THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS

AN Ordinary Meeting of the Society was held at the Chemical Society's Rooms, Burlington House, on Friday, March 6th, at 3 p.m., the President, Mr. John Evans, being in the chair.

Certificates were read in favour of Lewis G. S. Hebbs, A.I.C., William Charles Johnson, James Young, A.I.C.

The following were elected Members of the Society:—Archie Hector Cameron, B.Sc. (Glas.), A.I.C., A.R.T.C., Frederick T. W. Carman, Basil William Clarke, B.Sc., A.I.C., A.R.C.S., D.I.C., Evelyn Beryl Daw, B.Sc., A.I.C., William Edward James Hansford, Cyril Charles Harris, B.Sc., A.R.C.S., Arthur George Jones, B.Sc.(Hons.), A.I.C., Reginald William Money, M.Sc., A.I.C., Horace Edward Newton, Kenneth Sams, B.Sc., Ph.D. (Lond.), A.R.C.S., A.I.C., D.I.C., Winifred Edris Welton, B.Sc. (Lond.), A.I.C., Donald Major Wilson, M.C., B.Sc., A.I.C.

The Annual General Meeting then followed, when Special Resolutions were passed for the alteration of certain Articles of Association of the Society in connection with Area Sections. The Honorary Treasurer presented the accounts for the year, and the Honorary Secretary the Annual Report of the Council. The President delivered his Presidential Address.

The following were elected as Officers and Council for the year 1936:

President.—G. Roche Lynch, O.B.E., M.B., B.S., D.P.H., F.I.C.

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

Vice-Presidents.—A. L. Bacharach, H. E. Cox, L. H. Lampitt, A. R. Tankard (*Chairman*, North of England Section), R. T. Thomson (*Chairman*, Scottish Section).

Honorary Treasurer.—E. B. Hughes.

Honorary Secretary.—Lewis Eynon.

Other Members of Council.—P. S. Arup, B. S. Evans, R. C. Frederick, G. Hogan, B. G. McLellan, A. More, J. R. Nicholls, Miss M. Roberts, W. H. Roberts, W. H. Simmons, R. W. Sutton, J. R. Stubbs (*Hon. Secretary*, North of England Section), J. B. McKean (*Hon. Secretary*, Scottish Section).

SCOTTISH SECTION

THE First Annual General Meeting of the Section was held in the Royal (Dick) Veterinary College, Edinburgh, on 24th February, 1936.

The Report and Financial Statement of the Committee for 1935 were read and adopted.

The office bearers for 1936 were elected as follows:

Chairman—R. T. Thomson; *Vice-Chairman*—T. W. Drinkwater; *Committee*—A. M. Cameron, A. Dargie, Dr. A. Scott Dodd, Dr. H. Dryerre, A. R. Jamieson, and M. M. Love; *Honorary Secretary and Treasurer*—J. B. McKean.

The following paper was read and discussed:—"The Determination of Lead in Potable Waters," by Sidney L. Tompsett, Ph.D., B.Sc., F.I.C.

Anniversary Dinner

ON Friday, March 6th, 1936, the Society held a dinner at the Trocadero Restaurant to commemorate the sixty-second year of its foundation.

The members and guests, who numbered 134, were received by the President, Mr. John Evans, M.Sc., F.I.C., and Mrs. David Evans, and Mr. Evans afterwards took the chair at the dinner.

The guests of the Society included the Rt. Hon. Sir Kingsley Wood, P.C., M.P. (Minister of Health), Sir Edward Tindal Atkinson, K.C.B., C.B.E. (Director of Public Prosecutions), Sir William Willcox, K.C.I.E., C.B., M.D. (Master of the Society of Apothecaries of London), Sir Harry Lindsay, K.C.I.E., C.B.E., I.C.S. (Director of the Imperial Institute), Sir Samuel Roberts, Bart. (Master Cutler), Prof. N. V. Sidgwick, C.B.E., D.Sc., F.R.S. (President, Chemical Society), Dr. R. H. Pickard, D.Sc., Ph.D., F.I.C., F.R.S. (President, Institute of Chemistry), Dr. C. H. Hampshire, M.B., B.S., B.Sc., M.R.C.S., L.R.C.P., F.I.C. (Secretary of the Pharmacopoeia Commission), Dr. Edward Mellanby, M.D., D.Sc., F.R.S. (Secretary, Medical Research Council), Mr. E. Saville Peck, M.A. (President of the Pharmaceutical Society), Dr. E. F. Armstrong, Ph.D., D.Sc., LL.D., F.R.S., Mr. Norman Kendal, C.B.E., Dr. C. Ainsworth Mitchell, M.A., D.Sc., F.I.C. (President, Medico-Legal Society), Mr. R. B. Pilcher, O.B.E., F.C.I.S. (Registrar, Institute of Chemistry), Mr. R. A. Beck.

The President asked the members and guests of the Society to stand for a minute in memory of his late Majesty, King George V.

After the loyal toasts had been honoured, the President proposed the toast of H.M. Ministers, coupled with the name of the Minister of Health. He said that Sir Kingsley Wood had under his care the general health of millions of people leading a complex life. Modern science had shown that man's nutritional well-being was an affair of very delicate balance, and it behoved the large numbers of the members of the Society who were concerned with the manufacture, preparation and inspection of food to do their utmost to see that nothing which nature provided for our nourishment was withheld or damaged. In virtue of that duty it was their privilege to assist the Minister of Health in his onerous task of safeguarding the public health.

The Rt. Hon. Sir CHARLES KINGSLEY WOOD, P.C., M.P., in responding to the toast, said that it would be generally recognised that the responsibility of

those in positions of authority had not grown less in recent years. He would like to say that the extensive plans for the public health services formulated by the Government in their programme at the last election were being completely pursued, and would in no way be checked or hindered by the defence proposals now before Parliament. He regarded the defence proposals as an insurance against war, but certainly he also regarded their projects for the improvement of the conditions of the people as an insurance against disease and ill-health.

The consumption of food in the United Kingdom had grown considerably, and had probably reached a figure of over 25,000,000 tons a year. Every year we consumed 533,000 tons of butter, 886,000,000 gallons of milk, and 202,000 tons of cheese (to refer particularly to the group of foods for the purity of which the analyst was, in a specially large degree, responsible). He did not hesitate to say that for many years the analyst had been the chief defender of the people's food, and he would add that, with the advance of modern food chemistry, his work had not grown less or become easier.

Without doubt, in recent years there had been a considerable improvement in our food standards in this country, and he wished to testify that it had been achieved very largely by the work of the health authorities and their professional advisers. There was no doubt that in past years food frauds were rampant in this country, as might be gathered from the realistic and rather horrifying account of the state of impurity of food in this country, published by Frederick Accum in 1820. Largely owing to the work of many men present that evening, there was now very little gross adulteration or substitution of one article for another. Last year over 140,000 samples were submitted to Public Analysts (the highest on record), and those adulterated, or not up to standard, represented a little over 5 per cent. They had to be vigilant to see that the public had some sort of guarantee that it was getting what it asked and paid for and that the food did not contain any ingredients that were injurious to health. The consumer's interest must come first, from the point of view both of fair trading and of health.

Finally, he would like to say that, so far as his own Department was concerned, the relations of the Ministry of Health with the Public Analysts were both close and cordial. Although the appointment (and the removal) of the Public Analysts was subject to the approval of the Minister of Health, he would remind them that he had no say in their remuneration, and he made no comment upon this point, except to express the view that their duties and responsibilities had grown considerably.

Sir EDWARD TINDAL ATKINSON, proposing the toast of the Society, said that there was a close connection between forensic chemistry and law, and in his work he was greatly impressed by the reports of chemical analysis in cases with which he was concerned. He had tried to compare the work of the Society with his own work. Some might say that his work was destructive, but no one could say that the Society's work was other than magnificently constructive. In the short space of time that we had to live it was of the utmost importance that man should at least get health and happiness from having decent food, and he regarded the analysts as watch dogs against the activities of those who sought to adulterate human food and drink.

The PRESIDENT, responding to the toast of the Society, thanked Sir E. Tindal Atkinson for the kind way in which he had spoken of their work. Their Society was justifiably proud of the fact that a very large proportion of their members were engaged in work directly concerned with the health and well-being of the people. Some were Public Analysts and many others were concerned with the manufacture of food, and one of the chief functions of the Society was that it provided a meeting place for these two groups of chemical workers. The contamination of food with harmful substances due to careless manufacture was a

rare occurrence, and as time went on the Public Analyst and the high-minded food manufacturer were able to say with increasing confidence that their ideals were identical.

Mr. E. HINKS, proposing the toast of kindred Societies, said that they had present with them representatives of five Societies, one Commission, one Council, and one Institute.

Dr. R. H. PICKARD, President of the Institute of Chemistry, said that it was worth placing on record that over 80 per cent. of the members of the Society of Public Analysts were also members of the Institute of Chemistry. A mistake commonly made was to think of an analyst merely as a chemist; nowadays an analyst was expected also to carry out physical and biological tests. The extent to which the art and science of analysis progressed was largely dependent on the way in which the Society fostered research in analytical methods. The discussions and papers presented to the Society were of a very high standard, and the Society's journal, THE ANALYST, was the admiration of all kinds and sorts of chemists. He congratulated the Society on its very vigorous and flourishing condition.

Dr. MELLANBY, who also responded, said the great thing about learned societies was that they brought scientific men into contact with social life. The Society was working entirely in the public service, and it was the men brought into contact with administrators of local life who knew all forms of social life.

The health of the guests was proposed by Professor W. H. Roberts, the toasts being responded to by Sir Samuel Roberts and Sir Harry Lindsay.

Annual Report of the Council

FOR THE YEAR 1935–36

THE Roll of the Society stands at 755, an increase of 22 on the membership of last year.

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

L. A. Archbutt
S. T. Burford
H. C. H. Candy
C. F. Cross
C. T. Kingzett
F. T. Munton
T. H. Pope
P. A. Self
A. J. Starey
A. C. Wilson

Archbutt was in his 78th year. He joined the Society in 1894, and from then onwards took an active part in the proceedings, serving on the Council on several occasions, and, after being Vice-President, he was elected President in 1912. THE ANALYST contains many contributions from him, and he also established his position as a scientific author by his book on *Lubrication and Lubricants*, written in collaboration with Mr. R. M. Deeley. A sympathetic notice on his life, written by his old friend Mr. John White, was published in THE ANALYST (1935, p. 57).

Burford, who also reached the age of 78, was a Vice-President of the Society in 1924–5. He was Public Analyst for Leicester until 1929, when he retired from practice. His obituary notice appeared in THE ANALYST (p. 792).

Candy was for some years Lecturer on Physics and Chemistry at the London Hospital Medical School. He was also known outside the chemical profession

in connection with his literary work on Milton. An obituary notice was published in the November issue (ANALYST, p. 730).

Cross, who died at the age of 80, had been a member of the Society since 1905. He had gained an international reputation for his investigations into the chemistry of cellulose (obituary notice, ANALYST, July, p. 437).

Kingzett had become little more than a name to present members of the Society, although in his earlier years he had taken an active part in its work. He became a member in 1881, and was elected Vice-President in 1885 (obituary notice, ANALYST, October, p. 649).

Munton joined the Society in 1918, but was not personally known to many of our members.

Pope, who died early in the present year, had been an abstractor on the staff of THE ANALYST since he joined the Society in 1921. From 1931 onwards he had helped the Editor in his editorial work, and early in 1934 the Council recognised the value of his services by appointing him Assistant Editor. His quiet and unassuming manner endeared him to his colleagues on the Publication Committee, and in his death the Society has suffered a great loss (obituary notice, ANALYST, March, 1936).

Self was a comparatively recent member of the Society, which he joined in 1929. By his death we have lost an able chemist who was an authority on questions of pharmacognosy (obituary notice, ANALYST, July, p. 737).

Starey, who joined the Society in 1888 (obituary notice, ANALYST, June, p. 349), and Wilson, who became a member in 1885, were two of our oldest members, and the Council much regrets their loss.

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

"Commercial Ground Almonds and their Adulteration." By G. N. Grinling, F.I.C.

"The Application of Analysis to the Study of Liesegang Rings." By E. B. Hughes, M.Sc., F.I.C.

"The Detection of Japanese Mint Oil in other Peppermint Oils." By D. C. Garratt, B.Sc., Ph.D., F.I.C.

"Measurement of the Small Volumes of Nitrogen obtained by the Micro-Dumas Method." By H. C. Gull, M.Sc.

(i) "A Case of Meta Fuel Poisoning," (ii) "A Crystalline Putrefaction Product of Toxicological Significance." By G. Roche Lynch, O.B.E., M.B., B.S., D.P.H., F.I.C., and R. H. Slater, D.Sc., Ph.D., F.R.S.E., F.I.C.

"A Simple Form of Micro-counter." By T. E. Wallis, B.Sc., F.I.C.

"A Colorimetric Method for the Quantitative Measurement of Rancidity." By Magnus A. Pyke, B.Sc.

"The Determination of Total Alkaloids in Cocoa," and "The Determination of Cocoa Matter in Flour Confectionery." By D. D. Moir, M.Sc., F.I.C., and E. Hinks, B.Sc., F.I.C.

"Colour Measurement of Oils and Other Liquids." By E. R. Bolton, F.I.C., M.I.Chem.E., and K. A. Williams, B.Sc., F.I.C.

"The Chemical Examination of Fur in Relation to Dermatitis." Part VI.: "The Identification of Vegetable and other Dyes." By H. E. Cox, D.Sc., Ph.D., F.I.C.

"Testing for 'Sea-Water Damage.'" By W. M. Seaber, B.Sc., F.I.C.

"The Iodimetric Titration of Tin." By F. L. Okell, F.I.C., and John Lumsden, B.Sc., A.I.C.

"Characteristics of Halibut-liver Oils." By R. T. M. Haines and J. C. Drummond, D.Sc., F.I.C.

"Notes on Mendel and Goldschieder's Method for Determining Lactic Acid in Blood." By R. Milton, B.Sc.

"The Application of Controlled Potential to Microchemical Analysis." By A. J. Lindsey, M.Sc., A.I.C., and H. J. S. Sand, D.Sc., Ph.D., F.I.C.
"The Micro-electrolytic Determination of Bismuth and Lead and their Separation by Graded Potential." By A. J. Lindsey, M.Sc., A.I.C.
"Air-damped Balances." By W. N. Bond, M.A., D.Sc., F.Inst.P.
"Colorimetric Analysis by means of the Photo-electric Cell." By N. Strafford, M.Sc., F.I.C.
"Characteristics of Halibut-liver Oils of the 1935 Season." By Norman Evers, B.Sc., F.I.C., A. G. Jones, B.Sc., A.I.C., and Wilfred Smith, B.Sc., A.I.C.
"The Composition and Examination of Tanganyika Arrow Poison." By W. D. Raymond, B.Sc., A.I.C.
"The Constitution of Tannins, including those of Tea and Coffee." By Peter Maitland, B.Sc., Ph.D.
"A Survey of the Methods of Analysis for Tannins." By C. Ainsworth Mitchell, M.A., D.Sc., F.I.C.
"Experimental Work on Tea Tannin." By M. Nierenstein, D.Sc., Ph.D.
"The Pharmacology of Caffeine, and of Tea and Coffee." By G. Roche Lynch, O.B.E., M.B., B.S., F.I.C.
"The Tannins in Tea." By J. P. Norman, B.Sc., A.I.C., and E. B. Hughes, D.Sc., F.I.C.
"Coffee Extracts." By E. Hinks, B.Sc., F.I.C.
"A Note on 'Tanninless' Teas." By H. H. Bagnall, B.Sc., F.I.C.

The February meeting was a joint meeting with the Food Group of the Society of Chemical Industry, and was novel in that two sessions were held—5 to 6.45 p.m. and 8.15 to 10 p.m. Several papers were read, which dealt chiefly with the alkaloids and tannins in tea and coffee. The meeting was well attended, and the usefulness of these joint meetings was once again amply demonstrated.

At the other meetings papers of diverse interest have been read, and the Council believes that the high quality and interest of these papers reflect credit upon the activities of the Society.

The North of England Section reports that they have held five meetings, at which the following papers were read:

"The Mineral Waters of Harrogate." By A. Woodmansey, M.Sc., A.I.C.
"Colorimetric Determination by Photo-electric Cell." By N. Strafford, M.Sc., F.I.C.
"The Oxalates of Calcium, Barium, Strontium and Magnesium." By J. Haslam, M.Sc., F.I.C.
"Medicines, Ancient and Modern." By U. Aylmer Coates, M.P.S.
"The Estimation of Tartaric Acid as Lead Tartrate." By C. H. Manley, M.A., F.I.C.
"The Detection of Added Water in Milk by means of 'Constants.'" By G. D. Elsdon, B.Sc., F.I.C., and J. R. Stubbs, M.Sc., F.I.C.
"The Calculation of Added Water from the Freezing-point of Watered Milk." By G. D. Elsdon, B.Sc., F.I.C., and J. R. Stubbs, M.Sc., F.I.C.
"The Standardisation of Hortvet Thermometers." By J. R. Stubbs, M.Sc., F.I.C., and G. D. Elsdon, B.Sc., F.I.C.
"The Gravimetric Determination of Sulphur in some Pharmaceutical Preparations." By A. N. Leather, B.Sc., F.I.C.
"The New Poisons List and Rules." By H. Humphreys Jones, F.I.C.
"The Chlorine Content of Feathers." By F. Robertson Dodd, F.I.C.

THE ANALYST.—Once more THE ANALYST has maintained its size, the number of pages being 858 as compared with the previous highest total of 856 in 1934. In view of a probable still further increase in bulk, the Publication Committee

has given careful consideration to the possibility of using a paper thinner than that in use at present, which conforms to the requirements of the Library Association. It was found, however, that such a change was not possible without sacrificing permanency of material and the requisite opacity, and it was decided, therefore, to make no change. The Committee was indebted to Dr. Julius Grant for his specialised assistance in coming to this decision.

The papers published in THE ANALYST have shown the usual wide range of interest. Of the 66 papers that appeared in Volume 60, the largest number (24) related to the analysis of food and drugs. There were 12 papers on organic, 13 on inorganic analysis, 12 dealing with physical methods and apparatus (including the examination of milk by the freezing-point method), 4 on toxicological and forensic subjects, and one on a microchemical method. There were also 41 notes on subjects of analytical interest, 11 legal notes containing reports of cases in which points of special scientific or legal interest were raised, and a large number of extracts from the reports of Public Analysts and of Government Analysts in the Dominions and Colonies. Fifty-six books in all were reviewed, each review having been written by someone with specialised knowledge upon the subject of the particular book, and thus affording guidance to the readers of the journal.

TREASURER'S REPORT.—The Honorary Treasurer reports that the Society has maintained its usual satisfactory financial position, and that its income still slightly exceeds its expenditure.

STANDING COMMITTEE ON THE UNIFORMITY OF ANALYTICAL METHODS.—During this year the Council has reconstituted this committee, which henceforth will be known as the Analytical Methods Committee. The instructions from the Council to the new Committee are as follows:

(1) That the Standing Committee on the Uniformity of Analytical Methods shall in future be called the Analytical Methods Committee.

(2) That the functions of this Committee are:

- (*a*) To receive all enquiries through the Society concerning methods of analysis from recognised organisations.
- (*b*) To put forward such methods of analysis as they deem desirable.
- (*c*) To appoint, and direct the work of sub-committees as may be necessary for this purpose.
- (*d*) To take over the work of the Analytical Investigation Scheme.

The above-recorded changes should be noted in connection with the negotiations with the British Standards Institution mentioned in the Report of the Council for last year. They enable the British Standards Institution and other organisations that may so desire to consult the Society when methods of analysis are required for inclusion in specifications issued by the former body or for such purposes as may be required by the latter.

The following is a brief account of the activities of this Committee during the past year:

Three reports have been published, *viz.*:

"The Determination of Lead in Food Colouring Materials" (ANALYST, 1935, **60**, 541).

"The Determination of Unsaponified Fat in Soaps" (ANALYST, 1935, **60**, 537).

"The Determination of Water, of Total Solids, and of Fat in Dried Milk (ANALYST, 1936, **61**, 105).

With the publication of its fourth report, the Milk Products Sub-Committee considered that its work was completed, and this Sub-Committee has now been dissolved. The Council takes this opportunity of thanking the Committee for its arduous work over a period of eleven years, which it has brought to such a successful conclusion. The Sub-Committee on the Determination of Unsaponifiable Matter in Fats, and of Unsaponified Fat in Soaps, has been re-appointed as a Sub-Committee on Methods of Soap Analysis.

A new Sub-Committee has been appointed, in agreement with the British Standards Institution, to carry on the work of the Freezing-Point Sub-Committee of the Dairy Committee of the Empire Marketing Board, on the Determination of the Freezing-Point of Milk. The Chairman is Mr. A. More (*cf.* ANALYST, **1935**, **60**, 730).

The British Standards Institution has suggested the desirability of standardising the technique of the Reichert tests. A drafting panel of the Committee is now engaged on this work, and it was agreed that the methods reviewed, and the procedure recommended, should embrace the full Reichert–Meissl–Polenske–Kirschner procedure.

The request of the British Standards Institution for a specification for a standard light for Lovibond tintometer readings has been carefully considered, and it was finally reported to the Institution that no "simple light source of quite small size" could be satisfactorily standardised without further physical research, which is outside the scope of the Society. The necessary lines of research were indicated, and it was suggested that this might be undertaken by the National Physical Laboratory.

ANALYTICAL INVESTIGATION SCHEME.—At present there are five problems under investigation, and four papers giving the results of work done under the Scheme were published in THE ANALYST.

NORTH OF ENGLAND SECTION.—Five meetings have been held during the year. The good attendances of previous years have been fully maintained. Ten papers have been read.

In April a well-attended meeting was held in Hull, where, at the invitation of Mr. Arnold R. Tankard, the new Corporation Laboratories were inspected. The Summer Meeting was held in Harrogate in June. A very encouraging feature was the attendance of officials and members of the Parent Society and members of the projected Scottish Section. The meeting was very successful.

Seven new members have joined the Section, bringing the total to 755.

The Secretary of the Section wishes to acknowledge the constant support it has received from the Chairman (Prof. W. H. Roberts) and Committee, and to express its thanks for their co-operation.

SCOTTISH SECTION.—This Section, mindful of the fact that its official existence cannot come into being until the Articles of Association have been passed at the Annual General Meeting, held an informal meeting in Glasgow, on November 13th, under the acting Chairmanship of Mr. R. T. Thomson. The President attended, and, after dinner, the following papers were read:

"Some Properties of Sodium Hexametaphosphate," by R. T. Thomson, F.I.C.

"The Determination of Iodine in Kelp," by J. B. McKean, F.I.C.

On January 22nd the Section held a Joint Meeting with the Food Group of the Society of Chemical Industry, when the following papers were read:

"A System of Judging Flavour in Bread," by A. M. Maiden, B.Sc., Ph.D., A.I.C.

"The Determination of the Gel Strength of Weak Gels," by L. H. Lampitt, D.Sc., F.I.C., and R. W. Money, M.Sc., A.I.C.

"Some Observations on the Appreciation of Flavour in Foodstuffs," by H. C. Moir, B.Sc., A.I.C.

"Milk in Adult Nutrition," by Miss Mary Andross, B.Sc.

"The Composition of Scottish Raspberries," by A. Dargie, B.Sc., A.I.C.

The Acting Honorary Secretary of the Section is Mr. J. B. McKean, of 156, Bath Street, Glasgow, C.2, to whom all applications for membership should be made.

The Council wishes the new Section every success when it formally comes into being, and expresses the hope that the future may see other sections formed, as it is convinced that their social value, in addition to the advantages they offer of scientific knowledge, is inestimable.

ALTERATION OF ARTICLES OF ASSOCIATION.—The Articles of Association which the Council requests members to pass at the Annual General Meeting are concerned with the formation of other sections. At the present time our Articles permit of the existence of only one section, namely, the North of England Section, and, when the new Scottish Section was formed, the Council deemed it desirable to obtain wider powers, so that, in addition to regularising the position of the Scottish Section, it might be able to authorise the formation of other sections in the future without the expense of a further alteration of the Articles. Consequently, the new wording of the Articles of Association includes, for the first time, "area sections."

ANALYTICAL CHEMISTRY RESEARCH FUND.—One grant has been made to a member during the year. In connection with this Fund, the Council would remind members that there is available annually a small sum to assist members in the pursuit of their researches if working under the Analytical Investigation Scheme.

CONGRESSES.

Quinzième Congrès de Chimie Industrielle at Brussels.—The Society was represented at this Congress by the President.

International Commission for the Examination of Oils and Fats.—The Society's representative, Mr. Bolton, was President of the Congress held in London under the auspices of the British Standards Institution.

In conclusion, the Council desires to thank those members who have so kindly undertaken to assist it by serving on various committees within and without the Society, and appreciates the spirit which prompts them to devote their valuable time to furthering the interests of the Society.

JOHN EVANS, *President*
G. ROCHE LYNCH, *Hon. Secretary*

Address of the Retiring President

(MR. JOHN EVANS, M.Sc., F.I.C.)

(*Delivered at the Annual General Meeting, held on March* 6, 1936)

LADIES AND GENTLEMEN,

The Council of the Society of Public Analysts and Other Analytical Chemists, in its wisdom, has decreed that in future the President shall give only one Presidential Address, and that at the expiration of his second year of office. In place of the Presidential Address formerly given at the end of the first year, it has been decided to have a lecture by someone of outstanding eminence. Last year we had the privilege of hearing a highly interesting lecture by Dr. Bernard Dyer on "Reminiscences of Fifty Years of the Society of Public Analysts."

It is usual for the President to give a short summary of the events of his period of office, but, as all these events are given in the Annual Report of the Council, I will not repeat them, but will content myself with pointing out some of the more important of our activities.

I can venture to say, with confidence, that our Society continues to make progress. Our ordinary meetings have been well attended, and the subjects discussed have been very varied and important. From the Council's Report you will see that the membership of the Society is increasing—a sure sign that the Society is in a healthy condition. Not only is the Society healthy, it is also fertile. The Scottish Section came into being last year, under the Chairmanship of Mr. R. T. Thomson, and we hope it will prove as sturdy and vigorous a child as its older sister, the North of England Section. Your President, accompanied by the Hon. Secretary, the Hon. Treasurer and the Chairman of the North of England Section, was present at the Inaugural Meeting of the Scottish Section at Glasgow.

We are pleased to be able to congratulate Mr. A. R. Tankard upon his election to the Chairmanship of the North of England Section for the coming year, and we feel sure that under his guidance it will continue to thrive.

Among the deaths recorded in the Council's Report is the loss of one of our Past-Presidents, Mr. L. Archbutt. Mr. Archbutt started his career, first as an articled pupil, then as an assistant in the laboratory of the late Alfred H. Allen, the laboratory of which I have the honour of being the present Chief. Allen would have been pleased if he had been able to look into the future to see two of his pupils following in his footsteps as Presidents of this Society. We have suffered a great loss in the death, early this year, of Mr. T. H. Pope. He joined the Society in 1921 and became an Abstractor for the Journal, and ultimately its Assistant-Editor. To readers of THE ANALYST his ability is well known, and all the members of the Publication Committee regret the loss of a valued colleague. The comprehensive *Bibliography on Heavy Metals in Biological Material*, which he compiled at the request of the Publication Committee, will remain as a memorial to him.

The November Meeting in 1934 continued the series of joint meetings with the Food Group of the Society of Chemical Industry; on that occasion a number of

papers were read on the identification and qualities of common edible fish and some kindred subjects. In February of this year, at another joint meeting with the Food Group, papers were read on the alkaloids and tannins in tea and coffee. Our Society is linked with the Food Group by our Treasurer, Dr. Hughes, who sits on the Committee of the Food Group as a co-opted member. I am glad to say that our relations with the Group are cordial and our joint meetings are mutually beneficial.

Sub-Committees are being formed to work in conjunction with the British Standards Institution on matters concerned with analytical technique. Already a Sub-Committee has been appointed, under the Chairmanship of Mr. A. More, to work in agreement with the British Standards Institution on the Determination of the Freezing-point of Milk. All who are interested in this question are familiar with the pioneer work of Dr. Monier-Williams, and the subsequent work of Messrs. Elsdon and Stubbs, of the North of England Section. This work in itself would sufficiently justify the existence of the North of England Section. The British Standards Institution has suggested the desirability of standardising the technique of the Reichert–Wollny tests, and a drafting panel of the Committee is now engaged on this work.

The Report of the Departmental Committee on the Composition and Description of Food was presented to Parliament by the Minister of Health in April, 1934. Whatever may be our individual views regarding this Report, it is highly satisfactory to note that the main contention of the Society, namely, the necessity for extended statutory powers for establishing definitions and standards for articles of food, has been conceded.

It has been the custom in the past for the President, in the course of his Presidential Address, to select a topic of wide and modern interest for brief review. In considering the advances which have been made in the chemistry of food during the past few years, it appeared to me that the minute amounts of various elements which are being found in biological material are assuming great importance, and I propose to discuss very briefly the significance of these traces.

THE SIGNIFICANCE OF TRACES

The attention paid in recent years to the occurrence and significance of minute amounts of chemical substances in the living organism and in its food is the result, partly of advances in biochemistry and the chemistry of nutrition, and partly of refinements in analytical methods, whereby these minute amounts, previously undetectable, are now not only detectable, but determinable. Detection of these traces is often a matter of comparative ease, but the elucidation of the part they play in metabolism and nutrition, whether animal or vegetable, is a problem of much greater difficulty. A metallic element, for example, hitherto unsuspected, may be found to occur constantly in living matter not subject to external contamination. From this initial step the biochemist proceeds with the more difficult task of determining in what form it occurs in the organism; is it in the sap of the plant, in the blood of the animal; is it essential; is it assimilated in the inorganic or in the organic form; does an animal deprived of it cease to thrive; does a plant germinating in soil not containing it cease to grow? In some

instances more is known of the significance of such factors than of their nature, an obvious example being the vitamins. Up to a few years ago, although the dire effects of the absence of these bodies from food were well known, so little was known of their nature that they were all denoted arbitrarily by letters of the alphabet. The list of elements, not only occurring in the living organism, but essential to its well-being, is rapidly increasing in length, and I do not think that we shall be very much surprised if some day we arrive at the conclusion that all the elements are essential to some form of life or another. We are the children of Mother Earth—very infantile children, entirely dependent upon her for our whole material existence. We are never weaned. Deriving the whole of our sustenance from her, as we do, is it not likely that we draw upon all her resources—some in bulk, others in delicately adjusted amounts which must not be exceeded?

The state of science which I have outlined briefly has raised problems that affect the analyst, and, if my remarks appear at first sight to be addressed only to analysts, or even to a particular kind of analyst, let us remember that analytical chemistry is a very wide subject indeed. It has long been the Cinderella of the branches of practical chemistry—a drudge, denied the comfort of academic chairs, neglected and sometimes scorned by her more romantic sisters, whom she has silently served. I venture to think that this is changing with the progress of biochemistry, which, like the fairy godmother of the legend, and much in the same magical fashion with the aid of rats, mice and pumpkins, has produced the structure which will bear this Cinderella to a sphere of wider recognition and usefulness. As science progresses its devotees specialise more and more, but at the same time they become more and more dependent upon some knowledge of the branches in which they are not experts. Each newly-discovered fact causes a disturbance in its own particular sphere with eddies in other spheres. Let the biochemist discover that lack of small amounts of an element in certain foods causes a deficiency disease, and the food analyst immediately asks whether his methods will detect this essential trace, whilst the food-manufacturing chemist enquires whether his manufacturing processes remove it, and, if they do, how can it be put back. We have heard much recently of the occurrence of fluorides in certain water supplies and their effect upon the enamel of the teeth, particularly the deciduous teeth of children. Fluorides occur also in coal, and their presence is suspected to be due to infiltered water.[1] Their occurrence in coal leads to corrosion of gas-works scrubbers and trouble in glass manufacture.[1] They can be removed from water by filtration through active carbon, and recent work upon the adsorptive properties of the synthetic resins indicates the possibility of the use of the amino-resins for their removal from drinking water.[2] Here we have a link binding the physiological chemist, the water analyst, the gas-works chemist, the glass chemist and the synthetic-resin chemist.

To return to my original thesis, what problems raised by recent work on the significance of traces confront the analyst? There is, of course, the obvious problem of devising methods of detecting and determining these traces, but I do not think that this is as difficult as the more serious problem of distinguishing between the trace occurring naturally and the trace due to contamination. We are very apt, in devising analytical processes, to try to obtain the element we are seeking in the

form of an inorganic compound, partly because inorganic technique is easier and partly because inorganic reactions can be induced to proceed to completion—we are less troubled by equilibrium reactions, partial yields and dependence upon external conditions. But are we sufficiently careful of our language when we say that sultanas, for example, contain boric acid, when what we really mean is that the ash of sultanas contains borates? And, as our method for its detection depends upon its conversion into an inorganic compound, we cannot say with certainty whether the trace of boron we find in a sample of unknown origin is there as a natural constituent or as a contaminant. It is highly probable that boron occurs in the sultana in organic combination as a compound with no preservative action. It may even be essential to the life of the plant. It has been shown that the boron-content of apples affected by the disease known as "internal cork" is about one-third of that of healthy fruit, and that the severity of the disease is inversely proportional to the amount of boron in the fruit.[3] The sugar-cane is also peculiarly susceptible to boron deficiency.[4]

Traces of Metals in Foods.—During the last few years much work has been done on the significance of traces of metals in foods. Copper, especially, has been the subject of much research, and it appears to be an essential element in animal nutrition. If iron is added to the milk diet of anaemic rats depleted of their iron reserve in the liver and spleen, there is no increase in haemoglobin formation, although the iron-content of the liver and spleen is increased. If, however, the iron in the diet is replaced by copper, the iron stored in the liver becomes available for haemoglobin formation. Several liver preparations, the hydrogen sulphide fractions of their ash, and copper in the form of a solution of copper sulphate—all at the same levels of copper intake—have been shown to serve equally well for the curing of the nutritional anaemia produced by a basal diet consisting of whole milk and iron. It appears to be well established that the deficiency in this diet is inorganic in nature, and that this inorganic deficiency is copper only.[5,6] It is now suspected that the condition known as "milk anaemia" in young children is due to copper deficiency. Unfortunately, attempts to increase the copper-content of milk foods and other fatty foods by the addition of copper compounds are attended by some difficulties. Copper compounds have the property of accelerating the auto-oxidation of fats, and their addition to foods such as milk and ice-cream causes a tallowy flavour. It has been shown that the "off-flavour" of ice-cream is often due to traces of copper of the order of from 1 to 2 parts per million derived from the manufacturing plant.[7] Frequently, wrapping material made from wood pulp, the average copper-content of which is about 50 parts per million, and vegetable parchment which may contain 20 parts per million, introduces sufficient copper to affect the flavour of fatty-food material.[8] There is some evidence that traces of zinc have a protective action and retard oxidative changes in fats.[9] Very small amounts of metallic compounds in coffee can be tasted, and expert tasters claim to be able to detect a metallic flavour in coffee which has been prepared in stainless steel vessels.[9] Small amounts of aluminium affect the colour of tea, presumably by forming lakes with the tannins present.[9] Instances could be multiplied, but what I have said concerning the presence of copper in food indicates the difficulties confronting the food

manufacturer and the food analyst. If the public analyst were too insistent upon the removal of copper contamination from food the manufacturer might reply by removing from his products all trace of copper whether due to contamination or natural occurrence and the biochemist would lay his finger upon copper deficiency as the cause of a nutritional disease.

ARSENIC IN LIVING ORGANISMS.—Many of you will remember the arsenic-in-beer poisoning episode in 1901. In connection with investigations arising out of that episode, Chaston Chapman made the observation that the arsenic present in brewers' yeast did not exist in the simple form in which it occurred in the wort from which it was derived. He was led to the conclusion that the cell built up the element into a complex organic compound, either to provide a substance favourable to its development or to reduce its toxicity. In 1922 A. J. Jones[10] found up to 125 parts per million of arsenic in certain marine algae, and in 1926 Chaston Chapman reported the occurrence of up to 20 parts per million of arsenic in Irish moss. In 1925 a Joint Medical and Chemical Commission, set up by the Swedish Government to investigate arsenical poisoning, found that the urine of normal persons frequently contained arsenic in such amount that, were the contrary not known, chronic arsenical poisoning would be suspected. The well-known extensive investigation by Chaston Chapman[11] of the arsenic-content of shell fish and crustaceans produced some surprising results, such, for example, as the occurrence of 174 parts per million of arsenious oxide in prawns, this being the maximum figure obtained in the numerous kinds of fish examined. British oysters were found to contain up to 10 parts per million, mussels from 36 to 119 parts per million, whilst Portuguese oysters contained up to 70 parts per million. Cox,[12] in 1925, had made the observation that among ordinary edible fish, as distinct from shell-fish and crustaceans, plaice contained the largest amount of arsenic—an interesting observation, for the reason that, of the fish examined, the plaice is the one feeding largely on bi-valves. Many interesting points arise from this extraordinary occurrence of arsenic. Does the shell-fish or crustacean use the arsenic in its metabolism, or does it merely fail to excrete it as completely as the ordinary fish? Is the idiosyncrasy which some people exhibit towards shell fish due to their ability to break down the complex arsenical compound into simpler, more toxic, compounds during the digestion? So far as I know, these questions have not been completely answered, but from Chaston Chapman's investigation, one point emerges concerning which there is reasonable certainty. The arsenic occurs in these marine creatures as an organic compound, or mixture of compounds, soluble in water, alcohol and acetone, sufficiently stable to resist the action of hot dilute hydrochloric acid or 5 per cent. sodium hydroxide solution, obviously possessed of very slight toxic properties, and not reducible by hydrogen to arsenic trihydride. Further, when the flesh is artificially digested with trypsin and peptase, no breaking-down of the arsenical complex can be noted. It is undoubtedly in solution, but the digest does not give the Marsh-Berzelius test. This investigation indicates the importance of determining in what form the traces of so-called "noxious" elements occur, and the analyst, particularly the analyst concerned with the manufacture or inspection of food, is anxious to find means of distinguishing between the natural trace and the trace due to contamination.

ARSENIC AND MOULD FUNGI.—Before leaving the subject of arsenic I may mention some recent work which throws light upon what used to be known as the arsenical wall-paper danger. During the first half of the nineteenth century several cases of poisoning occurred through inhalation of air from rooms papered with wall-paper containing arsenical pigments. The earliest explanation was that particles of pigment floating in the air were responsible for the poisoning, but it was gradually realised that the toxic compound was gaseous, and that the presence of moulds in the paper or in the adhesive was essential to its formation. As early as 1891 it was discovered that some moulds were intensely active in producing this compound, especially *Penicillium brevicaule.* In 1931 Challenger, Higginbottom and Ellis[13] identified the gas as trimethylarsine, and pointed out that it is an interesting instance of methylation performed by a living organism. Although the danger from arsenical wallpapers has been removed by their disuse, a similar danger seems to exist in the use of concrete containing coke-breeze, which may, under abnormal conditions of damp and mould, produce toxic compounds.

The formation of trimethylarsine has recently been suggested as a qualitative test for minute amounts of arsenic. It is claimed that 1 part per million can be detected in a sample of 1 g. or less and that, for qualitative purposes, the test is more delicate than the Marsh test. The mould recommended is *Scopulariopsis brevicaulis* (Sacc.) Bainier. A suitable nutrient medium is sterilised, inoculated with the mould, and incubated until an obviously active growth develops. About a gram of the sample is then distributed over the surface of the growth, and incubation at room temperature is continued. In the presence of about a millionth of a gram of arsenic the garlic-like odour of trimethylarsine becomes apparent in from 2 to 5 hours. Appreciable amounts of antimony do not interfere with the test, but it is inhibited by inorganic mercury compounds. Selenium and tellurium give odours similar to that given by arsenic.[14]

DETECTION OF TRACES OF ELEMENTS.—It would be beyond the scope of my subject to discuss the methods now available for the detection and determination of minute traces of elements: I can only classify them as colorimetric, nephelometric, micro-chemical and spectrographic. It is probable that the spectroscope will ultimately be used to a much greater extent for this purpose than it has been in the past. Neither does my subject include a discussion of the methods available for the removal of traces of unwanted elements, but I do not think it would be out of place to mention the recent work of Adams and Holmes on the absorptive properties of synthetic resins.[15] By the judicious choice of one or more resins it is possible to effect the removal from solution of a number of anions and cations. Most of the objectionable cations, such as iron, manganese, lead, copper and zinc, can be removed from drinking water by their use, as well as anions such as fluorides, silicates, sulphates, chlorides, etc. By the consecutive use of phenolic and amino-resins it is possible to effect complete removal of dissolved salts from solution, thus obtaining a filtrate equivalent to distilled water. This has been proved for tap-water, a quebracho-tannin resin (which removed the cations) being used first, and then a *m*-phenylenediamine resin which completes the purification. The total solids are reduced from 33 parts to about 1 part per 100,000.

In this address I have discussed a few examples of the significance of traces of elements, as it is obviously impossible for me to summarise the whole of the work which has been done on the subject in recent years. We have seen that boron, the addition of whose compounds to food is prohibited by law, occurs naturally, and is even essential to certain forms of life; copper, which as a contaminant we regard as a mildly noxious element, not only occurs naturally in food, but its absence causes a deficiency disease; arsenic, which is a violent poison, occurs in shell-fish and crustaceans as a harmless organic compound. I said at the commencement that man, like all other forms of life, is the unweaned child of Mother Earth. She has nourished him in the past and will undoubtedly strive to do so in the future. But, with the progress of science, comes the uneasy feeling that man's nutritional well-being is an affair of very delicate balance. If certain substances occur in his food in small, but excessive amounts, he suffers; if they occur in small, but insufficient amount, again he suffers. In the broad sense he is well advised to take from the Universal Mother all that she provides and often in the state in which she provides it; but as a dutiful son he must return to her what she requires. Her need of nitrogen, phosphorus, potassium and calcium has long been known, but it is only recently that the significance of small amounts of other elements, such as iron, magnesium, manganese, boron and iodine, has been realised. To maintain the fertility of the soil, all these are necessary, and the bulk of our food is dependent upon the fertility of our pasture-lands. The biochemist is gradually explaining what part each element plays in the maintaining of vegetable, animal and human life. It is the duty of the food chemist to see that nothing that nature provides for our nourishment is withheld, even the traces whose significance I hope I have succeeded in demonstrating.

In conclusion, it is my pleasant duty to thank all members of the Council, and particularly the Hon. Treasurer, Dr. E. B. Hughes, and the Hon. Secretary, Dr. G. Roche Lynch, and the Secretary and Editor of THE ANALYST, Dr. C. A. Mitchell, for the generous help they have given me during my period of office. Living, as I do, in the North of England, I fear that my Presidency has occasioned them rather more labour than has that of some of my predecessors.

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Investigations into the Analytical Chemistry of Tantalum, Niobium, and their Mineral Associates

XXX. Observations on Beryllium

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(Work done under the Society's Analytical Investigation Scheme)

According to the published analyses, beryllia is an infrequent and always minor constituent of earth-acid minerals. It may have escaped detection by some of the earlier mineralogists, owing to the lack of specific and sensitive tests. The object of this Section is to ascertain the fate of beryllium in the more important analytical operations which we advocate for the separation of the various earths and the analysis of the minerals containing them. The paper also gives us an opportunity to discuss some recently-published methods, and to present an alternative procedure for the separation of beryllium from uranium.

Our thanks are due to Dr. H. Fischer, of Messrs. Siemens & Halske, Berlin, to whose courtesy we owe the preparation of pure beryllia used in this investigation.

A. Determination of Beryllium.—Before discussing beryllia as a constituent of earth-acid minerals, we wish to submit some observations on the question of its determination. In practice this is always done gravimetrically, the oxide being obtained by precipitation with ammonia and ignition of the precipitate, exactly as with alumina. The determination is subject to positive errors, due to co-precipitation of other elements, if present, and occlusion of alkali salts by the precipitate. Hillebrand and Lundell[1] prescribe double or triple precipitation in presence of non-volatile salts such as sodium chloride (*i.e.* in silicate analysis), and urge the need for a final purity test on the weighed precipitate. Again, the beryllium should be precipitated from chloride or nitrate, not sulphate, solution, as in the latter case the precipitate would carry down substantial amounts of sulphur trioxide, from which it cannot be freed by ignition and hardly so by re-precipitation, since the use of an excess of ammonia causes low beryllium results.

From the above considerations it follows that the solution obtained after bisulphate fusion is unsuitable for the direct gravimetric determination of beryllium; but, as bisulphate fusions are the rule in earth-acid work, the accurate determination of beryllium in presence of alkali sulphate became our first consideration. We finally adopted fusion of the ignited ammonia precipitate with sodium carbonate and lixiviation of the product with water, which furnished a residue of purified beryllia in a weighable form. The procedure removes, not only adsorbed alkali and sulphur trioxide, but also elements forming soluble sodium compounds in general, and alumina and minor quantities of silica in particular.

As the practical value of sodium carbonate fusion in beryllium analysis has not yet been generally recognised (Hillebrand and Lundell,[2] *e.g.* say that they have no personal experience with the method), a brief discussion of the subject may not be out of place. In 1912, Wunder and Wenger[3] published a new method for the separation of beryllium from aluminium, based on the fact that molten sodium carbonate reacts with alumina to form soluble aluminate, whilst beryllia remains insoluble. The process was re-investigated by Britton,[4] who found it "capable of yielding satisfactory separations by the use of a single fusion when sufficiently small amounts of the two earths are present" (*i.e.* less than 0·15 g.). It was subsequently incorporated by Dixon[5] in a method for the determination of beryllium in rocks, the ignited ammonia precipitate being fused with sodium carbonate and the melt extracted with water for the removal of alumina, together with chromium, vanadium, phosphorus, and residual silica. Hills[6] considers fusion of the ammonia precipitate with sodium carbonate "a very satisfactory technical scheme" in the analysis of beryllium minerals.

We have found two unfavourable references to Wunder and Wenger's method in the literature, neither of them supported by test analyses on synthetic oxide mixtures. (1) Moser and Niessner,[7] discussing the methods proposed for the separation of beryllium from aluminium, comment as follows: "The failure of Wunder and Wenger's method may be ascribed to a similar error." Of what nature this error may be is not made clear by the context, for the sentence preceding the above deals with Gibbs's method (precipitation of sodium fluoro-aluminate, which is not sufficiently insoluble for quantitative purposes), while the text following the translated sentence reads thus: "A re-investigation of this (Wunder and Wenger's) method by Stock, Priess, and Praetorius[8] gave high aluminium values." (2) Reference to the last-named authors' paper proved that it is concerned with the preparation of beryllium and contains an analytical section on the elaboration of a routine method for the determination of subordinate amounts of alumina in beryllia. The authors adopt a colorimetric process utilising alizarine, the sodium-carbonate fusion being dismissed in a single sentence with the statement that it "proved hardly worth recommending." A footnote reproduces a determination of aluminium in one sample by different methods, the lower value given by the alizarine method being considered the most reliable. That, apparently, is Moser and Niessner's authority for their statement that the fusion method gave high aluminium values.

We may sum up by saying that there is no serious experimental evidence against the method, but a good deal in its favour, whilst our own experience, both in the analysis of beryl and in the present investigation, confirms its reliability.

B. Recovery from Tartrate Solution.—Like every other earth, beryllia can be precipitated from tartrate solution by means of tannin. The precipitation must be carried out in ammoniacal solution, as the precipitate is readily soluble in dilute acid. In this respect, beryllium behaves like the other members (*i.e.* rare earths and manganese) of what we have previously designated as Tannin Group C.[9]

In Exps. 1 to 4, the bisulphate melt of the oxide was dissolved in 100 ml. of 4 per cent. tartaric acid solution; the liquid was treated with 30 ml. of strong

hydrochloric acid and 5 g. of ammonium acetate, made ammoniacal, and diluted to 250 ml. The boiling solution, containing 2 ml. of free ammonia, was precipitated with a fresh solution of 0·5 g. tannin (*i.e.* at least 30 times the weight of the beryllia). The liquid was filtered after cooling; the precipitate was washed with slightly ammoniacal 2 per cent. ammonium chloride solution containing a little tannin, and ignited in a platinum crucible.

The crude oxide was then fused with 2·5 g. of sodium carbonate for an hour, and the melt was extracted with 100 ml. of hot water in a porcelain basin. The insoluble residue was collected on a close-grained filter (Munktel No. 00) containing a pad of filter pulp, washed with hot water, ignited strongly, and weighed.

Exp.	BeO taken g.	†M_2O_5 added g.	BeO found g.	Error g.
1*	0·0110	None	0·0114	+0·0004
2*	0·0080	,,	0·0091	+0·0011
3*	0·0133	,,	0·0134	+0·0001
4*	0·0141	,,	0·0145	+0·0004
Ta 5	0·0402	0·2000	None	—
Nb 6	0·0501	0·2042	in HP^1§	—

* Quantities taken not known to operator.
† M_2O_5 represents $(Ta,Nb)_2O_5$. § HP^1 represents the first hydrolysis precipitate.

The tests prove that a quantitative recovery of beryllia from tartrate solution can be effected. We do not advocate this procedure for the determination of beryllium in preference to other methods; it will, however, prove useful in the recovery of mixed earths from tartrate solutions.

Generally speaking, tannin precipitations are more satisfactory in neutral, or faintly acid, than in ammoniacal solutions. Whilst the former yield light flocculent precipitates floating in a cloudless liquid, the latter (through co-precipitation of tannin) are apt to produce curdy or clotted precipitates and cloudy solutions which gradually darken by oxidation, with separation of a little black organic matter. Contamination of the tannin precipitates with silica from the glass vessels is more pronounced in ammoniacal solutions; and for beryllium precipitations, we find it necessary to let the solution cool before collecting the precipitate, otherwise the filtrate may become cloudy and deposit a slight precipitate containing a minute quantity of beryllia.

In an earlier Section[10] we have described a procedure for the precipitation of certain earths from neutralised tartrate and acetate solution, which we intended to apply also for their separation from beryllia, if present; but subsequent experience has caused us to question the suitability of tartrate solutions for tannin separations. Whether better separations can be achieved in tartrate solutions free from alkali acetate is a point worth further investigation; with oxalate solutions, we have found that it is not possible to carry out the important tannin separations described in Sections 23 and 24[11,12] in the presence of acetate. The adjustment of the acidity of the tartrate solution is difficult: this is exemplified in the attempted separation of the earth acids from the rare earths.[13] Even simple acetate solutions present difficulties in this respect, as in Moser and Niessner's proposed method[14] for the separation of aluminium from beryllium; Mitchell and Ward[15] state that they "did not obtain satisfactory separations by the method of Moser and Niessner, possibly owing to insufficiently definite specification of the acidity conditions in

their description." The method of Moser and Singer[16] for the separation of beryllium from iron by tannin in acetate solution has been criticised in the same way as the preceding, Dixon[5] stating that it is liable to lead to co-precipitation of beryllium at the reduced acidity required for the complete precipitation of the iron.

As a result of our experience in the analysis of beryl, we share the view of Mitchell and Ward, though we consider tannin a valuable reagent for the separation of small quantities of alumina from beryllia.

C. Behaviour in Tartaric Hydrolysis.—We have reported in an earlier Section[17] that beryllia could not be detected with certainty in the earth-acid precipitate, *HP*, obtained by boiling the tartrate solution with excess of mineral acid. Using more refined methods, we have once more tested such precipitates for small quantities of occluded beryllia, with entirely negative results. The technique was similar to that applied in the rare-earth investigation[13]: the carefully washed HP^1 was ignited and again submitted to the same sequence of operations. This furnished a second precipitate, HP^2 (which was rejected), and its filtrate, containing any beryllia occluded in HP^1, in addition to the few mg. of earth acid that normally escape precipitation. The earths were recovered by tannin precipitation from the ammoniacal tartrate solution, as under B above; the tannin precipitate was ignited and treated by the pyrosulphate tannin method,[18] the filtrate from the insoluble earth-acid complex being precipitated with ammonia. The small ferruginous precipitates thus obtained (Exps. 5 and 6) weighed 0·0008 and 0·0012 g., respectively. They were fused with a minute quantity of bisulphate, and the solution of the melt was submitted to Fischer's quinalizarine test[19]: no blue colour was observed. We conclude that tartaric hydrolysis gives an earth-acid precipitate free from beryllia.

D. Separation from Group A (Tantalum, Niobium and Titanium).—In view of the ready solubility of its tannin complex in dilute acids, the separation of beryllium from the members of Group A by the general procedure described in Section 23[11] seemed a foregone conclusion. Nevertheless, we carried out a number of test separations (Exps. 7 to 15) by that procedure, recovering the beryllia from the filtrates, as explained below.

The filtrate from the tannin precipitate of Group A (400 to 600 ml.) is boiled, treated with more tannin (30 times the weight of BeO) in freshly-made solution, and stirred, while ammonia (1 : 3) is added, drop by drop, until a decided excess is present. The liquid is left to cool to room temperature, filtered by suction through a 12·5-cm. Whatman filter (No. 40), and the precipitate is returned to the beaker as usual[20] with slightly ammoniacal ammonium chloride solution and churned up with filter-pulp. The precipitate is again collected, ignited wet in a platinum crucible, and fused with sodium carbonate, etc., as before (B). The residue from the lixiviation is collected, washed with water, ignited strongly, and weighed as BeO.

The errors observed in Exp. 10 made it appear probable that about 1 mg. of Group A oxides had not been precipitated with the bulk, and hence become included in the beryllia fraction. We therefore re-treated the latter in oxalate solution by careful addition of dilute ammonia in presence of tannin, and obtained

a slight precipitate in the still faintly-acid solution. After flocculation on a steam-bath this precipitate showed a red colour characteristic of titanium and niobium. Hence a re-treatment of the beryllia fraction by the same procedure would have reduced the errors reproduced below.

	Grams taken		Grams found		Error	
Exp.	BeO	Group A Oxide	BeO	Group A	BeO	Group A
7	0·0455	TiO_2 0·0183	0·0449	0·0186	−0·0006	+0·0003
8*	0·0538	Ta_2O_5 0·1132	0·0534	0·1132	−0·0004	0·0000
9*	0·0330	Nb_2O_5 0·1335	0·0333	0·1328	+0·0003	−0·0007
10*	0·0117	†M_2O_5 0·1132 TiO_2 0·0326	0·0128		+0·0011	
		0·1458		0·1444		−0·0014
11*	0·0120	M_2O_5 0·0309 TiO_2 0·1019	0·0125		+0·0005	
		0·1328		0·1329		+0·0001
12	0·0306	TiO_2 0·0250		0·0258		+0·0008
13	0·0455	,, 0·0183		0·0186		+0·0003
14*	0·0312	,, 0·0294		0·0297		+0·0003
15*	0·0466	,, 0·0214		0·0210		−0·0004

* Quantities taken not known to operator.
† M_2O_5 represents $(Ta,Nb)_2O_5$.

The separation of beryllia from Group A in general and titania in particular (as in Exps. 12 to 15) can be accomplished in one operation at the correct degree of acidity. Dixon, in the paper already referred to,[5] proposed the use of *p*-chloroaniline for the separation of titanium from beryllium. We do not wish to criticise Dixon's useful work when we say that we consider tannin more advantageous than *p*-chloroaniline because (1) titanium can be separated in one operation, (2) the titanium precipitate is strongly coloured, (3) the tannin separation can be carried out in sulphate solution, and (4) tannin is the more easily procurable and more generally applicable reagent, which has become indispensable in mineral analysis.

E. Separation from other Earths (Groups B and C).—As stated under B above, whilst a clean-cut separation of Group A from Group B can be effected by tannin in oxalate solution, we agree with other investigators in doubting the reliability of the tannin separation of Group B from beryllium (or other members of Group C) in acetate solution, a procedure included in Moser and List's scheme[21] for the quantitative separation of beryllium from other metals. No precise directions for such a separation are laid down in their paper, and no test separations from more than one member of Group B have been published (by Moser and Singer[16]). One element overlooked by Moser and List is uranium, which accompanies beryllium in a number of reactions, and is precipitated by tannin from neutralised acetate solution,[22] when co-precipitation of beryllia is almost certain to occur. The proposed tannin method might be made to yield serviceable results by a process of fractional precipitation or re-precipitation, but there is

no real need for such a process in the complete analysis of earth-acid minerals, in which all the constituents must be separated from each other. One of these separations will be considered here, *viz.* that of beryllium from uranium.

F. SEPARATION FROM URANIUM.—As both elements furnish soluble double alkali carbonates and bicarbonates, instances may occur when the uranic oxide recovered from carbonate solutions will have to be tested for beryllia. There seems to be no stated objection against separation by sodium hydroxide (which gives insoluble uranate and soluble beryllate), apart, perhaps, from the general tendency to avoid alkaline reagents.

Two other separation methods have been described. Wunder and Wenger[23] treat the chloride or nitrate solution with hydrogen peroxide, whereby the uranium is precipitated as a yellow higher oxide; double precipitation is prescribed unless the quantity of beryllia is small. The filtrate is boiled down, and the beryllia precipitated with ammonia. Brinton and Ellestad[24] precipitate beryllia from chloride solution with ammonium carbonate and hydroxylamine hydrochloride; the precipitate is free from uranium, but the precipitation is not quantitative; the filtrate must be re-treated for a minor beryllium fraction. This is hardly an attractive feature of the method if applied to the search for a minute quantity. Wunder and Wenger's method appears to give a better separation, but is not applicable in sulphate solution; from the purely practical point of view, it has the disadvantage that C.P. hydrogen peroxide is an unstable reagent.

We are able to suggest an alternative separation method, *viz.* by means of potassium ferrocyanide. It gives a clean-cut separation in one operation in weakly-acid sulphate solution, in which respect the procedure is superior to others, while the salt is a cheap, stable, and common reagent. Against this has to be set the disadvantage that iron is introduced into the solution, which necessitates another, though easier, separation. The difficulty of filtering the slimy uranyl ferrocyanide is easily and completely overcome by the use of filter pulp. The only application of ferrocyanide in gravimetric analysis known to us is Fresenius and Hintz's process[25] for the separation of uranium from phosphoric acid, in which it is proposed to saturate the solution with sodium chloride to facilitate filtration.

We fuse the mixed oxides with bisulphate, dissolve the product in water, and treat the cold solution (100 ml. per 0·1 g. U_3O_8) with 2 g. of ammonium chloride, a cream of filter pulp, and a solution of 0·3 g. of potassium ferrocyanide, stirring well to blend the precipitate with the fibre. The liquid is allowed to stand for about an hour, when the precipitate deposits completely, provided that enough pulp has been added. The liquid is filtered through close-grained paper; the matted pulp containing the red precipitate is returned to the beaker with 2 per cent. ammonium chloride solution, stirred up and returned to the filter, and the washing is completed. The filtrate is treated with a little filter pulp, made slightly ammoniacal, and heated to boiling. We find it advisable, for the quantitative recovery of the beryllia, to add a little tannin. The precipitate is collected, washed with ammonium chloride solution, and ignited in a platinum crucible. It is then fused with sodium carbonate, etc., as described under B. The weighed beryllia may contain a fraction of a mg. of ferric oxide, which must be determined and subtracted: the weighed oxide is fused with a little bisulphate, the melt dissolved

in water, and one-half of the solution is tested colorimetrically for iron. In the other half, the beryllium may be identified by Fischer's reagent.[19]

RESULTS OF TEST SEPARATIONS.—In the four experiments reproduced below, the beryllia was determined as described. In addition, we determined the uranium also, by returning the filter with the ferrocyanide precipitate to the beaker, destroying the paper with nitric and sulphuric acids, and precipitating the iron as sulphide in tartrate solution. The uranium was precipitated in the filtrate by our tannin method,[26] the weight of the uranic oxide being corrected for silica.

In Exps. 16 and 17, in which no tannin had been added in the beryllium precipitation, we accounted for the negative errors by re-treating the ammoniacal filtrates with tannin and a few drops of ammonia; the small precipitates thus obtained gave strong reactions with Fischer's reagent.

	Grams taken		Grams found		Error	
Exp.	U_3O_8	BeO	U_3O_8	BeO	U_3O_8	BeO
16*	0·1330	0·0237	0·1325	0·0226	−0·0005	−0·0011
17*	0·0406	0·0443	0·0409	0·0424	+0·0003	−0·0019
18*	0·1162	0·0206	0·1166	0·0208	+0·0004	+0·0002
19*	0·0652	0·0512	0·0662	0·0506	+0·0010	−0·0006

* Quantities taken not known to operator.

We believe that the principle of ferrocyanide precipitation can be extended to certain other separations, *e.g.* that of uranium from aluminium; so far we have not investigated the subject quantitatively.

SUMMARY.—For the gravimetric determination of beryllia obtained by ammonia or tannin precipitation in presence of alkali sulphate, we fuse the ignited oxide with sodium carbonate and extract the fused mass with water, which leaves pure beryllia as an insoluble residue. The fusion process was first proposed by Wunder and Wenger for the separation of beryllia from alumina, a method which we, in common with several other investigators, regard as reliable.

Beryllia is quantitatively precipitated by tannin from ammoniacal tartrate solution. The earth-acid precipitate obtained by boiling the tartrate solution with excess of mineral acid (tartaric hydrolysis) does not occlude beryllia, if present. Tannin precipitation from oxalate solution half-saturated with ammonium chloride separates titanium, niobium, and tantalum from beryllium as well as from zirconium, thorium, aluminium, uranium, etc.

Up to the present, tartrate and acetate solutions do not appear to have proved suitable media for quantitative tannin separations. Uranium can be quantitatively separated from beryllium by precipitation as ferrocyanide; the slimy precipitate is readily filtered off and washed when mixed with pulped filter fibre.

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The Detection and Colorimetric Determination of Tin by means of substituted 1:2-Dimercaptobenzenes. A Specific Reagent for Tin

BY ROBERT E. D. CLARK, M.A., PH.D.

PREVIOUS investigators (Pollak,[1] Guha and Chakladar,[2] Hurtley and Smiles[3] observed the formation of a red compound when benzene-1:2-disulphonic chloride is reduced with tin and hydrochloric acid. The nature of this substance has never been elucidated. Pollak recognised the presence of tin, but could obtain no consistent analytical figures, while Guha and Chakladar supposed the substance to be wholly organic. Recent work by Mills and Clark[4] has shown that the benzene-1:2-dithiols readily yield derivatives in which an atom of a metal forms part of a 5-membered ring, and it is possible that the red compound is similarly constituted.

In the course of the present investigation it has been found that the formation of red compounds with 1:2-dimercaptobenzenes constitutes a specific and very delicate test for tin. As the only other known sensitive tests for this metal, the cacotheline and molybdenum blue tests (*e.g.* that of Gutzeit[5]) depend upon the reducing action of stannous chloride, and are vitiated by the presence of many other reducing agents, the present method should find wide application.

REAGENTS.—The reagents employed were 4-methyl-1:2-dimercaptobenzene and 4-chloro-1:2-dimercaptobenzene; methods of preparation have been published elsewhere (Mills and Clark[4]).

It was found most convenient to employ the reagents in the form of solutions (about 0·2 per cent.) in sodium hydroxide solution. In the sequel these solutions are referred to as Reagent I and Reagent II, respectively. Since they readily oxidise in air, depositing the white disulphide, they were stored in an atmosphere of hydrogen or else prepared shortly before use.

USE OF THE REAGENT AS A SPOT-TEST FOR TIN.—The test is best applied by adding a few drops of a 0·2 per cent. solution of the mercaptan in aqueous sodium hydroxide solution to an acid solution (up to 15 per cent. of hydrochloric acid) containing tin, and warming the mixture. A pink or red colour develops within a few seconds if stannous tin is present in a quantity of 1 in 10^6 or more. With stannic tin the colour is also obtained, probably as a result of the reduction of the tin to the stannous state by the mercaptan, but the colour takes longer to develop, and the test is less sensitive. If, however, a trace of thioglycollic acid is added to the liquid to be tested, the whole of the tin is rapidly reduced, and the sensitivity of the test becomes independent of the initial state of oxidation of the tin. The use of 4-methyl-1:2-dimercaptobenzene causes the development of a red colour more rapidly than the 4-chloro-derivative, but the final colour intensity is identical, at least to within 10 per cent. If, after being boiled for ten minutes, the solution is filtered, readily visible pink stains are obtained for tin concentrations of $1{\cdot}2 \times 10^{-7}$ and 5×10^{-7} with the methyl- and chloro-mercaptan, respectively (50 ml. of solution). Much lower concentrations could probably be detected with longer boiling and larger volumes of liquid.

No other element or substance was found to give a red colour with the mercaptan under comparable conditions, with the exception of bismuth, which produces a brick-red precipitate, quite unlike the magenta-red of tin.

The reagent is invaluable in ordinary qualitative analysis. On acidifying the ammonium sulphide filtrate in Group II*b*, after addition of Reagent I or II, a pronounced red colour develops under conditions in which stannic sulphide is difficult or impossible to detect. It is essential that alkali sulphides should not be present in too large an excess (see below), but, when there is a doubt, hydrogen sulphide can easily be partly expelled by acidifying and warming, followed by the addition of alkali. Antimony and arsenic may both be present; although a yellowish precipitate of the derivatives of these metals will then be formed at first, this becomes red on addition of excess of the reagent.

In general, the reagent may be employed as a test for tin in presence of all other metals, except when the colour of the other metallic mercaptides is so intense as to mask the red colour of the tin. In the following list any metal is precipitated by 4-chloro-1:2-dimercaptobenzene in preference to those placed after it, the conditions applying to an acid solution at the boiling temperature. Silver (yellow) and mercury (pale yellow); copper (black); bismuth (brick red); cadmium (pale yellow); arsenic (pale yellow) and antimony (yellow); nickel (black); tin (red); cobalt (black); lead (bright yellow). Although this order is approximate only, it is seen that, provided the reagent is used in excess, copper, bismuth and nickel are the only metals likely to interfere. When these are present only in traces the characteristic scarlet of the tin compound is usually discernible.

When alloys containing tin, but not containing copper, bismuth or nickel, are treated with a drop of hydrochloric acid and a trace of Reagent I or II, they soon develop a scarlet stain. In this manner tin can easily be detected in commercial lead.

RELATIVE SOLUBILITY OF THE PRECIPITATE COMPARED WITH TIN SULPHIDE.—A trace of stannous chloride was added to standardised sodium sulphide solution

(0·5–2·0 per cent.), with or without thioglycollic acid to prevent atmospheric oxidation. The mixture was titrated with standard (0·25 per cent.) Reagent II, which was stored in an atmosphere of hydrogen. A few drops of the liquid were periodically placed in a test-tube and acidified with hydrochloric acid, the end-point being marked by the change from the yellow tin sulphide to the red tin mercaptide. A sharp end-point (1 to 2 per cent. of Reagent II added) could be obtained if 60 seconds were allowed for the development of the colour towards the end of the titration. Heating vitiated the result by removing free hydrogen sulphide and thus precipitating the red compound before the end-point was reached. The volume of mercaptan solution required was independent of the concentration of sodium sulphide over the range employed. In this manner it was found that 0·00358 g. of chloromercaptan just caused precipitation of the red mercaptide in presence of 0·104 g. of sodium sulphide (Na_2S), giving a ratio of 1:29 by weight. For the methyl mercaptan (Reagent I) figures of 0·0205 g. of mercaptan to 0·251 g. of sodium sulphide (ratio, 1:12) were obtained, but the end-point was much less sharp. Calculation of the absolute solubility of the tin mercaptides from these figures is not possible, owing to a lack of knowledge of the relative ionisation of hydrogen sulphide and mercaptides in acid solution. If it be assumed that ionisation of the two classes of compounds is affected similarly, and if Weigel's[6] figure of $1{\cdot}13 \times 10^{-6}$ gm. mol./l at 18° C. be taken for the solubility of stannic sulphide, then the solubilities of the mercaptides are of the order of $2{\cdot}5 \times 10^{-8}$ and $5{\cdot}7 \times 10^{-8}$ gm. mol./l, respectively, reckoned as metal.

THE COLORIMETRIC DETERMINATION OF TIN.—The unknown solution is diluted, after addition of a drop of thioglycollic acid, until the concentration of tin lies between 1·5 and 6 p.p.m. A standard solution of tin containing about 10 p.p.m., together with about 0·2 g. of thioglycollic acid per l. is diluted in a measuring cylinder until a colour-match is obtained in the following manner. Two test-tubes containing identical volumes (5 to 10 ml.) of the two solutions are treated with 0·5 ml. of hydrochloric acid, followed by an equal quantity of Reagent II. This at once causes the precipitation of a white suspension of the mercaptan. The tubes are immersed in boiling water for 10 seconds, by which time the pink colour has developed fully, and the two colours are compared directly by reflected light. The dilution of the standard tin is then varied until a match is obtained. An accuracy of about 10 per cent. is attainable. The following results refer to a series of consecutive experiments obtained after negligible practice:

Found, p.p.m. ..	3·1	5·35	4·7	2·3	2·2	1·5	0·9
Present, p.p.m. ..	2·8	4·2	4·2	2·3	1·9	1·5	1·2

The precipitate settles out in time, but on shaking it up with the mother liquor the original colour is approximately restored.

As already stated, an identical intensity of colour can be obtained by using 4-methyl-1:2-dimercaptobenzene. This reagent, however, is much more soluble than the chloro-derivative in hot water, with the result that the red precipitate may alone appear in the liquid, which makes colour matching more difficult. The precipitate also shows a greater tendency to coagulate, which is partly offset by the greater rapidity with which the colour develops. As, however, the methyl-

mercaptan is less costly to prepare it may be substituted for the chloro-compound without great loss in accuracy. No conditions have been found under which it would be feasible to compare colours by transmitted light.

Effect of Foreign Substances on the Colorimetric Determination of Tin.—For the purpose of the following tests the substances named were introduced into a standard solution containing 4 to 5 p.p.m. of tin, and the colours obtained with 4-chloro-1:2-dimercaptobenzene were compared, as in the preceding section, with the standard after 15 seconds' heating in boiling water.

The colours were identical, within the limits of experimental accuracy, for 2 per cent. solutions of ammonium chloride, sodium chloride, magnesium sulphate, zinc sulphate, calcium chloride, barium chloride, strontium chloride, potassium aluminium sulphate, urea, hyperol, potassium fluoride (delay in attainment of full colour), chloride, bromide, iodide, cyanide, thiocyanate, sodium tetraborate or sodium sulphite. Salts of iron (ferrous and ferric ammonium sulphate) sometimes gave a slightly different tint at 2 per cent. concentration, but 0·5 to 1·0 per cent. solutions had no effect. Manganese sulphate could be added up to 1·0 per cent. With lead (cold saturated lead chloride solution) no modification in the colour was observable, provided that sufficient (7 per cent.) hydrochloric acid was present.*

Of acid radicals, nitrites must be absent, since they at once give a red colour with thioglycollic acid. Nitric acid (1 per cent.) does not interfere under the conditions described, but on *prolonged* boiling it decomposes the red compound, producing a white precipitate. Persulphates may be present up to 1 per cent. Phosphates alter the nature of the precipitate, very little colour being produced in 1 per cent. sodium or potassium dihydrogen phosphate. They have no effect in amounts lower than 0·05 per cent.

Organic acids, when present in large amount, prevent ready precipitation. No effect on the colorimetric determination is, however, experienced in 2 per cent. solutions of oxalic, citric, malonic, formic, acetic, succinic or tartaric acids. Organic substances of a colloidal nature interfere seriously. Thus, if starch is present in a concentration higher than 0·002 per cent., there is either a considerable lessening of the colour or its complete obliteration. The same remark applies to dilute solutions of glue. The products of hydrolysis of these substances, however, appear to have no effect. Thus fructose and glucose do not alter the colour, even in 20 per cent. solution, and glycine and also sucrose are without effect in a concentration of 2 per cent.

I wish to express my thanks to the International Tin Research and Development Council for a grant, without which this investigation could not have been carried out.

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Notes

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

APPLICATION OF THE FREEZING-POINT TEST TO HEATED MILKS

A NUMBER of samples of hot milk, as served in restaurants, etc., have recently been analysed under the Food and Drugs (Adulteration) Act.

Whilst in certain cases the addition of excessive quantities of water was obvious from all the figures obtained, and successful prosecutions were instituted, in a few cases the freezing-point test indicated the presence of small proportions of added water when the solids-not-fat were in the neighbourhood of 8·5 per cent.

On consideration of the latter results, some doubt arose as to whether the normal inference from the freezing-point could be relied upon when the milk had been heated under unknown conditions.

It has been shown (Barille, ANALYST, 1910, **35**, 22; Bell, *id.*, 1925, **50**, 466, *et al.*) that salts, such as citrates and phosphates of calcium and magnesium, are precipitated when milk is heated, and the possibility of an accompanying rise in the freezing-point of the remaining milk had to be considered.

Both Monier-Williams (L.G.B. Report No. 22) and Elsdon and Stubbs (ANALYST, 1933, **58**, 7) have clearly shown that pasteurisation and sterilisation have little effect on the freezing-point of milk; the tendency was, however, for a slight rise to occur in the freezing-point after heating.

The heating and boiling of milk in an open or partly open vessel and keeping it hot for unlimited periods, accompanied at times by the formation and separation of skin and sediment, such as might occur in a restaurant where occasional glasses of hot milk are sold, is, however, another matter, and the need for information on the effect of such conditions on the freezing-point was manifest.

The following experiments were therefore carried out with a view to ascertaining the effect of such varying conditions:—A sample pint of fresh milk was purchased and its freezing-point determined; it was then heated in a small covered saucepan to 80° C. (*i.e.* about the lowest temperature suitable for preparing a glass of hot milk), decanted from the skin and sediment which had formed on the pan, and cooled; after determination of the freezing-point, this process was repeated twice on the same sample in the same (cleaned) saucepan, for confirmation.

A second sample was then purchased and, its freezing-point having been taken, it was heated in the same covered saucepan to 90° C., decanted, cooled and tested again; this process was repeated, as before.

A third sample was treated similarly, but heated to the boiling-point each time, and, after the three heat treatments, it was finally boiled for 15 minutes between the tests; it was then no longer suitable for serving, owing to its burnt taste and colour.

A fourth sample was then treated in the same way as the second one, *i.e.* heated to 90° C., except that it was heated in an open saucepan, no precautions being taken to minimise evaporation.

All freezing-points were determined in duplicate by the Hortvet process; in most instances the duplicates were identical, and in the others the means are given to the nearest third decimal place.

	Heated in covered saucepan						In open pan	
	(1) to 80° C.		(2) to 90° C.		(3) to boiling		(4) to 90° C.	
	F.pt.	Difference	F.pt.	Difference	F.pt.	Difference	F.pt.	Difference
Before heating	−0·537		−0·541		−0·544		−0·543	
		−0·004		−0·004		−0·011		−0·051
After once heating	−0·541		−0·545		−0·555		−0·594	
		−0·006		−0·006		−0·010		−0·032
After twice heating	−0·547		−0·551		−0·565		−0·626	
		−0·007		−0·011		−0·010		−0·035
After thrice heating	−0·554		−0·562		−0·575		−0·661	
						−0·031		
After boiling for 15 minutes					−0·606			

It will be seen that an appreciable lowering of the freezing-point results from heating milk under such conditions. As the greater lowerings follow greater heating, and still more so heating in an uncovered pan, the predominating factor is clearly evaporation.

The various conditions, however, under which milk may be heated and kept hot in cafés and restaurants are hardly reproducible in a laboratory, and it was felt that the only way to discover what changes might arise in those circumstances was to obtain, with the co-operation of the managements, actual samples of such milk, before and after heating.

In small cafés, coffee stalls, etc., where hot milk is infrequently asked for, it is usually heated in a saucepan as required, and the above results would be representative.

In medium-sized cafés, buffets, snack bars, etc., apparently the usual practice is for the milk to be heated and kept hot in a water- or steam-jacketed earthenware urn; whilst in the larger multiple restaurants, it appears to be the practice to fill and replenish from time to time a water-heated urn, kept at an almost constant temperature, with milk heated first in a saucepan.

Samples of milk, before and after heating, were accordingly obtained from various restaurants in the last two categories, and the results of analyses were as follows:

Source	No.	Fat Per Cent.	S.N.F. Per Cent.	F.pt.	Difference	Observations
Dairy snack bar	1*a*	2·89	8·85	−0·541		Before filling urn
					−0·003	
	1*b*	2·02	8·94	−0·544		After 2 hours' heating in urn; thick skin formed
Dairy snack bar	2*a*	3·81	8·84	−0·543		Before filling urn
					−0·002	
	2*b*	3·73	8·89	−0·545		After ½ hour's heating in urn; slight skin formed
Railway refreshment buffet	3*a*	4·02	9·03	−0·543		Before filling urn
					+0·023	
	3*b*	3·91	8·70	−0·520		Half-hour's heating; found to contain water from cleaning
The same, later	4*a*	3·87	8·94	−0·543		Immediately after last filling
					−0·003	
	4*b*	3·79	9·02	−0·546		After 1¼ hours' heating
Dairy snack bar	5*a*	3·81	8·82	−0·545		Before heating
					−0·001	
	5*b*	1·76	9·05	−0·546		After 1½ hours' heating; thick skin formed

Source	No.	Fat Per Cent.	S.N.F. Per Cent.	F.pt.	Difference	Observations
Public house	6*a*	3·71	8·72	−0·544		Before filling urn
					+0·039	
	6*b*	3·29	8·00	−0·505		After ¾ hour's heating; contained condensed steam
Large multiple restaurant	7*a*	3·67	8·47	−0·525		Before heating
					−0·001	
	7*b*	3·63	8·45	−0·526		Kept hot for an hour
Large multiple restaurant	8*a*	3·82	8·93	−0·545		Before heating
					−0·008	
	8*b*	3·83	9·02	−0·553		Kept hot for 1½ hours
Large multiple restaurant	9*a*	3·64	8·34	−0·516		Before heating
					0	
	9*b*	3·49	8·39	−0·516		Kept hot for an hour

In two cases, Nos. (3) and (6), where the results departed from the general trend, investigation proved that water had accidentally found its way into the hot milk; in (3) this was due to the outlet tap incompletely emptying the urn, with the result that a small amount of water was introduced into the first filling of the urn after cleaning, from which filling the sample had been taken; a second pair of samples, numbered (4), taken from the same urn (*a*) immediately after the last filling of the day, and (*b*) after heating for 1¼ hours, gave normal results. In the other case, (6), the flat earthenware lid covering the milk compartment was found to be broken, and steam from the outer jacket was condensing on the outer lid and percolating through the broken lid into the milk; as no new lid was provided, further samples were not taken.

The series of analyses showed several points of interest; in some instances, particularly where much skin had formed, the fat-content was lowered considerably by the heating; in others, only slightly—showing that from this aspect the method of heating adopted is of the greatest importance; and, further, that practically no fat need be lost when milk is heated under suitable conditions, and even kept hot for an hour or more.

In most cases, and even when a thick skin was observed, the solids-not-fat are slightly higher in the heated sample; the percentages, however, agree closely with figures which may be calculated from the solids-not-fat in the corresponding unheated sample, allowance being made for the difference in fat and the evaporation suggested by the freezing-points; this indicates but little loss in solids-not-fat through heating, in spite of skin and sediment formation.

As may be seen, the samples could not all have been considered genuine, but they serve nevertheless for the purposes of comparison, and show no instance where the heated sample gave a higher freezing-point than the unheated milk, excepting where it was proved to contain added water.

To summarise, therefore, it would appear that the normal inference from the freezing-point can be relied upon when a milk has been heated in any of the ways usual where hot milk is sold; except that under some conditions when the milk has been re-heated or allowed to evaporate to any extent, a proportion of added water may be somewhat under-estimated by this means.

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THE DETERMINATION OF NITRATE BY MEANS OF DEVARDA'S ALLOY

THE determination of nitrate, according to Devarda's original instructions,[1] is unsatisfactory because of the tendency of the hydrogen, which is being evolved from the alloy, to carry over an extremely fine alkaline fog during distillation. This fog is very difficult to remove by scrubbing, even with complicated bubble scrubbers.[2] The method of reducing nitrate to ammonia has obvious advantages over alternative methods from the theoretical point of view, since reduction with ferrous sulphate in carbon dioxide or steam is troublesome to operate and, moreover, Chilian nitrate, its liquors and raw caliche also contain other oxidising agents, such as iodates. The nitrometer method is not safe, on account of the pressures which may be developed with the strong acids in the mercury vessel. Further, if the amount of sodium chloride in the sample exceeds 17 per cent. of the weight of sodium nitrate, the method gives high results.[3]

The object of this research was to determine the easiest and most economical method of determining nitrate as a routine operation. Most writers appear to have substituted sodium hydroxide for the more expensive potassium hydroxide specified by Devarda, who required 40 ml. of potassium hydroxide solution of sp.gr. 1·3 and 2 to 2·5 g. of alloy (50 per cent. of copper, 45 per cent. of aluminium, 5 per cent. of zinc) for 0·5 g. of sodium nitrate.

The other extreme was advocated by Valmari,[4] quoted by Allen,[5] who used $N/10$ alkali and 6·5 g. of alloy per g. of nitrate. (The concentration given by Devarda works out at about 2 N, or twenty times as large.)

Butt[6] found that the particle-size of the alloy was immaterial so long as it would all pass through a 20-mesh sieve, and that about 200 ml. had to be distilled to ensure that all the ammonia had been transferred from the distilling flask into the standard acid. These results were confirmed in the present investigation.

EXPERIMENTAL.—The sodium hydroxide, which was guaranteed to contain less than 0·0015 per cent. of ammonia, was made up to give a solution of sp.gr. 1·32 (381 g. per l.). The distillation was carried out in litre flasks connected by means of Davisson scrubbers with condenser tubes made of tin, to eliminate any possibility of soda being dissolved from a glass tube by steam condensing on it. The quantity of sodium nitrate taken for analysis was 1 g. of the purest quality obtainable; the standard acid and alkali were made of the same normality, and were equivalent to 0·02 g. of nitrate ion per ml.; and the indicator was methyl red dissolved in alcohol. In the first test, 250 ml. of distilled water were distilled, and the blank test gave zero readings. The result of adding varying quantities of alkali and alloy is given in Table I. In every test the amount of water used was 250 ml.

TABLE I

Sodium hydroxide solution		Percentage reduction of the nitrate		
ml.	Normality	3·00 g. of alloy	2·5 g. of alloy	2·00 g. of alloy
1	0·035	35·2		
2	0·069	90·8	84·0	61·7
3	0·104	100·0	100·0	86·3
4	0·139	100·0	100·1	86·7
5	0·173	100·0	98·9	79·5
6	0·208	99·9	95·2	79·6
7	0·243	100·0		
8	0·277	97·7		69·7
9	0·312	95·0		
10	0·347	95·8		
20	0·694			66·2
30	1·040	92·5		
50	1·735	88·5		

The effect of doubling the quantity of water in the reaction was next tried, 525 ml. of water being added to the reaction flask. The results are given in Table II.

TABLE II

Sodium hydroxide solution		Percentage reduction of the nitrate	
ml.	Normality	3·00 g. of alloy	2·50 g. of alloy
2	0·035	85·1	
3	0·052	98·2	86·1
4	0·069	100·0	91·0
5	0·086	96·3	87·1
6	0·104		81·7
7	0·121		77·3
8	0·139	71·0	

These figures show that the only method of obtaining correct results with the original Devarda proportions of reagents was to get just sufficient alkaline spray to balance the lack of ammonia due to incomplete reduction of the nitrate. On comparing Table II with Table I it will be seen that it is also inadvisable to specify $N/10$ alkali, because the reduction depends, not on the concentration of alkali, but on the quantity of it used per g. of alloy.

The experience gained from these results was embodied in some instructions to assayers, published in 1928.[7] The quantity of reagents then recommended was 4 g. of alloy and 8 to 10 ml. of 25 per cent. caustic soda solution (sp.gr. 1·28) for 1 g. of sodium nitrate. In view of the fact that papers are still being published which indicate that it is not generally recognised that the amount of caustic soda originally recommended by Devarda is excessive and leads to inaccurate results, it was thought that the publication of the experimental work on which these instructions were based might serve a useful purpose. Thus, Cattelain[8] recommends for 0·5 g. of nitrate the use of 2·5 g. of alloy and 25 ml. of 30 per cent. caustic soda solution (sp.gr. 1·33) and 5 ml. of ethyl alcohol with 125 ml. of distilled water. Similarly, Meurice and Martens,[9] for 0·25 g. of nitrate, recommend 40 ml. of 40 per cent. caustic soda solution and 3 g. of Devarda alloy with 250 ml. of water. Both these papers are misleading, and the quantities that are now put forward are recommended as being productive of more accurate results, as well as being more economical.

SUMMARY.—The determination of nitrate by reduction to ammonia by means of Devarda alloy has been investigated by varying all the conditions and reagents one by one. As a result, it has been found that the limits are very different from those specified by Devarda in his instructions. The reduction is best accomplished by using only one-twentieth of the amount of alkali originally specified. It is inadvisable to specify concentration of alkali, because the reduction depends upon the quantity of alkali to a given amount of alloy. The optimum amount for the reduction of 1 g. of nitrate is 3 g. of alloy and 2 g. of caustic soda in about 250 ml. of distilled water.

MAXWELL BRUCE DONALD

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THE COMPOSITION OF SCOTTISH RASPBERRIES

AT first sight it appears to be a simple matter to determine whether the composition of a sample of jam complies with the standards agreed upon between the Society and the Food Manufacturers' Federation (*cf.* ANALYST, 1930, **55**, 694), but one must remember that we are dealing with a natural product subject to wide variations in composition, and that only a limited number of analyses of Scottish raspberries have been published.

The insoluble solids or the number of seeds in the raspberries are usually taken for computing the fruit-content of a jam, but both constituents vary within wide limits depending on various factors, such as the strain of cane, the climate and the soil conditions.

Macara (ANALYST, 1931, **56**, 39) found the insoluble solids to range from a minimum of 4·4 per cent. to a maximum of 9·2 per cent., with an average of 6·17 per cent. He also found that the number of seeds per 10 g. of fruit varied from 356 to a maximum of 490, with an average of 419.

Since this experiment was made I find that Macara (ANALYST, 1935, **60**, 592) has found a minimum of 3·29 per cent. of insoluble solids in Blairgowrie fruit.

In a disputed case before the Law Courts regarding the fruit content of a sample of raspberry jam taken under the Food and Drugs (Adulteration) Act, the above figures were taken as a basis of calculation of the fruit-content. From my experience of Scottish raspberries I was not satisfied that these figures represented their average composition. Accordingly, I arranged to visit a few fruit farms in Essendy and Craigie, near Blairgowrie, and collected the fruit direct from the canes. The raspberries were just in the right condition for pulling, and there had been no rain for a week previously. The samples were kept overnight in screw-capped glass jars, and the analyses were carried out next morning.

The results are as follows:

		Insoluble solids Per Cent.	Soluble solids Per Cent.	Seeds per 10 g.
Lloyd George	4 years canes ..	3·99	8·43	246
,, ,,	5 ,, ,, ..	4·95	8·20	326
Devons	2 ,, ,, ..	4·71	8·67	332
,,	6 ,, ,, ..	4·80	10·70	402
,,	6 ,, ,, ..	4·65	11·18	400
,,	10 ,, ,, ..	4·36	10·61	334
Antwerp	10 ,, ,, ..	3·93	8·21	288
Mitchells	3 ,, ,, ..	4·24	9·34	284
Pynes Red Cross	3 ,, ,, ..	4·94	9·23	274
Pynes Royal	10 ,, ,, ..	3·94	8·64	287
Maximum		4·95	11·18	402
Minimum		3·93	8·20	246
Average		4·45	9·32	317

It is quite true only 10 samples were taken, but at least they indicate appreciably lower values than those published at the time.

There is consequently no definite standard upon which to found an opinion, and one is left to decide what figure to adopt for purposes of calculation. This will be obvious if the respective minima stated above be taken in order to calculate the fruit-content of a jam, containing, say, 1·80 per cent. of insoluble solids. Macara's minimum of 4·4 would indicate 40·9 per cent. of fruit, and his later minimum of 3·29 indicates 54·7 per cent. of fruit. The minimum of 3·93 found in my experiments is intermediate, with 45·8 per cent. of fruit.

The difference between the highest and lowest result thus obtained is 13·8 per cent., and brings one to the conclusion that the amount of fruit in a raspberry jam can only be determined with accuracy if the composition of the raspberries from which the jam is made is known.

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THE CHLORINE-CONTENT OF FEATHERS

(Read at the Meeting of the North of England Section, February 1, 1936)

IN 1935 rumour spread among a section of the upholstery trade that the limit of chlorine, prescribed by the Rag Flock Act Regulations, 1912, was applicable to such articles as feathers, feather pillows, feather beds, down cushions, etc.

In spite of assurances to the contrary, the fear of legal action persisted, and it was decided to ascertain the amount of chlorine extracted with water in the usual way from feathers actually being used at the time.

As I was unable to discover any figures relative to this point in the literature, the results obtained in the investigation may be of interest to analysts.

The feathers from pillows (purchased locally) manufactured by rival firms, showed the possibility of fairly wide differences.

Pillow No. 1 yielded chlorine .. 499 parts per 100,000 (*cf.* Grade 6 below)
„ No. 2 „ „ .. 102 „ „ „

By the courtesy of the Scott Feather Co. I was enabled to obtain samples of the feathers as purchased wholesale and of the various grades in general use after treatment. Feathers are purchased from many sources and stored in a warehouse until wanted. To keep down offensive odours when in store, they are sometimes sprayed with dilute ammonia while awaiting treatment. Before being washed they are graded according to size, each grade being dealt with separately.

The treatment consists in a thorough washing with boiling water in a large tank fitted with a stirrer, the time of washing varying with the grade of feather. The feathers are then screened off and stoved to complete their sterilisation.

A sample of the untreated feathers direct from a warehouse yielded:

Chlorine .. .. .. 609 parts per 100,000

After treatment, the following figures were obtained for the various grades:

	Chlorine (parts per 100,000)
Grade 1 (down)	68
„ 2	51
„ 3	68
„ 4	85
„ 5 (larger whole feathers) ..	398
„ 6 „ „ ..	406

As the above results represented a mixture of feathers from all kinds of birds, I procured, from the C.W.S. Poultry Department, Armagh, samples of feathers plucked from selected birds during their preparation for the Christmas market, and a second lot a few weeks later. These had not been washed or treated in any way.

The results were as follows:

	Chlorine (parts per 100,000) Lot 1	Lot 2
Duck feathers	176	209 (grey duck)
Goose feathers	242	267
Chicken feathers	342	350
Turkey feathers	359	359
Pheasant feathers (Lancashire) ..	367	
Goose feathers from old feather bed	409	

The method adopted for the tests was that used for rag flock. The chlorine in the extracts was present chiefly as potassium and sodium chlorides, any excess being presumably due to ammonium chloride.

The following figures (expressed as percentages on the extracts) were obtained in the course of the work:

	Potassium chloride	Sodium chloride
Duck feathers	0·25	0·05
Geese feathers	0·24	0·21
Geese feathers (old bed) ..	0·23	0·26
Chicken feathers	0·24	0·15
Turkey feathers	0·28	0·28
Pheasant feathers	0·20	0·24

A comparison of the figures for fresh and old geese feathers indicates that the increase in the chlorine-content of the old feathers is mainly in the form of ammonium chloride.

F. ROBERTSON DODD

Erratum

MILK PRODUCTS REPORT, NO. 4 (ANALYST, 1936, p. 11). In the Figure the 20 cm. length should run to the shoulder and not to the lip of the tube.

Appointments

JOHN EVANS, as Public Analyst for the Borough of Chesterfield, in place of G. E. Scott-Smith (deceased), December 28th, 1935.

F. DIXON to be Deputy Agricultural Analyst for the County Borough of Stoke-on-Trent (February 11th, 1936).

E. T. SHELBOURN, as Chemist to the London County Council, in succession to J. H. Coste (retired), March 26th, 1936.

Notes from the Reports of Public Analysts

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

CITY OF BIRMINGHAM

Report of the City Analyst for the Fourth Quarter, 1935

Of the 1261 samples submitted, 65 were bought formally.

Chocolate Sponge Roll.—One sample contained no cocoa and another a mere trace; both were artificially coloured to simulate the appearance of a genuine chocolate roll. A reasonable standard would be that suggested by the British Research Association for the Confectionery Trade, namely, a minimum of 4 per cent. of dry fat-free cocoa material, corresponding with about 5·5 per cent. of cocoa. The local Master Bakers' Association was communicated with, and has called the attention of its members to the matter. Meanwhile the National Association of Master Bakers has been making a series of tests, with a view to finding a standard practicable in manufacture and acceptable to food and drug authorities.

Dried Mint.—A sample contained about 6 per cent. of extraneous mineral matter. In a communication from the packers it was stated that it was very difficult to remove the mineral matter entirely from mint, and that it was possible that in this case the extraneous matter had, owing to its weight, sunk to the bottom of the bulked material, so that the last few small packets had received nearly all of it.

Labelling of Raspberry Jam.—Two samples were labelled "Full Fruit Standard," with the addition of the words "Improved by the addition of other fruit juice." One contained not more than 22 per cent., and two other one-pound jars bought at the same shop contained only 15 and 10 per cent., respectively, of fruit. Letters were written to the Food Manufacturers' Federation and to the manufacturers of the jam, pointing out that the agreed standards had not been complied with. The manufacturers replied that, after taking legal advice, they had, in reply to a demand for a cheap jam, determined to set up their own guaranteed full fruit standard. This, they said, was not in any way giving either the distributor or the consumer a guarantee of Federation standard.

In view of the fact that there appears to be no satisfactory remedy against individual manufacturers who choose to adopt their own standards, it would appear desirable that the Ministry of Health should review the situation, with a view to the establishment of some binding standards.

In the absence of any definite legal standard of composition there is apparently nothing to prevent any member of the Federation setting up his own Full Fruit Standard, with the result that the entire significance of the term will be destroyed. From the consumer's point of view the position would be even worse than before the Federation standards came into force.

Jelly Cream.—This was stated on the label to contain milk, none being required to make it up. The butter-fat content was 0·1 per cent., and the jelly contained a considerable quantity of starch, which gave it a fictitious milky appearance. It is possible that some dried skimmed milk powder was present, but the amount, if any, was very small. The packers were approached on the matter, it being maintained that an article labelled in such a way should contain a substantial amount of cream. After considerable argument the makers agreed to alter the description to "Jelly Dessert," followed by the words, "No milk required—simply add water." Two months were allowed to clear the stocks of existing labels.

H. H. Bagnall

COUNTY OF KENT

REPORTS OF THE COUNTY ANALYST FOR THE FOURTH QUARTER, 1935

THE food and drug samples examined during the quarter amounted to 1355, of which 421 were informal. Under the Fertilisers and Feeding Stuffs Act, 222 samples were submitted for analysis, and of miscellaneous specimens 266 were examined.

FISH.—Two pieces of fish were examined for an institution on different dates; in each instance coal fish had been substituted for cod.

LEAD POISONING BY HOME-MADE CIDER.—An investigation of a case of lead poisoning was traced to home-made cider, all the cider found upon the premises containing lead in quantities varying from 0·025 grain to 1·02 grain per gallon.

IDENTIFICATION OF MUD.—In a case of larceny, mud on the shoes of a man charged with the crime was found to be identical with that in the vicinity of a certain house.

MINERAL MATTER IN SHODDY.—Of 59 shoddies sold with a warranty, 25 were unsatisfactory owing to low nitrogen-content. In several of the samples the low nitrogen was due to high amounts of mineral matter. Dirt in the form of mineral matter was present in rather large quantities in no less than 12 shoddies, and in these the mineral matter varied from 20·4 to 33·4 per cent. Many of these dirty shoddies consisted of fleece combings, and fleece combings almost always contain not only dirt, but weed seeds. Several producers are requesting that all shoddy samples shall be taken from the truck at the station before unloading, and this because of the practice of some farmers of refraining from covering unloaded shoddy, so that the consignment, or a considerable part of it, is unprotected from the weather. There are several arguments against shoddy being sampled in the truck, firstly, because it is often impossible for the samplers to attend at a station within, say, 48 hours, owing to a pressure of other duties, as well as those of sampling elsewhere. Nor is it always easy to obtain a representative sample from a truck, on account of the difficulty of drawing material from the truck floor. When shoddy has travelled several hundred miles in a truck there is always a tendency for the dirt to be shaken towards the bottom of a truck, and therefore for the uppermost shoddy to be of the better quality. Practically all shoddy now arrives in sheeted trucks, so that there is little danger of addition of moisture from rain in transit.

BONE CHARCOALS.—These are generally obtained from decolorising plant when partly charred bones have been used for taking colour from a liquid such as sugar solution. Bone charcoal is used for decolorisation purposes for a considerable time because, as soon as its capacity for absorbing colour is lost, it may again be made active by slight ignition. However, the time comes when there is little organic matter to burn off, and then the charcoal is given a final ignition, ground, and (it may be) placed on the market as a fertiliser or a feeding-stuff. It is essential, if the charcoal is to be used as a fertiliser, that it should be ground to a fairly fine condition, and often a sample will entirely pass the 1/25-inch sieve. The phosphates usually vary between 65 and 75 per cent., but may amount to as much as 80 per cent., the latter being equivalent to 37 per cent. of phosphoric acid. With these phosphates are always associated varying quantities of nitrogen, and these vary from 0·2 to about 1 per cent., and depend upon the thoroughness with which the bone charcoal has been ignited.

PIG FOOD WITH EXCESS OF FIBRE.—Excess of fibre in a feeding stuff is not encountered as commonly as might be expected, but one sample of pig food recently examined contained 2 per cent. more fibre than the guarantee of 5·7 per cent. The declaration of fibre in some feeding stuffs is compulsory.

F. W. F. ARNAUD

Legal Notes

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

LYSOL SOAP

ON January 22nd a retail tradesman was summoned at Salford under the Merchandise Marks Act for applying a false trade description to goods, designated as lysol soap, which did not contain lysol. The manufacturer of the soap was also charged with applying a false description to the soap and with "aiding, abetting and counselling" the first defendant "to sell goods to which a false trade description had been applied."

The Deputy Town Clerk of Salford, prosecuting, said that the soap had been analysed by the City Analyst, who had certified that it did not contain any lysol or cresol.

The managing director of the manufacturing company, giving evidence for the defence, said that the soap contained 0·2 per cent. of lysol, and agreed that this amount was so small as to escape detection in analysis. He said that he purposely placed only this small amount in the soap, as he did not want to run the risk of actions against him if larger quantities were put in and irritated the skin.

The Stipendiary (Mr. P. Macbeth), giving his decision, said that he was not prepared to state what the standard amount of lysol should be. It might be that a big combine would be prepared to take the risk of putting too much lysol in the soap, and would be prepared to face a series of actions which a smaller manufacturer might not be prepared to face. If he (the Stipendiary) had the right to fix a standard as to the quantity of cresol that should be put in a soap described as lysol soap, he would fix it at 1 per cent.—that is, 2 per cent. of lysol. If he was not entitled to fix a correct percentage, then he must find that so long as the soap contained some cresol, he was not entitled to convict. Therefore, he found that, as the soap did contain 0·1 per cent. of cresol, it could rightly be described as lysol soap.

The summonses were dismissed, and twenty guineas costs were allowed to the manufacturer and one guinea to the retailer (*cf.* ANALYST, 1935, **60**, 820).

MEAT AND MALT WINE

ON February 14th a shopkeeper was summoned at Bradford Police Court for selling as meat and malt wine a preparation containing less than 4 per cent. of meat extract and less than 25 per cent. of malt extract.

Mr. F. W. Richardson, F.I.C., City Analyst for Bradford, said that the sample bottle, for which the inspector had paid 2s. 9d., contained 84 per cent. of water, and not more than 1 per cent. of meat extract; it could be produced at a cost of 2½d.

For the defence it was submitted that there was no recognised commercial standard for meat and malt wine. The sample sold was genuine meat and malt wine, and had been on the market for some years.

Mr. C. H. Manley, F.I.C., City Analyst for Leeds, said that his standard for such a non-alcoholic preparation as that in question would be 1 per cent. of meat extract and 4 per cent. of malt extract. Accepting Mr. Richardson's maximum figure for the meat extract present, the present preparation contained 1 per cent. of meat extract and 6·3 per cent. of malt extract. A preparation containing more than 1 per cent. of meat extract would, in his opinion, be liable to putrefy when the bottle was opened.

Dr. D. M. McIntyre, of Leeds, said that, in his opinion, the product in question was a stimulating, energy-producing and heat-producing food.

The Magistrate said that it was difficult to know what anyone expected when buying meat and malt wine. Probably it was thought that something very much better than milk, wine or ox-tail soup was being supplied. He felt that he was not in a position to say that this was not meat and malt wine, that the proportions were below any reasonable proportion, or that he could definitely say that the compound fell below the true minimum. He therefore dismissed the summons.

Department of Scientific and Industrial Research

THE INVESTIGATION OF ATMOSPHERIC POLLUTION

REPORT AND OBSERVATIONS IN THE YEAR ENDED 31ST MARCH, 1935*

THIS Report (the 21st) follows the same lines as previous reports (*cf.* ANALYST, 1935, **60**, 409). It embodies the Report of the Standing Conference of Co-operating Bodies, the Report of the Atmospheric Pollution Research Committee (Chairman, Dr. G. M. B. Dobson), and the Report of the Superintendent of Observations (Dr. J. S. Owens). The results of a statistical examination of records of deposit gauges over a period of some 20 years (embodied in an appendix) point to the need of further knowledge through an amplification of air-pollution observations, particularly in rural and country areas. A special survey on a larger scale than has hitherto been carried out is in contemplation and involves the installation of instruments covering a specially selected industrial centre, if possible at a distance from the main industrial areas.

SULPHUR GASES IN AIR.—The lead peroxide method (ANALYST, 1933, **58**, 284; 1934, **59**, 280) for measuring the "activity" of sulphur pollution has been increasingly used, and further attempts are being made to develop it so as to provide information with regard to the main directions from which the pollution arrives. The distribution of sulphur pollution is being further studied experimentally at Oxford with the volumetric sulphur apparatus, to provide records of suspended impurities. The absorption of acid or alkaline particles by the filter-paper does not appear to be sufficient to affect the sulphur records.

DAYLIGHT MEASUREMENT.—It was found that the new apparatus referred to last year (ANALYST, 1935, **60**, 410) was affected by temperature in strong sunlight, but this difficulty has been met and the apparatus is now undergoing final tests.

MEASUREMENT OF ULTRA-VIOLET RADIATION BY THE ACETONE AND METHYLENE BLUE METHOD.—Results from a few stations have been collected, and the figures are reduced to a quartz-tube minus glass-tube reading in accordance with the finding that the solution of methylene blue is sensitive to the visible part of the spectrum as well as to ultra-violet (*cf.* ANALYST, 1935, **60**, 410). The small range of variation is somewhat remarkable—*e.g.* at Kingston-on-Hull, 0·25 to 2·0; at Oakwood Hall Sanatorium, Rotherham, 0·2 to 0·8; at Attercliffe, Sheffield, 0 to 1; and at Southport, 0·3 to 2·9; the highest reading was 3 at Stirling. It has been found that the fading of the blue solution in ultra-violet is reversible, and that there is a recovery of colour if tubes are kept in the dark, and a recovery at night of colour lost in daylight. Tubes should therefore always be read in the evening. The amount of ultra-violet received at a particular place depends not only upon

* Published February 24th, 1936, pp. 103. Obtainable at Adastral House, Kingsway, W.C.2. Price 5s. net.

the transparency of the sky, but also upon other factors, such as the condition of the sky as to clouds, the altitude of the sun, the effect of reflection from the ground, walls, and so on.

Results obtained with the Automatic Filter.—A table showing the highest concentrations of impurity in the last two years at various times on weekdays (that is, excluding Saturdays) for four London, five Glasgow stations, at Kew and at Coventry and Stoke-on-Trent, make it evident that no great change in the amount of impurity has occurred since last year.

RECORD OF OBSERVATIONS.—The number of deposit gauges has increased to 98, the new ones being in Bristol. Five new stations are now using the lead peroxide sulphur method, and the number of cylinders has been increased at three others; one new station is using the volumetric sulphur method, whilst observations have ceased at three stations. The maximum and minimum monthly deposits as metric tons per sq.km. (conversion tables are given) were:

Tar: London (Golden Lane), 250, Marple, 10; *Carbonaceous matter other than tar:* London (Archbishop's Park), 167, Marple, 36; *insoluble ash:* London (Archbishop's Park), 160, Huddersfield (Cooper Bridge), 40; *ash of soluble matter:* London (Finsbury Park), 183, Huddersfield (Cooper Bridge), 28; *total solids:* London (Finsbury Park), 153, Huddersfield (Cooper Bridge), 29; *rainfall:* Newcastle-upon-Tyne (Town Moor), 107 mm., London (Southwark Park), 59.

A reduction for tar is shown for 26 stations, an increase for 16, and 14 were the same as the general average, this being a slightly better result than last year. The figures for other carbonaceous matter are not so good, and those for insoluble and soluble ash vary little from those of the two preceding years. Compared with the general average, 41 stations show a reduction of total solids, compared with 45 last year, and 17 an increase, compared to 8 last year. There has been a definite increase in both tar and sulphates at several London stations, and at Castleford, Glasgow (Alexandra Park), Richmond Park, and Newcastle-upon-Tyne (Town Moor). The only stations showing a consistent reduction in all the components of the deposit are London (Southwark Park), Glasgow (Victoria Park), Rothamsted, Salford (Peel Park), and Wakefield (West Riding Rivers Board). Amongst outstanding figures are those for soluble loss on ignition at London (Finsbury Park), 3·27 times the average; ash at the same station, 1·83 times the average; sulphates, 3·05, and total solids 1·5 times the average. London (Golden Lane) also shows some abnormally high figures. Tar deposit in Glasgow (Alexandra Park), and in Kingston-on-Hull, Suburban, and Newcastle (Town Moor) were exceptionally high, whilst Rochdale and Rotherham also show high sulphur deposits.

Taking the whole of the tables into account, a slight set-back has to be recorded compared with last year, the only figure showing an improvement being that of tar. It is evident that great efforts are called for if improvement in atmospheric conditions is to be realised. D. G. H.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS

Food and Drugs

Volatile Sulphur-content and Pungency of Onions. H. Platenius. (*J. Agric. Res.*, 1935, **51**, 847–853.)—It is assumed that onion oil has a definite composition and that differences in pungency are due only to quantitative differences in the oil present and indirectly to the volatile sulphur-content. For the determination of volatile sulphur a representative sample is taken from at least 20 onions, a quarter or a half of each bulb being thinly sliced, the portions mixed and 500 g. weighed into a flask. The oil is hydrolysed by heating with 100 ml. of conc. hydrochloric acid and 250 ml. of water for 3 hours. The mixture is then partly neutralised to prevent distillation of any hydrochloric acid, but, since protein sulphur is readily hydrolysed in alkaline solution, the mixture must remain faintly acid. The mixture is then distilled with steam from an oil-bath at 145° C. Very little sulphur comes over after 750 or 1000 ml. have been distilled, and, if comparative values only are required, 800 ml. of distillate are sufficient. The distillate is evaporated to about 300 ml. and filtered, the filter-paper is washed, and 10 ml. of bromine water added to the filtrate. The excess of bromine is driven off, the volume is reduced to about 300 ml., and the sulphuric acid is determined as barium sulphate. Samples were found to vary in their content of volatile sulphur from 25 to 200 p.p.m., and it is doubtful whether differences of less than 10 p.p.m. are significant, owing to the variability of the samples (20 onions per sample) being greater than the experimental error. With slight modifications the method should be applicable to garlic and cabbage and, possibly, to other vegetables. D. G. H.

Detection of Caramel. A. Joszt and S. Molinski. (*Z. Unters. Lebensm.*, 1936, **71**, 19–32.)—Various methods are here investigated by means of standard caramel preparations—A, B and C, corresponding with caramelan, caramelen and caramelin, respectively. These were obtained from highly-refined sugar by heating it *in vacuo* (2 to 7 mm. of mercury) to 187·5–189·5° C. From the volatile liquid distilled a liquid preparation, D, was obtained. The composition of these is stated. A standard liquid sugar colour, here called "colour," was also used. *Conclusions.*—Good results were obtained only by the Jagerschmidt methods (particularly with resorcinol), by the methods of Amthor, Crampton and Simons (with adsorption earths), and the Griessmayer and Aubry methods for the examination of beer. The reactive substance formed by both the Jagerschmidt methods is ω-oxymethylfurfural. The reaction was most pronounced with preparation, D. This method must be controlled by other methods, as ω-oxymethylfurfural may be formed naturally in solutions examined, through the action of heat or acids. Hence the solutions in this, and in Amthor's method, are best evaporated *in vacuo*, at room temperature. For Amthor's method, B and "Colour" were most sensitive; D was not reactive. In Crampton and Simons' adsorption method, A, B, C, and sugar colour were completely decolorised by argillaceous Florida, and fullers'

earths. The quantitative colorimetric method of Griessmayer and Aubry gave satisfactory results for malt caramel and for "Colour." Lichthardt's method was more sensitive for the commercial sugar colour than for A, B, C, and D; it is not clear what compound is the active substance in this method. Crampton and Simons' ether-extraction method does not extract the artificial caramels; only 27 per cent. of the natural colour of a matured rye-spirit could be extracted. Straub's method gave coloured precipitates with all caramel preparations examined, as well as with some wines. In Nessler and Carles' method there was no decolorisation of caramel products by egg albumen; but matured rye-spirit and some wines were considerably decolorised. Fradiss' method was of little value; Ihl's method was less sensitive than the corresponding Jagerschmidt method, and the Magalhaes reaction was not found satisfactory for the detection of caramel. E. B. D.

Determination of Carbon Dioxide in Beer. E. C. Martin. (*J. Inst. Brewing*, 1936, **42**, 79–83.)—The gas absorbed in an excess of alkali is re-liberated by acidification and its volume then determined. The method takes only a few minutes, and it is sufficiently accurate for most purposes. Bottled beer should be cooled for 1 hour in ice, the level being then marked with a file, the stopper is removed and 10 ml. of 40 per cent. sodium hydroxide solution per pint are added rapidly from a pipette with a wide jet; the liquids are mixed by inversion and the volume of the beer is determined subsequently by filling the bottle again to the mark with water. Other beers are led (without cooling) from a sampling-cock through a rubber tube which dips below the surface of 25 ml. of the alkali in a 500-ml. flask which may then be filled slowly to the mark. The measuring apparatus consists of a spherical extraction vessel (150 ml.) with a glass-cock at the lower end, which is connected by means of rubber tubing with a mercury reservoir (220 ml.). The upper end leads to a tube 25 cm. long and graduated from 0·05 to 10 ml., above which is a glass-cock and then a funnel with a constricted opening and a small side-arm sloping downwards. The latter is sealed in at such a point that it will allow 4 to 5 ml. of liquid to remain in the funnel, and it is connected by rubber tubing with a piece of bent glass tubing which is hooked into the mouth of a 500-ml. separating funnel with a short stem. The apparatus is rinsed with the alkaline beer, exactly 2 ml. being then measured into the graduated tube and 1 ml. of 20 per cent. (v/v) sulphuric acid is pipetted into the funnel (the top tap being closed). The reservoir and taps are then manipulated so that the acid is in contact with the sample in the spherical vessel with both taps closed and only a few ml. of mercury present. The gas is liberated by gentle shaking, its volume is read after equalising the mercury levels, and the extraction is repeated. The difference should not exceed 0·05 ml., and it is advisable to avoid delay as the gas is slightly soluble in the acid. The reading is checked by adding 2 ml. of alkali through the funnel and noting the contraction; there is usually an unabsorbed volume of 0·05 ml. Control tests with a standard carbonate solution should be carried out for each batch of tests, or if the room temperature varies considerably, and the details of technique should be exactly the same in both cases. It is advisable to lower the mercury level before adding the reagents, so that any heat developed is absorbed by the mercury rather than by the glass

extraction vessel. The object of the side-arm in the funnel is to ensure complete removal of the excess of sample before addition of the acid; mercury is poured in, and the solution displaced by flotation is collected with the overflow of mercury in the separating funnel. If there is insufficient room for the alkali in the original bottle it should be opened in an inverted position in a vessel of alkali, some of which is forced up into the bottle by means of a bent pipette and a rubber bulb, to replace the beer removed. The maximum deviations between the usual gravimetric (soda-lime) method and the present procedure was 0·07 for "volumes" of carbon dioxide from 0·86 to 4·62; the former method always gave the higher result. J. G.

Component Fatty Acids of Goat Milk Fat. R. W. Riemenschneider and N. R. Ellis. (*J. Biol. Chem.*, 1936, **113**, 219–233.)—A composite sample of fat from goat milk was converted into the methyl esters which were fractionated into 63 fractions for the determination of the component fatty acids. The acids found were:—butyric, 2·1; caproic, 1·9; caprylic, 2·7; capric, 7·9; lauric, 3·5; myristic, 10·2; palmitic, 28·7; stearic, 8·1; decenoic, 0·2; tetradecenoic, 0·4; hexadecenoic, 2·1; and oleic, 31·2 per cent. In addition, 0·4 per cent. of a saturated acid of a higher molecular weight than stearic, 0·7 per cent. of arachidonic acid, and a trace of an unknown acid, probably a C_{22} acid or an isomer of arachidonic acid were also found. These results agree with those previously published, except for the presence of decenoic, tetradecenoic and hexadecenoic acids and the absence of linolic acid. S. G. S.

Moroccan Olive Oil. J. Valin. (*Ann. Falsif.*, 1936, **29**, 31–34.)—Tables are given showing the characteristics of more than 100 Moroccan olive oils over a period of 5 years. The figures given are mean weight of one fruit and of one kernel; percentage of shell and of kernel; of dry pulp and of dry fruit; proportion of oil in the pulp and in the fruit; iodine value of the oil and of the liquid acids, and the proportion of "stearine" in the oil. More than half the oils had iodine values over 90, and a few reached 95. It is suggested that the usual limits of 86 to 88 need revision for Moroccan oils. Attention is drawn to the fact that certain plantations yielded up to 50 kg. of fruit per tree, and that oil percentages reached 40 per cent. in the pulp, or 30 per cent. in the whole fruits. D. G. H.

Oil from *Ricinus Zanzibarinus*. A. Steger, J. Van Loon and C. Smelt. (*J. Soc. Chem. Ind.*, 1936, **55**, 41–42T.)—According to Bloemendaal, *Ricinus Zanzibarinus* is indigenous to East Africa, whence it was brought to equatorial America. It appears to bear more and larger fruits with higher oil-content than *Ricinus communis*, but a tropical or sub-tropical climate is needed for the seeds to ripen. A sample from Paraguay had deep black, glossy shells (26 per cent.) and white kernels (74 per cent.) covered with a silver-white seed-coat. The extracted oil had the following characteristics: sp.gr. at 78/4° C., 0·9211; n_D^{20}, 1·4788; n_D^{70}, 1·4610; saponification value, 179·2; iodine value (Wijs), 88·4; Reichert–Meissl value, 0·5; thiocyanogen value, 82·0; acetyl value, 145; unsaponifiable matter, 0·5 per cent. The oil contained 90·8 per cent. of total fatty acids, 4·7 per cent. of volatile products, and 4·0 per cent. of glyceryl residue. The fatty acids had

iodine value 92·0; n_D^{70}, 1·4537; neutralisation value, 187; mean mol. equiv., 300; and saturated acids, 0·5 per cent. The oil is thus seen to resemble that of *Ricinus communis* very closely. Calculation from the iodine and thiocyanogen values showed the total fatty acids to contain 6·6 per cent. of linolic acid. As 1·1 per cent. of saturated acids are present, the remaining 92·3 per cent. are acids with one double linking, and consist of ricinoleic acid alone or with a small quantity of Δ^9-oleic acid. D. G. H.

Carob Gum. W. A. Knight and M. M. Dowsett. (*Pharm. J.*, 1936, **136**, 35–36.)—Carob gum (*Ceratoniae gummi*) is obtained from the seeds of the locust-bean tree, *Ceratonia siliqua*, and has only now been satisfactorily separated from the other unpleasant constituents of the embryo by a series of mechanical processes. The powdered commercial gum showed, on analysis, galactan, 29·18; mannan, 58·42; pentosans, 2·75; proteins, 5·29; nitrogen, 0·83; cellular tissue, 3·64; and mineral matter, 0·82 per cent., together with an enzyme, ceratoniase. The gum gives a solution at least equal in viscosity to that of tragacanth, and is being used for such purposes as thickening sauces and pickles, for "smoothing" ice cream and in salad creams. The low price (1s. to 1s. 6d. per lb.) has led to its trial in various pharmaceutical preparations. *Mucilago ceratoniae* is prepared by mixing 0·15 g. of benzoic acid, 1·0 g. of carob gum, 3·0 ml. of glycerin, adding 80 ml. of water, and heating for 30 minutes to aid the formation of a good mucilage and also to destroy the enzyme which would otherwise cause loss of viscosity by hydrolysis. As an emulsifier used alone the mucilage is somewhat more efficient than tragacanth, but not equal to acacia. It may be used in Kesbar cream for radiographic work, but, if kept for long, more benzoic acid should be added. A concentrated mucilage is a suitable paste for labels and for lozenge-making, and is especially adapted for iodised throat lozenges. A mixture with glycerin and glucose forms a good universal pill excipient, and a paste of the formula glycerin 10 ml., boric acid 5 g., oil of lavender 0·5 ml., and 100 g. of carob mucilage (4 per cent.) is much easier to make than the Past. Trag. Co. of the Codex. A very effective toilet cream and a lotion may also be prepared with carob gum (*cf.* Williams, ANALYST, 1928, **53**, 411).
D. G. H.

Iodine-content of Foods. R. Balks. (*Z. Unters. Lebensm.*, 1936, **71**, 76–92.)—Potable waters and milk from all districts of Westphalia were examined. The iodine-content of the waters is, on the average, of the same order of magnitude as the values obtained in regions with "normal iodine standard." The average iodine-content of milk is comparatively high; it is about 74 per cent. higher than the normal value, 30γ per l., for inland cattle-farms. Potatoes, white cabbages, and carrots grown at the Munster Experimental Station, in typical different Westphalian soils, in the same climatic conditions, show no relation between iodine accumulated and the iodine-content of the soil. The iodine absorption of spinach was high on all soils, and was related to the natural iodine-content of the soils. It was greatly increased by a fertiliser of very dilute potassium iodide solution, used in amounts corresponding with 1·5 kg. per hectare, care being taken to avoid contact with young plants and direct feeding through the leaves. In two samples of spinach—one from natural soil and one from soil manured with potassium

iodide, the amounts of water-soluble iodine were very similar, indicating that the iodine in both cases exists almost entirely in similar, almost insoluble compounds. For carrots, also, potassium iodide fertiliser greatly increases the iodine absorption; the action of the fertiliser depends on the nature of the soil. In Westphalia, goitre occurs most frequently in districts where the iodine-content of the soil, and therefore of the food of the population, is lowest. E. B. D.

Metal Utensils in Food Industries and in Kitchens. A. Beythien. (*Chem.-Ztg.*, 1936, **60**, 107–109.)—New German food laws are being prepared to replace the "so-called" Lead-Zinc Laws, 1887, which refer to metal utensils, etc., coming in contact with food. These laws deal principally with the lead and zinc content of these utensils, but as questions about the toxicity of other metals are also to be raised, a review of the properties of these metals is made, and the following recommendations are advanced:—*Silver.*—This is completely insoluble in foods, and small amounts mechanically removed from the utensils are harmless. *Tin.*—Practically insoluble in the liquids contained in foods; the lead-zinc laws permit the use of alloys with 1 per cent. (tinplate) and 10 per cent. (solder) of lead. Appreciable amounts of tin are dissolved from tinned copper kettles by boiling 40 per cent. sugar solutions containing 1 per cent. of citric acid, and from canned vegetable material, especially if acid (lacquering prevents solution of tin from canned goods). Opinions differ about toxicity of these amounts. *Nickel.*—Small amounts are dissolved by foods prepared and stored in nickel utensils, but these amounts are considered non-toxic, and the Imperial Board of Health "Health-booklet" does not oppose the use of nickel utensils for foods. It is considered superior to copper for the preserved foods industries; the turning bitter of tomatoes and the browning of spinach is avoided by boiling in nickel vessels. *Aluminium.*—Not attacked, or only very slightly, by most foods; more strongly attacked by acid or alkaline liquids, *e.g.* sugar solution containing 1·5 per cent. citric acid, and by pickled meats. Apart from acid or alkaline foods, the amounts of aluminium dissolved are harmless, being no more than are usually taken in food alone. There is no truth in the rumour that aluminium causes or spreads cancer. *Copper.*—Only comparatively small amounts are dissolved by most foods, during preparation and storage. By careless preparation in copper utensils, quantities of the order of 304 mg. copper may be taken, per person, daily, but this can be tasted. Opinion is divided regarding the toxicity of copper in foods; it is recognised that the green from vegetable conserves is due to very stable complex copper-protein compounds, and that copper salts are largely excreted. *Cadmium.*—Cadmium-plating of food utensils should be prohibited. Cadmium is readily attacked by cold, 0·5 to 2·5 per cent., acetic acid, and also by boiling jam. Its toxic properties have been considered similar to those of mercury, and very small amounts are injurious. *Chromium.*—Chromium-plating for saucepans, etc., is considered harmless, provided that the plating is non-porous. Chromium is only slightly soluble, and almost non-toxic in small amounts. Fruit boiled in chromium-plated copper or brass kettles is not discoloured as in tinplate. *Zinc* is readily attacked by acids, and large amounts are dissolved, *e.g.* by acid sugar solutions; these amounts are harmful. Opinions differ regarding the toxicity of

small amounts. Zinc occurs in small proportions in foods themselves. The possibility of cumulative poisoning through storage of zinc in the liver is mentioned. Prohibition of zinc for food utensils by the laws of various countries is reviewed; German law is lax, but the new laws proposed regarding zinc-plate are stricter. Articles wholly of zinc will be permitted, because their range for daily use is limited. The new milk laws proposed will prohibit the use of articles wholly or partly of zinc in contact with milk or its products, unless tinned or coated with enamel or aluminium; cheese and curds are explicitly exempt from the milk regulations. *Zinc alloys.*—Brass (24 to 36 per cent. of zinc), pinchbeck (under 18 per cent. of zinc), and German silver (20 to 30 per cent. of nickel, 15–20 per cent. of zinc) are used for tea and coffee-pots, etc. The presence of other metals protects zinc from the action of foods, but these alloys will probably not be permitted for milk and its products. *Antimony.*—As a constituent of various alloys, especially Britannia metal (91 to 94 per cent. of tin, 6 to 9 per cent. of antimony, with copper, zinc, lead and bismuth), is completely insoluble in foods. The lead-zinc laws make conditions for its use in enamelled utensils. *Lead* is regarded as very dangerous, even in small quantities. E. B. D.

Variations in Caffeine-content of Commercial Coffee Extracts. A. Guillaume and Ch. Lefranc. (*Ann. Falsif.*, 1936, **29**, 10–16.)—A large number of coffee extracts were analysed for caffeine, dry extract at 100° C., ash and phosphates, total nitrogen, fatty matter and sugars. Assuming that 10 g. of ground coffee containing 1 per cent. of caffeine are taken for 1 cup (100 ml.) of coffee, the cup will contain a maximum of 0·1 g. of caffeine, and a laboratory extract made by percolation with boiling water did, in fact, yield 96·6 per cent. of this amount. By the same procedure for the commercial samples only 1 sample of 26 yielded a comparative figure (0·110), and the average yield was 0·0539, showing that most commercial extracts are liquids made with 5 per cent., not 10 per cent., of ground coffee. The other analytical figures confirm this, except that the average for nitrogenous matter was only a quarter of that obtained with 10 per cent. of ground coffee. D. G. H.

Contribution to the Study of the Identification of the Alkaloids and of Antipyrine as Picrates. A. Jonescu-Matiu and E. Iliesco. (*J. Pharm. Chim.*, 1936, **23**, 117–141.)—Reagents which may be used for all alkaloids are discussed. The disadvantages of picric acid are the slow rate of formation of characteristic crystals and the solubility of these in an excess of the alkaloid, and the modified reagents preferred are:—(1) A solution, saturated in the cold, of picric acid in 96° ethyl alcohol to which is added 5 per cent. of glycerin; or (2) picramic acid, prepared by reducing a 5 per cent. solution of picric acid by the action of 2 g. of pure powdered dextrose in the presence of sufficient sodium carbonate to produce an alkaline reaction, cooling and filtering. The former is preferable in most cases. In general, crystallisation is allowed to take place on the microscope slide, and the minimum quantity of alkaloid is used. The micro-reactions of the following alkaloids are discussed, the crystals produced with the above reagents being illustrated:—*Atropine.*—Groups of rectangles and rosettes, m.p. 165° to 166° C. in both cases, and, after crystallisation, rectangular tablets; sensitiveness, 0·01 mg.

(0·06 per cent.). *Hyoscyamine.*—The crystals are similar to those produced by atropine, but melt at 162° to 163° C., forming a red liquid. Reagent (1) is sensitive to 0·5 mg. (0·1 per cent.). *Nicotine.*—(1) Feathery crystals, m.p. 208° C. (approx.), are produced after 5 minutes with 0·001 mg. (2) No precipitate. *Strychnine.*—Characteristic feather-shaped groups of crystals are obtained, reagent (1) being sensitive to 0·002 mg. of strychnine sulphate, although reagent (2) is preferable for the base, crystals (which turn red at 200° C. without melting) being produced with a drop of solution containing 0·0002 mg. *Brucine.*—Reagent (1) produces bunches of radiating long prisms with a 0·005 per cent. solution of brucine after 15 hours, whilst (2) gives negative results for all concentrations, and picric acid is sensitive to 0·01 per cent. *Morphine, Codeine, Dionin and Heroin.*—Details of the reactions with reagent (1) are, in order:—Groups of rectangular plates with a 0·5 per cent. solution after 24 to 40 hours; rosette- or fan-shaped groups of prisms, 0·1 per cent.; and hedgehog-shaped groups of needles, hexagonal plates or octahedra, 0·1 per cent. In all cases the crystals dissolve in an excess of alkaloid or of reagent. *Papaverine.*—(1) Radiating prisms with 0·01 mg. per ml. in 17 hours, m.p. 154° C.; (2) an amorphous precipitate. *Sparteine.*—(1) Produced needles, m.p. 199° C., with 0·03 mg. *Hydrastinine.*—(1) Produces feathery leaf-shaped crystals with 0·12 per cent. solutions. *Cocaine.*—(2) Is most sensitive and produces brush-shaped groups of crystals, m.p. 154° C., with 0·002 mg. *Ephedrine* (0·8 per cent.) forms radiating needles with (1). The limiting concentrations for *novocaine* are 0·02 with picric acid and 0·4 per cent. with (1), and the m.p. of the picrate is 146° to 147° C. The crystals produced by (1) with 0·02 per cent. of *stovaine* have m.p. 110° to 112° C. (*cf.* cocaine), and consist of radiating prisms. *Antipyrine* may be distinguished from *pyramidone* by the difference in appearance of their picrates; by their m.p. (180° to 182° C. and 168° to 170° C., respectively); and by the fact that reagent (1) precipitates even 0·12 per cent. of the former at once, whilst precipitation of the latter is delayed even when the concentration is 1 per cent.

J. G.

Analysis of Drugs, Extracts and Preparations containing Pyrethrin. D. Mann. (*Chem. Ztg.*, 1936, **60**, 147–149.)—It is claimed that the following modification of Ripert's method avoids errors occurring in the older methods (*cf.* ANALYST, 1933, **58**, 300). Pyrethrum extract is saponified with *N*-alcoholic potash, the alcohol is removed by evaporation on a water-bath under reduced pressure, the residue is dissolved in water, and the solution is saturated with sodium chloride, after which barium chloride is added and the precipitated substances filtered off. Chrysanthemum mono- and di-carboxylic acids, which are present in the filtrate as barium salts, are liberated by addition of hydrochloric acid, extracted with ether, and washed three times with 10-ml. portions of sodium chloride solution; the ether is then removed by evaporation and the residue dissolved in neutral alcohol. The chrysanthemum carboxylic acids are titrated with *N*/5 alcoholic potash, phenolphthalein being used as indicator. The neutral solution is acidified with sulphuric acid and distilled in steam (180° to 200° C.), two 100-ml. portions of the distillate being collected. The first portion is extracted with 100 ml. of petroleum spirit, the aqueous layer is separated, and the spirit

layer is washed with brine. The aqueous and brine washings are combined (A). Twenty-five ml. of water are added to the petroleum spirit layer, which is then titrated with $N/50$ alkali. The neutralised aqueous layer is added to (A), and the petroleum spirit layer is used for extraction of the second portion of the distillate. The petroleum spirit layer is mixed with 25 ml. of water and titrated. The aqueous and brine layers are combined as before (B). Finally, (A) and (B) are titrated with $N/50$ alkali. If, for example, the extract from 40 g. of flowers requires 9·5 ml. of $N/5$ alkali for neutralisation, the first distillate requiring 27·4 ml., the second distillate 3·0 ml., and the wash-waters (A) and (B) 8·1 ml. and 5·0 ml. of $N/50$ alkali, respectively, there remains for neutralisation of the dicarboxylic acid $95 - (30{\cdot}4 + 13{\cdot}1) = 51{\cdot}5$ ml. of $N/50$ alkali. Since 1 ml. of $N/50$ alkali is equivalent to 6·6 mg. of pyrethrin I and 3·7 mg. of pyrethrin II, the percentages of pyrethrin I and II, respectively, are 0·5 and 0·475. If the extract contains a volatile solvent, it is removed on the water-bath before saponification; if it contains perfume, this is removed by a preliminary distillation with water. If non-volatile oils (*e.g.* petroleum) are present, the preparation is heated with N alcoholic potash for 1½ hours, and then extracted with water in a separating funnel, the aqueous extract being used for the analysis. Ripert and Gauder (*Compt. rend.*, 1935, **200**, 2219) have shown that, for frogs, pyrethrin II is somewhat more toxic than pyrethrin I, and that a mixture of equal parts of the two principles is more toxic than either alone. A. O. J.

Biochemical

Copper-content of Some Human and Animal Tissues. P. F. Hahn and E. Fairman. (*J. Biol. Chem.*, 1936, **113**, 161–165.)—Tissues obtained from normal and anaemic dogs, and from humans at autopsy or operation have been examined for their copper-content. Weighed samples of fresh material were placed in Kjeldahl flasks, and 40 ml. of fuming nitric acid and 20 ml. of concentrated sulphuric acid were added. The material was heated to boiling, and heating was continued until charring became apparent. Perchloric acid (60 per cent.) was then added, 2 ml. at a time, at intervals (from 2 to 10 ml. in all) until the solution was colourless or pale yellow, becoming colourless on cooling. The cooled digestion products were then transferred to volumetric flasks by means of water distilled from glass. Aliquot portions were examined for copper by the chromotropic method of Ansbacher, Remington and Culp (*Ind. Eng. Chem., Anal. Ed.*, 1931, **3**, 314; Abst., ANALYST, 1931, **56**, 684). For anaemic dogs it was found that the copper stores in the spleen and liver rose to very high levels as the iron-content fell to the lowest levels. In the small series examined disease did not appear to modify the copper reserves in humans, but the *normal* base-line is none too securely established. In cases of Mediterranean anaemia the liver contained a large amount of both copper and iron. The copper-content of the tissues from normal dogs in mg. of copper per kg. was:—liver, 15 to 23; spleen, 1·4 to 3·1; kidney, 4 to 11; and lung, 1·4. Human foetal livers contained from 42 to 78 mg. of copper per kg. of body weight, and those from young infants from 4·2 to 55 mg. of copper per kg. S. G. S.

Citric Acid formed in Animal Metabolism. C. C. Sherman, L. B. Mendel and A. H. Smith. (*J. Biol. Chem.*, 1936, **113**, 247–263.)—Citric acid was invariably found in normal urine of human subjects, of rats and of dogs, and in the blood, faeces and body tissues of dogs. The observations on man, *viz.* that the citric acid excretion varies directly with the urinary *p*H for a given individual, confirms the findings of Ostberg. This is true, regardless of the reason for the alteration of the *p*H value. When sodium carbonate (10 per cent. of the dry mixture) is added to the basal citrate-low diet of rats, the citrate elimination increases one hundred-fold. In some dogs, the excretion of citric acid was greater on a low protein, high sucrose diet than on a low carbohydrate, high casein diet, although, in other dogs, dietary differences produced no consistent change. A basal diet with a low citrate-content usually caused an increase in the citrate excretion, and often caused a rise in the blood citrate concentration. Repeated daily administration of alkali produced a twenty-fold to one-hundred-fold increase in citrate elimination, and this was helped by the substitution of sucrose for casein in the diet, although sucrose *per se* produced no increase. From a consideration of the large amounts of citrate which are secreted by dogs on a citrate-low diet during prolonged alkalosis, and the absence of reserves of pre-formed citrate in blood, liver, muscle and kidney, it is concluded that the dog can synthesise citric acid. S. G. S.

Determination of Small Amounts of Citric Acid in Biological Material. G. W. Pucher, C. C. Sherman and H. B. Vickery. (*J. Biol. Chem.*, 1936, **113**, 235–245.)—Quantities of citric acid of the order of 0·1 to 1·0 mg. may be determined with an accuracy of ± 5 per cent. by oxidation to pentabromoacetone and conversion of this substance by means of sodium sulphide into a coloured material that is suitable for estimation in the Pulfrich spectrophotometer. The solution to be analysed, containing not more than 1·0 mg. of citric acid, is transferred to a 150-ml. beaker, together with water to make a volume of about 75 ml.; 3 ml. of 50 per cent. sulphuric acid and a few quartz pebbles are added, and the solution is boiled for about 10 minutes. The solution, which should now have a volume of about 40 ml., is cooled to room temperature, an excess of saturated bromine water (usually 3 ml.) is added, and the mixture is allowed to stand for 10 minutes. If a precipitate forms, the solution is allowed to stand 20 minutes more, bromine water being added from time to time as necessary to ensure an excess. The solution is then transferred to a 50-ml. centrifuge tube, the precipitate is centrifuged down, and the supernatant fluid is poured into a 125-ml. separating funnel, after which 2 ml. of 1·0 *M* potassium bromide solution and 10 ml. of potassium permanganate solution (1·5 *N*) are added. When the solution has stood for 10 minutes it is decolorised by the addition of ferrous sulphate solution (20 g. of crystalline ferrous sulphate and 1 ml. of conc. sulphuric acid in 100 ml.). The mixture is shaken with 25 ml. of petroleum spirit, the aqueous layer is drawn off and the spirit layer is washed once with 5 to 10 ml. of water, the wash-liquid being added to the aqueous solution, which is again extracted with petroleum spirit. The combined extracts are then washed four times with 5-ml. portions of water. The washed solution is shaken successively with 3, 2 and 1-ml. portions of filtered

sodium sulphide solution, these being drawn off into a 10-ml. graduated flask containing 3·5 ml. of pyridine. The solution is made up to volume with 50 per cent. pyridine and, by means of a Pulfrich spectrophotometer, the extinction coefficient is determined within 30 minutes in a cell of appropriate length, with a light-filter No. S.-43. Water is used in the control cell. A calibration curve, from which the amount of citric acid present in the original solution is determined, is made from a series of solutions containing 0·1 to 1·0 mg. of citric acid, the preliminary boiling and treatment with bromine being omitted. Portions of 5 to 10 ml. of dog's urine or of 0·2 to 1·0 ml. of human urine usually contain suitable quantities of citric acid. When blood is examined, one volume of whole blood or plasma is added to 4 to 9 volumes of 10 per cent. trichloroacetic acid, and the mixture is stirred and allowed to stand for 10 minutes before being filtered or centrifuged. The aliquot portion removed should contain about 0·1 mg. of citric acid; that is, 10 ml. or less of whole blood. It is important that the blood be added to the trichloroacetic acid immediately after being drawn from an animal, as large losses occur within a short space of time. Faecal matter is ground with water which has been acidified to Congo red with sulphuric acid, and an aliquot portion is mixed with an equal volume of the trichloroacetic acid. After filtration, an aliquot portion, representing one-fifth to one-tenth of a day's collection, is taken for analysis. Animal tissue is ground with sand in a mortar with several portions of 10 per cent. trichloroacetic acid, and an aliquot part, representing 10 g. of the original material, is used. The recommended method for dealing with plant tissues is preparation of the "organic acid fraction" outlined by Pucher, Vickery and Wakeman (*Ind. Eng. Chem., Anal. Ed.*, 1934, **6**, 140). The following amounts of citric acid were obtained from dogs:—blood, 0·9 to 1·9 mg. per 100 ml.; faeces, 0·4 to 0·8 mg. per 24 hours; tissues, 0·6 to 1·2 mg. per 100 g. Tobacco plants contained up to 0·87 per cent. of citric acid. S. G. S.

Metabolism of Orally Administered Citric Acid. C. C. Sherman, L. B. Mendel and A. H. Smith. (*J. Biol. Chem.*, 1936, **113**, 265–271.)—The dog possesses the power of destroying almost entirely large amounts of orally administered citric acid. When 0·5 to 2·0 g. of citric acid per kg. of body-weight was ingested, only about 0·7 per cent. escaped oxidation and appeared in the urine. At the same time, a rise in the citrate concentration in the blood was maintained for 3½ to 7½ hours, no extra citric acid appeared in the faeces; nor were the *p*H and total nitrogen in the 24-hour collection of the urine affected. Apparent renal threshold values per 100 ml. of whole blood varied from 2·2 to 6·0 mg. of citric acid. S. G. S.

New Crystalline Derivative of Blood Pigment. M. Wagenaar. (*Z. anal. Chem.*, 1935, **103**, 417–418.)—A survey of the usual methods for identifying blood was given in an earlier paper (*Z. anal. Chem.*, 1930, **79**, 101; Abst., ANALYST, 1930, **55**, 405). A new crystalline derivative of blood pigment, acetone-haemin, is now described, and its preparation, used as a test for the identification of blood, is free from the disadvantages of other tests. A trace of blood plasma (or a particle of dried blood or a portion of fabric stained with blood) is placed upon a microscope slide and covered with a cover-glass, a small object such as a grain of sand or a

hair being interposed to prevent direct contact of the cover glass with the slide. A drop of acetone is allowed to flow under the cover glass so that it surrounds the particles of blood, and a drop of dilute mineral acid is then added. Crystals of acetone-haemin are soon formed from the blood pigment. Under high magnification innumerable small, often minute but quite characteristic, dichroic needle-crystals are seen. If only a trace of blood is present abundant crystals appear, even if the blood is old and partly decomposed. Preliminary extraction of the blood is not necessary. It is sufficient to soak blood-stained fabric in acetone and mineral acid. The material is soon covered with tufts of black needles. It is easy to detect 0·05 mg. of dried blood on fabric in this manner. A. O. J.

Colorimetric Determination of Urea in Blood and Biological Material, Cerebro-spinal Fluid and Tissues. J. A. Sanchez. (*J. Pharm. Chim.*, 1936, **128**, 188–189.)—Since nitrous acid and urea in equimolecular quantities are mutually destructive under defined conditions of temperature and time, any excess of nitrous acid will remain at the end of the reaction and may be determined. One ml. of a solution of sodium nitrite (2 in 10,000) and increasing quantities of a 1 in 10,000 solution of urea are placed in each of a series of test-tubes. The solutions are diluted to the same volume with water, and a layer of melted vaseline 2 cm. thick is added to each test-tube, followed by 30 drops of nitrite-free conc. sulphuric acid. After their contents are mixed the tubes are left for 25 minutes in a water-bath at 60° C., during which time the urea and nitrous acid react. Any excess of nitrous acid, which is out of contact of air, may then be colorimetrically determined by adding phenol sulphonic reagent and rendering alkaline with ammonia. D. G. H.

Application of the *o*-Phthaldialdehyde-reagent of W. Zimmerman to the detection of Small Amounts of Glycocoll and to the Determination of its Presence in Polypeptides. E. Aberhalden and A. Neumann. (*Hoppe-Seyler's Z. phys. Chem.*, 1936, **238**, 177–182.)—Glycocoll has been found to give a colour reaction with the orthophthaldialdehyde reagent of Zimmerman. As little as 2 ml. of a 0·1 per cent. solution gave an opaque blue colour which became a dark flocculation on standing. Increased amounts of glycocoll gave a dark violet, finely divided suspension which tended to flocculate on standing. Other amino acids gave either no colour or yellowish or reddish ones. Mixtures of glycocoll and other amino acids gave the characteristic glycocoll colour, and the same reaction was obtained with polypeptides containing this substance. S. G. S.

Biological Decomposition of Fatty Acids, Esters and Dicarboxylic Fatty Acids. B. Flaschenträger and K. Bernhard. (*Hoppe-Seyler's Z. phys. Chem.*, 1936, **238**, 221–232.)—Coconut oil and cooking fat (coconut oil, 80; and dripping, 20 per cent.) produced small amounts of sebacic and suberic acids, when ingested by the dog. The body fats, after such feeding tests, contained fatty acids of the C_8, C_9 and C_{10} series. Salts, methyl esters and ethyl esters of the C_6, C_7, C_8, C_9, C_{10}, C_{11}, C_{12}, C_{14}, C_{16}, and C_{18} fatty acids gave caprylic, nonoic, capric, and small amounts of dicarboxylic acids. When sebacic and other dicarboxylic acids were used in the diet, most of them were recovered in the urine unchanged, although some had partly undergone β-oxidation. S. G. S.

Fat Metabolism in Plants, with Special Reference to Sterols. P. L. MacLachlan. (*J. Biol. Chem.*, 1936, **113**, 197–204.)—The changes in the sterol-content have been compared with those of the total fatty acid content, before and after germination, in the light and in the dark, for the mammoth yellow and black Wilson soya beans. It was found that, although the total fat-content diminished as germination proceeded, there was a continuous production of sterol, which was greater during growth in the dark than during growth in the light. It was also found that the sterol became esterified during the period of rapid fat utilisation. These results indicate a relationship between the fat and sterol metabolisms in plants similar to that which is known to exist in animals.

S. G. S.

Some Colourless Substances found with Plant Carotenoids. L. Zechmeister and P. Tuzson. (*Hoppe-Seyler's Z. phys. Chem.*, 1936, **238**, 204–209.)—Daucosterol, first isolated from the carrot by v. Euler and Nordenson, has been found, on hydrolysis, to yield one molecule of sitosterol ($C_{29}H_{50}O$) and one molecule of glucose, and is therefore a sitosterol-*d*-glucoside, $C_{35}H_{60}O_6$. From the petals of the sunflower five crystalline, colourless substances have been isolated. These are hentriacontane ($C_{31}H_{64}$), a wax-alcohol ($C_{24}H_{49}OH$), a univalent sterol, the glucoside of another univalent sterol and a bivalent sterol. The univalent sterol, $C_{30}H_{52}O$, is different from sitosterol or stigmasterol. The glucoside $C_{33}H_{56}O_6$ gave, on hydrolysis, one molecule of glucose, an aglucone, $C_{27}H_{46}O$, and smaller amounts of similar bodies. The bivalent sterol probably has the formula $C_{21}H_{36}O_2$, but is similar to that obtained from calendula flowers, and named "helisterol," $C_{26}H_{44}O_2$. It has two hydroxyl groups and gives a crystalline di-acetate. S. G. S.

Glutamine and Asparagine in Tobacco Leaves. H. B. Vickery and G. W. Pucher. (*J. Biol. Chem.*, 1936, **113**, 157–160.)—The presence of glutamine and asparagine in tobacco leaves has been established; from 13·4 kg. of mature leaves, 17·9 g. of asparagine and 6·04 g. of glutamine were obtained. The presence of these substances explains the production of ammonia when tobacco-plant tissue is boiled with water; but this does not exclude the possibility of the presence of other amides or amide-like substances. The need for further study for the understanding of the amide metabolism of plants is emphasised. S. G. S.

Toxicological

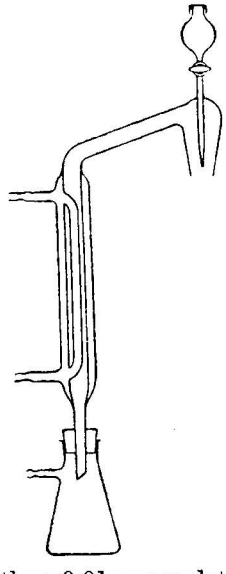
Toxicology of Selenium. I. Study of the Distribution of Selenium in Acute and Chronic Cases of Selenium Poisoning. II. Urinary Excretion of Selenium. H. C. Dudley. (*Amer. J. Hyg.*, 1936, **23**, 169–180; 181–186.)—I. Various animals (hog, horse, cow, steer, calf, and sheep) were fed with sodium selenite or with selenium-bearing plants so as to produce fatal results in 6 hours to 3 days. Selenium was then found distributed throughout the organism in widely varying proportions, the liver, kidneys and spleen (4·0 to 25·0 p.p.m.), and the liver and kidneys (3·0 to 25·0 p.p.m.), carrying the greatest quantities in the stages of acute and chronic poisoning, respectively; in the latter case

concentrations of 8·0 to 20·0 p.p.m. were also sometimes found in the hoofs. The presence of 7·0 to 27·0 p.p.m. of selenium in the blood suggests that by this medium the element is transported to all parts of the body and is deposited mainly in the above organs. Since in both acute and chronic cases 1·0 to 6·0 p.p.m. of selenium were found in the bile and 0·1 to 5·0 p.p.m. in the urine, it is concluded that elimination occurs mainly by the hepatic and renal pathways. The whole blood of a horse containing 0·2 p.p.m. of selenium (derived from sodium selenite) was fractionated, when the centrifuged corpuscles contained 0·3 p.p.m., and the clot, after prolonged standing in the cold, 1·0 p.p.m., whilst selenium was absent from the serum, plasma or fibrin. Haden's modification of the Folin–Wu method (ANALYST, 1923, **48**, 501) showed that the selenium in the hoofs was present as a protein complex, and extraction and fractional distillation of the urine proved that in this case the selenium had formed a volatile compound soluble in ether. It is considered that hoof deformities observed in range stock pastured on "alkali" land may be due to the replacement, by selenium, of sulphur in certain amino acids which are utilised to form modified proteins. Less than 0·2 p.p.m. of selenium was found in the organs and body fluids of animals receiving normal food (*vide infra* II). The determinations (which are calculated on the fresh body-weight of the sample) are made on 20 to 100 g. of fresh diced sample by the method described by Dudley, Byers and others (*Ind. Eng. Chem., Anal. Ed.*, 1934, **6**, 274; 1935, **7**, 3), the organic matter being oxidised by 30 per cent. hydrogen peroxide and nitric acid, and the solution evaporated almost to dryness. The mixture is then heated with 10 ml. of strong sulphuric acid to remove the nitric acid and to carbonise any residual organic matter, and the residue is distilled in the presence of hydrobromic acid and an excess of bromine. The selenium in the distillate is precipitated by sulphur dioxide and hydroxylamine hydrochloride, and, after filtration, the precipitate is re-dissolved in a 0·2 per cent. solution of free bromine in hydrobromic acid, the total volume being 25 ml. The pink colour produced when the selenium is re-precipitated, as described above, in the presence of gum arabic is matched against standards prepared in a similar way; an allowance should be made for the result of a blank test on the reagents.

II. The method of analysis previously described (*vide supra* I) has been used to demonstrate the presence of selenium in the urine of 20 workers exposed to selenium or selenium oxide dust from a process for the extraction of selenium from the electrode slimes of a copper refinery. The amounts found varied from a trace to 6·9 p.p.m. (expressed on the whole urine), but they bore no apparent relationship to the occurrence or otherwise of the usual symptoms, *viz.* marked pallor, coated tongues, gastro-intestinal disorders, nervousness, and a garlic odour of the breath; in general, the effects are less pronounced with older men of heavy stocky build. Normal urine contains no trace of selenium, but 6 p.p.m. were found in the urine of a worker 2 days after he had received a bad burn on the face, hands and arms with a hot mixture of sulphuric and hydrobromic acids containing large quantities of selenium. This shows that selenium can be absorbed through the skin, although in this case no symptoms were produced, and no selenium was detectable in the urine after 2 weeks. J. G.

Application of the Nitro-sulpho-perchloric Acid Method of Destruction of Organic Matter to the Toxicological Determination of Arsenic. E. Kahane. (*J. Pharm. Chim.*, 1936, **23**, 5–22.)—During the destruction of biological material by nitro-sulpho-perchloric acid, preliminary to the determination of arsenic, some arsenic is volatilised with the acid. In the determination of arsenic in medicinal organic compounds (cf. *J. Pharm. Chim.*, 1934, **19**, 116–123; Abst., ANALYST, 1934, **59**, 356), this loss is negligible, but in toxicological analysis, in which about 200 g. of viscera are used and very small amounts of arsenic are present, the method must be modified.

Destruction of Organic Matter.—A vertical condenser is fixed on a vacuum flask connected with a water-pump. The upper part of the inner tube of the condenser is bent twice; below the second bend it forms a pear-shaped bulb, which dips into a 2 l.-Pyrex flask, but is not attached to it. In the flask are placed 200 g. of the substance to be analysed and 50 ml. of concentrated sulphuric acid, and the mixture is heated while concentrated nitric acid is added, drop by drop, from a dropping funnel fixed in the bulb of the condenser tube (see Figure). As the acid turns brown with rise of temperature, when 180 to 200° C. is reached the nitric acid is replaced by pure perchloric acid or a mixture of 2 vols. of perchloric acid and 1 vol. of nitric acid. The total volumes of acid used are approximately 150 ml. of nitric acid and 30 ml. of perchloric acid. The maximum amounts of arsenic permissible in the sulphuric, nitric, and perchloric acids used are 0·02, 0·01, and 0·2 parts per million, respectively. Any traces of arsenic carried over during the distillation are recovered by adding the condensed liquid to the residue and re-distilling.



Determination of Arsenic.—Amounts of arsenic greater than 0·01 g. are determined in the sulphuric acid residue by a modified bromate process (I) (Abst., ANALYST, 1934, **59**, 356). Amounts from 0·001 to 0·01 g. are determined by precipitating as sulphide after dilution, filtration and washing on an ashless filter; a blank determination, by (I), is made on the residual acid after destruction of filter paper by the nitro-sulpho-perchloric acid method. For amounts of arsenic below 0·001 g., the arsenic is obtained by the magnesium-ammonium-phosphate precipitation, which carries down the arsenic as arsenate (cf. *Ind. Eng. Chem., Anal. Ed.*, 1931, **5**, 58–60), and the determination is made (*a*):—for 0·01 mg. to 1 mg., by Bougault's method (*cf.* Abst., ANALYST, 1907, **32**, 325), or (*b*):—for 0·001 to 0·05 mg., by Cribier's method (*cf.* Abst., ANALYST, 1921, **46**, 517). E. B. D.

Organic

Grouping of Organic Solvents and Compounds by means of Magdala Red. H. Eichler. (*Z. anal. Chem.*, 1935, **103**, 425–427.)—At ordinary temperatures organic solvents either cause a certain degree of dissociation of dissolved substances (*e.g.* the lower alcohols) or develop, themselves, a definite degree of

hydrogen- or hydroxyl ion concentration (*e.g.* the lower fatty acids). Solvents developing definite *p*H values may be detected by adding as indicator a dye which gives specifically coloured or fluorescent solutions. Magdala red is especially suitable for this purpose. A solution of the dye is yellowish-red and not fluorescent at *p*H 2, red and fluorescent at *p*H 4, whilst in alkaline solutions it loses its fluorescence. (Salm, *Z. phys. Chem.*, 1907, **57**, 500; *cf.* Eichler, Abst., ANALYST, 1934, **59**, 303). In the Magdala red reaction the fluorescence depends, therefore, upon the solvent properties of the liquid (in the substances of Group II, which, as a rule, do not dissolve the dye, the fluorescence does not appear), and upon the *p*H value of the solution, since the substances of Group I (which lie in the acid region of the *p*H scale) give a fluorescence, whilst those of Group III, which lie in the alkaline region, give no fluorescence. The fact that Magdala red gives fluorescent solutions in water and in dilute acids at higher, but not at ordinary, temperatures, is explained by variations in the degree of dispersion of the dye and in the degree of dissociation of the solvent with temperature (Schoorl, *Rec. Trav. chim. Pays-Bas*, 1921, **40**, 616). The behaviour of solvents towards filter-paper stained with an aqueous suspension of Magdala red is also specific (Eichler, *Z. anal. Chem.*, 1935, **100**, 183; Abst., ANALYST, 1935, **60**, 274). If the solvents are associated with coloured or turbid substances, they may be separated by distillation, the condensate being then tested with Magdala red or with the paper. Since the presence of water affects the test, it is necessary to fix it by adding dehydrating agents before distillation, and due regard must be paid to the action of the dehydrating agents upon the solvent. Phosphorus pentoxide, for example, fixes basic substances, but does not affect the detection of fatty acids. Alkaline dehydrating agents, such as sodium and potassium hydroxides and calcium oxide, fix fatty acids and saponify esters, so that it is the alcoholic components of the latter which appear in the distillate. The caustic alkalis convert many aldehydes into alcohols and salts of the corresponding acids (Cannizaro's reaction), but do not affect the detection of basic substances. Anhydrous copper sulphate may also be used as a dehydrating agent. Alcohols, ketones nitrobenzene, aliphatic, and aromatic hydrocarbons are not usually affected by the dehydrating agents mentioned. By their reaction to Magdala red or Magdala red paper, substances can be differentiated into the following groups:—(I) Compounds which dissolve the dye forming fluorescent solutions at ordinary temperature and which redden Magdala red paper:—Simple and polybasic alcohols, lower ketones, fatty acids, aldehydes, Turkey red oil, molten mono- or poly-basic phenols and their aqueous solutions; nitrobenzene dissolves Magdala red, giving a solution only faintly fluorescent. Addition of substances of Group III, especially the aromatic amines, causes the fluorescence to disappear. Aqueous solutions of these substances, such, for example, as the alcohols and fatty acids do not give the Magdala red reaction, at least not at the ordinary temperature. (II) Substances which do not dissolve Magdala red either at ordinary or at higher temperatures and which do not affect Magdala red paper:—Aliphatic and aromatic hydrocarbons and their halogen derivatives, fats, carbon disulphide, ether, fatty and essential oils. Mixtures of these substances with sufficient ethyl alcohol dissolve the dye, with red fluorescence. If the substances are immiscible with alcohol, the dye dissolves only in the alcohol

layer, giving a fluorescent solution. (III) Compounds which dissolve the dye without fluorescence, and redden Magdala red paper:—Aromatic amines and heterocyclic bases. On acidifying these with sufficient glacial acetic acid or concentrated formic acid the fluorescence appears. After separation of substances into these three groups there still remain substances which do not affect Magdala red paper, and in which the dye dissolves with fluorescence only at higher temperatures. These are water, aqueous fatty acid solutions and dilute mineral acids having a *p*H value of about 4. (*Cf.* ANALYST, 1935, **60**, 274.) A. O. J.

Action of Periodic Acid on Tartaric Acid. P. Fleury and G. Bon-Bernatets. (*J. Pharm. Chim.*, 1936, **23**, 85–98.)—Periodic acid acts rapidly on tartaric acid, yielding glyoxylic acid according to the equation

(1) $COOH–CHOH–CHOH–COOH + HIO_4 = 2COOH–CHO + HIO_3 + H_2O$

A further reaction has now been detected, in which glyoxylic acid is slowly oxidised by periodic acid, giving carbonic and formic acids as products

(2) $COOH–CHO + HIO_4 = CO_2 + HCOOH + HIO_3$.

The first reaction occupies 5 to 10 minutes, and the second is complete in 36 to 48 hours at ordinary temperature or in 2 hours at 37° C. These reactions proceed concurrently, and it was not found possible to suppress reaction (2). It is pointed out that the occurrence of reaction (2) does not invalidate Malprade's method (*Bull. Soc. Chim.*, 1934, **1**, 833), in which potassium periodate is employed in neutral solution, and the amount of this reagent consumed in the oxidation of the tartrate is determined by titrating (to thymolphthalein indicator) the alkali liberated according to the reaction (3) $K_2H_3IO_6 \rightarrow O_2 + KIO_3 + H_2O + KOH$. In Malprade's method oxidation of glyoxylate is not revealed, since the potassium hydroxide liberated is automatically neutralised by the equivalent amount of formic acid produced, thus: $COOK–CHO + O + H_2O = KHCO_3 + HCOOH$.

S. G. C.

Determination of the Refractive Index of Fats in Oilseeds by means of Bromonaphthalene. W. Leithe. (*Z. Unters. Lebensm.*, 1936, **71**, 33–44.)—The method is as follows:—Two g. of seed are very finely ground for 2 minutes, with sand, in a porcelain mortar. Exactly 3 ml. of bromonaphthalene are added, the mixture is ground for a further 2 minutes and then filtered through a porcelain suction-filter, and some drops of the filtrate are placed in the Abbé refractometer, and their refractive index is determined; that of the solvent is also determined at the beginning and end of a series of measurements at the same temperature. For temperatures between 15 and 25° C., the difference is usually independent of the actual temperature; it is therefore sufficient to keep the temperature constant (± 0·1–0·2° C.) at room temperature. From the results, and by the use of the known refractive index of the oil, the amount of oil in the solution can be found by the method of calculation for mixtures. Hence the oil-content of the seed can be obtained. The refractive index of the oil, n_D^{20}, is corrected to room temperature. If the value for the particular sample taken differs considerably from the average value for that oil, determination of the refractive index of the sample would be necessary. This method gives results from 0·1 to 0·3 per cent. lower than the extraction method,

in which difficultly-soluble constituents of seed are extracted in amounts depending on the time of extraction. The method can also be used for the determination of fat in cakes, etc. Bromonaphthalene is practically non-volatile at room temperature, and its refractive index differs considerably from those of edible oils. E. B. D.

Determination of Semicarbazide and Semicarbazones. V. Harlay. (*J. Pharm. Chem.*, 1936, **128**, 199–204.)—The hydrazine group is determined iodimetrically after the hydrolysis of the semicarbazide by heating with a dilute mineral acid. The substance to be hydrolysed and 30 ml. of 20 per cent. sulphuric acid are heated nearly to b.p., and steam is passed through to remove any aldehyde liberated. Hydrolysis may be regarded as complete in 8 to 10 hours. The hydrazine is then oxidised by means of 0·1 *N* iodine solution, and, after 20 minutes, the unused iodine is titrated with 0·1 *N* sodium thiosulphate solution. Very satisfactory recoveries were obtained by this method with semicarbazide hydrochloride, semicarbazones of the ketones, acetone, acetophenone, methylheptenone, methylnaphthylketone, carvone, cyclohexanone, trimethylcyclohexanone and camphor, and with the semicarbazones of benzaldehyde, acetaldehyde, anisaldehyde and vanillin. In general, the nearer the ketone grouping is to the carboxyl, the more difficult is hydrolysis, and the method cannot be used for the determination of formaldehyde, cinnamic aldehyde or citral, nor can it, owing to secondary reactions, be regarded as a method of general application. D. G. H.

Inorganic

Gravimetric Determination of Lead as Chromate. L. Guzelj. (*Z. anal. Chem.*, 1936, **104**, 107–119.)—The precipitation of lead as chromate was found to give accurate results when applied to dilute nitrate solutions, even in presence of large quantities—exceeding those usually introduced in the course of a determination—of ammonium acetate or acetic acid (*cf.* ANALYST, 1934, **59**, 61). Precipitation in nitric acid solution gave correct results at acidities not exceeding 0·1 *N* (*cf.* ANALYST, 1936, 61). The addition of ammonia in presence of copper or silver is not to be recommended, as it leads to the formation of basic lead chromate and consequent low results, although this can be counteracted by an excess of precipitant or by ammonium acetate. Potassium chloride or bromide, ammonium sulphate or nitrate, do not influence the results provided ammonium acetate is present. The determination of about 1 g. of lead sulphate dissolved in ammonium acetate solution (10 parts of salt to 1 of precipitate) gave slightly positive errors (a little less than 0·1 per cent.). Larger quantities of lead sulphate could be determined with a maximum error of +0·3 per cent. The technique followed consisted in adding the precipitant (2 per cent. ammonium chromate or 0·25 *M* potassium dichromate, solution), drop by drop, to the boiling lead solution, the operation lasting about 5 minutes. After 2 minutes' boiling, the liquid was left overnight, filtered through a porous porcelain crucible, washed with hot water until the washings were colourless, and ignited for 10 minutes at about 600° C. W. R. S.

Volumetric Determination of Lead and of Molybdate with Eosin as Adsorption Indicator. C. Candea and I. G. Murgulescu. (*Ann. Chim. anal.*, 1936, **18**, 33–36.)—Eosin acts for practical purposes as a reversible adsorption indicator for detecting the equivalence-point of the reaction $Pb^{++} + MoO_4^{--} = PbMoO_4$. With lead ions in slight excess the precipitate is red, whilst with excess of molybdate the precipitate is yellow. The precipitate does not flocculate before the equivalence-point is reached, so that the colour-change is easy to observe. The nature of other ions present has an effect on the sensitiveness of the colour change. *Titration of Lead.*—The neutral solution of lead as nitrate (0·1 to 0·2 g. in 100 ml.) is acidified with nitric acid, 2 or 3 drops of methyl orange solution (0·02 per cent.) being added as indicator (the free acid present should not exceed 0·0005 *N* as otherwise the adsorption indicator fails to act). Twenty drops of eosin A solution (0·5 per cent. in water) are added, and the solution is titrated with *N*/20 sodium molybdate solution until the precipitate, which is reddish at first, changes to yellow. In the titration of lead acetate, the solution should be acidified with acetic acid to make it 0·01 *N* in free acid. Lead acetate solutions may also be titrated with ammonium molybdate solution, but this last reagent does not give a satisfactory colour-change with lead nitrate. *Titration of Molybdate.*—The sodium molybdate solution (100 ml. containing 0·025 to 0·05 g. of molybdenum) is neutralised to phenolphthalein with nitric acid, 20 drops of eosin indicator are added, and the liquid is titrated with *N*/20 lead acetate solution until the precipitate changes to red. With ammonium molybdate the quantity of molybdenum present should be within 0·01 to 0·03 g. of molybdenum in 100 ml., larger amounts having been found to give low results. Tests of the above titration methods gave results close to the theoretical. S. G. C.

Corrosion of Tinplate. T. P. Hoar. (*Tech. Pub. International Tin Research and Dev. Council, Series A, No.* 30, 1936, 11 *pp. Reprinted from Proc. Swansea Tech. College Met. Soc.*, 1936.)—Corrosion of tinplate vessels by various foodstuffs and liquids in domestic use is discussed from the standpoint of (*a*) a perfect tin surface, (*b*) tin coatings having small discontinuities at which the steel base is exposed, (*c*) the steel base. In fruit canning a number of factors incidental to the composition and processing are favourable to prolonged resistance of sealed cans to corrosion. As oxygen present is kept small in amount by exhausting the can before sealing, the oxygen-depolarisation type of attack is minimised. The attack of tin by dilute organic acids is practically negligible in absence of air. Tin is electrochemically anodic to iron in presence of fruit acids, and therefore any corrosion of tin which may occur will result in a cathodic (reducing) condition being set up at steel exposed at discontinuities in the tin coating and tend to suppress corrosion at these points, reducing danger of perforation. Proteins and carbohydrates tend to restrain corrosion of steel by acids, and tin ions present in solution are also known to have a similar effect. On the other hand, fruits which contain reducible substances, such as the anthocyanin colouring matter in certain red fruits, or nitrate contained in the pumpkin, give trouble by stimulating corrosion; cans are lacquered internally to prevent this. Discontinuities in the lacquer film are often coincident with a break in the tin coating, and at these points the steel

base is denied cathodic protection as the tin is insulated. Lacquered cans are thus liable to perforate more readily than plain cans, although they are superior in other respects. Attack of unlacquered fruit cans gives an etched appearance to the tin surface, which is objectionable because consumers frequently regard it as an indication of lack of quality in the fruit; it is thus important to canners that corrosion should be rendered as small as possible. Discoloration by a blackish film of cans containing fish, certain vegetables, and cream, is attributed to iron sulphide generated at pores in the coating. In water and neutral saline liquids tin is highly resistant to attack, and is generally cathodic to steel; as a result rust-spots appear at pores. Tin coatings of minimum porosity are therefore advantageous, and in this connection a process has recently been devised for reducing or eliminating porosity (for special applications), consisting in electrodeposition of a thin layer of tin on top of the hot-dipped coating. S. G. C.

Volumetric Determination of Palladium. M. Gahide. (*Bull. Soc. Chim. Belg.*, 1936, **45**, 9–14.)—The solution is acidified with 15 ml. of 12 *N* sulphuric acid and treated with 15·0 ml. of a 1 per cent. solution of the reagent (1 g. of salicylaldoxime in 5 ml. of alcohol, diluted to 100 ml. with distilled water). After 6 to 10 minutes' settling the liquid is filtered into a Kjeldahl flask, and the precipitate is washed 10 times with cold water. The filtrate is boiled for 15 minutes, 50 ml. of a 4 per cent. ferric sulphate solution being added after 10 minutes while ebullition is continued. The flask is cooled under the tap, 3 ml. of phosphoric acid are added, and the solution is titrated with 0·1 *N* permanganate. Another 15-ml. portion of the reagent is titrated after addition of 45 ml. of water, 15 ml. of 12 *N* sulphuric acid, and boiling with ferric sulphate as before. The difference between the two titrations gives the equivalent of the palladium (0·002665 g. per ml. of 0·1 *N* permanganate). Indirect titration is necessary on account of the very slight solubility of the precipitate in mineral acid. Copper, if present, causes high results. Dimethylglyoxime is not suitable for the volumetric method. W. R. S.

Volumetric Determination of Nickel in Presence of Cobalt. G. Charlot. (*Bull. Soc. Chim.*, 1936, **3**, 324–326.)—The solution is neutralised, and potassium cyanide solution is added in amount sufficient just to re-dissolve the precipitate first formed, and then half as much again. Bromine is added, drop by drop, with shaking, until a slight excess is present (3 or 4 ml. of bromine are usually sufficient). This results in the precipitation of a mixture of nickel cyanide and nickel cobalticyanide. Then 40 to 50 ml. of potassium hydroxide solution (36° Bé.) are added, which precipitates black nickel oxide; bromine is added until this precipitation is complete; the liquid should remain strongly alkaline. The temperature must be kept below 50° C. in order to avoid destruction of cobalticyanide. The liquid is just acidified with hydrochloric acid to dissolve the nickel oxide, and subsequently boiled to remove liberated bromine. Oxalic acid is now added, in small portions at a time, until gas is no longer evolved; the solution is boiled for 5 minutes, cooled, rendered slightly ammoniacal, and diluted to 200 ml. *Titration of nickel.*—A measured excess of standard potassium cyanide solution (5 per cent.; 30 ml. are sufficient for about 0·2 g. of nickel) is added, and the solution is titrated with

standard nickel solution (a solution of 10 g. of nickel in nitric acid, rendered slightly ammoniacal and diluted to 1 l.) until a drop of the liquid withdrawn and placed on dimethylgloxime test paper (filter-paper impregnated with an alcoholic solution of the reagent and dried) gives a pink colour. The nickel equivalent of the potassium cyanide solution is determined by diluting a suitable volume of it to about 200 ml., making it slightly ammoniacal, and titrating with the standard nickel solution, as described above. The degree of precision is stated to be within 1 mg. of nickel. When only small amounts of nickel (4 to 5 mg.) are present, more dilute titrating solutions should be used, *viz.* potassium cyanide, 0·5 per cent.; nickel, 1 g. per l.; the accuracy is within 0·2 mg. of nickel. S. G. C.

Gravimetric Determination of Selenates. P. Spacu. (*Bull. Soc. Chim.*, 1936, **3**, 159).—To the hot solution (100 to 150 ml.), feebly acid with nitric or acetic acid, is added concentrated lead acetate solution acidified with a few drops of acetic acid. The precipitate of lead selenate is allowed to settle, and, after cooling, about 5 per cent. of alcohol is added; the precipitate is filtered off, washed with water (either hot or cold), then with 3 or 4 2-ml. portions of 96 per cent. alcohol, and finally with 3 or 4 2-ml. portions of ether. The precipitate is dried for 10 minutes *in vacuo* and weighed as lead selenate ($PbSeO_4$). In tests with pure solutions of potassium selenate results very close to the theoretical were obtained. S. G. C.

Microchemical

Microchemical Studies of Artificial Sweetening Substances. I. Saccharin. II. Dulcin. Vl. Staněk and P. Pavlas. (*Mikrochem.*, 1934–5, **16**, 211–222; 1935, **17**, 22–28; *Listy Cukrovarnicke*, 1934–35, **53**, 33.)—I. (SACCHARIN). —A simple method for the detection of saccharin has been described (*Z. Zuckerind. d. Tschech. Rep.*, 1933–4, **58**, 313), in which the saccharin diffuses from an acid solution into ether, where it is absorbed on paper impregnated with magnesia, and is identified by its sweet taste. This requires from 18 to 24 hours, as the rate of diffusion is very slow, only about 0·33 mg. diffusing in 24 hours from a solution containing 1 mg. in 100 ml. To accelerate the separation of saccharin the sample should be extracted several times with a suitable solvent (ether or ether and petroleum spirit), which can then be distilled off. With beer and other strongly emulsifying solutions this is impracticable, and diffusion with stirring is applied. The apparatus is a 200-ml. wide-mouthed bottle, closed with a cork impregnated with calcium chloride solution, through which is fitted a glass tube. The stirrer passes through the tube and is made of some non-corrodible metal, such as monel metal or silver. This is bent up twice at right angles, and the second horizontal portion extends into the upper part of the ethereal layer, and should support the magnesia paper. Varying concentrations of saccharin, from 0·25 to 1 mgm. in 100 ml. of beer, covered with a layer of 50 ml. of ether were tested for the time required to give a positive test when the stirrer was set at 80 revs. per minute and the paper contained 20 per cent. of magnesium oxide; it was found that 0·25 mg. of saccharin required 1 hour and 1 mgm. ¼ hour.

Quantitative Determination.—The saccharin is hydrolysed by heating with 20 per cent. sulphuric acid for 2 hours under a reflux condenser. On cooling, the liquid is neutralised with 30 per cent. alkali solution (free from carbonate), a slight excess of alkali is added, and the ammonia is distilled off in a 150 to 200-ml. flask through a steel or stainless steel condenser tube. The stoppers of the apparatus are best made of ground-glass. When less than 2 to 3 mg. of ammonia are present the receiver may contain water and methyl red as indicator; for larger amounts N/70 acid is used. For complete extraction of saccharin from the original solution the stirring should be carried out for 11 hours for quantities of saccharin up to 1 mg., whilst 33 hours is sufficient for amounts up to 5 mg. in 100 ml. of beer, 80 per cent. magnesia paper being used. The paper is prepared by shaking 20 g. of cut filter-paper in 500 ml. of 5 per cent. sodium hydroxide solution for 1 hour. It is then stirred with 80 g. of magnesia mixed with 300 ml. of water and coloured with litmus. The mixture is then drawn by suction on a cloth filter into a 2-mm. layer. This is well washed, dried, and cut into sections of 20 × 20 mm. Blank tests on similar material, saccharin-free, should be carried out to ascertain the absence of other substances that liberate ammonia. The presence of large amounts of ether-soluble acids (*e.g.* lactic acid in many lemonades) is indicated by the blue magnesia paper turning red. In that case more filter-paper must be used, otherwise the results will be too low. Errors of the order of a few per cent. were obtained in a number of determinations of saccharin in beer. *Chemical tests for saccharin.*—These are less sensitive than the tasting test. Schmidt's test (*Pharmaz. Zentralhalle*, 1887, **28**, 466) is applicable. Magnesia paper, on which the saccharin is absorbed, is placed on a watch-glass, moistened with a solution of magnesium permanganate (prepared from calcium permanganate and magnesium sulphate), and left on the water-bath until dry. The paper is then moistened with alcohol and re-dried. In this way any glycyrrhizin, salicylic or acetyl-salicylic acid is decomposed. If the tasting test is positive, the brown mass is stirred with water, the filtrate is taken up in a nickel or silver crucible and evaporated to dryness, and the residue is heated for 10 to 15 minutes at 220 to 230° C. with about 1 g. of a mixture of equal parts of sodium and potassium hydroxides. The melt is dissolved in a little water, acidified and extracted with ether. The ethereal solution is filtered and evaporated, the residue is dissolved in water, and the solution is neutralised with alkali (to litmus) and treated with a drop of 0·5 per cent. ferric chloride solution. The salicylic acid formed from the saccharin gives a violet colour. In the absence of salicylic acid or acetyl-salicylic acid the permanganate treatment and extraction are omitted. The amount detectable is 0·25 mg. of saccharin. Another test (sensitive to 0·1 mg.) depends on the formation of insoluble bromphenol. The solution of the ethereal extract, obtained as described above, is treated with bromine vapour until it turns yellow; the salicylic acid present shows a milkiness, and needles of bromphenol can be seen under the microscope.

II (DULCIN).—Jorissen's reaction (*Chem. Tech. Gärung. Nahrungs u. Genussm.*, 1922, **2**, 1462) is applied after extraction and concentration of the saccharin. One-hundred ml. of the sample (*e.g.* as beer) are clarified with 10 ml. of a saturated solution of copper sulphate and 20 g. of dried slaked lime. The precipitate is

filtered off and washed with 30 ml. of water. The filtrate is neutralised with acetic acid, treated with excess of sodium hydroxide, filtered, and shaken out three times with 50 ml. of ethyl acetate. The last time the solution is saturated with sodium chloride in order to salt out any dissolved ethyl acetate. The ethyl acetate is distilled off, the residue is dissolved in 2 to 3 ml. of alcohol, a minute quantity of yellow lead oxide is added, and the mixture is stirred and evaporated to dryness. The powdered residue is extracted with three successive portions of 5 ml. of ether, the combined extract is filtered and evaporated to dryness, and the residue is warmed on the water-bath with 1 ml. of water and 3 drops of Jonissen's reagent (mercuric nitrate). After 3 minutes' heating, 2 drops of cerium acetate solution are added. In the presence of dulcin a violet colour appears. Sometimes a yellow precipitate forms on heating the sample with mercuric nitrate; this must be filtered off through porcelain, while the liquid is hot, before adding the cerium acetate. It is also advisable to add a few drops of dilute acetic acid before the test; this dissolves some of the precipitate and renders the reaction clearer. The violet colour fades rapidly, but can be fixed with 1 to 2 drops of benzyl alcohol. *Reagents.*—(i) *Mercuric nitrate.*—Four g. of yellow mercuric oxide are dissolved in dilute nitric acid, and the solution is mixed with dilute alkali until a perceptible precipitate forms; this is filtered off and the solution is diluted to 25 ml. It is stable. (ii) *Ceric acetate.*—One g. of ceric nitrate or sulphate is dissolved in water, the solution is acidified with nitric or sulphuric acid, and treated with excess of ammonia. A spoonful of kieselguhr is added, the mixture is filtered, and the precipitate is well washed. The precipitate and filter are stirred up with water, 2 to 3 ml. of acetic acid are added, and the mixture is filtered. The filtrate and washings should make 50 ml. This solution, which should be stored in dark bottles, will not keep longer than a month. Alternatively, benzoyl peroxide may be used for the oxidation, but the test is then slightly less sensitive. The sensitivity, with ceric acetate, is about 0·25 mg. in 100 ml. of beer, or 0·05 mg. in 1 to 2 ml. of pure solutions of dulcin. Dulcin and saccharin may be identified together, as the above-described methods do not interfere with each other. A further reaction for dulcin is the formation of an intense yellow colour on heating the solution with a saturated solution of potassium nitrate in glacial acetic acid. This is very sensitive, but vanillin, phenol, salicylic acid, proteins and tyrosine also give the reaction. J. W. M.

Spot Tests for Organic Compounds. VIII. F. Feigl, V. Anger and O. Frehden. (*Mikrochem.*, 1934–5, **17**, 29–37.)—*Detection of dicarboxylic acids by conversion into fluorescein dyes.*—Dicarboxylic acids, of which the carboxyl groups are separated by at most two carbon atoms form fluorescein dyes on heating with resorcinol in concentrated sulphuric acid, and these fluoresce intensely in alkaline solution. The peri-dicarboxylic acids and their salts, esters, anhydrides, amides, imides and nitrites behave similarly. With nuclear nitrated aromatic *o*-dicarboxylic acids non-fluorescing dyes are formed; these can be made to fluoresce if the interfering nitro group is reduced to an amino group before the reaction. Maleinic acid and its derivatives give red to violet dyes with resorcinol, the fluorescence of which is visible only under the quartz mercury-vapour lamp.

Detail.—A few mg. of the substance under examination, or a few drops of the solution evaporated to dryness, are treated with a little freshly-sublimed resorcinol and a few drops of pure sulphuric acid and heated for 5 minutes at 130° C. On cooling, the crucible is placed in a 50-ml. beaker, the contents are washed out, and the solution is rendered alkaline with sodium hydroxide, when the green fluorescence is apparent in daylight. A blank test should be made.

Compound	Colour	Fluorescence (daylight)	Limit of identification γ
Oxalic acid	rose	yellow-green	15
Malonic ester	yellow	green	10
Succinic acid	yellow	green	5
Succinic anhydride	yellow	green	5
Succinimide	yellow	green	5
Potassium succinate	yellow	green	5
Asparagine	dark wine-red	dark green	5
Tartaric acid	red	blue-green	50
Tricarballylic acid	rose	grass-green	5
Phthalic acid	yellow	light green	5
Trimellitic acid methyl ester	yellow	light green	2·5
Naphthalic acid anhydride	yellow	dark green	5
Saccharin	yellow	green-yellow	10

Detection of β-keto carboxylic acid and α-hydroxycarboxylic acids.—When heated with formic-sulphuric acid hydroxy 1-2-dicarboxylic acids are converted into β-ketocarboxylic acids which react in their enolic form with resorcinol and concentrated sulphuric acid, forming umbelliferone compounds, which are colourless or yellow, and fluoresce blue in alkaline solution in ultra-violet light.

Name	Colour	Fluorescence (U-V. light)	Limit of identification γ
Acetic acid ester	pale yellow	sky-blue	2
Malic acid	yellow	bright blue	1
Citric acid	yellow	sky-blue	1
Tartaric acid	yellow	green-blue	25

Detection of citric acid by conversion into the fluorescent ammonium salts of citraconic acid.—A drop of the test solution is evaporated in a micro-crucible, and the residue is mixed with 4 drops of thionyl chloride and heated until fumes appear; then about 8 drops of a conc. aqueous solution of ammonia are added, and the mixture is boiled until only 2 drops remain. On cooling, 6 drops of conc. sulphuric acid are added, and the mixture is heated until fumes appear; the contents of the micro-crucible are then poured into a test-tube and rendered ammoniacal. When very large amounts of citric acid are present the fluorescence is apparent in daylight, otherwise a quartz mercury-vapour lamp is required. *Limit of identification.*—1 γ citric acid. *Concentration limit.*—1 : 50,000. J. W. M.

Microchemical Reactions of Novocaine. M. Wagenaar. (*Pharm. Weekblad*, 1936, **73**, 122–128.)—The free base (*p*-amino benzoyl diethylamino-ethanol, m.p. 62° C.) forms a white crystalline mass, insoluble in water, but soluble in alcohol, ether or benzene. Novocaine (hydrochloride, m.p. 51° C.), when crystallised from alcohol, forms white needles and prisms having a top angle of 120° (*vide infra*); its solubility is 1 : 1 in cold water, and 1 : 30 in cold alcohol. It does not crystallise readily when sublimed, even if the sublimate is treated with acetone, but it is precipitated from a solution in water by addition of ammonium chloride or hydrochloric acid, and the resulting crystals have a top angle of 60°, and show polarisation effects (sensitiveness, 0·1 mg., 1 : 50). Addition of a solution of a gold salt in the presence of hydrochloric acid and a small crystal of sodium acetate, produces fern-shaped crystals which are strongly doubly-refracting, and if sodium bromide also is present characteristic dark brown crystals result; platinum salts behave similarly to gold salts. Mercury salts produce a group of crystals and, in the presence of hydrochloric acid and sodium acetate, the sensitiveness is 0·025 mg. (1 : 1000). With picrolonic acid, bundles of star-shaped doubly-refracting crystals are formed, and if the ammonium salt is used, the sensitiveness is 0·002 mg. (1 : 1000). An amorphous precipitate, in which block-shaped crystals subsequently develop, is produced by ammonium picrate with 0·02 mg. (1 : 200), and if a mixture of drops of potassium dichromate and sample (0·025 mg., 1 : 100) is scratched on a microscope slide, characteristic crystals having a top angle of 78° are produced. One drop of bromine water forms a yellow amorphous precipitate, and 2 drops produce bundles of fine crystals, but if the solution of the sample is very dilute, a drop of it should be exposed on a microscope slide to the bromine vapour produced by adding hydrochloric acid to a mixture of potassium bromide and potassium bromate. The theory of the formation of coloured reaction products with furfural (*cf.* ANALYST, 1932, **57**, 579) is discussed. A 15 per cent. solution of furfural in oleic acid (in which novocaine is soluble) should be used, and if a crystal of the alkaloid is added to this, a red tinge is produced around it; this, incidentally, enables the top angle of 120° (*vide supra*) to be seen more easily.

J. G.

Physical Methods, Apparatus, etc.

Explosibility of Agricultural and other Dusts as Indicated by Maximum Pressure and Rates of Pressure Rise. P. W. Edwards and L. R. Leinbach. (*U.S. Dept. Agric., Tech. Bull.*, No. 490, Oct. 1935, 1–24.)—Methods of determination of explosibility are:—(1) The open system in which the explosion travels into an open gallery, the length of the flame produced when the dust in the gallery is ignited being measured; alternatively, the quantity of inert dust necessary to prevent or limit propagation of the flame may be estimated. (2) The closed system, in which the maximum pressure developed when a dust-cloud inside a gas-tight bomb is ignited is measured; in this case the average and maximum rates of rise in pressure are also valuable indications of explosibility. The latter type of apparatus, which was used in the present instance, consisted of a spherical bronze bomb (1,417 ml.) fitted with ignition electrodes and connected by a tube

with a piston in a recording manometer (*cf.* Rice, Frazer, Larsen, Haas and Scholz, *U.S. Bur. Mines Bull.*, 1911, **20**, 204). This piston actuated a calibrated flat steel spring attached to a stylus which, in turn, produced records on a chart on a revolving manometer drum, enabling time intervals to be read with an accuracy of 0·001 second. Investigations were made with 100 and 500 mg. of dust per l. of air, the weighed material being placed in a small hemispherical dust-cup, and blown as a uniform cloud into the bomb by an air-jet (*cf.* Trostel and Frevert, *Chem. and Met. Eng.*, 1924, **30**, 141). The trigger actuating this operation simultaneously closed the circuit for igniting the dust-cloud (at about 1,800° C.) by means of a pellet consisting of 0·1 g. of an equimolecular mixture of magnesium and barium peroxide; since the former is oxidised and the latter is reduced, the oxygen-content of the atmosphere is unaffected, while the solid products of combustion form a drop of slag between the electrodes. The 133 dusts tested comprised 50 food-products or by-products (flours, milk powders, casein, grain-elevator dusts, etc.), 25 spices, drugs and insecticides, 23 wood, paper and tanning materials, 5 fertilisers (bones, flours and meals), 8 resins, waxes and soaps, 11 carbon and coal products, 5 metals (aluminium being used as a check to ensure that the apparatus always gave consistent results), and rubbers, sulphur, ivory nuts and indigo. Tables show these materials classified into categories in terms of their explosibility, account being taken of the maximum pressure, and the maximum and average rates of rise in pressure; the importance of the concentration of the dust is emphasised, since the ratios of the values for the 500-mg. dust to the 100-mg. dust vary from 0·5 to 7·5, 0·5 to 5·3, and 0·4 to 4·8, respectively. The rate of rise in pressure determines the amount of damage done, and if it is not too high the load on the structure may be released adequately by breakages of windows or through other vents. Alkali starch, lycopodium, soap powder, sodium resinate and candelilla wax head the list with explosibility values of 10; aluminium is rated at 7, and steamed bone, tobacco-stem dust, animal charcoal, anthracite, graphite, gold bronzë and indigo have values of zero. J. G.

Reviews

REACTIONS OF ORGANIC COMPOUNDS. By W. J. HICKINBOTTOM, D.Sc. Pp. x + 449. Longmans, Green & Co., Ltd. 1936. Price 16s. net.

This is a first-rate book; its aim is to present the facts of organic chemistry from the point of view of laboratory practice. In the opinion of the reviewer this is a sound method of approach to the science, and the author has been eminently successful in working out his idea.

A comprehensive account is given of the reactions of typical groups, and includes some instructive paragraphs dealing with the limitations of "general" reactions.

The book abounds in detailed accounts of methods of preparation of a wide variety of substances—some chosen from quite recent literature, *e.g.* Chattaway's convenient method of acetylating phenols (1931).

A large amount of information has been rendered easily accessible by the 24 tables, which include data of the physical properties of classes of compounds and of their commoner derivatives. The great utility of the book is enhanced by the copious and well-arranged index.

It is inevitable that some recent references and improvements in procedure should have escaped even the eagle eye of the author, and the following have been noticed:—The improved method of preparing resorcylic acid (*Organic Syntheses*, X, 94) might have been given (p. 101); in the section on acid anhydrides (p. 195–6) one of the most convenient methods of preparation, the treatment of an ethereal suspension of the acid containing 1 mol. of pyridine with 1 mol. of thionyl chloride (D.R.P. 201,325; *cf. J. Chem. Soc.*, 1929, 69) is not mentioned; in dealing with Gabriel's method of preparing primary amines (p. 227) no reference is made to the modifications devised by Ing and Manske (*J. Chem. Soc.*, 1926, 2348), which avoid the preparation of potassium phthalimide and facilitate hydrolysis of the alkyl phthalimide.

An appendix of 20 pages provides a scheme for the identification of organic substances, which will doubtless prove a great help to the examinee.

Many text-books are borrowed by the student from college libraries; this is one which he should buy—*and use.* J. KENYON

ELECTROLYTIC OXIDATION AND REDUCTION. By S. GLASSTONE, D.Sc., Ph.D., F.I.C., and A. HICKLING, M.Sc., Ph.D. (Volume IX of a Series of Monographs on Applied Chemistry, edited by E. HOWARD TRIPP, Ph.D.). Pp. ix + 420. London: Chapman & Hall, Ltd. 1935. Price 25s. net.

In view of the increasing interest that has been shown during recent years in electrolytic oxidation and reduction, this volume should be of great value to research workers and others engaged in chemical industry. No attempt has been made to give practical methods in detail, and the reader is recommended to consult original papers or other sources for information upon experimental procedure. Emphasis has been laid upon "the basic theoretical significance of the observations and . . . the optimum conditions for any process."

The first three chapters, occupying 87 pages, are devoted to a discussion of fundamental facts and theories. The principles enunciated in these chapters are intended to act as a guide to the processes discussed in the subsequent systematic treatment of oxidation and reduction phenomena. All the important aspects of reversible electrode potentials, polarisation, over-voltage and diffusion phenomena in electrolysis are dealt with in a lucid manner and in sufficient detail to enable them to be applied to the problems of practical electrolytic work.

The remainder of the book, consisting of eight chapters, deals systematically with the study of reversible and irreversible electrolytic processes in both inorganic and organic systems.

Three chapters are devoted to the special subjects of the polymerisation of anions, the anodic behaviour of fatty acids, and anodic substitution.

The comprehensive bibliographies appended to each chapter should be particularly valuable to the reader who desires further information. In all there

are over 1130 entries giving references to original papers, text-books and patent specifications.

The underlying principles of many industrial processes are discussed, but chlorine-alkali manufacturing methods have not been described, since they are "adequately discussed elsewhere."

A peculiar error has escaped detection on pages 28, 112 and 113, where the valencies of ceric and cerous ions are erroneously indicated as $Ce^{\cdots}$ and $Ce^{\cdot\cdot}$, respectively. The book is well produced and can be recommended to serious workers in chemistry, especially to those engaged in electro-chemical work.

H. J. LINDSEY

THE CHEMICAL CONTROL OF CONCEPTION. By JOHN R. BAKER. Pp. x + 173. London: Chapman & Hall, Ltd. 1935. Price 15s.

This book is concerned with discussing the experimental methods and the results obtained in what was mainly a chemo-therapeutic investigation. Dr. Baker has been engaged for many years in these studies, in which he is, of course, the pioneer.

His description of the technique required for making comparable suspensions of highly active live sperm (of which the guinea-pig is almost the only source used in experimental work) and of the standard test designed to eliminate as far as possible every anticipated variable *except* the composition of the compound being investigated, and his discussion of the mode of action of spermicides occupy Chapters II, III and IV of this book; they afford an excellent example of the scientific way to approach a laboratory problem, as well as of the proper relationship between experimental aims, technique and conclusions.

In Chapters I, V, VI, and VIII, Dr. Baker is concerned with certain applied aspects of his work, and these chapters will consequently be of rather less interest to analysts in their purely professional capacity. Dr. H. M. Carleton's chapter discusses the pathological bearings of part of Dr. Baker's book, which concludes with a postscript containing certain "stop-press" results, and with a number of useful appendixes. There is a good index, and the production of the book calls for little but praise. Its price, however, does not show a normal correlation with size.

A. L. BACHARACH

THORPE'S DICTIONARY OF APPLIED CHEMISTRY SUPPLEMENT. Vol. III. GLOSSARY AND INDEX. By J. F. THORPE, C.B.E., D.Sc., F.R.S., F.I.C., and M. A. WHITELEY, O.B.E., D.Sc., F.I.C. Assisted by Eminent Contributors. Pp. vii + 166. London: Longmans, Green & Co. 1936. Price 21s. net.

Every industry acquires a vocabulary of technical words which are familiar everyday terms within the industry, but the exact meaning of which is often a matter of guesswork to those outside. It was, therefore, a happy thought of the editors of "Thorpe's Dictionary" to issue a glossary of such words and phrases, and in this work they have had the assistance of a large number of specialists, whose help is acknowledged in the preface.

The need for such a glossary is amply shown by a single reference to this volume, for the word "bloom" has a different connotation according to whether

it is applied to leather, cocoa, milling and baking products, oils, rubber or varnish. In addition to terms, such as this, with specialised industrial usages, the glossary also defines numerous chemical terms of comparatively recent introduction, many of which will be unfamiliar to those who are not working on the respective subjects. For example, "Bömer's difference number" will usually convey as little to the chemist who has not specialised in oils, as will "epimerism" to those who are not sugar chemists. There is also included a large selection of terms used in modern conceptions of physical and organic chemistry, such as, for example, "Spin isomerism," "L-radiations," "Hofmann degradation," and "Chelate groups."

From what has been said it will be seen that the volume will be found a useful supplement to any chemical dictionary; in a sense, it is itself a dictionary in miniature.

The last part (pp. 101–166) contains a full index to the previous two supplementary volumes of "Thorpe" for which the present editors are responsible. They are to be congratulated on the completion of their task of bringing the main work up to date. EDITOR

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