trouble in the service of the Society, and there have been few whose influence in our Council was greater or whose loss has been more deeply felt. Roberts and Evans also had been active in promoting the interests of the Society and were particularly associated with the work of the North of England Section.

I cannot let this occasion pass without expressing the great pleasure we feel in Dr. Bernard Dyer's continued presence among us. We were able the other day to congratulate him on his 91st birthday, and I can assure him, on behalf of every member of the Society, that we are

extremely proud of our distinguished original member and grateful to him for the close and practical interest which he continues to take in the affairs of the Society.

The past year has been remarkable for the success which has attended the proceedings of the three subject groups. The Microchemical Group under the chairmanship of Professor Briscoe, the Physical Methods Group under Mr. Chirnside, and the Biological Methods Group under Mr. Bacharach, have been ably guided through the hazards of infancy and have become firmly established as active and progressive members of the analytical body.

In the coming years the problem of chemical publications is bound to become more and more acute, and the task of our Publication Committee, under Dr. Nicholls, in maintaining the high standard and reputation of THE ANALYST, will become still more exacting. A particularly difficult problem is that of abstracts of papers published in other journals. Our policy is to select our material very carefully over the whole range of analytical chemistry and to make the abstracts full enough to be of real value in the laboratory. This policy makes increasing demands upon the skill and judgment of the editorial staff. We have lately enlarged this staff considerably, and I think we may feel confident that they will prove fully equal to the task before them. In this matter of abstracts we shall be wise to co-operate closely with other publishing bodies, and members of our publication committee have already held preliminary discussions on this subject with representatives of the Bureau of Abstracts. Another important step in the direction of co-operation is the co-option of the Society to membership of the Chemical Council, a position which secures to us a voice in matters of general policy and administration.

The Public Analysts Committee, under the chairmanship of Dr. Cox, recently drafted recommendations on terms and conditions of appointment, for issue by the Council to local authorities. These recommendations have gained sympathetic response and improved conditions in a number of cases, but many authorities still seem unwilling or unable to recognise the heavy responsibility and exacting nature of the work and the long scientific training required. With the emphasis now laid upon sound nutrition as the basis of health, a highly qualified and efficient public analyst service, working under conditions which will attract the ablest men, will become of ever greater importance to the country.

With the end of the war the Analytical Methods Committee has again become active. Some valuable reports have been published, notably one on the microbiological determination of riboflavine and nicotinic acid. A sub-committee is also engaged on a bibliography of published approved and standard methods, which should be extremely useful to analysts in this country. The thanks of the Society are due to Dr. Hughes and his colleagues on the committee, and also to the members of the many sub-committees, for the immense amount of arduous voluntary work which they give so willingly. The reports of this committee contribute very greatly to the high reputation of THE ANALYST both in this country and abroad.

We have been concerned during the past two years with the teaching, or, as some would say, the lack of teaching, of analytical chemistry in university courses. This is a very difficult question and perhaps we, as analysts, run the risk of being accused of undue bias. We may remember, however, that our illustrious former honorary member, Professor Fresenius of Wiesbaden, in the introduction to his well-known book on chemical analysis, emphasises the fact that "chemistry owes to quantitative analysis its elevation to the rank of a science." Further he says that "quantitative analysis forms the strongest and most powerful lever for chemistry as a science, and not less so for chemistry in its applications to the practical purposes of life, to trades, arts and manufactures." I am sure that the truth of these words, written over 70 years ago, is recognised by those responsible for chemical education in this country. Many of us feel, however, that more attention should be given to the systematic teaching of accurate quantitative analysis. Perhaps in these days of overloaded undergraduate courses the answer to the question is to be found in Professor Fresenius' further observation that "the pursuit of this branch of chemistry requires considerable expenditure of time."

I should like to take this opportunity of thanking those who replied so fully to my inquiries upon this subject. Their views have been submitted for consideration to the Chemistry Education Advisory Board, and there are indications that they may meet with a favourable response.

The year 1945 saw the end of the war and it would have been appropriate for me to have attempted a comprehensive survey of the impact of the war years upon this Society and its work, such a survey as was given in a masterly way by Dr. Samuel Rideal at the end of the previous war. Even from the food standpoint alone, however, it would be difficult to do

justice to this theme. One would have to deal with food substitutes, food standards, food labelling, the contributions of analytical chemistry to the solution of nutrition problems—all the scientific and administrative activities of the last seven years which have added so greatly to the work and responsibilities of the public analyst. Perhaps it will be more useful if, instead of attempting to cover such a wide field, I make some observations, and I hope some constructive observations, upon one aspect of food administration which has not received much attention during the war.

In all food legislation we can distinguish two distinct and separate objects, firstly the promotion of fair trading without any particular health significance other than that of adequate nutrition, and secondly the protection of the public against possible injury to health from harmful substances. It is this latter object that I want to discuss—the prevention of risk to health from questionable substances which may gain access to food during its preparation, storage or treatment.

Under Section 8 of the Food and Drugs Act of 1938, power was given to the Minister of Health to make regulations prohibiting or restricting the addition of any substance to food and regulating generally its composition. In 1943, under the Defence (Sale of Food) Regulations, similar powers were given to the Minister of Food, but it is not quite clear from the wording of the regulations, or of the explanatory White Paper, which of these Ministers was expected to take action where contamination is concerned. Practically all the orders made by the Minister of Food during the war have been concerned with fair trading or adequate nutrition. There has been some relaxation of the Preservative Regulations of 1925, and quite recently an order concerning edible gelatin, but with these exceptions no orders controlling potentially toxic substances in food have been made either under the 1938 Act or under the Defence (Sale of Food) Regulations. It seems doubtful whether this purely health function will in future be discharged by the Ministry of Health or whether it will be handed over to the Ministry of Food, as appears to have been done with edible gelatin and with fluorine in acid calcium phosphate. Whichever department is ultimately responsible for control of this kind there are, I think, three main principles which should govern any action taken.

If we set out to devise limits of tolerance for toxic substances in food, the chief consideration, obviously, is the effect on health of the small quantities in question, and here we meet with the first and greatest difficulty—what is to be regarded as injurious to health? Owing to the complexity of the processes taking place in the body and the extreme difficulty of relating cause and effect, there is usually a very wide range between that which can be accepted without question as being harmless and that which can be shown conclusively to be injurious. A limit might be fixed anywhere within this range and the claim made that the consumer had been adequately protected.

There are many people concerned with the chemistry of food and nutrition who deprecate the placing of any restraint upon trade in these matters unless it can be clearly shown to be necessary in order to prevent injury to health and who hold strictly to the experimental method. If any limit is set for a toxic substance they would put it high in this doubtful range on the ground that there is no experimental evidence that the substance in question is injurious in that amount. At the other extreme there are many who think that any suspicion that a substance may be injurious, even to a minority of the less resistant persons, should be enough to warrant its exclusion altogether from food. They would condemn any treatment of food which may involve the slightest contamination with questionable substances.

As an example of the difficulty of deciding what is or is not injurious we may take lead. We have first to consider what is the safe daily intake of lead on the assumption that it is all absorbed, for this will affect the safety limits for different foods. In spite of the long experience gained with water supplies this point is not yet definitely settled. Some people are more susceptible than others to lead poisoning, women for instance much more so than men. The effects of lead are cumulative: even when ingested in minute amounts it can be stored in the body and again become active under different conditions of health and of dietary habits. Its ultimate effects may not be evident for several years. Then again there is the difficulty of recognising with certainty the milder forms of lead poisoning. Some authorities hold that it is responsible for many anomalous nervous conditions and slight ill health. On the other hand it seems that many people who come into contact with lead industrially may excrete it in relatively large amounts and yet show none of the recognised symptoms of lead poisoning.

When we have arrived at some conclusion as to the limit which should be set for the amount of lead absorbed into the system over any given period we have still to consider what proportion of the lead in food is actually so absorbed. Water, beer and cider seem to give it up much more readily than solid foods. In particular a high intake of calcium in the diet hinders absorption; from foods rich in milk or dried milk, lead, if present, is absorbed only slightly. Phytic acid, also, and pectin may combine with lead and remove it from solution, and there are probably other constituents of food which have a similar effect. Absorption of lead may thus depend to some extent upon the nature of the food, and even upon the sequence of different foods, from day to day.

These are problems for the physiologist, the nutrition specialist and the chemist together, but even the most expert team would be hard put to it to prepare a schedule of limits for lead in different foods which would be free from criticism. A similar uncertainty applies more or less to other potentially toxic substances in food; for instance, antimony or zinc, certain azo dyes as colouring matters, thio-urea as an antioxidant, or methyl bromide as a fumigating agent. Methyl bromide in small doses over a long period has been found to hinder the growth of animals. Can we disentangle from the multitude of factors affecting growth in human beings a possible slight effect due to the use of methyl bromide for fumigating food and its retention by fat? How are we to say at what point a contamination or treatment of food loses its harmless character and becomes suspect?

The Minister of Health has often been asked to give a ruling on specific cases but has rarely, if ever, done so. There is a clear distinction between giving an interpretation of the meaning of the word "injurious" in Section 1 of the Act, which is the prerogative of the Courts, and embodying that interpretation in a regulation under Section 8, which is the privilege of the Minister. Section 7 of the Act lays it down that any addition to food which has been forbidden by regulations is *ipso facto* to be regarded as injurious to health, which seems to solve the problem quite neatly, but unfortunately regulations are always several laps behind public health requirements. The object of Section 7 is, presumably, to uphold the regulations in a possible conflict of expert evidence. I am not aware of any similar provision in respect of orders made under the Defence (Sale of Food) Regulations.

This Society, in a memorandum submitted to the Departmental Committee on the Composition and Description of Food, urged the setting up of a permanent statutory advisory committee consisting of representatives of the Ministry of Health, public analysts, manufacturers and consumers which, among other duties, "should on request state definitely with regard to any ingredient of a food, the interpretation of the term 'injurious to health'." This suggestion, possibly for the reasons I have given, was not favoured by the Departmental Committee, which made an alternative recommendation that any controversial question on which an order is applied for should be referred to an *ad hoc* committee of three independent persons for public inquiry and report. Neither of these suggestions has been adopted. There seems to be a tendency now to refer these questions to the Medical Research Council, but I doubt whether this is the proper body to deal with them.

In difficult and contentious matters of this kind the first instinct of the administrator is to compromise, but we may question whether this instinct is sound where food is concerned. One need not elaborate the fact that food is in a category altogether different from that of any other commodity. We are bound to take into consideration the chances of remote action beyond the reach of experimental proof and to insist upon a wide margin in favour of the consumer. The proper course is to ensure that, within the range in which doubt exists as to the toxicity of any substance in food, the limit of tolerance shall be placed at the lowest possible point, and this, I suggest, should be regarded as a first principle. It is precisely that lack of knowledge, that difficulty of experimental proof, which should make us chary of compromising in this matter.

If this principle is accepted, and if we have succeeded in defining the doubtful range in respect of a particular substance, we have to decide what are the very lowest limits within that range which can reasonably be insisted upon as a practical measure. It must be remembered that food manufacture, packing and distribution are by no means confined to large concerns with ample resources for regular scientific control. Many of the smaller concerns find it hard to keep a constant check on raw materials and to maintain a consistently high standard. In deciding upon appropriate limits we must consider carefully what can be reasonably demanded in all circumstances. I do not suggest for a moment that there should be any allowance for inefficiency; rather that every endeavour should be made to discover

what can and what can not be done by different types of producers working under different conditions. It may be that a requirement which seems at the time impossible of fulfilment will act as a stimulus to improvement and will soon become normal practice. It is of course very difficult to arrive at a sound conclusion. The necessity for careful and detailed inquiry has been recognised by successive departmental committees. In the old days of the Foods Department of the Local Government Board, and later of the Ministry of Health, someone with the requisite experience was detailed to investigate and report on a particular contamination or treatment of food as fully as possible, by visits to factories and by making contact with anyone who could give trustworthy information on the subject. Probably this method, in the hands of a competent investigator, gets at the facts more surely than the alternative one of taking evidence from, and cross-examining, interested parties at a committee table. The Interdepartmental Committee on Preservatives in 1923–24 employed both methods, and the subsequent regulations were the result of as full an inquiry as was possible with the limited staff available Even so, several points were missed but, considering the wide field covered by the inquiry, this was perhaps inevitable. We may remember that the Royal Commission on Arsenical Poisoning, whose report stands out as a model of thorough and exhaustive investigation, failed to appreciate the partiality of shell-fish for arsenic.

My second principle then, although it sounds self-evident, is that, when it has been decided on health grounds that control is necessary, the fullest possible investigation should first be made of all the commercial factors involved. This implies of course that an adequate staff should be made available for the purpose, but nowadays one hesitates to suggest any further addition to the public service.

When a decision has been reached on the practical limits to be adopted for any foreign substance in a particular food or foods there remains the question whether these limits or prohibitions should be embodied in regulations, or whether there is any other way of securing their acceptance by the trade and by those concerned with administration. Statutory standards have an immense advantage from the viewpoint of administrative convenience and greatly simplify the whole procedure of Food and Drugs Act work. They are also a safeguard for the manufacturer and distributor, who thus know exactly where they stand and are not so liable to become involved in unforeseen litigation. On the other hand it is very difficult, when framing regulations, to take every factor into account, and clearly impossible to make adequate allowance for changing circumstances. Legal standards have the disadvantage that they are not readily altered and may remain in force long after scientific research and industrial progress have made them virtually obsolete. Nominally it should be an easy matter to amend a regulation—and this is one of the advantages claimed for departmental regulations as compared with Acts of Parliament—but in practice it has proved difficult, even when the weight of evidence has been overwhelmingly in favour of amendment. I am not speaking, of course, of war-time orders which have to deal quickly with constantly changing conditions of supply.

The third principle, therefore, is that any regulations on food matters must allow of ready revision, not only in theory but in practice, as unforeseen contingencies arise.

A common charge against standards is that they tend to become the normal and that while they may induce a general levelling up of poor quality articles there is also a tendency to a levelling down of the better qualities, to the disadvantage of the consumer. If, however, we are able to place the limit for a toxic substance low enough, this objection loses much of its force. Another objection, of course, is the instinctive dislike of official interference in our daily lives. The experience of the last six years has not made us anxious for further restrictions. It will be agreed, I think, that no standard of the type we are discussing should be imposed unless a reasonable case has been made out for it on health grounds. There is always a danger that a demand for standards may be prompted more by a desire for administrative convenience than by health considerations.

There has been much criticism of government by shoals of departmental regulations having the force of law, and it is worth while considering whether in certain cases official pronouncements or recommendations might carry enough weight to make such regulations unnecessary. Arsenic provides a case in point. The limits proposed for arsenic in food by the Royal Commission over forty years ago have never yet been given statutory force except for edible gelatin, and yet they have been accepted without serious question for administrative purposes. In fact the absence of a rigid standard has on occasions been undeniably convenient, as for instance when arsenic of natural origin was discovered in fish and more recently in parsley.

Would similar pronouncements in respect of other potentially injurious substances in food, backed by a sufficient weight of authority, be accepted in the same way as the arsenic limits? I very much doubt it. The recommendations of the Royal Commission followed a serious and widespread outbreak of poisoning in Lancashire, and the general acceptance of the limits then proposed was due more to the shock of that occurrence and the fear of further trouble than to the authority of Lord Kelvin and his colleagues on the Commission. The questions of lead, copper, and antimony, of preservatives and colours, of flavouring vehicles, of fumigating agents, and of antioxidants are more controversial and less clear cut than that of arsenic, and the possible effects on health are not nearly so spectacular. The report of the Departmental Committee on Preservatives, a body just as competent as the Royal Commission to decide the health question, would have had little influence on the use of preservatives if its findings had not been crystallised into regulations. The report by itself would undoubtedly have been of assistance to those whose task it is to interpret the meaning of the word "injurious" in the Act, and indeed several of the Local Government Board and Ministry of Health reports have been of great value in this way; but the regulations and standards which followed the findings of the Preservatives Committee have had a definite and salutory effect on the quality of food in this country and on the conditions under which it is prepared and stored. Moreover, it is doubtful whether any advisory body would give sufficiently close attention to these problems, especially the more controversial of them, if their advice was to be treated only as an opinion to be contested and possibly over-ruled. Such advice would have to be definitely associated with a proposal for an order or regulation.

In the absence of statutory standards certain administrative methods have been adopted with more or less success to secure some sort of control. Thus medical officers at the ports, using their powers under the Imported Food Regulations, agreed a few years ago on limits for toxic metals in some imported foods. The importer could, of course, appeal to the Courts if he felt aggrieved, but in practice he seldom did so. This Society has, in a few instances, formulated its own standards, and local authorities often bring pressure to bear upon manufacturers and distributors to accept certain criteria of purity in respect of particular foods.

These isolated efforts only touch the fringe of the problem. During the recent period of stringency and short supply many things have of necessity been tolerated which will demand careful reconsideration on return to more normal conditions. It is clear that if any contamination or treatment of food is suspected of being in any way injurious, the consumer's interest demands that it should be effectively regulated. Public analysts have always been insistent upon this. At present they are in the invidious position of having, in the first instance, to apply their own standards in order to carry out their duties under the Act. They are justified in asking that they should be relieved of this responsibility and that it should be undertaken by some department of State through an advisory body combining the necessary scientific, commercial and administrative experience and authority.

In conclusion, I should like to express my thanks to the officers, council, and members of this Society for the kindness and consideration which they have at all times shown to me, and to accord to you, Mr. President, my best wishes for your term of office.

The Oxidation of Nitrogen during the Micro-Combustion of Organic Substances

By A. E. HERON

(*Read at the Annual General Meeting of the Microchemistry Group on January* 31*st*, 1947)

According to Pregl,[1] amino, imino, and similar compounds yield on combustion, in addition to carbon dioxide and water, elementary nitrogen, or at the most, nitrous oxide. In their paper on "A New Technique for the Ultimate Micro Analysis of Organic Compounds," Belcher and Spooner[2] state that it depends on how the nitrogen is combined whether or not oxides of nitrogen are formed, but recommend the use of a bubbler containing acid dichromate or permanganate, with all compounds containing nitrogen, until information is available as to which compounds do and which do not form oxides of nitrogen.

In March, 1944, we were comparing the standard macro- and the Pregl micro-method for the analysis of coal, and found that the results obtained with 5- to 7-mg. samples and

Pregl's method were not greatly divergent from those obtained with 150-mg. samples by the standard macro-method. The results obtained with a modified form of the combustion technique described in the paper referred to,[2] but without the chromic and sulphuric acid bubbler, were all somewhat higher than those obtained by our standard macro-method. These results were published in *J. Inst. Fuel*,[3] and the suggestion was made that the discrepancy might be due to retention, by the reagent in the carbon dioxide absorption tube, of oxides of nitrogen produced from the nitrogen contained in the coal. It was subsequently shown that oxides of nitrogen could be readily detected in the products of combustion of a 5-mg. sample. Analyses of the same sample of coal when a bubbler containing chromic and sulphuric acids was used gave results that agreed tolerably well with those obtained by the Pregl micro- and our standard macro-method. In all the experiments on the micro scale the sample was burned in a platinum boat. In the method of Belcher and Spooner[4] for the rapid simultaneous determination of carbon, hydrogen, sulphur, and chlorine in coal, on the *macro* scale, samples are burned in a rapid stream of oxygen at a temperature of 1300° C. No special provision is made for the removal of oxides of nitrogen, for, according to the authors, under the conditions employed, no oxides of nitrogen are formed, the nitrogen being evolved completely as elementary nitrogen gas.[3] Thus, although the influence of nitrogen on the macro scale is apparently insignificant, our results suggested that the effect of nitrogen on the micro scale was important and required further investigation.

In a private communication, Belcher stated that he had carried out some experiments on the macro scale, using porcelain and platinum boats to contain the sample of coal, and had found that if platinum was used oxides of nitrogen could be detected in the products of combustion, but that he could find none when porcelain was used. He also said that tests on the micro scale, with use of a train packed with silica chips, such as we were using in our modification of the unpacked tube technique, indicated that nitrogen oxide formation could be detected to a small extent when coal was burnt in a porcelain boat, and more readily when a platinum boat was used.

It was thought that the problem should be investigated quantitatively and that the results obtained by examining the products of combustion of various nitrogen-containing organic substances would be of real value. According to Kirner,[5] "oxidation of the nitrogen-containing thermal decomposition products by the excess of oxygen present in the combustion tube at temperatures from 600° C. down to 180° C. would yield only nitrogen peroxide and nitrogen. The proportion of these two substances which finally results under a given set of conditions should primarily be a function of the manner in which the nitrogen is linked in the molecule." The purpose of our investigation was therefore to find out how much oxidation of nitrogen occurs during the combustion of compounds containing nitrogen. The determination of the extent to which this occurs resolves itself into the finding of a suitable absorbent, the oxidation of the absorbed gas, and the evaluation, by a suitable method, of the nitrate produced.

Experimental

The silica combustion tube was of the usual type with side arm; it was heated by an electric furnace giving a temperature of 1000° C. at the centre. Toward the exit end of the tube was a 2½-inch roll of silver gauze, so placed that about half of it was heated directly by the furnace winding. The exit end of the tube was heated by a copper block, maintained at 130° to 160° C. to prevent condensation of water. In some of the experiments the portion of the tube that was heated in the furnace was packed with silica chips, but in others the tube was free from packing. A pre-heater packed with silica chips was used. For the determination of the "oxides of nitrogen" it was decided to use a colorimetric method which depended upon the nitration of an organic compound, rather than upon an oxidation effect as does the diphenylamine test. Soda asbestos as used for the carbon determination was tried as absorbent for the nitrogen peroxide, but replicate tests for nitrate on weighed amounts gave widely different results. It was found that when the gases were passed through a spiral bubbler containing sodium hydroxide solution, absorption of carbon dioxide was not complete; it could be detected in the gases after they had passed through the spiral bubbler. A vacuum technique was also tried by means of which all the gases obtained during the combustion could be submitted to the action of an absorbent solution. Heavy walled bottles of about 750 ml. capacity were obtained and each fitted with a rubber bung carrying a single-way tap, the end of which was gradually tapered to the same diameter as the exit of the combustion tube. When a determination was to be made, a small volume of suitable absorbent was

measured into a bottle, which was then evacuated with a good water-pump, and connected to the train. The rate of flow of oxygen was controlled by adjustment of the tap fitted to the bottle. In the earlier experiments a bubble-counter was used to indicate the rate of flow of oxygen, but later a White - Wright flow meter[6] was substituted.

Alkaline permanganate was first tried as absorbent. This is a recognised absorbent for nitric oxide and nitrogen peroxide. After the gases had been in contact with this solution for 3 hours, it was acidified with 20 per cent. sulphuric acid and warmed to about 40° C. for 15 minutes to oxidise nitrite. Hydrogen peroxide was then added to destroy the excess of permanganate and followed by a slight excess of sodium hydroxide. The sample was now boiled and filtered, and the filtrate evaporated to dryness. Two ml. of a 1·25 per cent. solution of sulphosalicylic acid in 50 per cent. sulphuric acid were added and the solution was heated on a water-bath for 30 minutes. It was then diluted, 25 ml. of 10 per cent. sodium hydroxide solution were added, the volume was made up to 50 ml. and the optical density was measured on the Spekker absorptiometer at 4360A. In a number of tests trouble was experienced owing to precipitation of manganese after the final addition of sodium hydroxide, and the blank values obtained were not consistent. A similar lack of agreement on control tests was observed when the absorbent used was 80 per cent. sulphuric acid containing sulphosalicylic acid. It is possible that in this case there was some carbonisation of the reagent.

The use of 2:4-xylenol was then considered. When this reagent is used, the nitro compound may be steam distilled from the solution in which it is formed, and thus separated from substances that might interfere with the measurement of its optical density. At the same time, in order to reduce the number of reagents employed to a minimum and to keep the technique as simple as possible, it was decided to use as absorbent ceric sulphate in sulphuric acid, a reagent which rapidly oxidises nitrite to nitrate at room temperature. With these modifications to the method, there was no difficulty in obtaining agreement in blank tests.

The procedure was as follows—

The electric furnaces were switched on and the temperature of the copper block adjusted. The oxygen flow was adjusted to a rate of about 35 ml. per minute. Three ml. of 0·1 *N* ceric sulphate solution in 12 per cent. sulphuric acid were measured into the vacuum bottle, which was evacuated by means of a good water pump, and connected to the train. The weighed sample was then introduced into the combustion tube and this was securely stoppered with the rubber bung. The tap of the vacuum bottle was cautiously opened until a rate of flow of oxygen of 25 ml. per minute was indicated by the bubbler or flow meter. The sample was then burned off and the products of combustion swept forward in the usual way, the oxygen rate being maintained at 25 to 30 ml. per minute throughout, by adjustment of the tap. At the end of twenty minutes the tap was closed and the bottle disconnected from the train. The inlet tube was washed with 2 ml. of 20 per cent. sulphuric acid without removal of the stopper from the bottle, the contents of which were still under slightly reduced pressure. The bottle was then rotated frequently over a period of 1 hour, and at the end of this time the stopper was removed. 0·2 *N* Ferrous sulphate was added till the colour of the ceric sulphate was just discharged, and then followed a dropwise addition of 0·1 *N* potassium permanganate until a barely perceptible pink colour was obtained. The permanganate, of which only 1 or 2 drops are required, was used as an indicator, ceric sulphate itself not being sufficiently intensely coloured. Five ml. of a 1 per cent. solution of 2:4-xylenol in dilute sodium hydroxide solution were added, followed by 35 ml. of sulphuric acid of sp.gr. 1·76. The temperature while this was being added was not allowed to exceed 35° C.; the solution was at this temperature for 30 minutes. At the end of this time the contents of the bottle were washed into a steam distillation apparatus, with approximately 150 ml. of water. The nitro compound and excess of 2:4-xylenol were distilled into 1 ml. of *N* sodium hydroxide. The distillate was made up to a given volume and its optical density determined on a Spekker absorptiometer. The mercury lamp was used with Chance No. 6 and Wratten No. 50 filters (4360A).

RECOVERY OF NITROGEN—

Dinitrobenzene was burned, the products of combustion were collected and the oxides of nitrogen formed determined as described above. Five determinations were made, two with a boat of fireclay, and three with a boat of platinum. The tube contained only the silver coil. The results are given in Table I. This and the subsequent tables show the conditions of each experiment, the weight of sample taken and the quantity of "oxides of nitrogen," calculated as NO_2, which was collected in the ceric sulphate solution. This is also shown as a

percentage of nitrogen on the sample. Finally the proportion of nitrogen collected as NO_2 is shown as a percentage of the total nitrogen present.

TABLE I

COMBUSTION OF DINITROBENZENE, 16·7 PER CENT. N

Expt. No.	Conditions of test	Boat	Wt. of sample mg.	Nitrogen absorbed: Calc. as mg. NO_2	Nitrogen absorbed: Calc. as N % of sample	Nitrogen absorbed: Calc. as N % of total N
1	Combustion tube contained only silver coil. Oxygen rate 25 to 30 ml. per min.	Fireclay	6·376	3·29	15·7	94·2
2		,,	7·045	3·75	16·2	97·2
3		Platinum	7·310	3·88	16·1	96·6
4		,,	8·678	4·54	15·8	94·8
5		,,	8·960	4·05	13·7	82·2

These results were considered satisfactory for the purpose of this investigation and the method was used for all the subsequent work.

THE COMBUSTION OF COAL—

A sample of Craghead coking coal which contained 1·26 per cent. of nitrogen, as determined by the Kjeldahl method, was used for the work on coal. Weighed amounts of this sample were burned under different conditions, and the results are recorded in Table II. Oxides of nitrogen were produced in all experiments. The results when the tube was packed

TABLE II

COMBUSTION OF CRAGHEAD COKING COAL, 1·26 PER CENT. N

Expt. No.	Conditions of test	Boat	Weight of sample mg.	Nitrogen absorbed: Calc. as mg. NO_2	Nitrogen absorbed: Calc. as N % of sample	Nitrogen absorbed: Calc. as N % of total N
6	Tube packed with silica chips and containing silver coil. Oxygen rate 25 to 30 ml./min.	Fireclay	10·843	0·105	0·30	23·8
7		,,	9·822	0·039	0·12	9·5
8		,,	9·864	0·063	0·19	15·1
9		,,	10·022	0·066	0·20	15·9
10		,,	10·412	0·049	0·15	11·9
11		,,	10·466	0·095	0·28	22·3
12		,,	9·749	0·079	0·25	19·9
13		,,	10·822	0·102	0·28	22·3
14		,,	12·240	0·151	0·38	30·2
15		Platinum	9·640	0·098	0·31	25·0
16		,,	10·050	0·066	0·20	15·9
17		,,	8·654	0·049	0·18	13·5
18		,,	10·068	0·043	0·13	10·3
19		,,	10·083	0·085	0·26	20·6
20		,,	9·843	0·138	0·38	30·2
21		,,	10·039	0·121	0·37	29·4
22		,,	9·840	0·128	0·40	31·4
23		,,	10·422	0·135	0·39	30·9
24	No silica chips, otherwise as above.	Fireclay	9·642	0·068	0·22	17·5
25		,,	10·625	0·061	0·19	14·3
26		,,	10·053	0·032	0·10	7·9
27		Platinum	10·383	0·059	0·17	13·5
28		,,	10·545	0·106	0·31	24·6
29		,,	9·842	0·049	0·15	11·8

with silica chips and contained the silver coil ranged from 9·5 to 30·2 per cent., when the boat was of fireclay. When the platinum boat was used, the results ranged from 10·3 to 31·4 per cent. In each case the distribution of results was fairly even over the whole range. When the tube contained only the silver spiral, the results with the porcelain boat ranged from 7·9 to 17·5 per cent. and with the platinum boat from 11·8 to 24·6 per cent. It is of interest that W. R. Kirner,[7] in a series of experiments for the direct simultaneous micro-determination of carbon, hydrogen, and oxygen in coal, found that correct oxygen values were obtained when it was postulated that 59 per cent. of the nitrogen present was converted into nitrogen peroxide and 41 per cent. to elemental nitrogen. This figure for nitrogen peroxide is much higher than any of the results we have obtained.

COMBUSTION OF PURE ORGANIC COMPOUNDS—

Table III shows the results of combustions carried out on various pure organic compounds: (*a*) carbazole, (*b*) pyridine, (*c*) xylidine, (*d*) dimethylaminoazobenzene (dimethyl yellow) and (*e*) urea. With all of them oxides of nitrogen were formed in variable amounts. Carbazole, for example, yielded from 7·4 to 32·2 of its total nitrogen as oxides and there was

TABLE III

COMBUSTION OF PURE ORGANIC COMPOUNDS

Expt. No.	Conditions of test	Boat	Weight of sample mg.	Nitrogen absorbed: Calc. as mg. NO_2	Nitrogen absorbed: Calc. as N % of sample	Nitrogen absorbed: Calc. as N % of total N
	(*a*) COMBUSTION OF CARBAZOLE, $C_6H_4.NH.C_6H_4$, 8·4% N					
30	Tube packed with silica chips and containing silver coil and boat with sample.	Fireclay	10·328	0·490	1·43	17·0
31		,,	9·851	0·259	0·80	9·5
32		,,	8·502	0·756	2·70	32·2
33		,,	8·306	0·665	2·44	29·2
34		Platinum	8·511	0·598	2·14	25·4
35		,,	10·522	0·216	0·62	7·4
36	Tube contained only silver spiral and boat with sample.	Fireclay	10·310	0·484	1·40	16·7
37		,,	12·780	0·319	0·76	9·1
38		Platinum	10·550	0·484	1·39	16·5
39		,,	11·890	0·298	0·74	8·8
40	Tube contained only boat with sample.	Fireclay	10·497	0·362	1·04	12·4
41		Platinum	10·287	0·328	0·98	11·7
	(*b*) COMBUSTION OF PYRIDINE, C_5H_5N, 17·7% N					
42	Tube packed with silica chips and containing silver coil and boat with sample.	Fireclay	10·684	1·260	3·60	20·3
43		Platinum	6·990	0·415	1·80	10·3
44		,,	8·690	1·015	3·58	20·2
45	Tube contained silver coil and boat with sample.	Fireclay	10·420	0·756	2·20	12·5
46		,,	8·116	1·882	7·08	40·0
47		Platinum	5·424	0·684	3·84	20·5
	(*c*) COMBUSTION OF XYLIDINE $(CH_3)_2C_6H_3NH_2$, 11·5% N					
48	Tube packed with silica chips and containing silver coil and boat with sample.	Platinum	11·100	0·342	0·94	8·1
49		,,	8·624	0·495	1·75	15·2
50		Fireclay	9·961	0·645	1·96	17·0
51		,,	10·727	0·529	1·50	13·1
	(*d*) COMBUSTION OF DIMETHYL YELLOW, $C_6H_5N:N.C_6H_4N(CH_3)_2$, 18·65% N					
52	Tube containing silver coil and boat with sample.	Fireclay	7·504	0·379	1·54	8·3
53		,,	6·760	0·551	1·98	10·6
54		Platinum	8·520	0·437	1·98	10·6
	(*e*) COMBUSTION OF UREA, $CO(NH_2)_2$, 46·6% N					
55	Tube packed with silica chips and containing silver coil and boat with sample.	Fireclay	9·186	0·349	1·31	2·8
56		,,	11·045	0·402	1·07	2·3
57		Platinum	9·900	0·414	1·27	2·8
58		,,	8·765	0·609	2·11	4·6
59	Tube contained silver coil and boat with sample.	Fireclay	10·740	0·168	0·48	1·0
60		,,	12·928	0·312	0·73	1·6
61		Platinum	11·701	0·282	0·73	1·6
62		,,	9·854	0·175	0·54	1·2

no marked difference in the order of magnitude of the results according as fireclay or platinum boats were used. With pyridine, again, the results show no marked differences attributable to any particular condition in the tests. Dimethyl yellow contains two types of nitrogen linkage, *viz.*, C.N:N.C and C.N$\langle^{C}_{C}$. The amounts of nitrogen oxides formed from urea were consistently small. One result amounting to 48 per cent. of the total nitrogen was obtained, but as this figure was not approached in any subsequent test it has been rejected

Finally, several experiments were carried out in which air was passed through the train with a platinum or fireclay boat in position. With a platinum boat the results obtained when 500 ml. of air were passed along the tube at a rate of 25 to 30 ml. per minute showed that oxides of nitrogen equivalent to 0·01 to 0·02 mg. of NO_2 were produced and that when fireclay boats were used the results varied between 0·003 and 0·01 mgm. It should be added here, that the control tests on reagents and blank tests for the combustions in oxygen were deducted in every case, in the calculation of results.

DISCUSSION—

The investigation has shown that when compounds of different types containing carbon, hydrogen and nitrogen are burned in a stream of oxygen, appreciable amounts of oxides of nitrogen are produced. The results obtained from the combustion of coal indicate that although the material used for the boat may have some effect, it is of minor importance, and the effect of the surface provided by the silica chips is somewhat greater.

In the written discussion on the papers by Belcher and Fenton on "Methods for the Determination of Carbon and Hydrogen in Coal and their Practical Utility"[3] results obtained on the same sample of coal by the B.S. method[8] and the open tube technique[4] are quoted by Edwards and Vahrman. These results are in substantial agreement, but it should be noted that in neither of these methods is special provision made for the removal of oxides of nitrogen from the products of combustion, and thus it is unsound to deduce from these results that oxidation does not occur. Oxidation to the extent indicated in Table II would result in the carbon values being high by approximately 0·9 times the amount of nitrogen absorbed (*i.e.*, from 0·11 to 0·36 per cent.). An error of this magnitude would be of no special significance in the analysis of coal.

The subordinate effect of the silica packing can also be traced in the results obtained by the combustion of the pure organic compounds, but with these the effect of replacing the fireclay boat by one of platinum was not marked. The oxidation of the nitrogen probably takes place at the instant of ignition. The rapidity with which a given weight of sample is consumed is not easy to control as it is influenced by several factors, *e.g.*, the thickness and length of the layer in the boat and the temperature of the tube at the instant of ignition. These factors may be responsible for the variation in the results obtained on each compound. Up to 40 per cent. of the total nitrogen may be oxidised during the combustion.

Kirner,[5] who used a combustion tube packed with copper oxide, states that in cases where nitrogen is linked to hydrogen and carbon, the conversion to nitrogen peroxide is 26 per cent. and for nitriles, heterocyclic nitrogen compounds and nitro compounds, the conversion is 59 per cent. Our results indicate that for the amino compounds examined oxidation may vary between 1 and 17 per cent. of the total nitrogen, and for heterocyclic compounds the range lies between 7 and 40 per cent. This represents less conversion of nitrogen to nitrogen dioxide than Kirner's work suggests should take place when this type of compound is burned in oxygen. For dinitrobenzene the amount of nitrogen dioxide absorbed represented between 82 and 97 per cent. of the nitrogen present, a much higher value than that quoted by Kirner.

Our experiments amply confirm Pregl's statement[1] that "metallic silver is quite unsuitable for the reliable absorption of the nitrogen oxides," but we have not confirmed his statement that "amino and similar compounds yield on combustion elementary nitrogen only, or at most possibly nitrous oxides."

It is evident that if carbon and hydrogen are to be determined on organic substances containing amino, heterocyclic, diazo or nitro nitrogen provision must be made to remove oxides of nitrogen from the products of combustion, when combustions are carried out on the micro scale.

SUMMARY—

Experiments on the micro scale have been performed to determine the extent to which oxides of nitrogen are produced when organic compounds containing carbon, hydrogen, and nitrogen are burned in oxygen. Almost the whole of the nitrogen present in nitro compounds is recovered as oxides of nitrogen; when nitrogen is not linked to oxygen, between one and forty per cent. of the total is oxidised. Thus, when carbon and hydrogen are to be determined on compounds containing nitrogen on the micro scale, provision must be made to remove oxides of nitrogen completely from the products of combustion.

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Discussion

Mr. R. Belcher said that Mr. Heron's investigation constituted a valuable contribution to our knowledge of the behaviour of nitrogen-containing compounds when burnt in oxygen. He felt, however, that much still remained to be explained. The combustion technique used by Mr. Heron was one of a series of methods that have been developed, using what was called the "empty-tube" technique. The first of these methods to be developed was that for the simultaneous determination of sulphur and chlorine in coals (A. E. Beet and R. Belcher, *Fuel*, 1940, **19**, 42). This was a macro method using 0·5 to 1·0 g. of sample. In the initial stages of the work it was assumed that nitrogen oxides would be formed from oxidation of the nitrogen in the coal, and a titrimetric method was sought that would be specific for the sulphate ion, rather than an alkalimetric method, because it was believed that nitric acid would be formed. Since none of the titrimetric methods for sulphate examined was reliable it was decided to test whether or not nitrogen oxides were formed when the coal was burnt under these conditions, that is, in an empty porcelain tube at a temperature of 1350° to 1400° C. No trace of nitrogen oxides was detected by qualitative tests with the brucine reagent. In the method finally adopted sulphur oxides and chlorine were absorbed in hydrogen peroxide to give sulphuric and hydrochloric acids respectively. After determination of the total acidity, chloride was determined titrimetrically and the sulphur figure found by the difference between the two readings. Since the results gave good agreement with the B.S. methods, further evidence—although indirect—was thus supplied that nitric acid was absent, for otherwise the results would have been high. That there was no loss of chlorine or sulphur oxides, compensated by nitric acid absorption was shown by carrying out gravimetric determinations for sulphate and chloride. This method had been adopted by many laboratories in the coking industry and had been recommended as a standard method for routine testing within the industry. Attention was next turned to the determination of carbon and hydrogen in coals and a macro-method in which carbon, hydrogen, sulphur, and chlorine were determined simultaneously was evolved (R. Belcher and C. E. Spooner, *Fuel*, 1941, **20**, 130). If nitrogen oxides were formed, one would have expected the carbon figures to have been unduly high, whereas all results were in accord with those obtained by the B.S. method. Again, this method or a modification of it was used in other laboratories, which, he believed, had satisfied themselves, both by qualitative tests and indirectly by comparing the results with those given by the B.S. method, that nitrogen oxides are not formed. This method had given so much satisfaction and possessed so many advantages over the B.S. method that the British Standards Institution was to examine it with a view to its recommendation in the standard specifications. So far the methods discussed were carried out on the macro-scale and only applied to coals. Later the same technique was used for the analysis of organic compounds on both the macro- and the micro-scale at a temperature of 800° C. during the combustion. The speaker and his collaborators concerned themselves only with the application of the method to organic compounds and did not apply it to coals. Had they done so they would certainly have assumed that nitrogen oxide formation was non-existent, for it was reasonable to assume that a mere reduction in the scale of working would not cause nitrogen oxide to be formed. The few nitrogen-containing compounds that were examined appeared to bear out Pregl's statement, as quoted by Mr. Heron. When the nitrogen was already linked to oxygen, nitrogen oxides were formed. With other combinations such as an amino group, there was no evidence of nitrogen oxide formation. However, it was felt that a more thorough investigation of this aspect was required, hence the cautious statement with which Mr. Heron prefaces his account, that a bubbler containing an absorbent for nitrogen oxides should always be included if the compound contains nitrogen. Mr. Heron's investigation appeared to indicate that this precaution must now always be taken. It was the behaviour of coal on combustion which presented some mystery. When Mr. Heron, using the micro method of the speaker and his collaborators, first communicated his results with coal, some fundamental difference between the two scales of working was sought, because it was thought that their own evidence

and that of many other workers supported the view that nitrogen oxides were not formed from coal on the macro-scale or at least they were formed in negligible quantities. On the macro-scale a porcelain boat had been used, whereas Mr. Heron had used a platinum boat on the micro-scale. This seemed to be a likely cause of the differences because of the catalytic effect of the platinum. Some qualitative tests were done with coal burnt in porcelain and platinum boats, with the result that no nitrogen oxides were found in the former case, but the latter conditions showed that some formation had occurred. When the tube was packed with silica chips the formation also seemed to be enhanced slightly. Mr. Ingram carried out some similar tests at the speaker's suggestion and confirmed these findings as regards the effect of platinum and porcelain boats. An explanation was needed, therefore, as to why nitrogen oxides were formed when micro quantities of coal are burnt in the combustion tube, while the effect on the macro-scale is negligible. It might be that the same amount was formed regardless of the quantity of sample taken and that this amount, whilst significant on the micro-scale, was not apparent on the macro-scale. Against this, however, there were the very variable quantities that Mr. Heron found when using similar weights of coal. When the reason for this phenomenon had been explained we should have a clear picture of the whole process. As far as the various methods were concerned, the original instructions still held; namely, for coals on the macro-scale, nitrogen oxide formation could be ignored. On the micro-scale provision must always be made for removing nitrogen oxides when nitrogen-containing substances were present in a combustion. The only difference appeared to be that what the speaker and his collaborators had recommended as a precautionary measure, Mr. Heron has shown to be a necessity.

Mr. G. INGRAM said that with an empty combustion tube he had found that no nitrogen dioxide was produced by burning coal in a porcelain boat, but with a platinum boat some was formed. Silica chips had but little effect; only occasionally were traces of nitrogen dioxide produced. Organic substances containing nitro groups yielded traces of nitrogen dioxide when burnt in a porcelain boat; under the same conditions, those containing the amino group did not. With a platinum boat high carbon figures were obtained from amino compounds, showing that some nitrogen dioxide was formed.

The Polarographic Determination of Tin, Lead and Zinc in Phenol

BY H. N. WILSON AND W. HUTCHINSON

(*Read at a meeting of the North of England Section on October 19th, 1946*)

INTRODUCTION—Zinc, tin, and—occasionally—lead may be present in "pure" phenol, and methods are required for their determination. In manufacture the phenol is distilled, and a "block-tin" or tin-lined condenser is sometimes used, as it is known that phenol condensed in this way does not discolour so rapidly as that condensed in mild steel or iron. The phenol, whilst still liquid, is run into drums, which may be "galvanised," or zinc-lined. The metals are present in a few parts per million, with perhaps lead and iron also. A rapid and simple method of analysis is required.

Several variations of the dithizone reaction can be used for the colorimetric determination of zinc and lead, but these were found to be troublesome and slow; difficulties were also encountered in the colorimetric determination of tin. As polarographic methods for the determination of these elements have been described,[1,2] it was decided to attempt to devise modifications suitable for our purpose. A rapid method of sufficient accuracy was worked out. This is described below, and followed by a brief description of the experimental data on which the accuracy of the method may be assessed. The quantity of iron commonly present does not interfere, and a determination of the three metals may be completed in 3 hours. The method has now been in routine use for about eighteen months, and has proved very satisfactory.

METHOD

REAGENTS—

Hydrochloric acid, 10 per cent. w/w (sp.gr. 1·050).
Hydrobromic acid A.R. containing 46 to 48 per cent. of HBr.
Bromine A.R.
Sodium hydroxide solution, N.

Benzene, pure. Redistilled in glass apparatus.
Phenol. B.P. phenol doubly distilled in glass apparatus.
Gelatin, 0·1 per cent. solution in water.
Tin standard solution—Dissolve 1·00 g. of pure tin in a minimum of concentrated hydrochloric acid, evaporate to low bulk and make up to 1 litre with 10 per cent. (w/w) hydrochloric acid; (1 ml. ≡ 1·00 mg. of tin).
Lead standard solution—Dissolve 1·29 g. of pure lead carbonate in a minimum of dilute hydrochloric acid, evaporate to low bulk and make up to 1 litre with 10 per cent. (w/w) hydrochloric acid.
Zinc standard solution—Dissolve 1·00 g. of pure zinc in a minimum of dilute hydrochloric acid, evaporate to low bulk and make up to 1 litre with distilled water; (1 ml. ≡ 1·00 mg. of zinc.)
Prepare suitable dilutions of these standards for use as required.

PROCEDURE—

Weigh 25 g. of the phenol into a 150-ml. beaker. Add 50 ml. of benzene, warm gently until dissolved and wash into a stoppered 250-ml. separating funnel with a further 50 ml. of benzene. Extract this solution with five separate 5-ml. portions of 10 per cent. hydrochloric acid, washing each extract twice with 20 ml. of benzene and finally collect the extracts in a 25-ml. measuring cylinder. Adjust the volume to 25 ml. with 10 per cent. hydrochloric acid. It is necessary to shake vigorously and allow to settle well between washes as traces of benzene interfere with the regularity of the tin polarogram.

TIN AND LEAD—Pipette 2 or 5 ml. of the hydrochloric acid extract into a suitable polarographic cell, de-oxygenate with electrolytic hydrogen for 5 minutes, adjust the sensitivity to 1/5 and make a polarogram over the voltage range −0·2 to −0·7 volt, using the mercury pool as the anode. The half-wave potential for tin and lead occurs at −0·46 volts. Measure the step height.

LEAD AND ZINC—Pipette 10 ml. of the extract into a 30-ml. squat beaker and evaporate carefully just to dryness on a hot plate. Add 1 ml. of hydrobromic acid and two or three drops of elemental bromine, evaporate to dryness and repeat the treatment with hydrobromic acid and bromine. This treatment completely removes tin. Cool, extract the residue four times with 1 ml. of boiling *N* sodium hydroxide and combine the extracts in a 10-ml. measuring cylinder. Cool the combined extracts to room temperature, add a further 1 ml. of *N* sodium hydroxide and one drop of 0·1 per cent. gelatin solution and make up to 10 ml. with distilled water. Pipette 2 or 5 ml. of the solution into a suitable polarographic cell, de-oxygenate with electrolytic hydrogen for five minutes, adjust the sensitivity to 1/5 and make a polarogram over the voltage range −0·5 to −0·1 volt, using a saturated calomel cell as the anode. With the same photographic paper, solution and saturated calomel cell, adjust the sensitivity to 1/10, reset the zero and make a polarogram over the voltage range −1·2 to −1·8 volt. Measure the step height in both instances.

The half-wave potential for lead in *N* sodium hydroxide solution is −0·75 volt and that for zinc in the same solution is −1·50 volt.

Carry out a "blank" on the reagents and deduct the step height found for any of the metals from the corresponding step height of the particular metal polarogram. The lead and zinc contents can be read direct from graphs giving the step height in alkaline solution as concentration of the respective metals. In the case of tin, deduct the step height of the lead in alkaline solution from the combined tin and lead step height in acid solution; this gives the step height due to the tin alone, from which the tin content can then be read from the appropriate graph.

A control experiment may be run on a solution containing 0·020 g. of phenol and 0·0005 g. of tin, lead, and zinc per 25 ml. of 10 per cent. hydrochloric acid, and, if necessary, a correction applied to compensate for temperature changes. This solution is equivalent to 20 parts of tin, lead, and zinc per million, when a 25-g. sample is used for analysis.

EXPERIMENTAL

APPARATUS—The polarograph used in this work was the Cambridge instrument, the relationship between galvanometer deflection and current being $5{\cdot}3 \times 10^{-3}$ micro-amps. per

division. All the polarograph work was done in a room with the temperature 20° C. ± 1·0° C. Hydrogen was used for de-oxygenating the solutions by bubbling the gas through for 5 minutes. A mercury anode was used for tin and lead in acid solution, and a saturated calomel anode for lead and zinc in alkaline solution. The capillary constant was $m^{2/3} t^{1/6} = 2{\cdot}09$ mg.$^{2/3}$ sec.$^{-\frac{1}{2}}$ at −0·45 volt in 10 per cent. hydrochloric acid (w/w), 1·88 mg.$^{2/3}$ sec.$^{-\frac{1}{2}}$ at −0·75 volt in 0·5 *N* sodium hydroxide, and 1·80 mg.$^{2/3}$ sec.$^{-\frac{1}{2}}$ at −1·5 volt in 0·5 *N* sodium hydroxide. The step height was taken as the vertical height in divisions between the points of intersection obtained by drawing straight lines through the photographic trace before, through and in line with, and after the step.

DETAIL OF EXPERIMENTAL WORK—

Much time was spent in unsuccessful attempts to find a base solution in which all three metals could be determined polarographically and, after trials of various solutions, we decided upon an acid "base" solution for the tin, and an alkaline "base" solution for the lead and zinc after removal of the tin with hydrobromic acid and bromine.

The extraction of the metals from the phenol was next investigated. As extraction is much quicker than ashing we dissolved the sample in benzene, extracted with dilute hydrochloric acid and determined the amount of the metals in the extracts. Three extractions were sufficient to remove the whole of the metallic constituents. This was proved on samples of phenol containing known amounts of tin, zinc, and lead. Pure phenol (m.p. 40·4° C.) was distilled twice in all-glass apparatus; the final material showed no trace whatever of any of the three metals when examined by the method given above.

Tin was introduced by boiling a known weight of tin foil in a weighed quantity of phenol for three hours, after which the tin foil was removed, carefully washed with benzene and methanol, dried and reweighed. It was thus ascertained that tin equivalent to 20 parts per million had dissolved in the phenol. By the method of analysis described above, duplicate tests indicated 18 and 19 parts of tin per million, a result of adequate accuracy.

Similarly, a solution of lead in phenol was prepared by boiling redistilled phenol with a known weight of bright sheet lead in exactly the same manner as with tin. This gave a figure of 30 parts per million based on loss of weight, and a polarographically determined figure of 31 parts per million.

A solution of zinc in phenol was prepared by boiling the redistilled phenol with a weighed amount of zinc dust, allowing the excess of zinc to settle out from the hot phenol, pouring off into a beaker as much phenol as possible, then recovering the zinc dust on a Gooch filter crucible, washing with methanol and finally drying at 105° C. This showed that the phenol had dissolved zinc equivalent to 75 parts per million. Analysis by the polarographic method given above showed 71 parts per million. As the zinc dust contained some zinc oxide the results are in good agreement, and as the phenol to be examined seldom contains more than 8 parts per million the accuracy is quite adequate.

It was found that small amounts of phenol had an effect on the step height of tin in 10 per cent. hydrochloric acid solution but had little, if any, effect on the step height of lead in the same acid solution. In the extraction of the phenol in benzene solution with 10 per cent. hydrochloric acid under the conditions of the analysis, approximately 0·020 g. of phenol was found in the final extract, and this amount of phenol must be added in the preparation of standards for tin. Since the lead and zinc are determined after evaporation to dryness and treatment with hydrobromic acid and bromine, phenol is absent from the solution polarographed for these metals.

In acid solution, tin and lead are reduced at the same half-wave potential of −0·45 volt against a mercury anode; therefore, in order to determine the lead, the tin must be separated from the lead either by using another base solution in which the two waves are separated and can be measured, or by removing the tin chemically. The latter method was used, as it was found extremely difficult to separate the two waves sufficiently for accurate measurement. By means of hydrobromic acid and bromine, tin (0·5 mg. in 25 ml. of the hydrochloric acid extract, ≡ 20 p.p.m. in the phenol) was completely removed in two treatments. Generally, one treatment was sufficient, but occasionally a trace of tin was left; hence we have prescribed two treatments in the final method. After removal of tin, the lead and zinc were extracted from the residue with *N* sodium hydroxide; three extractions are necessary to remove completely the whole of these metals; and the final solution is made 0·5 *N* before polarographing.

At a sensitivity of 1/10, we found that the following relationships hold. We realise that they depend on the dimensions of the capillary used, etc., but include them as a guide to other workers.

Tin in 10 per cent. HCl by weight, containing 0·02 g. of phenol per 25 ml.—
1 division of step height ≡ 0·029 mg. of tin per 25 ml.
Lead in 10 per cent. HCl by weight, containing 0·02 g. of phenol per 25 ml.—
1 division of step height ≡ 0·0415 mg. of lead per 25 ml.
Lead in 0·5 *N* sodium hydroxide solution—
1 division of step height ≡ 0·0415 mg. of lead per 25 ml.
Zinc in 0·5 *N* sodium hydroxide solution—
1 division of step height ≡ 0·015 mg. of zinc per 25 ml.

ACCURACY OF METHOD—

To establish the accuracy of the method, mixtures were made up from doubly distilled phenol and the solutions of the three metals already described, so as to contain known amounts of the metals, in about the same quantities as the samples under examination—tin about 10 parts per million, zinc less than 5 parts per million, and lead less than 1 part per million. Results are shown in the Table below.

TABLE

	Present, parts per million			Found, parts per million		
Mixture	Tin	Lead	Zinc	Tin	Lead	Zinc
1	20	nil	10	21	nil	11
2	15	5	15	14·5	4	14
3	10	10	20	11	9	21
4	5	15	nil	5	14	nil
5	10	5	15	11	4	14

The method is thus entirely adequate for this type of analysis, and compares well with any other method for traces of such metals in material of this type.

SUMMARY—

Traces of tin, lead, and zinc in phenol may be conveniently determined by the polarograph. Twenty-five g. of the phenol are dissolved in benzene and the solution is extracted with dilute hydrochloric acid. Aliquots are taken for the polarographic determination of the three metals, tin in acid solution, and lead and zinc in alkaline solution after removal of tin with hydrobromic acid and bromine. The three determinations can be completed in less than three hours.

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3. Cholik, J., Hubbard, D. M., and Burkey, K. E., *Ind. Eng. Chem., Anal. Ed.*, **1943**, **15**, 754–759.
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IMPERIAL CHEMICAL INDUSTRIES LIMITED
BILLINGHAM DIVISION
RESEARCH DEPARTMENT

Official Appointments

PUBLIC ANALYST APPOINTMENTS

NOTIFICATION of the following appointments of Public Analysts has been received from the Ministry of Health since the last record in THE ANALYST (1947, **72**, 64).

Public Analysts	*Appointments*
COOMBES, A. H. (Deputy)	City of Birmingham.
HAWKINS, Ernest Stephen	County Borough of Canterbury.
WORDSWORTH, C. H.	Borough of Luton.

The Urban District of Solihull has been constituted a Food and Drugs Area, and the U.D. Council propose to appoint Mr. F. G. D. Chalmers as their Public Analyst.

OFFICIAL AGRICULTURAL ANALYST APPOINTMENTS

NOTIFICATION of the following appointments of Official Agricultural Analysts has been received from the Ministry of Agriculture and Fisheries since the last record in THE ANALYST (1946, 71, 586).

Official Agricultural Analysts	*Appointments*
FLINT, John Walter (Deputy)	Administrative County of Kent.
HAWKINS, Ernest Stephen	County Borough of Canterbury.
WHITTLE, Ernest George	County Borough of Bristol.

Ministry of Food

STATUTORY RULES AND ORDERS*

1947—No. 478. The Soft Drinks (Amendment) Order, 1947. Dated March 18, 1947. Pp. 2 Price 1d.

The Soft Drinks Order, 1946 (S.R. & O., 945 of 1946; ANALYST, 1946, **71**, 381) *is hereby amended* (*a*) *by modifications in the definition of "soft drinks" and* (*b*) *by providing that some soft drinks may contain tartaric or phosphoric acid.*

In the first paragraph of the definition of "soft drink" the words "suitable for or" *are inserted before* "intended for use after dilution as a drink for human consumption." *Dandelion coffee and simple and mixed herbs are now included among the products excepted from the definition.*

In Part II of the Second Schedule, dealing with the ingredients of soft drinks, the phrase "acid content" *is now replaced by* "free acid content." *As before, the acid is expressed as citric acid monohydrate, but it may now include, besides the sources of acid mentioned in the principal Order, added tartaric or phosphoric acid*; provided that no part of such acid content shall be derived from tartaric acid in the case of any description of soft drink containing citrus fruit juice, or from phosphoric acid in the case of any description of soft drink containing any fruit juice. *No change is made in the Table appended to Part II of the Second Schedule of the principal Order, except that in the heading of column 2 the phrase* "minimum acid content . . ." *is changed to* "minimum free acid content . . . ."

— **No. 545 The Meat Products and Canned Meat (Control and Maximum Prices) (Amendment) Order. Dated March 28, 1947.** Pp. 3. Price 1d.

Besides making various changes in maximum prices, this Order, by an insertion at the end of Article 3 of the principal Order (S.R. & O., No. 2046 of 1946; ANALYST, 1947, **72**, 64) *provides* that any fat of vegetable origin used in the manufacture of beef sausages, beef sausage meat, or beef slicing sausage, shall be deemed to be meat for the purpose of assessing the meat content of any of these products, if the total quantity of such fat so used does not exceed 25 per cent. of the prescribed minimum meat content of the product.

Ministry of Health

STATUTORY RULES AND ORDERS*

1947 No. 612. The Ice Cream (Heat Treatment, etc.) Regulations. Dated April 2, 1947, made by the Minister of Health under the Food and Drugs Act, 1938. Price 1d.

1. *These regulations come into operation on the first day of May,* 1947, *with the exception of* 3 (*b*) (*iv*), *which becomes operative on a date to be fixed later.*

2. In these regulations—"ice-cream" includes water ices and any article, under whatever description it is sold, which is so similar to ice-cream as to constitute a substitute therefor:
   "ingredients" includes sugar and dried egg, but does not include colouring or flavouring materials or fruit, nuts, chocolate, and other similar substances; and
   "complete cold mix" means a product which is capable of manufacture into ice-cream with the addition of water only, is sent out by the manufacturer in airtight containers, and has been made by evaporating a liquid mixture which has already been submitted to heat treatment comparable with that prescribed in these regulations.

3. The following requirements shall be observed in the manufacture of ice-cream intended for sale for human consumption:
   (*a*) Where a complete cold mix is used which is reconstituted with wholesome drinking water and to which nothing is added other than colouring or flavouring materials, fruit,

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

nuts, chocolate, or other similar substances, the reconstituted product shall be converted into ice-cream within one hour of reconstitution.

(*b*) In any other case, after the ingredients have been mixed together, the following provisions shall apply:—

(i) the mixture shall not be kept for more than one hour at any temperature which exceeds 45° F. before being subjected to heat treatment in accordance with the next following sub-paragraph;

(ii) the mixture shall be subjected to heat treatment as follows:—

It shall be raised to and kept at a temperature of not less than 150° F. for 30 minutes or alternatively of not less than 160° F. for 10 minutes;

(iii) after the mixture has been subjected to heat treatment as aforesaid it shall be reduced to a temperature of not more than 45° F. within 1½ hours and shall be kept at such a temperature until the freezing process is begun;

(iv), (v), and (vi) *the local authority is given power to specify the necessary indicating and recording thermometers and to exercise supervision over all apparatus. Any temperature charts shall be kept for at least a month.*

4. Ice-cream shall not be sold or offered for sale unless either—

(*a*) it has been kept at a temperature not exceeding 28° F. since it was frozen, or

(*b*) if its temperature has risen above 28° F. at any time since it was frozen, it has again been subjected to the treatment prescribed by sub-paragraphs (i), (ii), and (iii) of regulation 3 (*b*) and, after having again been frozen, has been kept at a temperature not exceeding 28° F.

5. Ice-cream shall be protected from dirt, dust or other contamination at all times during its manufacture, storage and distribution, and all apparatus and utensils brought into contact with ice-cream during its manufacture, storage or distribution shall be thoroughly cleansed immediately after use and shall be kept clean at all times.

CIRCULAR 69/47

This circular explaining the above regulations was issued on the 10th April, 1947, by the Ministry of Health. It states that the regulations referred to in Circular 183/46, ANALYST, 1946, **71**, 539, *have now been made (see above) and come into force, with the exception of those relating to thermometers, on the 1st of May, 1947. They define the conditions under which the ingredients of ice-cream shall be heat treated, cooled, frozen, and stored previous to sale. An exception is made for "complete cold mix" requiring the addition of no ingredient other than water, for which heat treatment is not required, but which must be frozen within an hour of its being reconstituted. Provided that all practicable steps have been taken to procure the necessary apparatus, failure to obtain it before the 1st of May, 1948, is held to be a defence to a charge of non-compliance with regulation 3 (b) (iii). The date from which the indicating and recording thermometers considered necessary by local authorities are to be put into compulsory use remains to be announced by the Minister; but the charts of any recording instruments in use are to be kept for at least a month,* even though the compulsory requirement of such thermometers has not come into operation. *The general cleanliness of all materials and apparatus, together with the operation of the plant, is subject to supervision by the local authority.* The Minister has given further consideration to the prescription in the Regulations of a bacteriological standard of cleanliness for ice-cream, but he is still not satisfied that there is any test, the reliability of which is sufficiently established to justify its use as a statutory test, non-compliance with which would constitute an offence. He desires, however, to draw attention to a form of methylene blue test, adapted for testing ice-cream, particulars of which have been published in the Ministry of Health's Monthly Bulletin for March, 1947. The Bulletin is sent to Medical Officers of Health. Application for these particulars by other persons interested should be made to the Secretary, Ministry of Health, Whitehall, S.W.1. As pointed out in the Bulletin, the conclusions and suggested grading are at present provisional, but the Minister is advised that this test of bacterial cleanliness appears to provide the best available for the present purpose. At the same time it is simple and cheap to perform. It is suggested that if, out of the four grades recommended, ice-cream consistently fails to reach grades one and two, it would be reasonable to regard this as indicating defects of manufacture or of handling which call for further investigation. The Minister has also had regard to the representations which have been made to him on a number of other points with a view to strengthening the Regulations, particularly in relation to the protection of ice-cream after manufacture. While, however, he fully appreciates the importance of the latter, he has come to the conclusion that the Regulations as now made represent as much as it is at present practicable to require.

April 10, 1947

The Dysonian Notation

THE following account of the Dyson system for the nomenclature of organic compounds has been condensed from the record of the lecture delivered by G. Malcolm Dyson, M.A., Ph.D., M.I.Chem.E., F.Inst.Pet., F.R.I.C., on October 21st, 1946, under the joint auspices of the Chemical Society, the Royal Institute of Chemistry, the Society of Chemical Industry, and the Bureau of Abstracts, and published by the Royal Institute of Chemistry.*

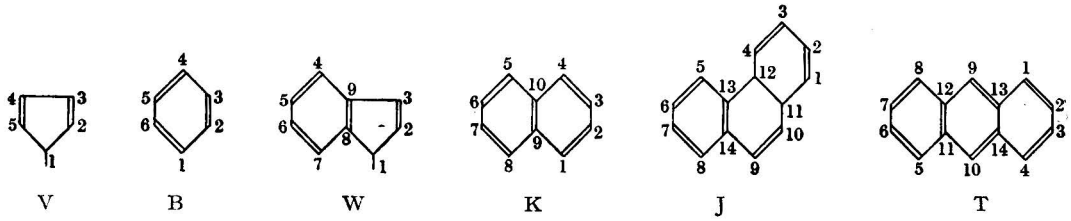
The rules governing the system are here given in the form of instructions for expressing the structural formula or ideograph of an organic compound as a CIPHER, or line of capital letters, numbers, stops, and symbols.

Describe each feature of the formula in turn as a separate OPERATION, dividing operations from each other by STOPS. Number every carbon atom in order as it appears in the ideograph, progressing first through the longest straight chain, following outwards from this along the next longest chain and proceeding in turn along the remaining branches in decreasing order of length. If *polyfunctional groups* do not occur, *i.e.*, groups having an acyclic carbon chain broken by non-carbon atoms as in esters, ethers, etc., start each section of the cipher with a description of the *basic carbon skeleton*. If this is *acyclic*, cipher it in terms of the longest carbon chain present, using C to denote the chain, followed by a MODULANT or number corresponding to the number of carbon atoms in the chain (if the modulant is 1, omit it). Thus, methane is C, ethane is C2, octane is C8. Cipher branched chains in terms of the longest straight chain, adding the branches in subsequent operations, the longest first. Start the operation describing a branch chain with a LOCANT, a number showing to which carbon atom of the main chain the branch is attached. Follow the locant with a description of the branch modulated similarly to that of the main chain; thus, *iso*-butane is C3.2C. Make all additions to the main chain in order of decreasing size. Group together in a single operation two or more similar fragments, separating their locants by a COMMA; thus 2,2,4C indicates three methyl groups in the 2, 2 and 4 positions. Cite a series of locants forming an arithmetical series of four or more terms in the form $a(b)c$, where a and c define the limiting carbon atoms of the series and b defines the constant difference; thus, one writes 4(2)10C and not 4,6,8,10C.

Denote *unsaturation* by the letter E, modulated thus: E, ordinary double bond; E1, double bond with *cis*- arrangement of groups; E2, double bond with *trans*- arrangement; E3, triple bond. Ethylene is C2.E; butadiene is C4.1,3E; *cis*-butene is C4.2E1. Whatever the unsaturation, the initial operation must delineate the longest carbon chain. Cite E operations in order of their modulants, *e.g.*, E before E3.

Cipher *saturated cyclic hydrocarbons* by the letter A, which shows that a ring has been created; thus, AC3 is *cyclo*-propane, AC6 is *cyclo*-hexane. Show a second or third ring as a subsequent operation, indicating the two carbon atoms of the main ring between which the subsidiary ring lies by HYPHENED locants; thus, thujane is AC6.1–3A.C2.4–7C. Indicate carbon atoms in the subsidiary rings in the appropriate A operation; thus, adamantane is AC8.1–5,2–7AC, since each subsidiary ring contains a CH_2 group.

Relate all *aromatic rings* to the six conventional rings set out below with the letters used for them.



Thus, toluene is B.C.; *p*-xylene is B.1,4C; 5-methyl-4-ethyl-1-butyl-phenanthrene is J.C4.4C2.5C. Signify the extra hydrogen with V and W by a modulant as in V1, W1, W2. Cite rings in the same structure in this order of seniority: T, J, K, W, B, V, AC; enclose subsidiary rings in SQUARE BRACKETS, beginning enumeration afresh for each symbol so enclosed. Note that the locants of the conventional rings are numbered anti-clockwise, except in T, where the traditional enumeration is preserved.

Indicate *partial hydrogenation* of a ring by the letter H; thus, dihydrophenanthrene is J.9,10H. Place H before a ring symbol to indicate *complete hydrogenation* as in HT, tetradecahydroanthracene. *Intermediate hydrogenated rings* may be shown by inserting E operations into the fully hydrogenated ring or by a separate operation in H conjoined with the aromatic ring, using whichever leads to the shorter cipher.

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* A fuller account of the system will be found in the book by Dr. Dyson, reviewed on p. 176.

Use the following modulated forms of T, J, K, and W to delineate the more commonly encountered formulae:

Cipher	Extended cipher	Cipher	Extended cipher
T1	T.1–2B4	J3	J.9–10B4
T2	T.2–3B4	J4	J.1–2,3–4B4
T3	T.1–2,3–4B4	J5	J.1–2,5–6B4
T4	T.1–2,5–6B4	J6	J.1–2,7–8B4
T5	T.1–2,6–7B4	J7	J.3–4,5–6B4
T6	T.1–2,7–8B4	J8	J.1–2B3
T7	T.2–3,6–7B4		
T8	T.1–2,15–16B4	K1	K.1–2B3
T9	T.1–2,16–17B4	K2	K.2–3B3
T10	T.1–2,17–18B4	K3	K.1–8B3
		K4	K.1–8B2
J1	J.1–2B4		
J2	J.3–4B4	W1/1	W1.2–3B4

Enumerate carbon atoms of subsidiary rings in ascending order as one proceeds round the ring from the carbon atom of the main ring having the lower locant to that having the higher. Number the carbon atoms of the subsidiary rings in T1 to T10 and J1 to J7 inclusive in this manner: for the subsidiary ring first mentioned in the cipher, 15, 16, 17, 18; for the second, 19, 20, 21, 22. Number those in the subsidiary ring of J8 similarly, 15, 16, 17; those of that in K1, K2, K3, 11, 12, 13· of that in K4, 11, 12; of that in W1/1, 10, 11, 12, 13.

Start the cipher of a *fused ring* from its most senior constituent ring. If it comprises elements of structure over and above those of the six conventional rings, build up its cipher by adduction; thus, J.4–5AC indicates that annellation has taken place at the 4–5 position, and that the adduct is a methylene group. Adducts may be saturated as in AC, AC2; or unsaturated portions of the benzene ring as in B1, B2, B3; odd numbered B adducts need an additional hydrogen citation to complete the structure.

If a hydrocarbon comprises two or more identical rings joined either directly or through a single carbon atom, θ is used to show duplication, 3θ triplication, 4θ quadruplication and so on. θ may be used indefinitely in handling functional links but only once in citing a hydrocarbon structure. Delineate hydrocarbons in which several rings are joined to a chain of carbon atoms or to another and different ring by citing first the chain or ring and treating the several rings as substituents.

Two types of *heterocyclic compound* are recognised. In the first, adducts to hetero-derivatives of conventional rings are more than half carbon; in the second the adducts are more than half heterogeneous. Cipher the first type by adding operations in ZQ (*hetero-oxygen*), ZS (*hetero-sulphur*), ZN (*hetero-nitrogen*), etc., after citation of the corresponding homogeneous structure; thus, pyridine is B.ZN. · Cite in this order: ZQ, ZS, ZSE (selenium), ZTE (tellurium), ZN, ZP, ZAS (arsenic), ZSB (antimony), ZBI (bismuth), using capital letters for the hetero-elements. Cipher the second type in a sequential operation as in B.1–2AQCQ, methylenedioxy-benzene.

Use the following modulated forms of N to denote nitrogenous fractions: N, *amino*; N1, *nitroso*; N2, *nitro*; N3, *azido*; N4, *azo*; N5, *pentacovalent nitrogen*; N6, *alidiazo*.

Cite *functional groups* with Q to denote *oxygen* and *hydroxyl*, EQ for *carbonyl*, X for *carboxyl*; thus, ethanol is C2.Q; acetaldehyde is C2.EQ; acetic acid is C2.X; acetone is C3.2EQ. This suffices for all *alcohols*, *glycols*, *aldehydes*, *ketones*, and *acids*, and compounds containing any or all such groups together.

Cipher simple *ethers* and *esters* from the side containing the larger hydrocarbon residue; thus $CH_3CH_2OCH_2CH_3$ is C2.Q[C2]; $CH_3CH_2COOC_2H_5$ is C3.X[C2]; C_6H_5–$OCH_2CH=CH_2$ is B.Q[3C3.E]; $CH_3COOC_3H_7$ is C3.[XC2]. If an ester is ciphered from the acid side, cite X outside a SQUARE BRACKET containing the cipher of the alcoholic residue; if from the alcohol side, cite X within a SQUARE BRACKET containing that of the acid residue. Cipher esters of polyhydric alcohols from the alcohol side, and esters of polycarboxy acids from the acid side; thus tripalmitin is C3.1,2,3[XC16]; tri-ethyl tricarballylate is C5.3C.1,5,6X[C2].

If the compound is *polyfunctional*, containing a non-cyclic carbon chain broken by non-carbon atoms other than nitrogen in amines, deal first with that portion showing the largest number of operative polyfunctions; if two such portions are equal in length, choose that with the larger number of potential polyfunctions to start with. Thus, in ciphering $C_2H_5O.CO\ CH.CH_2OH\ CH_2O\ CO\ CH_2\ CH_2\ COOH$ start with (1),

1 2 3 4

(1)

a portion having two operative and one potential polyfunctions: C4.X[C2].4[XC4.4X].3Q. Again, glyceryl α-succino-dipalmitate is C3.[X.C4.4X].2,3,[XC16], because (*a*) glycerol has three operative functions and (*b*) the succinic acid has one operative and one potential polyfunction. K. A. W.

OFFICIAL AND TENTATIVE METHODS OF THE AMERICAN OIL CHEMISTS' SOCIETY*

THE methods, bound in a loose-leaf binder, are divided into nine sections and are as follows:—

Section A: Vegetable Source Materials—Sampling of cottonseed, peanuts and soyabeans; determinations of moisture and volatile matter, oil, nitrogen-protein-ammonia, free fatty acids in these three materials; determination of residual lint in cottonseed.

Section B: Oilseed By-Products—Sampling of cake, meal, meats, linters, hull fibre; determination of oil content of these materials; determination of moisture and volatile matter and nitrogen-protein-ammonia in cake, meals, meats; determination of cellulose yield in linters, hull fibre.

Section C: Commercial Fats and Oils—Sampling; determination of moisture by distillation, hot plate, air oven, and vacuum oven methods; determination of soluble mineral matter and fatty acids combined as mineral soap, of free fatty acids, of unsaponifiable matter in marine and other oils; detection of chlorinated solvents (Beilstein test), of sulphur (coin test) (silver benzoate test); determination of refining loss for extracted and de-gummed soyabean oils and for other oils; break test; Halphen test; detection of sesame and teaseed oils; Crismer test; detection of foreign fats in pork fat; determination of melting point by capillary tube and Wiley methods, of softening point and slipping point; flow test; cloud test; determination of refractive index; bleaching tests for cottonseed oil, soyabean oil and animal fats; determination of smoke, flash and fire points; determination of specific gravity of oils and liquid fats, of solid fats and waxes; cold test, titre test; determination of colour by F.A.C. and Wesson methods, of iodine value, of saponification value, of thiocyanogen value, of acetyl and hydroxyl values, of Reichert–Meissl, Polenske, Kirschner values, and of liquid and solid fatty acids.

Section D: Soap and Soap Products—Sampling; determination of moisture and volatile matter by air oven method, of moisture by distillation, of alcohol-soluble and alcohol-insoluble matter, of free acid or alkali in soda soaps, of free alkali or carbonate in potash paste soaps, of water-insoluble matter, of the total alkalinity of alcohol-insoluble matter, of total anhydrous soap and combined alkali, of total anhydrous soap in the presence of synthetic detergents, of chlorides, of unsaponified plus unsaponifiable matter, of unsaponifiable matter, of rosin. Titre test; determination of acid value of fatty acids, of iodine value, of saponification value, of borax, of alkaline silicates, of carbonates by gravimetric absorption method, and by volumetric evolution method, of phosphates, of tetrasodium pyrophosphate, of sulphates, of glycerol, of sugars, of starch, of volatile hydrocarbons, of combined sodium and potassium oxides; screen test; determination of fatty matter in soaps containing synthetic detergents.

Section E: Glycerin—Sampling; determination of ash, of alkalinity or acidity, of sodium chloride, of total and organic residue, of glycerol by acetin and dichromate methods.

Section F: Sulphonated and Sulphated Oils—Determination of moisture by distillation, of moisture and volatile matter by hot plate method, of organically-combined sulphuric anhydride by titration, extraction-titration and ash-gravimetric methods, of total de-sulphated fatty matter, of active ingredients, of unsaponifiable non-volatile matter, of inorganic salts, of total alkalinity, of total ammonia, of acidity in the presence of ammonium or triethanolamine soaps and in their absence, of acidity in dark coloured oils, of water-immiscible solvents.

Section G: Soap Stock—Sampling; determination of total fatty acids by wet and dry extraction methods; of total fatty acids in coconut oil soap stock; of neutral oil; titre test.

Section H: Specifications—Forced draught oven; petroleum ether; air oven; vacuum oven; smoke point thermometer; thermometers for titre test, Crismer test, flow test; desiccants.

Section I: Tables—Tables are presented showing the physical and chemical characteristics of 109 different kinds of fats, oils and waxes, and of 14 authentic samples of cottonseed oil.

All the methods above are described as Official except that for determining the refining loss from extracted soyabean oils, which is a Tentative method, and that for the cellulose yield of cottonseed linters and hull fibre, which is neither Official nor Tentative.

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* Second Edition. Published by the American Oil Chemists' Society, 35, East Wacker Drive, Chicago 1, Illinois, U.S.A. 1946. Price $6.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS

Food and Drugs

Determination of Egg Yolk in Egg White. J. H. Cook and V. C. Mehlenbacher (*Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 785–788)—As little as 0·05 per cent. of yolk in egg white has an adverse effect upon its baking qualities. Owing to difficulties in the extraction of small amounts of fat, estimation of yolk contamination by determination of fat proved unsatisfactory, and the possibility of determining the cholesterol carried into the white by the yolk was investigated. For this purpose the colour development in the Liebermann-Burchard reaction was studied. Five ml. of a solution in chloroform of pure cholesterol were treated with 2 ml. of a 10 to 1 mixture of acetic anhydride and sulphuric acid, and the mixture was placed in the dark for 24 min. at 35° C. The resulting green colour was measured in a Coleman Universal Model 11 spectrophotometer against the colour of pure chloroform in the solvent cell. The transmittance was determined over the range 400 to 800 mμ with a PC-4 filter. The point of maximum absorption, *viz.*, 640 mμ, was used in all subsequent measurements. The intensity of the colour was found to depend not only upon the concentration of cholesterol but also upon the temperature and the time of standing. Ireland (*Biochem. J.*, 1941, **35**, 283; ANALYST, 1941, **66**, 345) showed that the use of lower temperatures during colour development leads to better results, and it was found that colour development at 18° C. in the dark gave the best results with the necessary stability at the point of maximum absorption. The change in transmittance between readings taken at 20- and 30-min. intervals is 0·5 per cent., and, consequently, it is not necessary to make the measurements after the lapse of an exact period of time. In applying the Mojonnier modification of the Röse-Gottlieb method to the extraction of cholesterol it was found necessary to add 25 ml. of aqueous ammonia to the sample before adding any of the extracting solvents; otherwise the alcohol coagulates the albumin to a gelatinous mass that resists extraction. In determining the cholesterol in egg white a 10-g. sample is required,but with yolk a 1-g. sample is used, the extracted cholesterol being dissolved in 250 ml. of chloroform and a 5-ml. aliquot taken. In numerous individual yolks the amount of cholesterol ranged from 1·2 to 1·4 per cent. with an average value of 1·32, and this agrees with reports in the literature. Eggs with varying histories were separated and the whites, uncontaminated with yolk, were analysed to determine the blank value obtained when no yolk was present. This value appears to be constant for all egg whites, ranging only from 0·00011 to 0·00012 (average 0·000114) g. of cholesterol per 10 g. of egg white. Mixtures of yolk and white in varying amounts were then made and analysed.

Method—To prepare the reagent, add 10 ml. of sulphuric acid (sp.gr. 1·84) slowly and with cooling to 100 ml. of freshly distilled acetic anhydride in a 200-ml., glass-stoppered flask. The reagent should be clear and colourless. To prepare the standard curve, dissolve 50 ± 0·2 mg. of pure cholesterol in 500 ml. of chloroform and pipette 1, 2, 3, 4, and 5 ml. of the solution into test tubes (15 × 150 mm.), and make the total volume in each tube 5 ml. by addition of chloroform. Place the tubes in a light-proof water-bath maintained at a constant temperature of 18° C. for 5 min. Add 2 ml. of the acetic anhydride and sulphuric acid mixture, also at 18° C., to each tube, mix thoroughly with a glass rod, and allow the tubes to stand for 25 min. at 18° C. Determine the transmittance in a spectrophotometer at 640 mμ with a solvent cell of pure chloroform set at 100 per cent. transmittance. Plot transmittance (logarithmic ordinate) against concentration on semi-logarithmic paper, thus establishing the standard curve.

To 10 ± 0·1 g. of liquid egg white in a 200-ml., glass-stoppered flask, add 25 ml. of aqueous ammonia (sp.gr. 0·90), swirl the mixture, add 10 ml. of 95 per cent. alcohol, and shake for 1 min. Add successively 25 ml. of ethyl ether (A.C.S. specification) and 10 ml. of light petroleum (b.p. 35° to 38° C.), shaking for 1 min. after each addition, and allow the mixture to stand until the upper layer is clear. Break emulsions, if necessary, with 5 ml. of alcohol. Decant the clear solvent layer into a 250-ml. beaker, repeat the extraction of the residue twice, beginning with the addition of alcohol but using 5 ml. of alcohol each time and adding the final solvent layers to the original extract in the beaker. Evaporate the extract on the water-bath until only a small amount of alcohol remains, cool, add 15 ml. of alcohol, boil on the hot plate, add 1 ml. of 50 per cent. aqueous potassium hydroxide solution, and boil gently for 10 min. Cool, rinse down the sides of the beaker with 30 ml. of ether, and transfer the liquid into a 250-ml. separating funnel. Rinse out the beaker with two 25-ml. portions of water and transfer each into the separating funnel. Rotate the funnel without shaking, break any emulsion that forms with 5 ml. of alcohol, withdraw the lower soap layer, and wash the ether layer with 50-ml. portions of water three times or until neutral to phenolphthalein. Evaporate the ether layer to complete dryness on a water-bath, cool, and pipette 5 ml. of chloroform into the beaker, swirling the mixture gently to ensure solution. Transfer the solution into a test tube (15 × 150 mm.) and place

it in the light-proof water-bath maintained at 18° C. for 5 min. Proceed as in the construction of the standard curve and read the transmittance of each solution. From the standard curve find the concentration of cholesterol corresponding to the transmittance. A blank determination should be made by evaporating to dryness a mixture of all the reagents used, and subjecting the residue to the whole process of analysis; the value so obtained should be deducted from that obtained with the sample. A table is provided for conversion of cholesterol found into percentage of egg yolk in the egg white. The tabular values do not differ widely from the figures obtained by deducting the apparent cholesterol content of egg white (0·00011 g. per 10 g.) from the determined content of the sample and calculating the amount of yolk on the basis of 1·32 per cent. of cholesterol in egg yolk.

A simple, constant-temperature water-bath can be made by wrapping several sheets of asbestos paper round a suitable tin can, lining the lid with asbestos, and piercing it to form a hole only just large enough to take a thermometer. A. O. J.

Semi-micro Determination of Calcium in Foodstuffs. L. C. E. Kniphorst (*Chem. Weekblad*, 1946, **42**, 54–59, 66–70)—The chief difficulties in the determination of calcium in foodstuffs result from contamination of the calcium oxalate precipitate by magnesium, interference by phosphates or pyrophosphates at a *p*H of about 5, and delayed or incomplete precipitation of calcium oxalate under certain conditions. The method is applied to an ash containing 5 to 12 mg. of calcium oxide, not more than 90 mg. of magnesium oxide or 400 mg. of phosphorus pentoxide, and, at most, 5 mg. of iron + aluminium + manganese + copper.

Procedure—Dissolve the ash by repeated extraction with hot 4 *N* hydrochloric acid (in all 35 ml.), re-ignite any carbonaceous residue, and extract again. Dilute the filtered solution to 100 ml., heat to 100° C., and keep on the water-bath for one hour to decompose pyrophosphate. After the addition of bromocresol purple, neutralise the liquid by means of aqueous ammonia, acidify to a yellow colour with acetic acid, and dilute to 200 ml. Heat to boiling, remove the flame, and add 10 ml. of boiling ammonium oxalate solution (saturated at ordinary temperature), with stirring, to the clear liquid. If, however, the liquid is not clear, owing to the presence of phosphates, add bromocresol green, followed by just sufficient 4 *N* hydrochloric acid to clear it. Add then the hot ammonium oxalate solution, followed by 60 per cent. ammonium acetate solution added drop by drop until the *p*H is about 5 (a definite blue colour). Small quantities of iron, aluminium, copper, zinc, and manganese are held in solution by the excess of ammonium oxalate. The quantities of acid, ammonia, and ammonium acetate added must be kept at a minimum, since they are liable to delay the precipitation of the calcium. In general, 5 ml. of ammonium acetate should be sufficient; 8 ml. is the maximum permissible. Keep the mixture on the water-bath for four hours, and stir and scratch with a glass rod at intervals during the first 30 min. If the precipitate appears within ten minutes, filtration may be carried out after four hours; but otherwise the liquid should be left overnight, and then filtered cold. Wash the precipitate twice with a little cold water, then about six times with saturated calcium oxalate solution, until the filtrate shows only a weak reaction for chloride ion. Transfer the precipitate to a beaker, and wash the filter with 25 ml. of hot 4 *N* sulphuric acid. Dilute to 250 ml. with boiling water, treat with 15 ml. of sulphuric acid (1+1), and titrate with 0·05 *N* potassium permanganate added from a microburette. Carry out a blank titration with the same quantity of water and sulphuric acid. The maximum error of this method is 5 per cent.

An alternative method, which gives greater precision, is to ignite the precipitated calcium oxalate to lime, which is then dissolved in 10 ml. of hot, 10 per cent. boric acid solution, diluted with 50 ml. of water, and titrated with 0·05 *N* hydrochloric acid. The indicator is methyl red - methylene blue (Patterson, *Biochem. J.*, 1925, **19**, 601) or methyl red - bromocresol green (Reith and Klazinga, *Chem. Weekblad*, 1941, p. 122), and the end-point is observed by comparison with a solution containing 10 ml. of boric acid solution diluted to 65 ml., and indicator. G. M.

Source of Error in the Gutzeit Method for Arsenic. N. I. Goldstone (*Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 797–799)—Suggested modifications of the official and empirical Gutzeit method have not been widely adopted since they involve either special apparatus or complex procedure. The official method specifies standards of reagents, apparatus, and procedure, but leaves the form of the hydrogen-generating zinc undefined. Stick zinc can be selected only by a visual comparison of the rates of gas evolution, whilst granular zinc does not produce a constant stream of gas. The use of a pellet, the constant surface area of which would produce a uniform rate of gas evolution, thus increasing reproducibility of length and intensity of stain, is suggested.

Method. Preparation of pellets—Select a number of Pyrex tubes of identical internal diameter (approximately 15 mm.) and heat them carefully in a vertical position. Fill the test tubes with molten, arsenic-free zinc and, after tapping out air bubbles, allow to cool slowly. The upper portion should be maintained liquid until last, thus preventing the formation of a hollow core. Break

away the tubes, and cut the rods into suitable lengths, the ends being smoothed with emery. Cover the smooth ends with a paste of magnesium carbonate in gum arabic and allow to dry. Dip the pellet into a suitable wax to give a coat about 0·16 cm. thick. Scrape the ends free of wax and soak off the paste. Activate the pellets with a solution of diluted stannous chloride - hydrochloric acid solution (1+7) and store under water containing a drop of hydrochloric acid. A pellet 3·75 cm. long can be used for about 15 determinations.

Sensitisation of paper strips—Cut a sheet of 32 Hanford-Pratt paper strips into 9-cm. lengths and suspend them in alcoholic mercuric bromide solution in a 10-ml., glass-stoppered graduated cylinder in the dark.

Reagents—Solutions containing 0·002 mg. of arsenic per ml., 0·005 mg. of arsenious oxide and 5 ml. of hydrochloric acid in 35 ml., and 0·10 mg. of arsenious oxide and 5 ml. of hydrochloric acid in 35 ml. were used to test the pellets. A solution of digested oysters was prepared and acidified to the extent of 5 ml. of hydrochloric acid in 35 ml.

Alcoholic mercuric bromide. A 4 per cent. solution in 95 per cent. ethanol.

Potassium iodide. A 15 per cent. aqueous solution.

Stannous chloride. A 40 per cent. solution of the dihydrate in concentrated hydrochloric acid.

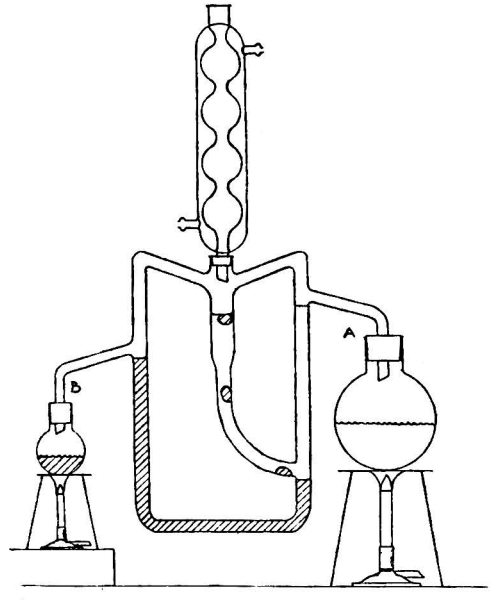
Clean sand moistened with 10 per cent. lead acetate was used in the absorption tower to remove hydrogen sulphide.

Procedure—Sets of six determinations on each solution were made on different days. 35-ml. portions were transferred to generating bottles and 5 ml. of potassium iodide solution added, then 4 drops of stannous chloride solution, and the solutions were left to stand for 0·5 hr. at 25° C. The zinc pellets were then added, the absorption tubes connected, and the generators submerged for 1·5 hr. in a bath at 25° C. The strips were then removed and the stains measured by the usual method.

Discussion—The pellets gave an accuracy comparable with that of the most carefully controlled conditions used by How (*Ibid.*, 1938, **10**, 226) using a zinc alloy and sensitised cotton threads, *i.e.*, 3 per cent. on a single determination. No previous attempt has been made to standardise the exposed area or the uniformity of gas evolution. The pellet surface becomes coated with tin, but the uniformity of rate of evolution of gas is apparently not affected. The pellets wear evenly without pitting of the surface, and there appeared to be no difference between the activities of the six pellets used during the experiments. A combination of the constant area pellet with sensitised cotton threads would probably greatly increase the accuracy of arsenic determinations in the range of 1 μg. of arsenious oxide.

M. E. D.

Titrimetric Determination of Di-acidic Bases (Nicotine, Quinine) in Mixtures with Mono-acidic Bases, and a Simplified Assay of Nicotine. E. M. Trautner and O. E. Neufeld (*Australian Chem. Inst. J.*, 1946, **13**, 70–74)—Of the numerous methods available for the determination of nicotine in the presence of lower amines, which are often liberated from the drug during the assay procedure, it is stated that the simple ones are unreliable, while the reliable ones are complicated and time-consuming. Advantage is usually taken



of the fact that the nicotine bases, like the cinchona bases, can be titrated as mono-acidic or di-acidic depending on the conditions. In chloroform solution, the cinchona bases can be titrated accurately as di-acidic bases with *p*-toluene sulphonic acid solution, dimethyl yellow being used as indicator. In aqueous-alcoholic solution, they react as mono-acidic bases to mineral acids when methyl red is used as indicator. Thus, the difference between the titration figures corresponds to the second nitrogen of the di-acidic bases, while twice the aqueous-alcoholic titration figure, less that found for the chloroform solution, is equivalent to the total nitrogen of all the bases present that have not altered their titration values in the two solvents. Nicotine in solutions in chloroform cannot be titrated satisfactorily with *p*-toluene sulphonic acid, but 0·05 *N* picric acid with dimethyl yellow as indicator gives accurate figures corresponding to a di-acidic base. The solution should be warmed to about 40° C. to prevent precipitation of the mono-picrate as it is formed; precipitation of the di-picrate in no way interferes with the accuracy of the titration. Tested on synthetic mixtures of nicotine with ammonia and with methylamine, the method

gives an accurate indication of the amount of diacidic base present.

The determination of nicotine in tobacco, etc., can be rapidly carried out by extracting the drug by distillation in the apparatus shown. *In the illustration the condenser and the flasks are drawn to half the scale of the separator.* The inner diameter of the separator tubes is 7 to 8 mm. The upper side arm, *A*, is connected to a flask containing the sample under examination immersed in aqueous alkali, and chloroform is placed in the other flask. The contents of both vessels are kept gently boiling, and the mixed vapours are condensed and separated, each being returned to its respective flask. The transfer of the nicotine to the chloroform proceeds smoothly and quickly. *Procedure for tobacco*—Transfer to the water flask such an amount of the drug or the extract, powder, etc., as might be expected to contain between 0·1 and 1·0 g. of nicotine, add 100 ml. of water, 3 g. of sodium hydroxide, and 20 to 30 g. of sodium chloride. A small amount of paraffin wax may be added to inhibit frothing. Charge the other flask with 50 to 75 ml. of chloroform, and heat the contents of both flasks to boiling. After the heating has continued for 1·5 to 2 hr., filter the chloroform through a dry filter, dilute to a known volume, and titrate an aliquot portion with 0·05 *N* picric acid.

With most samples of drug, separation from the volatile bases is sufficiently complete for the aqueous titration to be omitted. The procedure has been checked against that employing precipitation of nicotine silicotungstate, and close agreement between the two methods is indicated. J. A.

Biochemical

Estimation of Vitamin A and Carotene in Small Quantities of Blood Serum. O. A. Bessey, O. H. Lowry, M. J. Brock, and J. A. Lopez (*J. Biol. Chem.*, 1946, **166**, 177–188)—In this method the serum is saponified and extracted on a micro-scale with solvents of low volatility. The light absorption at 328 and 460 mμ is measured, the vitamin A absorption at 328 mμ is then destroyed by irradiation, and the absorption at 328 mμ is again measured.

Method—Into test tubes, 10 cm. × 3 mm., put 60 c.mm. of serum and 60 c.mm. of *N* potassium hydroxide in 90 per cent. ethanol (freshly prepared), immerse the tubes in a water-bath at 60° C. for 20 min., cool, and add 60 c.mm. of a 1:1 mixture of kerosene and xylene. Mix the contents of the tube by holding the tubes at an angle of 45° against a rapidly rotating metal rod slightly flattened at one point, and then centrifuge for 10 min. at 3,000 r.p.m. Cut the tube with a file just above the kerosene-xylene layer and transfer this by means of a pipette into the micro-cuvette of a Beckman spectrophotometer. Measure the absorption at 460 and 328 mμ, transfer the sample to a tube, 4 cm. × 2·5 mm., and irradiate by means of a mercury vapour lamp for 6 or 8 times the time necessary to destroy 50 per cent. of the vitamin A present, as determined by previous experiments with solutions of known vitamin A content. Take a second reading at 328 mμ. In order to eliminate interference due to turbidity, rinse with anhydrous propionic acid, for 1/3 to 1/2 of its length below the constriction, the pipette used to transfer the sample back to the cuvette after irradiation.

Calculation—Calculate the amount of carotene present (in μg. per 100 ml.) by multiplying the absorption at 460 mμ by 480, and the amount of vitamin A (in μg. per 100 ml.) by subtracting the second reading at 328 mμ from the first, and multiplying the difference by 637. The factor 637 is based on a value of 1720 for $E^{1\%}_{1\,cm.}$ at 328 mμ of vitamin A palmitate in alcohol, calculated as the free alcohol. Since vitamin A ester has only 96 per cent. of this value in kerosene-xylene and still retains 3 per cent. of its initial absorption after irradiation, and since furthermore the absorption is reduced 2 per cent. owing to the use of a wide spectral band, the nett value of $E^{1\%}_{1\,cm.}$ is

$$1720 \times 0{\cdot}96 \times 0{\cdot}97 \times 0{\cdot}98 = 1570$$

and

$$1{,}000{,}000/1570 = 637.$$

The method gave essentially the same values for carotene and vitamin A as did a macro-procedure using the Carr-Price reagent. F. A. R.

Tocopherol Content of Skeletal Muscle: Comparison of Chemical and Bio-assay Methods. H. Kaunitz and J. J. Beaver (*J. Biol. Chem.*, 1946, **166**, 205–217)—*Procedure*—Emulsify 30 to 50 g. of muscle with about 100 ml. of acetone in a Waring blendor for 6 min. and transfer to a centrifuge tube with an additional 350 ml. of acetone. After 24 hours, centrifuge, extract the residue once more with acetone, then once with a 1:1 mixture of acetone and light petroleum, and finally with a 1:2 mixture of acetone and light petroleum. Transfer the extracts to a 3-litre separating funnel and add water until 2 layers are formed. Remove the petroleum layer and extract the aqueous phase with two 200-ml. portions of petroleum. Wash the combined petroleum extracts with three 500-ml. portions of water, centrifuge, and evaporate in an all-glass apparatus under reduced pressure in an atmosphere of nitrogen. Transfer the residue to a 25-ml. flask with about 20 ml. of light petroleum. Add 1 to 2 ml. of sesame oil and make up to 25 ml. with petroleum. (The addition of an oil reduces the time required for development of the maximum colour. Sesame oil was selected because it contained only negligible amounts of reducing substances: 0·1 ml. dissolved in 2 ml. of light petroleum should give an extinction

value of less than 0·002 when mixed with 10 ml. of the iron-dipyridyl reagent). The mixture should be slightly yellow and quite clear. To estimate the carotene present, evaluate the colour in a spectrophotometer, with light petroleum as blank, at 440 mμ, and calculate the carotene concentration from a calibration curve.

To estimate the tocopherol content, pipette 1-ml. aliquots of the muscle extract into 3 test tubes, and to one add 1 ml. of light petroleum, to another add 1 ml. of a standard solution containing 40 to 50 μg. of synthetic *dl*-α-tocopherol per ml. of light petroleum, and to the third add 1 ml. of a standard solution containing 80 to 100 μg. per ml. Next add to each of the 3 tubes, 10 ml. of iron-dipyridyl reagent (250 mg. of $FeCl_3.6H_2O$ and 500 mg. of αα′-dipyridyl in 1 litre of glacial acetic acid). Use as a blank 2 ml. of light petroleum containing the same amount as the unknown of the same sesame oil plus 10 ml. of the iron-dipyridyl reagent. Keep the solutions in the dark and evaluate the colours every minute until a maximum reading is obtained. The dilutions of the extract should be such that none of the readings exceeds 0·4. Calculate the results (μg. per ml.) from the expression $e_1n/(e_2-e_1)$ where e_1 is the value obtained for the unknown, e_2 is the value obtained for the unknown plus n μg. of tocopherol. For each μg. of carotene present per ml., subtract 4·8 from the result so obtained. With different amounts of tocopherol and different concentrations of extract, the maximum error observed did not exceed ± 10 per cent. The results were compared with those obtained by bio-assay, and the agreement was within ± 10 per cent.

F. A. R.

Rapid Photocolorimetric Micro-procedure for Blood Sugar. B. D. Polis and M. Sortwell (*Arch. Biochem.*, 1946, **11**, 229–233)—The use of perchloric acid as a protein precipitant gives a convenient method of producing protein-free filtrates having, when mixed with alkaline copper reagent, a *p*H that renders them insensitive to impurities. A modified phosphomolybdic acid reagent has been devised that gives a blue colour stable over a period of 24 hours.

Method—Pipette 0·1 ml. of whole blood into 4·9 ml. of 3 per cent. perchloric acid, shake the mixture, and filter or centrifuge. Put 1 ml. of the clear filtrate into a Folin-Wu sugar tube and add 1 ml. of water and 2 ml. of copper reagent. (To prepare this reagent, dissolve 140 g. of anhydrous sodium carbonate in 800 ml. of water, add 52 g. of sodium tartrate and 44 g. of sodium bicarbonate. Dilute to about 3 litres and shake until the solid is dissolved. Dissolve 20 g. of $CuSO_4.5H_2O$ in a small amount of water, add the solution to the alkaline tartrate solution and dilute the solution to 4 litres. Mix and leave for 1 to 3 weeks and decant from the precipitate of cuprous oxide.) Run a reagent blank at the same time by substituting 1 ml. of 3 per cent. perchloric acid for the blood filtrate. Immerse the tubes in boiling water, heat for 8 min., and then cool for 2 min. in water at room temperature. Add 2 ml. of phosphomolybdic acid reagent (prepared by dissolving 160 g. of reagent-grade phosphomolybdic acid in 2 *N* sulphuric acid and diluting to 2 litres with 2 *N* sulphuric acid; leave for 10 days at room temperature and use the supernatant liquid) and allow the reaction to proceed for 2 min. Dilute the solutions to 25 ml. with *N* sulphuric acid. Maximum colour develops within 5 min. Evaluate the colour in a photoelectric colorimeter using a filter that transmits maximally at 660 mμ after setting the reagent blank to read 100 per cent. transmission. With this method, standards in the range 10 to 80 μg. of glucose fall on a straight line with a deviation of ± 5 mg. per 100 ml. The precision of the procedure under routine conditions was ± 9·4 and ± 12·6 mg. per 100 ml. in the ranges 0 to 150 and 151 to 480 mg. per 100 ml., respectively. F. A. R.

Estimation of Glycogen in Whole Blood and White Blood Cells. R. Wagner (*Arch. Biochem.*, 1946, **11**, 249–258)—The estimation of glycogen in blood requires certain modifications of the usual technique, and special precautions have to be taken.

Method—As an anti-coagulant use 2 mg. of dry potassium oxalate per ml. of blood or, with larger amounts of blood as in the study of isolated white blood cells, 0·2 ml. of a mixture of 3 per cent. ammonium oxalate and 2 per cent. potassium oxalate solutions per 5 ml. of blood; allow this to dry on the bottom of the container in which the blood is collected. Haemolyse 1 ml. of whole blood by adding 2 ml. of water, add 3 ml. of 60 per cent. potassium hydroxide solution and heat for about 30 min. in boiling water. Cool to room temperature, add 6 ml. of water, and precipitate the glycogen with 18 ml. of 95 per cent. ethanol. Allow the precipitate to settle overnight and then centrifuge for 25 min. at 2000 r.p.m. Wash with 60 per cent. alcohol until the supernatant liquid is colourless, and hydrolyse the precipitate with 4 ml. of 2·2 per cent. hydrochloric acid for 3 hours. Neutralise with 2 *N* sodium hydroxide, using one drop of phenol red as indicator and dilute the hydrolysate to 10 ml. Centrifuge and estimate the sugar in a 9-ml. portion by Somogyi's method. Repeat the procedure with another 1-ml. sample of the whole blood, but ferment the hydrolysate for one hour with 0·5 ml. of a 10 per cent. aqueous suspension of baker's yeast prior to determining the sugar. In the analysis of isolated blood cells, transfer the total hydrolysate to a 10-ml. flask, and use 5 ml. for the direct determination and 5 ml. for the determination after fermentation. The difference between the two results is a measure of the glycogen

content of whole blood or of the isolated blood cells. The fermentation is best carried out at *p*H 4 to 6, a convenient method being to neutralise the hydrolysate with 2 *N* sodium hydroxide until the phenol red turns red and then to add a 1 per cent. solution of potassium dihydrogen phosphate until a yellow-pink tinge is formed (*p*H 4·9).

F. A. R.

Organic

Qualitative Elementary Analysis [of Organic Substances] by Hydrogenation. A. Slooff (*Chem. Weekblad*, 1946, **42**, 34)—*Procedure*—Heat the organic compound (20 mg.) in a transparent silica tube, and carry the evolved gases by means of a current of hydrogen over a platinum spiral heated to 800° C. Pass the gases over a boat containing sodium hydroxide at 400 to 450° C., and finally through a trap containing a drop of 0·1 *N* hydrochloric acid coloured with methyl orange. *Carbon* is shown by a black residue from the ignition, and often by a black sublimate. The odour of naphthalene or diphenyl in the issuing gases indicates an aromatic compound. *Oxygen*—Water condensed in the connecting tube shows the presence of *oxygen*. In order to exclude false indications due to the presence of traces of moisture, the heating of the organic compound is temporarily interrupted, when, if oxygen is present, there is a rapid accumulation of small drops of water. *Nitrogen*—Ammonia is formed and caught in the trap, the contents of which may be titrated. If more than 0·1 ml. of *N* acid is consumed, nitrogen is present. *Sulphur*—Hydrogen sulphide is partially absorbed by the sodium hydroxde, and partially evolved in the issuing gases, where it may be detected by lead acetate paper. *Halogens*—These are held by the sodium hydroxide, and may be detected by means of silver nitrate. *Phosphorus*—Phosphorus is partially converted to phosphoretted hydrogen, and partially to phosphorus pentoxide. The former is detected in the issuing gases by moist silver nitrate paper (a positive reaction is also given by sulphur); the latter, in the sodium hydroxide. *Volatile metals*—Arsenic, antimony, cadmium, mercury, and zinc form mirrors before the platinum spiral, or behind the sodium hydroxide. Arsenic and antimony also form hydrides, which may be detected by their odours, whilst the mirrors may be dissolved in *aqua regia* and tested in the usual way. *Non-volatile metals*—These remain in the ash, which is extracted with warm water and tested for alkalies and alkaline-earths. The residue is dissolved in *aqua regia* and tested for other metals. The method may be used for liquids, and, if they are adsorbed on charcoal before analysis, for gases. Certain elements may sometimes remain in the ash in the form of salts, and the ash should therefore be tested for halides, phosphate, sulphate, and nitrate. G. M.

Kjeldahl Determination of Nitrogen. Elimination of the Distillation. K. Marcali and W. Rieman 3rd (*Ind. Eng. Chem., Anal Ed.*, 1946, **18**, 709–710)—The determination of ammonia without distillation by the use of standard sodium hypobromite solution is inconvenient since the hypobromite solution is unstable at room temperature and must be kept between 0° and 5° C. Since ammonia reacts readily with formaldehyde to form hexamethylenetetramine, ammonium salts may be titrated with sodium hydroxide solution in the presence of formaldehyde with phenolphthalein as indicator. The method consists of oxidising the organic matter in the sample by concentrated sulphuric acid, potassium sulphate, and a mercury catalyst; neutralisation of the excess acid with alkali in the presence of sodium bromide to prevent precipitation of mercury compounds; and titration of the ammonium salt with standard alkali to the phenolphthalein end-point in the presence of formaldehyde.

Method. Reagents—0·1 *N* sodium hydroxide standardised against potassium biphthalate and stored in alkali-resistant glass. Neutral formaldehyde solution, approximately 18 per cent. prepared by diluting the reagent grade solution (36 to 38 per cent.) with water, adding 2 drops of 1 per cent. phenolphthalein solution, and neutralising with 0·1 *N* sodium hydroxide just before use.

Procedure—Take a sample containing about 4 mg.-equivalents of nitrogen, and treat it with 10 g. of anhydrous potassium sulphate, 0·6 to 0·7 g. of mercuric oxide, and 15 ml. of concentrated sulphuric acid. Heat cautiously, as usual, until 20 min. after the solution clears. Cool, and dilute with 50 ml. of water. Add 10 ml. of 60 per cent. sodium bromide solution, 2 drops of 0·1 per cent. methyl red, and neutralise with 10 *N* sodium hydroxide. Boil gently for 3 min. to expel carbon dioxide, cool, and add 10 *N* alkali until the solution is just yellow. Add *N* sulphuric acid until the pink colour is just restored, and titrate with standard 0·1 *N* sodium hydroxide; read the burette. Add 30 ml. of the formaldehyde solution, disregard any slight pink colour of the solution, and continue the titration until the solution is yellow. Add 8 drops of phenolphthalein solution and complete the titration to the first distinct pink colour. The alkali used between the methyl red and phenolphthalein end-points is equivalent to the nitrogen present. If more than 17 ml. of concentrated sulphuric acid are used, enough silica is introduced as an impurity in the sodium hydroxide during the neutralisation to buffer the phenolphthalein end-point and to prevent a sharp colour change. Determine and apply a blank correction, usually 0·20 to 0·30 ml.

Results—On eleven carefully purified organic substances a maximum mean deviation of 1 in 220

is recorded, from 2 to 6 determinations being made on each substance. Standard procedures give similar accuracy on the three compounds for which results are given by each method. Agreement between the methods is good in samples with both low and high nitrogen contents. Calcium, barium, copper, and iron interfere by forming precipitates that obscure the end-point; phosphorus interferes since it is converted from primary to secondary phosphate between the end-points, so the method is inapplicable to fertilisers and many biological samples. Use of the sulphuric acid-salicylic acid modification before digestion renders the method applicable to samples containing nitrate-nitrogen.

M. E. D.

Estimation of Pentose in the Presence of Large Quantities of Glucose. A. H. Brown (*Arch. Biochem.*, 1946, **11**, 269–278)—Hexoses interfere with the orcinol reaction for the colorimetric determination of pentose. The procedure now described eliminates the interference due to as much as a 15-fold excess of hexose. The method employs light of different wavelengths in a conventional dichromatic assay.

Method—Into a series of tubes put 0·5, 1·0, and 1·5 ml. of a standard solution containing 0·1 μM of xylose or other pentose per ml., and the same volumes of a solution containing 3 μM of glucose per ml.; include with the series a distilled water blank. Make up the volume in each tube to 1·5 ml. with water. Put into other tubes the unknown solutions diluted so that the pentose concentration of each is in the range 0·03 to 0·07 μM per ml. Biological extracts should first be clarified by means of the barium - zinc method of Somogyi (*J. Biol. Chem.*, 1945, **160**, 69), rather than by the more usual lead oxalate method, which should be avoided. Dilute the unknowns to 1·5 ml., and to both standards and unknowns add 4·5 ml. of orcinol reagent. (This is prepared by dissolving 4 g. of orcinol in 100 ml. of 1·5 per cent. $FeCl_3.6H_2O$ solution and diluting 1 vol. to 20 vols. with 30 per cent. hydrochloric acid.) Immerse the tubes in boiling water for 20 min., cool, and evaluate the colour with a red filter, *e.g.*, Klett No. 66, or Corning No. 2403 alone or with Corning No. 3962, to eliminate infra-red. Repeat the colour measurement with a green filter, *e.g.*, Klett No. 54, or a combination of the three Corning filters No. 5120, 4303, and 3484. It is advisable to measure the colours of any one tube with the two filters within 30 min. The results can be calculated from the simultaneous equation resulting from the two estimations, but the method is time-consuming, and it is simpler to adopt a graphical method of calculation as follows: From the series of standard hexose samples prepare for both sets of filters calibration curves relating colorimeter readings to glucose concentrations. From the readings obtained with the unknown, determine its apparent glucose content for each wavelength from this calibration curve. If pentose is present, the apparent glucose content will be different in the two instances, and the difference can be converted graphically into true pentose content from the curve obtained by plotting the colorimeter readings of the pentose standards against the difference in the apparent glucose content. The error of the method is less than ± 5 per cent. for a single estimation within the range 0·03 to 0·20 μM of pentose in 1·5 ml. of sample when not more than 1·0 μM of glucose is present. The method is applicable to the estimation of pentose in fructose-pentose mixtures.

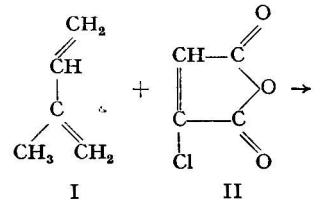
F. A. R.

Colorimetric Estimation of Glycogen. D. L. Morris (*J. Biol. Chem.*, 1946, **166**, 199–203)—The red-brown colour formed by glycogen with iodine varies in intensity not only with the concentration of the glycogen, but also with the temperature, the iodine concentration, and the source of the glycogen. Extraordinary precautions must be used when glycogen is estimated by measurement of this colour.

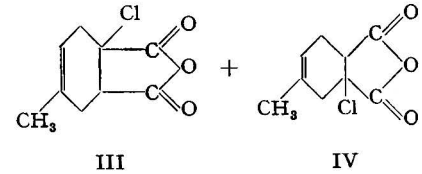
Glycogen from animal and vegetable sources appeared to produce the same colour when ammonium sulphate and iodine were added, and it was impossible to distinguish between them in this way.

F. A. R.

Determination of Conjugated Diolefines with Chloromaleic Anhydride. S. T. Putnam, M. L. Moss, and R. T. Hall (*Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 628–630)—Chloromaleic anhydride reacts with conjugated diolefines in the same manner as maleic anhydride to form a Diels-Alder adduct. Thus, isoprene (I) reacts with chloromaleic anhydride (II) to give 1-chloro-4-methyl-4-cyclohexene-1:2-dicarboxylic anhydride (III) and 2-chloro-4-methyl-4-cyclohexene-1:2-dicarboxylic anhydride (IV).



I II



III IV

Owing to the difference in reactivity between the highly active tertiary chlorine of the adduct and the unreactive vinyl chlorine of the reagent, a quantitative determination of the adduct in presence of

excess of the reagent can be made by heating under reflux with aqueous silver nitrate, which removes the chlorine completely from the adduct, leaving the chlorine of the reagent untouched.

Preliminary investigation showed that purification of commercial chloromaleic anhydride is necessary, chloromaleic acid, formed by hydrolysis, being a very troublesome impurity, probably catalysing the dimerisation and polymerisation of the conjugated dienes, and thus rendering them unavailable for adduct formation. Even with extensive purification it is difficult to obtain a reagent giving theoretical results, and it is advisable to use a purified reagent giving values between 95 and 100 per cent. of the theoretical, and to apply an empirical correction factor determined by analysing a sample of known diene content. Low and variable results are also obtained in presence of peroxides, which catalyse the co-polymerisation of conjugated dienes with chloromaleic anhydride, and consequently a small amount of inhibitor, *e.g.*, *p.*-tert.-butylcatechol, is added as a precautionary measure in all analyses.

Procedure—To purify the commercial reagent, separate the crystals from the mother liquor by filtration, wash them with dry hexane, dissolve them in the minimum amount of dry benzene required, and add hexane to incipient turbidity at room temperature. Seed the solution and cool in an ice bath (+3° C.). Collect the recrystallised product by filtration, and distil it under reduced pressure in an atmosphere of dry nitrogen, or carbon dioxide. Collect the fraction boiling at 110° C. under 44 mm. of mercury, and seal it in small glass ampoules in dry nitrogen or carbon dioxide until required. The purified product melts at 32° to 34° C., and is completely soluble in dry benzene (chloromaleic acid is insoluble). If the correction factor, determined by analysis of a known sample, exceeds 1·05, further purification is recommended.

Introduce volatile samples into a small, tared glass bulb having a long, finely drawn-out neck by inverting the orifice in the liquid and cooling the bulb with dry ice until 0·1 to 0·2 g. of sample has been drawn into the bulb. Seal the bulb quickly in a small flame, holding a piece of dry ice just below the tip of the stem to prevent carbonisation of the sample. Reweigh the bulb at room temperature and place it in a pressure bottle with 1 ± 0·1 g. of chloromaleic anhydride. Add 1 drop of a 10 per cent. solution of *p-tert*-butylcatechol in nitrobenzene, cap the pressure bottle by means of a household bottle capper, and break the bulb by striking the bottle sharply against a suitable surface. Heat for 2 hr. at 55° C., cool, open the bottle, and transfer the contents into a 250-ml. Erlenmeyer flask through a funnel with the aid of 10 ml. of acetone and 50 to 75 ml. of water. Rinse the cap with 2 ml. of acetone. Add 20 ml. of 0·2 *N* silver nitrate, and boil the mixture under reflux for 1 hr. After cooling, filter, wash the precipitate thoroughly with water, add 5 ml. of diluted nitric acid (1 + 1), and 1 ml. of ferric alum solution to the filtrate, and titrate the excess of silver nitrate with 0·1 *N* potassium thiocyanate. From the amount of silver nitrate converted into silver chloride, calculate the amount of diene in the sample, applying the necessary correction factor. If the molecular weight of the substance is not known, an approximate molecular weight may be assumed, or the result may be expressed as diene numbers.

Non-volatile samples may be weighed directly into the pressure bottle from a weighing burette. Otherwise the procedure is the same.

Heating at 55° C. for 2 hr. is sufficient for samples containing over 25 per cent. of isoprene, or for *cyclo*pentadiene. Samples containing smaller amounts of isoprene should be heated at higher temperatures and/or for longer periods, *e.g.*, samples containing from 5 to 10 per cent. required 4 hr. at 75° C. Butadiene reacts more slowly than isoprene and samples must be heated at 55° C. for 12 hr. to obtain satisfactory results. Isoprene - butadiene mixtures containing from 5 to 10 per cent. of butadiene require 6 to 8 hr. at 55° C., and mixtures containing 20 to 25 per cent., 8 to 12 hr. The method should be applicable to samples containing less than 5 per cent. of diene, but the optimum conditions have not been established experimentally.

Styrene interferes in the determination owing to the formation of a co-polymer with chloromaleic anhydride, even in absence of peroxides. The co-polymer contains tertiary chlorine, which is determined with the chlorine of the adduct. The method has been used most extensively for the determination of isoprene, but has been applied successfully to the determination of butadiene, *cyclo*pentadiene, and other conjugated dienes. Preliminary experiments show that *trans*-piperylene and 2-methyl-1:3-pentadiene can be determined, but not 4-methyl-1:3-pentadiene, because this compound forms no Diels-Alder adduct with the reagent. It is probable that the method can be applied to other conjugated dienes by varying the conditions of adduct formation to obtain a quantitative reaction. A. O. J.

Analysis of Furfural-Water Solutions. J. Griswald, M. E. Klecka, and R. V. O. West, jun. (*Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 696–698)—Water in furfural is determined by observation of the cloud-point of equal volumes of furfural and a reagent consisting of a mixture of 1-hexanol and cottonseed oil. Cloud-points with cottonseed oil alone occur at undesirably high temperatures and are lowered by the presence of the hexanol; by adjusting the ratio of oil to hexanol the cloud-point may be brought to a convenient temperature level. By substituting dry furfural for 1-hexanol in the

reagent the method should be adaptable to the determination of water in any other liquid that does not react with furfural.

Hughes and Acree (*Ibid.*, 1934, **6**, 123; ANALYST, 1934, **59**, 430) reported that furfural reacts with a molecular proportion of bromine rapidly and with a second molecular proportion slowly, and they developed a satisfactory bromometric method for determining furfural depending upon this principle. In applying this method to the determination of furfural in aqueous solution, it was found that the first reaction was far too slow to be satisfactory, but that in presence of hydrochloric acid and mercuric chloride it occurred sufficiently rapidly. The end-point is conveniently determined electrometrically as the e.m.f. of a calomel electrode. The e.m.f. rises sharply from about 250 to about 800 millivolts and persists at this value for a limited time when the end-point is reached. A certain excess of bromine is consumed over the stoicheiometric equivalent, but, with proper precautions and attention to details, the end-point is reproducible. Graphs of the stoicheiometric bromine equivalent plotted against the bromine consumption indicated by the potentiometric end-point are straight lines for any given concentration of bromine water, and so the reagent may be standardised by titration against pure furfural in terms of its "potentiometric normality."

Determination of water in furfural—The 1-hexanol is dried by removal of the water by distillation. Furfural for standardising is dried and redistilled under reduced pressure, the first quarter of the distillate being rejected. The cloud-point apparatus is a test tube (1 in. × 4 in.) mounted in a 250-ml. beaker of water warmed by a Bunsen burner. The test tube contains an A.S.T.M. titre test thermometer mounted in a cork and extending to about 1·25 cm. from the bottom, and a stirrer forming a loop round the thermometer. The A.S.T.M. aniline point apparatus may be used with advantage.

Procedure—To prepare the cloud-point reagent mix 0·4 part of dry 1-hexanol with 1 part of cottonseed oil (or commercial Wesson oil) in a glass-stoppered flask. Charge the apparatus with 10 ml. of the furfural sample and 10 ml. of the reagent, warm the stirred mixture until it is perfectly clear and transparent, then cool slowly with stirring until the cloud-point is observed. A haze appears as the cloud-point is approached and the liquid suddenly becomes opaque at about 0·75° C. lower. The "opaque point" is reproducible to within ± 0·1° C. and is taken as the cloud-point. Calibrate the reagent against samples of furfural containing known amounts of water. Samples containing more than 3 per cent. of water should be mixed with 1, 2, or 3 parts of dry furfural for testing. With samples containing less than 1 per cent. of water, a slight improvement in accuracy and convenience is obtained by using a reagent containing 0·3 part of 1-hexanol and 1 part of cottonseed oil.

Bromometric titration of furfural in water—The titration cell consists of a 250-ml. beaker marked at 100-ml. and equipped with a stirrer, a thermometer, a normal calomel electrode, and a reference electrode of a spiral of platinum wire sealed in a glass tube. The cell is mounted in a water-bath (*e.g.*, a 1-litre beaker) since the temperature must be kept constant to ± 0·1° C. during the titration. The e.m.f. is observed by means of a sensitive potentiometer, an electronic voltmeter or a *p*H meter of the proper range (0 to 1 volt). The bromine reagent is made approximately 0·005 *N* in bromine, 0·01 *N* in potassium bromide, and with 10 ml. of conc. hydrochloric acid per 100 ml. It should be made daily and standardised against furfural. A saturated solution of mercuric chloride in water is run through the electrode into the cell for each titration.

Procedure—Calibrate the bromine reagent against 5-mg. samples of furfural using the same titration procedure as for unknown samples. At any constant temperature the volume of reagent plotted against amounts of furfural gives a straight line with an intercept of −0·4 ml. After subtraction of 0·4 ml. from the burette reading at the end-point calculate the potentiometric normality of the reagent. With unknown samples make two titrations, the first to determine the approximate furfural content and the second with an amount of sample containing about 5 mg. of furfural. For the second titration, charge the beaker with the sample, dilute to 100 ml., add 3 ml. of saturated mercuric chloride solution, and start the stirrer. When the temperature is constant add bromine reagent equivalent to about 95 per cent. of the total amount required as fast as the burette will drain, or within 60 sec. This will cause a temporary false end-point potential. As soon as the e.m.f. has fallen below 0·75 volt, add bromine reagent in increments of about 1 per cent. of the expected total until the e.m.f. remains above 750 millivolts for 90 sec. and record this as the end-point. The bromine required depends upon the rate of bromine addition and to a great extent upon temperature. A curve is provided whereby correction may be made for differences in temperature between standard and sample. The necessity for this may be avoided by provision of a thermostat to maintain a constant temperature in the water bath.

With care and attention to detail titrations are reproducible to ± 0·5 per cent. of the total furfural in a 5-mg. sample. Samples containing 4 or 6 mg. of furfural titrated with reagent standardised against 5 mg. of furfural yield results with an accuracy of ± 1·0 per cent. The method will obviously give high results with samples containing impurities that react with bromine. A. O. J.

Inorganic

Determination of Sodium and Potassium in Silicates. S. Kallmann (*Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 678–680)—When a 20 per cent. solution of hydrogen chloride in dry *n*-butyl alcohol (Willard and Smith reagent) is added to a butyl alcohol solution of the perchlorates of sodium and potassium, the alkali metals are precipitated quantitatively as a mixture of sodium chloride, potassium chloride, and potassium perchlorate. The precipitation may be carried out in the presence of most of the elements encountered in silicate analysis and is proposed as the basis of a new method of determining sodium and potassium.

Method. Reagent—Pass dry hydrogen chloride into cooled, anhydrous *n*-butyl alcohol until the solution is practically saturated. The specific gravity of a 20 per cent. solution is about 0·905.

Procedure. Decomposition of the sample—Weigh 0·5 g. or more of finely ground sample into a platinum dish, moisten, add 20 ml. of 47 per cent. hydrofluoric acid and 15 ml. of 72 per cent. perchloric acid, stir, and heat until copious fumes of perchloric acid are evolved. Cool somewhat, rinse down the sides of the dish, and heat again until about half the perchloric acid has been expelled. Cool, and dissolve the salts by adding 20 ml. of hot water and heating. An insoluble residue at this stage may be unattacked sample, or may indicate the presence of large amounts of titanium, or of barium sulphate. Filter and wash the residue with hot water. If it is suspected that the residue contains undecomposed sample ignite it in the dish, treat again with hydrofluoric and perchloric acids, evaporate, dilute, and filter into the main solution. Evaporate the filtrate and heat gently until most of the perchloric acid is expelled.

Some silicates, especially those of high alumina content, or containing much titanium, barium, or lead, are not readily decomposed by hydrofluoric acid. For these, conduct a Lawrence Smith decomposition, add 10 ml. of perchloric acid to the water extract, and evaporate almost to dryness.

Precipitation of sodium and potassium—Add 25 ml. of *n*-butyl alcohol to the perchlorates and boil. (A precipitate at this stage consists of potassium perchlorate and its amount may give a useful guide to the potassium content of the sample.) Add 3 ml. of Willard and Smith reagent dropwise to the boiling solution, and then a further 8 ml. Simmer for 3 minutes, cool, and decant the liquid through a dry Gooch crucible. Rinse the bulk of the precipitate into the crucible and wash 8 to 10 times with 1- to 2-ml. portions of wash solution (40 ml. of reagent diluted with 100 ml. of butyl alcohol.

Separation of sodium and potassium—Dry the beaker and crucible and dissolve the precipitate in the minimum amount of hot water. Add 5 ml. of perchloric acid and evaporate almost to dryness. Cool, add 15 to 20 ml. of a mixture of equal volumes of butyl alcohol and ethyl acetate, heat nearly to boiling for 2 to 3 min., and cool. Decant the liquid into a dry Gooch crucible, catching the filtrate in a small beaker. Wash the residue of potassium perchlorate 3 times by decantation with the mixed solvent. Dissolve the residue in the crucible in the minimum amount of hot water, catching the solution in the precipitation beaker. Add 1 ml. of perchloric acid and evaporate to dryness, expelling any acid condensed on the beaker walls by brushing with a flame. Cool, add 3 to 5 ml. of water, and again evaporate to dryness. Add 10 to 15 ml. of the mixed solvent, heat nearly to boiling for 2 to 3 min., and cool. Filter through the Gooch crucible which has meanwhile been dried, ignited, and weighed. Wash 8 to 10 times with 1-ml. portions of mixed solvent. Add the filtrate and washings to the filtrate from the previous precipitation. Dry the Gooch crucible and precipitation beaker and brush any remaining particles of potassium perchlorate into the Gooch crucible. Dry the crucible for 1 hour at 110° C. and finally, covered by a watch-glass, for 15 min. at 350° C. Cool, and weigh as potassium perchlorate.

Determination of sodium—An estimate of the amount of sodium present may be made by observing the effect of the addition of the Willard and Smith reagent in separating the sodium and potassium from the non-alkali constituents of the sample. If the amount is small, add one-third of its volume of water to the combined filtrate from the potassium precipitation and evaporate to dryness. Add 10 ml. of water, 1 ml. of nitric acid, and 1 ml. of sulphuric acid and evaporate to fuming. Add more nitric acid, if necessary, to destroy organic matter and then heat until all the sulphuric acid is removed. Add water, filter if necessary, evaporate to 1 ml. and determine the sodium by a standard triple acetate method.

If the amount of sodium present is large, evaporate the filtrate to dryness as above. Add 10 ml. of water and 3 ml. of perchloric acid, cover the beaker, and heat until fumes of perchloric acid are copiously evolved. When all organic matter is destroyed, evaporate almost to dryness. Add 15 to 20 ml. of butyl alcohol and boil. Add 7 to 9 ml. of Willard and Smith reagent, the first 2 ml. dropwise, simmer for 1 min. and filter through a dry Gooch crucible and wash 5 times with 1- to 2-ml. portions of wash solution. Dry the crucible at 110° C. for 0·5 hr., and then heat for 5 min. at 650° C. Cool and weigh. Dissolve the precipitate in hot water, wash, dry, and reweigh the crucible. The loss of weight represents the sodium chloride.

The solution may be tested for barium or lead and purified either by adding ammonium carbonate, filtering and recovering sodium as the chloride or by

adding sulphuric acid, filtering and recovering the sodium as the sulphate.

For a rapid routine method the mixed alkali precipitate may be collected on a Gooch crucible and converted to chlorides by heating at 650° C. The residue is then dissolved and either potassium or sodium determined by any convenient method, the other element being found by difference. L. A. D.

Determination of Calcium in Magnesite and Fused Magnesia. W. M. Hazel and W. K. Eglof (*Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 759–760)—In the method proposed for the determination of calcium oxide in materials containing much magnesium oxide, mannitol is used to prevent precipitation of the calcium during precipitation of the magnesium as hydroxide. The calcium is finally precipitated as oxalate and weighed as lime.

Procedure—In a 100-ml. porcelain dish dissolve a weighed portion of about 2 g. of the magnesite or fused magnesia in 35 ml. of diluted hydrochloric acid (1+1). If the material is not completely soluble in the acid, mix a 2-g. sample with 3 to 5 g. of sodium carbonate in a platinum crucible, and heat at 900° to 1000° C. for 30 min. Dissolve the cold melt by adding to it 50 to 60 ml. of diluted hydrochloric acid (1+1), in the platinum crucible. After dissolution of the sample, evaporate the solution to dryness on a steam-bath, and bake the residue for one hour at 120° C. Digest the residue with 5 ml. of concentrated hydrochloric acid and 50 to 75 ml. of hot water. Filter off the silica and wash the precipitate with hot, diluted hydrochloric acid (1+4) and finally with hot water. Transfer the cold filtrate to a 500-ml. graduated flask, add 15 to 20 g. of mannitol, 6 drops of phenolphthalein solution, and then add dropwise, carbonate-free, 9 *N* sodium hydroxide until the solution is coloured pink. Add dropwise, while shaking the flask, a further 5 ml. of the sodium hydroxide solution. Cool to room temperature, dilute to 500 ml., allow to stand overnight, pour the liquid through a loose, dry filter paper, and transfer a 200-ml. portion of the filtrate to a 400-ml. beaker. Add 5 drops of methyl red and acidify with hydrochloric acid, adding 5 ml. in excess. If much magnesium is present in this solution, add 5 g. of ammonium chloride. To the boiling solution add 0·5 g. of ammonium oxalate, and aqueous ammonia until the solution is neutral. Digest on a steam-bath until the calcium oxalate is well formed, remove, add dropwise 2 ml. of aqueous ammonia, and allow to stand for 4 hr. Filter through a No. 40 Whatman paper, and wash the precipitate with cold, 1 per cent. ammonium oxalate solution. Dissolve the precipitate by washing the paper with 50 ml. of hot, diluted hydrochloric acid (1+2). Reprecipitate the calcium oxalate, collect the precipitate, and wash it, following the method used for the first precipitation. Ignite the oxalate in platinum at 1100° to 1200° C. and weigh as calcium oxide (*cf.* Scott, W., "*Standard Methods of Chemical Analysis*," New York, 1939).

Solutions equivalent to those of samples of magnesia containing known weights of from 0·5 to 5 per cent. of calcium oxide were analysed by the method. For weights of lime from 4 to 40 mg., the errors in the analyses were not greater than ±0·2 mg. Several samples of magnesite, including Bureau of Standards Samples, were analysed and the highest probable error found was ±0·6 mg. for 26·8 mg. of lime, the results obtained showing the method to be accurate and trustworthy. The proposed method gives results slightly higher than those obtained by the usual method in which calcium sulphate is precipitated in the presence of alcohol. Small quantities of aluminium do not interfere. Spectrographic analyses of a number of samples of lime obtained by this method showed them to be of analytical purity and free from co-precipitated constituents of the materials investigated. B. A.

Improved Analysis of Fuming and Concentrated Sulphuric Acid by Water Titration. J. C. D. Brand (*J. Chem. Soc.*, 1946, 585–588)—The alkalimetric determination of "free" sulphur trioxide in oleum incurs relatively large errors since most of the alkali is required by the sulphuric acid and only a small fraction is consumed by the sulphur trioxide. Titration with water until the cooled liquid no longer fumes in air can be used to determine the sulphur trioxide directly and more accurately. Modifications of Parker's procedure (*J. Soc. Chem. Ind.*, 1917, **36**, 692) now suggested are (1) a smaller titration drop and (2) a temperature below 10° C. at the end-point of the titration (anhydrous sulphuric acid fumes perceptibly at 13° C.)

Procedure—Using water in a micro-burette graduated to 0·02 ml. and with a jet drawn out to deliver 0·01-ml. drops, titrate 25 to 150 g. of oleum, according to its strength, in a glass-stoppered conical flask. Add the water in such an amount at a time that there is no loss of fume, and absorb the fume within the flask by vigorous shaking after each addition. Cool the flask in ice so that the temperature at the end-point of a titration is less than 10° C. Near the end-point fuming decreases, and is tested for by blowing a puff of air through a tube held a few millimeters above the surface of the liquid and inspecting the surface in bright daylight, the flask being held against a dark background. Immediately before the end-point a wisp of fume develops and lies on the centre of the oleum surface. If v ml. of water are consumed by 100 g. of oleum, then the strength of the oleum is $(100+v)$ per cent. of sulphuric acid, or the percentage of "free" sulphur trioxide is $v/0{\cdot}225$.

Agreement between this method and that involving titration with alkali is satisfactory, but the validity of the new method has been established by determinations of the freezing points of the acids obtained by adding water to an oleum containing 1·00 per cent. of "free" sulphur trioxide, and by showing that the maximum point obtained at 10·5° C. on the freezing point - water content curve corresponds to the point at which fuming ceases abruptly, the freezing point of anhydrous sulphuric acid being lowered by the addition of either water or sulphur trioxide.

Determination of the strength of concentrated sulphuric acid—Attempts to analyse concentrated sulphuric acid by direct titration with oleum to the first appearance of a fume were unsatisfactory, but the back-titration method can be used.

Procedure—Mix a weighed quantity of oleum of known strength with a weighed amount of concentrated sulphuric acid, and determine the remaining free sulphur trioxide by titration with water. The difference between the titration of the oleum and of the mixture of oleum and concentrated acid is equal to the volume of water contained in the aqueous acid, from which its strength can be calculated. The method gives the water content of concentrated sulphuric acid to within 0·2 per cent. of sulphuric acid, an accuracy several times greater than that of the volumetric alkalimetric method.

Preparation of standard aqueous sulphuric acid—Suitable dilution of a weighed quantity of a weak oleum containing about 1 per cent. of "free" sulphur trioxide, which can be done without significant loss of fume, gives approximately *N* solutions of a concentration known accurately to 1 part in 10,000, since this is the accuracy with which an oleum can be analysed by the new water-titration method.

M. E. D.

Nitration in Sulphuric Acid. Part III. Influence of Nitric Acid and other Substances on the Oleum-Water Titration. J. C. D. Brand (*J. Chem. Soc.*, 1946, 880–884)—Water titrations have been carried out on oleum solutions of various solutes and the resulting variations in titre studied. Potassium sulphate, potassium bisulphate, barium sulphate, potassium dihydrogen phosphate, nitric acid, potassium nitrate, sodium nitrite, and barium nitrate all reduce the titre by an amount directly proportional to the solute concentration, and independent of the initial free sulphur trioxide concentration; 2:4:6-trinitrotoluene and 1:3:5-trinitrobenzene have no significant effect. It is found that the reduction in moles of water required per mole of solute added is 0 for the trinitro compounds, 0·5 for potassium bisulphate, 1·0 for potassium and barium sulphates and potassium dihydrogen phosphate, 1·5 for nitric acid, 2 for potassium nitrate and sodium nitrite, and 4 for barium nitrate. The diminution in titre is a measure of the combination between the solute and the sulphur trioxide of the oleum, and is used in interpreting the reactions that may occur when nitrate, nitrite, and hydrogen sulphate ions are added to oleum. M. E. D.

Rare-Earth Metal Oxides. Part II. New Study of Oxide Precipitation by Nitrate Fusion. J. K. Marsh (*J. Chem. Soc.*, 1946, 17–20)—If anhydrous cerous nitrate is added to a cerium-free melt of rare-earth nitrates and alkali nitrates, cerium dioxide is precipitated in the melt and carries down with it a large proportion of the praseodymium present. By precipitation of praseodymium in this manner, it is possible to obtain 96 per cent. pure lanthanum compound from a mixture of lanthanum and praseodymium nitrates.

Praseodymium and neodymium nitrates were estimated with the Hilger "Spekker" Absorptiometer, 1-cm. cells being used. For neodymium, a green light filter (Ilford 604) was used, and it was possible to estimate 0 to 100 g. per l. with an accuracy of 1 g. per l. In the estimation of praseodymium, yellow and orange filters (Ilford 606 and 607) were used together, and a water setting of 0·5 was employed. Amounts of 0 to 100 g. per l. could be estimated with an accuracy of 2 g. per l. For the estimation of praseodymium in the presence of neodymium, filters of a solution of neodymium nitrate, of concentration equivalent to 400 g. per l. of neodymium oxide, were used with a violet filter (Ilford 601). Praseodymium has its main absorption band in the middle of the spectral range, 4540 to 4340 A, covered by the band of light transmitted by this combination of filters. The absorption effect of neodymium was about 3 per cent. of that of praseodymium and a correction for this was applied. From 0 to 30 g. of praseodymium per l. could be estimated to within ± 1 g. per litre.

B. A.

Rare-Earth Metal Oxides. Part III. Their Precipitation from Potassium Hydroxide Melts. J. K. Marsh (*J. Chem. Soc.*, 1946, 20–23)—Rare-earth hydroxides can be peptised in molten potassium hydroxide containing 16 per cent. of water. On desiccation of the melt at high temperatures, and with oxidising conditions, praseodymium dioxide and terbium dioxide are the first oxides to be precipitated. If a dysprosium hydroxide containing terbium hydroxide is peptised in this type of melt and the melt desiccated, an efficient elimination of the terbium from the dysprosium is obtained. A rapid, partial separation of praseodymium from neodymium can be effected by the same method.

B. A.

Use of Ion Exchange Resins in the Study of Chrome Liquors. R. S. Adams (*J. Amer. Leather Chem. Assoc.*, 1946, **41**, 552–573)—This paper describes an investigation of the possibility of using ion exchange resins in the analysis of the

solutions of chromium salts that are used in tanning. Two columns containing ion exchange resins were used, one containing a cation exchanger that removed chromium ions, and cationic complexes containing chromium, from the solutions, and the other containing an anion exchanger that gave up chloride ions in exchange for other anions, including anionic chromium complexes. Solutions of chromium chloride, chromium sulphate, and chromium oxalate, alone, and with sodium salts added, were used in the experiments, one of the chromium sulphate solutions being prepared by reduction of sodium chromate solution by glucose. The solutions were passed through the cation exchanger, the effluent was analysed for chromium, the anions present, and for total acidity; and the amount of cationic chromium present and the amount of anion bound in cationic complexes were calculated. Other portions of solutions were passed through the anion exchanger resin and analysed for chromium, the amount of chromium bound in anionic complexes being so determined.

Method—The resin is contained in a Pyrex glass tube 625 mm. long and 25 mm. in diameter, constricted at the bottom and attached to a Y-tube, both arms of which are closed by rubber tubing and screw clips. Near the top of the main tube is a side tube for overflow. The column of 55 to 60 g. of resin is supported by a piece of porcelain plate, a pad of glass wool, and a 25-mm. layer of washed gravel. The column is filled with cation exchanger resin (Amberlite 1R–100), or anion exchange resin (Amberlite 1R–4).

Procedure—Add a measured volume of the chromium solution at a rate of about 5 ml. per minute for the cation exchanger, or 20 ml. per minute for the anion exchanger, the rate being so adjusted that the liquid level remains just above the top of the column of resin. When all the chromium solution has been added, maintain the level of the liquid until the desired amount of effluent has been collected, by adding distilled water. The first 50 ml. of effluent contain no chromium and may be discarded. Collect and analyse the next 200 ml. of effluent. If the anion and cation columns are run in series, collect the effluent from the first column in 25-ml. portions and pass those portions that show a chromium colour through the second column first. To regenerate the columns pass, at the rate of 5 ml. per minute until all the chromium is removed, 10 per cent. sulphuric acid solution through the cation exchanger resin, and 10 per cent. hydrochloric acid solution through the anion exchanger resin. Wash at a rate of 30 to 40 ml. per minute with distilled water until all excess of acid is removed; and finally, force 500 ml. of distilled water up the column from the side arm of the Y-tube at such a rate as to cause a 50 per cent. expansion of the resin column.

Results for the amounts of chromium ions absorbed are reproducible to within about 1 part in 35, both columns being efficient absorbers, although the anion column did not give up chloride ions quantitatively in exchange for the anions absorbed. When cationic chromium is removed from a chromium sulphate solution, the anionic chromium complexes present dissociate slowly in an attempt to re-establish equilibrium. The rate of dissociation is such as to lead to an appreciable error in the analyses of chromium sulphate solutions by this method. The method is capable of showing the approximate distribution of the chromium in a solution between the anionic and the cationic states and of revealing the approximate proportion of cation to anion in the cationic complex. B. A.

Volumetric Determination of Water in Paints and Varnishes. M. H. Swann (*Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 799–800)—The water content of paints and varnishes can be determined accurately and quickly by a volumetric method that uses the Karl Fischer reagent (Fischer, *Angew. Chem.*, 1935, **48**, 394).

Method—The apparatus used to determine the end-point of the titration consists of a tungsten-platinum electrode connected to a *p*H-meter. The electrode holder carries a stirrer.

Reagent—Prepare a 0·1 per cent. solution of water in pyridine and standardise this solution by titrating it into the Karl Fischer reagent to be used in the determination.

Procedure—Weigh 10 to 20 g. of the paint into a 250-ml., glass-stoppered flask. From a burette add an amount of anhydrous pyridine not less than 10 ml. for thinned samples and up to 25 ml. for unthinned samples. Swirl the mixture, add immediately 25 to 50 ml. of the Karl Fischer reagent, stopper the flask, and allow it to stand in an anhydrous glycol bath at 50° C. for 45 min.; swirl at 10-min. intervals. When the flask is cold, titrate the unreacted Karl Fischer reagent with the standard 0·1 per cent. solution of water in pyridine.

Weighed quantities of a 1 per cent. solution of water in pyridine were added to samples of paints and varnishes and the water contents of the mixtures determined by this method. The values obtained for the percentages of water recovered were correct to within ±1 part in 100. They were much more accurate than those obtained by the azeotropic distillation method. Zinc oxide was the only constituent of enamels found to interfere.

B. A.

Agricultural

Determination of 1:2-*bis*(*p'*-Chlorophenyl) Ethane in DDT Dusts and Oil Solutions. J. B. LaClair (*Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 763–766)—Cristol (*J. Amer. Chem. Soc.*, 1945, **67**, 1494) has suggested that the difference in reaction

rates of *p,p'*-DDT and *o,p'*-DDT when subjected to dehydrochlorination might be used for determination of the *p,p'*-isomer in technical DDT. This reaction has been investigated and procedures applicable to most commercial DDT dusts and sprays have been developed. Samples (1 g.) of technical and purified DDT of known *p,p'*-DDT content were dissolved in 75 ml. of 95 per cent. alcohol and were allowed to react for 15 min. with 20 ml. of *N* alcoholic sodium hydroxide at 25° ± 0·1° C. Graphs of the amount of chloride ion formed (ordinate) and the percentage of *p,p'*-DDT gave straight lines corresponding to the equation (I) (chloride ion per cent. × 11) − 9·3 = *p,p'*-DDT per cent. Samples (0·5 g.) dissolved in 100 ml. of kerosene and allowed to react with 20 ml. of *N* alcoholic sodium hydroxide at 25° ± 0·1° C. while being stirred at 700 to 800 r.p.m. also gave linear graphs corresponding to the equation (II) (chloride ion per cent. × 23) − 16·9 = *p,p'*-DDT per cent.

Procedure for purified and technical DDT—Heat under refluxing conditions a 1-g. sample with 75 ml. of 95 per cent. alcohol until solution is effected, cool, maintain in a thermostatically controlled water-bath at 25° ± 0·1° C., add 20 ml. of *N* alcoholic sodium hydroxide also at the same temperature, and mix by rotating the flask at 5-min. intervals. After 15 min., quickly stop the reaction by adding 15 ml. of diluted nitric acid (1 + 3), remove the flask from the bath, add 30 ml. of 0·1 *N* silver nitrate, 3 ml. of 10 per cent. ferric alum solution, and 5 ml. of nitrobenzene. Shake the stoppered flask for a few seconds, rinse the stopper, and titrate the excess of silver nitrate with 0·1 *N* ammonium thiocyanate. From the percentage of chloride ion found calculate the percentage of *p,p'*-DDT from equation (I).

Procedure for dust mixtures not containing sulphur or organic thiocyanates—The total DDT content must first be found. Samples containing from 5 to 80 per cent. of total DDT can be ignited in a Parr peroxide bomb, or heated under refluxing conditions with metallic sodium and *iso*propyl alcohol (Umhoefer, *Ind. Eng. Chem., Anal. Ed.*, **1943**, **15**, 383), and the total DDT calculated from the chloride ion formed. Samples containing 1 to 5 per cent. are most conveniently analysed by the method of Gunther (*Ibid.*, **1945**, **17**, 149; ANALYST, 1945, **70**, 270), 5 to 10 g. of sample being heated under refluxing conditions with 30 ml. of *N* alcoholic sodium hydroxide, cooled, and acidified with nitric acid, the chloride ion being then determined as already described. The percentage of total DDT divided into 100 gives the sample weight in grams equivalent to 1 g. of total DDT. This sample weight is submitted to the procedure described for analysing technical and purified DDT, and the amount of *p,p'*-DDT in the sample is thus obtained.

Procedure for dust mixtures containing sulphur—These are first analysed for total DDT, a Parr bomb being used. With samples containing only 1 to 10 per cent. of DDT with 50 to 90 per cent. of sulphur the Gunther method is modified as follows. Heat under refluxing conditions a sample, large enough to give an accurate titration, for 10 min. with 25 to 40 ml. of acetone, cool it in an ice bath or refrigerator for 1 hr. to reduce the solubility of sulphur in acetone, remove the sulphur by suction through a small Buchner funnel, and wash it twice with cold acetone. Heat the filtrate on a steam bath with the aid of a current of air only long enough to remove the acetone. Heat under a refluxing condenser the residue of DDT with 30 ml. of *N* alcoholic sodium hydroxide for 15 min., cool, add a measured excess of 0·1 *N* silver nitrate, 50 ml. of water, and a few carborundum chips, and boil gently until most of the alcohol has been removed. Cool slightly, add 20 ml. of diluted nitric acid (1+1) and boil the mixture until all the silver sulphide has been decomposed. Determine the chloride ion by titration with 0·1 *N* ammonium thiocyanate as before, and calculate the total DDT content by means of the factor 3·546. To determine the *p,p'*-DDT content heat under refluxing conditions a sample weight equivalent to 1 g. of total DDT with 40 ml. of acetone for 10 min., cool in an ice bath or refrigerator, remove the sulphur as before, and remove acetone from the filtrate. Heat the residue of DDT under reflux with 75 ml. of 95 per cent. alcohol, cool, and dehydrochlorinate at 25° ± 0·1° C. for 15 min. with 20 ml. of *N* alcoholic sodium hydroxide. Stop the reaction by adding 15 ml. of diluted nitric acid (1+3), add 30 ml. of 0·1 *N* silver nitrate, 50 ml. of water and a few carborundum chips, heat to remove most of the alcohol, add 20 ml. of diluted nitric acid (1+1), and boil to decompose silver sulphide. Determine the chloride ion by titration with 0·1 *N* ammonium thiocyanate and calculate the amount of *p,p'*-DDT as in mixtures not containing sulphur.

Procedure for dust mixtures containing organic thiocyanates—Total DDT may be determined by ignition in a Parr bomb. With samples of low DDT content the Gunther method must be modified to prevent inclusion of cyanide and cyanate ions in the determination of chloride ions. Heat the sample under reflux with alcoholic sodium hydroxide, filter the liquid into a separating funnel and wash the filter thoroughly. Acidify the filtrate slightly with diluted nitric acid and extract twice with light petroleum to remove the dichloroethylene derivative of DDT, which would otherwise yield chloride in the subsequent boiling with nitric acid. Drain the aqueous layer into a 400- to 600-ml. beaker and combine with it two aqueous washings of the petroleum layer. Add a measured amount of 0·1 *N* silver nitrate, 25 ml. of concentrated nitric

acid, and a few carborundum chips. Cover the beaker with a watch glass, boil and evaporate the solution nearly to dryness to decompose silver cyanide and cyanate. Cool, and wash the liquid into a 250-ml. flask and titrate with 0·1 *N* ammonium thiocyanate as before, and calculate the percentage of total DDT as for mixtures not containing sulphur. To determine the content of *p,p'*-DDT, dehydrochlorinate a sample weight equivalent to 1 g. of total DDT with 20 ml. of alcoholic sodium hydroxide at 25° ± 0·1° C., stopping the reaction after 15 min. with diluted nitric acid. Filter into a separating funnel without suction, wash the filter free from soluble chloride, and extract the combined filtrate and washings twice with light pertroleum to remove the DDT olefine. Draw the aqueous layer into a 600-ml. beaker and add to it two aqueous washings of the petroleum layer. Add 30 ml. of 0·1 *N* silver nitrate, 25 ml. of concentrated nitric acid, a few carborundum chips, boil almost to dryness, cool slightly, and repeat the evaporation with 10 ml. of nitric acid. Cool, wash the liquid into a flask with water, and titrate the excess of silver nitrate as before and calculate the percentage of *p,p'*-DDT.

Procedure for oil solutions—Heat a sample of the oil solution weighing from 5 to 20 g. with 30 ml. of *N* alcoholic sodium hydroxide for 15 min. under reflux, cool, acidify slightly with nitric acid, and transfer into a separating funnel. Drain the lower layer into a 250-ml. Erlenmeyer flask, adding to it two aqueous washings of the upper layer. Determine the chloride ion in the aqueous layer and calculate the total DDT by means of the factor 3·546. In presence of organic thiocyanates, heat the sample under reflux as described, cool, transfer to a separating funnel, acidify with nitric acid, and extract twice with light petroleum to remove the DDT olefine. Drain the lower layer into a 600-ml. beaker together with two subsequent aqueous washings of the upper layer, cover the beaker, boil, and evaporate the liquid nearly to dryness. Determine the chloride ion and calculate the total DDT by means of the factor 3·546. To determine *p,p'*-DDT in the absence of organic thiocyanates weigh an amount of sample equivalent to 0·5 g. of total DDT, calculate its volume from its density, and make it up to 100 ml. with kerosene. Maintain the liquid at 25° ± 0·1° C. and, while it is stirred at 700 to 800 r.p.m., add 20 ml. of *N* alcoholic sodium hydroxide at the same temperature and allow the reaction to proceed for 15 min. before adding diluted nitric acid (1+3). Stop the stirrer, rinse the liquid into a separating funnel, and determine the chloride content of the lower layer as already described. Obtain the percentage of *p,p'*-DDT by means of equation (II). For determination of *p,p'*-DDT in presence of organic thiocyanates the sample and kerosene are dehydrochlorinated as already described, 20 ml. of *N* alcoholic sodium hydroxide being added while the liquid is stirred at 700 to 800 r.p.m. The reaction is allowed to proceed for 15 min. and then stopped by addition of 15 ml. of diluted nitric acid (1+3). The reaction mixture is washed into a separating funnel and the separated aqueous layer is treated as in the determination of total DDT in presence of organic thiocyanates. The *p,p'*-DDT content is calculated from the chloride formed by means of equation (II).

The values for *p,p'*-DDT obtained by the method are relative to the crystallisation method (Cristol *et al.*, *J. Amer. Chem. Soc.*, 1945, **17**, 470) as standard. An accuracy of 2 per cent. of *p,p'*-DDT can be expected with dry mixtures and 4 per cent. with oil mixtures. Pyrethrins can cause an error of 5 per cent. in dust mixtures. Water soluble chlorides, when present, must be determined and allowed for; if they occur in large amounts it is advisable to extract the DDT with benzene and determine the total DDT in the benzene extract.

A. O. J.

Gas Analysis

Determination of Hydrogen Sulphide in Gases. E. Field and C. S. Oldach (*Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 665–667)—A sensitive quantitative test for hydrogen sulphide is furnished by the technique described, which enables the optical detection of 1·4 μg. of hydrogen sulphide in aqueous solution, the two methods of procedure being particularly suited to the identification and determination of organic sulphur compounds present as impurities in commercial gases and solvents. The sulphide-bearing gas is scrubbed with 6 per cent. sodium hydroxide solution, and the resulting sulphide is precipitated as bismuth sulphide, the concentration of the suspension being determined by measuring the transmission of monochromatic light with an ultra-violet spectrophotometer. The accuracy is ± 10 per cent. when 7 μg. of hydrogen sulphide are collected, but by increasing the amount absorbed, the accuracy can be raised to ± 3 per cent. A sensitivity to 7 μg. can be attained by precipitating cadmium sulphide in the presence of uranyl ions, and comparing the colour and opacity with a blank in a visual comparator.

Method. Reagents—Prepare a 6 per cent., oxygen-free sodium hydroxide solution, and a solution of bismuth nitrate in diluted acetic acid (1+5), containing about 1 g. of bismuth per litre.

Uranyl-cadmium reagent—Dissolve 44·4 g. of uranyl nitrate hexahydrate and 31·4 g. of cadmium acetate dihydrate in 24 l. of diluted acetic acid (1+5).

For calibration purposes prepare a solution of about 3 g. of sodium sulphide nonahydrate in 1 litre of 6 per cent. sodium hydroxide solution, and

determine its concentration iodimetrically (Shaw, *Ibid.*, 1940, **12**, 668). Dilute aliquot portions with 6 per cent. sodium hydroxide solution to provide solutions of known concentration for the calibrations.

Scrubbing hydrogen sulphide from the gas stream—Oxidation of the sulphide is avoided by the absence of oxygen, but up to 1 per cent. of oxygen is removed by the conversion procedure described by Field and Oldach (*Ibid.*, 1946, **18**, 668, ANALYST, 1947, **72**, 115). Hydrogen, nitrogen, carbon monoxide, methane, ethylene, and up to 2 per cent. of carbon dioxide do not interfere. Absorption of hydrogen sulphide is complete using 6 per cent. sodium hydroxide solution while maintaining a gas rate of 0·6 litre per min. For the comparator method 100 ml. of solution in a Milligan bubbler are used, but 10 ml. are sufficient in the spectrophotometric method.

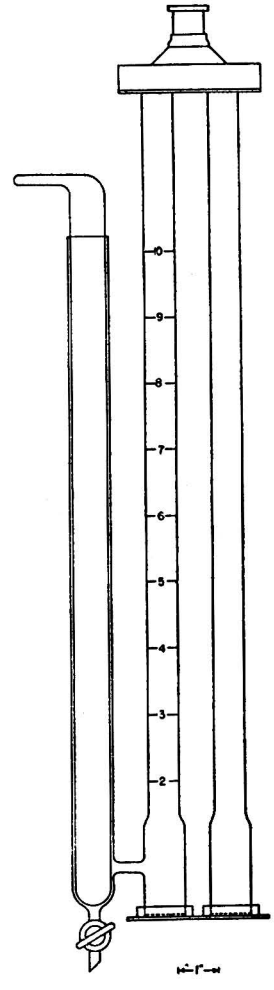
General precautions—Greased joints must be avoided. Use of a nitrogen atmosphere in transferring the solutions is also an advantage. On mixing the solutions, considerable changes occur due to reaction effects, but they become slow after about 5 min. The determination takes about 10 min. to complete, but any tendency for the precipitate to settle during that time can be reduced by adding 0·2 per cent. of clear gelatin to the bismuth reagent (J. K. Fogo, private communication). The final reagent concentrations are not critical, but the solution should be weakly acid.

Spectrophotometric method. Procedure—To the alkaline sulphide solution add an equal volume of bismuth reagent, mix thoroughly by passing oxygen-free nitrogen for 30 sec., and set aside for exactly 5 min. Transfer 10 ml. of the suspension to a spectrophotometer cuvette, the spectrophotometer being arbitrarily balanced at 100 per cent. transmission without the cuvette. Exactly 7 min. after precipitation determine the transmittance at wavelength 350 to 355 mμ with the cuvette in place, and compare with the calibration chart. The alkaline solution should contain between 6 and 40 μg. of sulphur in 10 ml.

A straight line is obtained when the sulphur concentrations of the calibration solutions are plotted against the logarithm of the percentage transmittance. The slope of the line is constant, but the intercept at zero sulphur concentration varies slightly. Thus, once the slope is determined the unit may be calibrated for zero sulphur content by mixing equal volumes of 6 per cent. alkali and the bismuth reagent.

Visual method. Apparatus—A standard comparator tube was used for the blank solution, while the second tube was of special design with a levelling tube and plunger (see Fig.). The light-source was a mercury-vapour lamp, and mounted between it and the comparator were two 2-in. square filters, resulting in the transmission of only blue and green light.

Procedure—Prepare a blank solution by mixing equal volumes of 6 per cent. sodium hydroxide solution and the uranyl-cadmium reagent, and pour it into the blank tube to the top of the scale.



Take a portion of the standard solution containing from 30 to 200 μg. of sulphur, and dilute to 70 ml. with 6 per cent. alkali; mix the solutions by passing nitrogen. Add 70 ml. of the uranyl reagent and stir as before. Pour into the comparator tube and leave for 3 min. Then adjust the plunger so that the colour and intensity match. Repeat with aliquot portions of different sizes. A graph of the logarithm of the balancing height against the sulphur content gives a straight line with a gentle curve near the origin. The curve varies slightly and should be prepared each day. The absorbing solution should contain 30 to 200 μg. of sulphur in 70 ml. to fall in the range of the comparator. Results are reproducible, but a personal factor is

involved. Figures obtained by both methods are given for each sample analysed, and show deviations ranging from −1·9 to +2·3 on values of about 22. Most of the results are within ± 1·0, the units being arbitrary.

The improvement of the accuracy in the presence of uranyl ions occurs only in the use of the comparator. The sulphide-free alkali solution transmits both the green and blue light penetrating the filters, but addition of sulphide causes absorption of the blue, the degree depending on the concentration and height of the liquid column. The use of a plunger is advantageous in that the balance point can be approached from both sides, thus increasing the accuracy over methods in which stop-cocks are used to establish liquid levels. M. E. D.

Physical Methods, Apparatus, etc.

Flame Method of Spectrochemical Analysis. W. A. Roach (*J. Soc. Chem. Ind.*, 1946, **65**, 33–39)—A rapid routine procedure, based on a method due to Ramage (*Nature*, 1929, **123**, 601), is described for the determination of certain elements in plants.

The leaves are the most useful parts for analysis; they are first cleaned, wet methods being avoided if possible owing to the risk of leaching action of the distilled water or solvents used. The cleaned leaves are slowly dried and then ground to a fine powder while still warm. A weighed amount, usually 25 mg., of plant powder is folded into Whatman No. 541 filter paper to form spills which are made to pre-determined dimensions by the use of special jigs. These jigs are designed so that there is no risk of the filter paper coming in contact with the fingers.

The spills are then charred in ammonium chloride vapour at approximately 350° C. This treatment converts the constituents of the material to the corresponding chlorides, thus increasing the sensitivity of the method. The spill, placed in a platinum trough, is pushed into an oxy-acetylene blowpipe flame at a constant speed by a wooden pusher geared to a synchronous motor, a constant length of the spill being burnt during each operation. The spectrogram is prepared by normal methods, and the usual procedures for treating spectrograms quantitatively are applied.

This method can be used for quantitative and comparative diagnostic purposes. Spectrograms obtained from a synthetic standard containing 0·3 mg. of potassium, 0·3 mg. of calcium, 0·06 mg. of magnesium, 0·06 mg. of sodium, 0·006 mg. of iron, and 0·001 mg. of manganese are reproduced to illustrate the sensitivity of the method.

D. A. P.

Identification of Organic Compounds by Use of Chromium Target X-Ray Diffraction Powder Patterns. F. W. Matthews and J. H. Michell (*Ind. Eng. Chem., Anal. Ed.*, 1946, **18**, 662–665)—The identification of organic compounds by X-ray diffraction methods is made difficult by the complexity of the powder pattern. Greater dispersion of the pattern should improve this condition, and this can be accomplished by the use of longer wavelength radiation and/or larger camera diameter. The latter necessitates a refinement of the camera collimator system and would result in longer exposure times. In the work now described, the former method has been used and the patterns of the anilides of the normal saturated fatty acids (C_1 to C_{18}) are shown to be sufficiently characteristic to enable individual identification.

Details of X-ray apparatus, technique, and preparation of the anilides are given.

Powder diffraction data—Lattice spacings and intensities are given in the conventional way for the samples examined. The diffraction data show that the patterns of the anilides of the normal acids C_1 to C_8 are readily distinguishable. The patterns of the even numbers of the series C_8 to C_{18} exhibit marked similarity, suggesting a uniformity of crystal structure, but each pattern can be distinguished; in particular, the longest spacing for each shows a stepwise change with increasing chain length of the acid. Two acids with an odd number of carbon atoms greater than C_8 were also examined and a separate "isostructural" series is indicated for these compounds.

The patterns of the anilides of the *normal* and *iso* isomers of butyric, valeric, and caproic acids could be readily distinguished. Four structural isomers of the C_5 acid were examined; *viz.*, *n*-valeric, *iso*-valeric, α-methylbutyric, and pivalic acids; the anilides of these could also be readily distinguished.

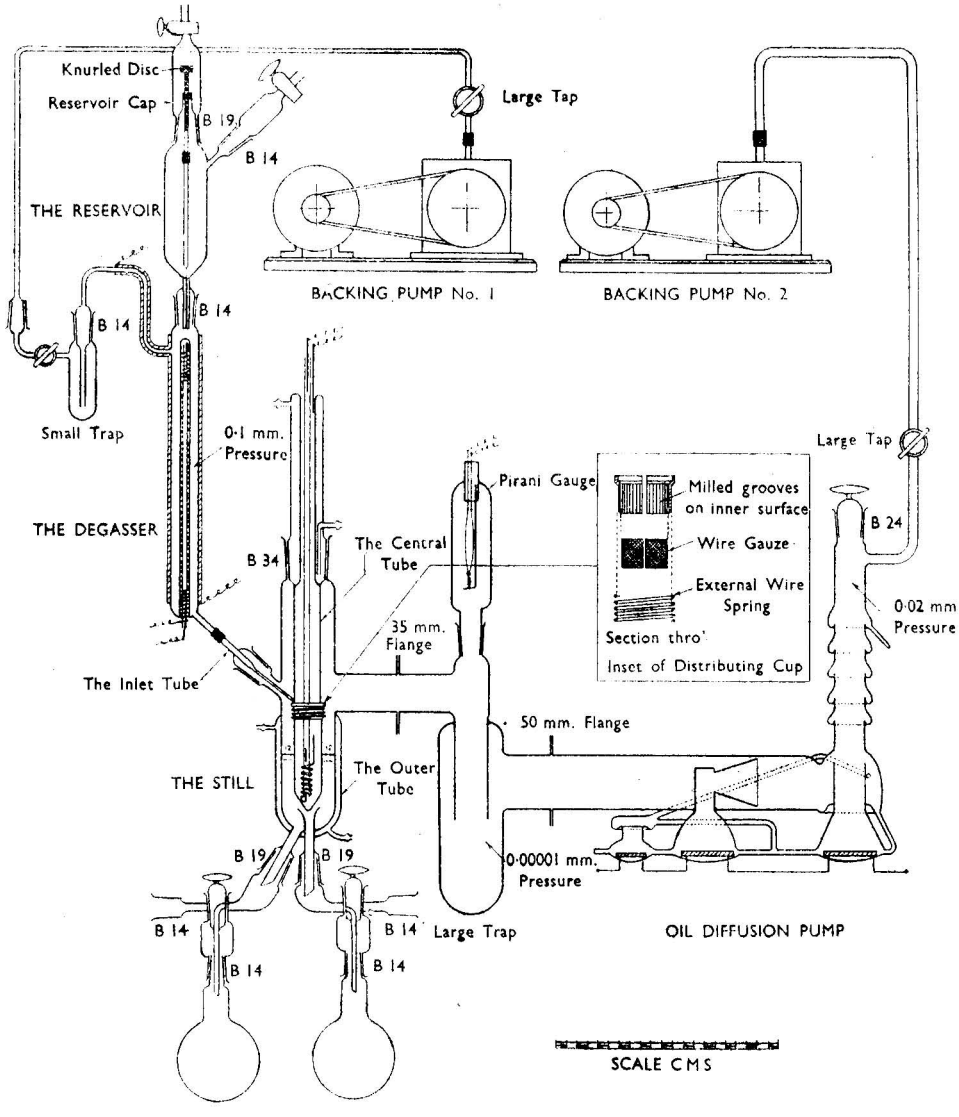
No evidence of polymorphic changes was observed in these compounds, even after several months. All samples were crystallised from solvents at room temperature, and these conditions would tend to give a product stable at room temperature.

Extreme purity of sample is not necessary in obtaining distinctive diffraction patterns.

E. G. S.

Simple Laboratory Falling-film Molecular Still. E. H. Farmer and D. A. Sutton (*J. Soc. Chem. Ind.*, 1946, **65**, 164–166)—A robust and easily operated, falling-film, molecular still in glass is described. It is of non-cyclic type, and has a wide range of usefulness for laboratory work in which moderate quantities of material are to be dealt with. Viscous liquids that do not flow, or mobile fluids that do not spread evenly over the glass distilling surface at the desired distillation temperature, are unsuitable for use in the apparatus. The assembly is made as completely as possible in Pyrex glass whence pin-holes may be readily detected by use of a high-frequency discharge, and the still may be left evacuated indefinitely without leakage. Backing-pump No. 1 should be capable of maintaining a

pressure of about 0·1 mm. of mercury in the degasser, while No. 2 should maintain a pressure of 0·02 mm. in the oil-diffusion pump, trap, and still. The thick rubber bungs connecting the metal nozzles of the pumps with the apparatus should be waxed over with "Apiezon Wax W." A two-jet Hickman all-glass diffusion pump with a nominal pumping speed of 12 litres per sec. at 10^{-4} mm. is employed:



a three-jet pump of the same type is recommended. The large trap is immersed in a Dewar vessel containing liquid air during use, and the gauge attached to it is a Pirani instrument having a range of 0·5 mm. to 0·0001 mm. This may be replaced with advantage by a Philip's Ionisation gauge reading to 10^{-6} mm. The still consists essentially of two concentric glass tubes, the inner one comprising a reflux condenser and a heated distilling tube joined in one piece. The source of heat is a boiling solvent or suitable mixture of solvents electrically heated, the condenser serving to return the solvent to the tube. The distilling (outer) surface of this tube may be roughened to aid the distribution of the distilland over its surface. The even formation of a thin film of the distilland over the distilling surface is achieved by the distributing cup shown separated and in section in the inset diagram. It consists of a stainless steel cup with many grooves, either straight or spirally disposed, milled into its inner surface. This fits over a piece of stainless steel wire gauze of 50- to 150-mesh, and is made to spring tightly over the distilling tube and gauze, and kept in place by a strong external wire spring. The

outer tube of the still has a water-jacket, but this is unnecessary for distilling temperatures over 125° C. The degasser consists of an electrically heated outer tube to the base of which is sealed a smaller concentric tube also electrically heated by an internal heating-coil. The crude distilland drops from the reservoir on to the top of the inner tube at a rate readily controlled by turning the knurled disc, thereby altering the length of stout tungsten wire partially obstructing the glass capillary connecting the reservoir to the degasser as desired. The high vacuum space inside the still is effectively sealed from the relatively low vacuum in the degasser by the distilland flowing through a constriction near the end of the inlet tube. Reference to the original paper must be made for complete details. J. A.

Reviews

A NEW NOTATION AND ENUMERATION SYSTEM FOR ORGANIC COMPOUNDS. By G. MALCOLM DYSON. Pp. 63+iv. London: Longmans, Green & Co., Ltd. 1947. Price 7s. 6d.

For the best part of a century the biggest problem confronting organic chemists has been the naming and indexing of the vast number of compounds they have discovered, made, or envisaged. The Geneva system of 1889, modernised in 1930, and the Ring Index arising therefrom, leave many classes of compound out of consideration, often fail to provide a usable name, and have now diverged far from current usage. The complexity of the system has led to grave arrears in the indexing of information and the compilation of reference works.

Dr. Dyson has tackled the problem as Dr. Clarence Smith hoped it would be tackled when he lectured to the Chemical Society in 1936, by making a fresh start and discarding the old terminology. The basis of the new system is described elsewhere in this issue of THE ANALYST; it will be seen that the ideograph of each compound is dissected in a logical and straightforward manner, each part or attribute of it being successively delineated by a combination of letters, numbers, and symbols unique for each substance. No longer is there confusion in the numbering of rings, or chains, or carbon atoms; no longer may one use alternatively numbers or Greek letters. The cipher can be readily and strictly classified and indexed; "trivial" names engendered by the complexities of the old systematic nomenclature need no more appear in indexes; and there is a strong basis for believing that within a reasonable time it will be possible to produce a lexicon of organic chemistry only a few months behind the then-current wave-front of publication.

Such a development in the history of organic chemistry is obviously of the utmost importance. Dr. Dyson truly merits the profound gratitude of his fellow chemists for his remarkable work, so ably described in this booklet, of replacing the turgid complexities of the Geneva system by a design of modern elegance, precision, and proportion most fitting to the times.

K. A. WILLIAMS

SUPPLY OF LABORATORY APPARATUS AND CHEMICALS

THE Council of the Society will be pleased if any member who is experiencing difficulty in obtaining delivery of laboratory apparatus or chemicals will send details of such difficulties to the Hon. Secretary, Mr. K. A. Williams, 6, Milner Street, London, S.W.3, in order that specific instances may be brought to the notice of the Controlling Authority.

The Council would also be pleased to receive the views of any member on his requirements of apparatus and chemicals for the coming year.

MICROCHEMISTRY GROUP

A MEETING of the Group will be held jointly with the South Yorks. Section of the Royal Institute of Chemistry and the Sheffield Metallurgical Association on Friday, May 16th, 1947, at Sheffield. There will be a visit to the Bragg Laboratory of the Admiralty in the afternoon. At the evening meeting, which will begin at 5.45 p.m. in the Chemical Department of the University, the following papers will be read:

"The Determination of Carbon, Hydrogen, and Nitrogen in Aliphatic Nitro Compounds," by A. E. Heron.

"A Review of Micro Methods for the Determination of Oxygen in Organic Compounds," by C. E. Spooner.

"A New Spot-test for the Detection of Sulphites and Sulphur Dioxide," by R. Belcher and G. Ingram.

"The Microchemical Analysis of Aluminium Base Alloys," by J. Townend and C. Whalley.