THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

THE Annual General Meeting of the Society was held at the Chemical Society's Rooms, Burlington House, on Wednesday, March 4th, when the President, Dr. J. T. Dunn, delivered his Annual Address.

The following were elected as Officers and Council for the year 1931:

President.—Dr. J. T. Dunn.

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

Vice-Presidents.—A. More, W. Partridge, G. Roche Lynch.

Hon. Treasurer.—E. B. Hughes.

Hon. Secretary.—F. W. F. Arnaud.

Members of Council.—A. L. Bacharach, F. H. Carr, C. H. Cribb, H. E. Cox, E. M. Hawkins, A. E. Johnson, D. W. Kent-Jones, H. M. Mason, W. G. Messenger, G. W. Monier-Williams, G. Stubbs, A. R. Tankard.

An Ordinary Meeting of the Society then followed, the President, Dr. J. T. Dunn, being in the chair.

Certificates were read for the first time in favour of:—K. N. Bagchi, B.Sc., M.B., D.T.M., William Nelson Bradshaw, B.Sc., Adrian Joseph Clifford Lickorish, F.I.C., Ernest Grenville Purser, B.Sc., A.I.C., and William Waddell Robson.

Certificates were read for the second time in favour of:—Cecil Chilvers, B.Sc., F.I.C., Jack Hubert Hamence, M.Sc., A.I.C., Cecil John House, B.Sc., A.R.C.Sc., F.I.C., and Henry George Rees, B.Sc., A.R.C.Sc., A.I.C.

The following were elected Members of the Society:—Kenneth Bullock, M.Sc., Ph.D., and Frederick Cecil Hymas, B.Sc., A.I.C.

The following papers were read:—"The Investigation of Japanese Beeswax," by H. Ikuta; and "The Denigès-Oliver Test for Morphine," by J. Bamford.

Annual Report of Council

March, 1931.

THE Roll of the Society stands at 661, an increase of 41 over the membership of last year.

With great regret the Council has had to report the death of:

Edward William Voelcker, who served the Society as Treasurer for many years, and as President during 1910–1911.

The Council also deplores the loss of two honorary members and three ordinary members:—

H. W. Wiley ("Obituary," ANALYST, 1930, **55**, 728).
Ludwig Moser.
Arthur Angell ("Obituary," ANALYST, 1930, **55**, 308).
Henry Leffmann.
A. MacLean Wright.

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

"The Determination of Minute Amounts of Iodine in Soils and Waters." By R. L. Andrew, F.I.C.

"Preliminary Studies in the Bacteriology of Wheat and Flour." By D. W. Kent-Jones, Ph.D., B.Sc., F.I.C., and A. J. Amos, B.Sc., A.I.C.

"The Separation of Metals by 'Internal Electrolysis'." By H. J. S. Sand, D.Sc., Ph.D., F.I.C.

"The Rapid Determination of Bismuth and Copper in Lead Bullion by Internal Electrolysis." By Ella M. Collin, B.Sc., A.I.C.

"Notes on the Thiocyanate Method of Determining Iron. Influence of Different Classes of Phosphates." By G. Winthrop Leeper, M.Sc.

(i) "The Spectroscopic Investigation of Jams and the like." (ii) "A Simple Polarimetric Test for Sugars in Jams." (iii) "A New Kjeldahl Distillation Apparatus." By S. Judd Lewis, D.Sc., F.I.C.

*"The Determination of Cadmium and Copper in Spelter and Zinc Ores by Internal Electrolysis." By Ella M. Collin, B.Sc., A.I.C.

"The Routine Detection of Nitrates in Milk." By A. F. Lerrigo, B.Sc., F.I.C.

"A Method for the Determination of Titanium as Phosphate." By J. C. Ghosh, D.Sc.

"The Diastatic Activity of Honey." By L. H. Lampitt, D.Sc., F.I.C., E. B. Hughes, M.Sc., F.I.C., and H. S. Rooke, M.Sc., A.I.C.

*"A New Method for the Separation of Titanium from Zirconium and Hafnium." By A. R. Powell and W. R. Schoeller, Ph.D.

"The Composition and Polymerisation of Chinese Wood (Tung) Oil." By E. R. Bolton, F.I.C., and K. A. Williams, B.Sc., A.I.C.

"The Examination of Milk for Tubercle Bacilli." By D. R. Wood, F.I.C.

"Scientific Evidence relating to Firearms, with Special Reference to a Recent Murder Trial." By G. W. Baker, F.I.C.

*"The Composition of Rye Oil." By J. W. Croxford, A.I.C.

* Work done under the Society's Analytical Investigation Scheme.

"The Determination of Unsaponified Oil in Soap or Fatty Acids." By G. E. Lester Smith, M.Sc., A.I.C.
"The Analysis and Composition of Vegetable Parchment used for Packing Dairy Products." By Paul Arup, M.Sc., F.I.C.
"The Determination of Milk Proteins." By George M. Moir, M.Sc., Ph.D., A.I.C., Pedler Research Scholar of the Institute of Chemistry.
"The Lead Reduction Method for the Volumetric Determination of Tin, and the Interference by Copper and Antimony with it." By S. G. Clarke, B.Sc., Ph.D., A.I.C.
"A Storage and Delivery Apparatus for Antimony Chloride and other Corrosive Reagents." By G. Middleton, B.Sc., A.I.C.
"Tests for Impurities in Ether." Parts II and III. By G. Middleton, B.Sc., A.I.C., and F. C. Hymas, B.Sc., A.I.C.
"The Determination of Small Quantities of Calcium in Magnesium Salts." By Norman Evers, B.Sc., F.I.C.
"A New Method for the Detection of Nitro-Group in Organic Compounds." By P. K. Bose, D.Sc.

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

"Drinking Waters for Cattle." By T. MacLachlan, F.I.C.
"The Freezing Point of Milk as a means of Detecting added Water." By G. D. Elsdon, B.Sc., F.I.C., and J. R. Stubbs, M.Sc., F.I.C.
"Gas Fatalities due to the Slow Oxidation of Vegetable Refuse." By A. R. Tankard, F.I.C., and D. J. T. Bagnall, A.I.C.
"The Examination and Commercial Analysis of Cotton Cloths." By R. H. Kay, A.T.I.
"The Reichert–Polenske–Kirschner Values of Rancid Butters and Margarines." By G. D. Elsdon, B.Sc., F.I.C., R. J. Taylor and P. Smith.
"The Detection of Benzoic Acid in Food." By A. N. Leather, B.Sc., F.I.C.

The pages of the ANALYST also contained a considerable number of other original papers and notes.

The Editor of the ANALYST, Dr. C. A. Mitchell, reports an increased sale of the journal, the size of which equalled the pages in the volume for the previous year, which was the largest volume published up to that date.

Reference to the Treasurer's statement, which is circulated to members separately, shows that expenses, as in the past, have been successfully met.

Reports were received from three of the Sub-Committees of the Standing Committee on Uniformity of Analytical Methods, namely:

Essential Oils Sub-Committee (two Reports).
Milk Products Sub-Committee.
Metallic Impurities in Food Colours Sub-Committee.

These have been published in the pages of the ANALYST. They have necessitated a very large amount of work by members of Sub-Committees, and an expression of appreciation of the work was forwarded, on behalf of the Council and of the Society, to the Standing Committee.

The Council express their appreciation of the facilities afforded to members of the Society for the use of the library of the Chemical Society during the year.

The Council received a communication from the Association of British Chemical Manufacturers with regard to the formation of a British Chemical Standardising Body. The Society was represented at a Conference at which this question was discussed, and a small Committee was appointed by the Conference to report to them.

A representative International Committee met at Geneva to discuss International Methods of Oil Analysis, Mr. E. R. Bolton being appointed representative of the Society. The Committee decided that a Conference should be held in different countries annually, and that each country should be represented by one individual only.

A Dinner held by the Society on the 4th March, at the Savoy Hotel, was largely attended and very successful. Among the many important guests were :— The Rt. Hon. Arthur Greenwood, M.P., Minister of Health ; The Rt. Hon. Lord Tomlin, Lord of Appeal ; The Rt. Hon. Lord Riddell ; and the Presidents of the Chemical Society, the Institute of Chemistry, the Society of Chemical Industry, the Institute of Brewing, the Institution of Chemical Engineers, and the Institution of Petroleum Technologists.

A letter was received from the Ministry of Health asking for observations with respect to the standardisation of glassware for testing milk and milk products. The Council supported the specifications contained in the publication of the National Physical Laboratory issued in April, 1927, but at the same time expressed the view that it did not approve of centrifugal methods for the determination of fat in milk, except as approximate methods.

A Cheese Bill was presented to Parliament. The composition of cheese had consequently been considered by the Council, who made the following proposals :

(1) That a statutory standard for fat in cheese was desirable.

(2) That the legal minimum of fat in cheese should be 45 per cent. of milk fat in the dry substance.

(3) That all cheese made from skimmed milk should be labelled "Skim Milk Cheese," with a further declaration of "three-quarter fat," "half fat," or "one-quarter fat," according to the proportion of milk fat contained in the milk from which the "Skim Milk Cheese" was prepared. Minimum limits for the fat occurring in the dry substance of "three-quarter fat," etc., "Skim Milk Cheese" should be laid down.

The Conference of Members of the Food Manufacturers' Federation and Public Analysts has issued its report on the standards for jam, and these standards have been circulated in the form of a final memorandum. This has also been published in the ANALYST.

J. T. DUNN, *President.*

F. W. F. ARNAUD, *Honorary Secretary.*

Annual Address of the President.

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

Delivered at the Annual General Meeting, held on March 4, 1931.

LADIES AND GENTLEMEN,

It has long been customary for the President to begin his address by an account of the position of the Society in membership and activities, quoting statistics and enumerating papers read during the year. All this is contained in the report of Council, and it seems unnecessary to repeat it in detail; but it is gratifying to find that we are steadily gaining numerical strength in membership. Both this circumstance, and the fact that the sales of THE ANALYST are increasing, are testimony to the excellence of our journal, and a tribute to the solid and careful work of the Publication Committee, and especially to the ability and untiring industry of our Editor, Dr. Mitchell, to whom the Society owes so much.

A Society like ours, which has passed its jubilee, must expect every year to have to record the passing of some of its older members; and this year is no exception to the rule. But of those who have come within "Time's bending sickle's compass," whose names we see in the Council's report, and among whom are some of considerable eminence, particularly in connexion with the analysis of foods, there is one in especial whom I must mention, because of his long and intimate association with our Society. Edward William Voelcker was elected a member in April, 1889, forty-two years ago; and during the whole of that long period, as member, as Member of Council, as President, and, above all, as Treasurer, he has given of his best to the Society. His financial ability, and the wisdom and moderation of his counsel—ability and wisdom never blatantly displayed, but perhaps even a little hidden from immediate recognition by the modesty of his demeanour, and the geniality and kindliness of his nature—have undoubtedly largely helped towards the growth of the Society and the attainment of its present prosperous position. We miss not only his ready help and advice, but his personal charm which all of us must have felt. He is become for us a fragrant memory, and the contemplation of his life and work cannot but be a stimulus and an inspiration.

The papers read during the Session are enumerated in the Council's report. I should like here only to draw attention to the wide range of the subjects treated in them, and to point out that almost half of them have no connection with Food and Drugs or the official work of the Public Analyst. The subjects cover practically the whole range of analytical chemistry, and testify to the wisdom of the policy which, a few years ago, added "Other Analytical Chemists" to the membership of the Society of Public Analysts.

THE NORTH OF ENGLAND SECTION.—This Section, I am glad to think, continues a vigorous existence with an increasing membership, and is becoming an affluent towards the membership of the parent Society. It held last July a week-end summer meeting at Scarborough, which was well attended and very successful. Glancing through old volumes of the ANALYST, I see that the Society was in the habit of holding a country meeting once a year, and it is worth while, I think, asking whether the practice might not usefully be revived. The opportunities for social intercourse which such a meeting gives are very valuable. Another feature of the Section's session was a visit to one of the factories of the Co-operative Wholesale Society, who entertained the members to lunch and tea; which indicates that the C.W.S. was not ashamed to submit its processes to the gaze of the Public Analysts, nor its products to the judgment of their palates.

CO-OPERATION WITH MANUFACTURERS.—The Council's report for last year mentioned that, at the invitation of the Food Manufacturers' Federation, conferences between their representatives and the Public Analysts' Committee were taking place, on the question of standards for jam, and you now know that agreement has been arrived at, and that a memorandum has been circulated to the members of the Federation, setting forth the standards to be adhered to, and the mode of labelling the jam. All of the members of the Federation have signed the declaration agreeing to abide by these standards; and I take it that our position as Public Analysts will be to look upon any sample of jam, not labelled "Lower Fruit Standard," as purporting to be "Full Fruit Standard," and to report against it if it should fall short of the requirements of that standard.

I regard this arrangement as of very great importance and with a considerable degree of satisfaction. In the early days of the Food and Drugs Act, and indeed for many years after its institution, the Public Analyst was looked on as more or less the natural enemy of the manufacturer—the earlier volumes of the ANALYST contain frequent references to this attitude—and any sort of co-operation between them would have been thought almost an impossibility. Gradually, as the various branches of the manufacture of food products have become more scientific—as manufacturers in increasing numbers have themselves called in the aid of the chemist to control and extend their work, and have more and more realised that success lies in the direction of raising the standard of excellence of their wares rather than in actually (though not professedly) lowering it, that feeling has died out; and of late years, and especially since the chief producers of different food products have banded themselves into associations, and have seen that it is to their advantage not only to keep up the standard of their own products, but to endeavour to prevent others of lower morality from spoiling their markets by the presentation of inferior or adulterated articles at lower prices, we find the manufacturers helping the Public Analysts, and in turn asking for their help. Three years ago we had the chemists of the Food Manufacturers' Federation placing unreservedly before us the results of their investigations into methods for the determination of the preservatives used in foodstuffs; and now we have the

Federation asking for our co-operation in fixing reasonable standards of quality for jam.

And this example seems likely to be followed. During last year the National Farmers' Union and the Cheshire County Council endeavoured to promote a bill in Parliament with the view of checking the sale in this country of inferior cheeses as Cheshire cheese; and they approached your Council to know whether or not it would give them backing. The Public Analysts' Committee considered the Bill, and, whilst giving it general approval, suggested alterations and additions for its improvement, and their proposals were approved by Council. No definite result has yet been arrived at, but the fact that the help of the Society was sought by the producers is very significant.

Quite lately the Council has been approached by another branch of the Food Manufacturers' Federation to see whether agreement can be arrived at on the question of rice flour in suet. The Public Analysts' Committee is now considering this, and I can, of course, say nothing on the question itself, but again chronicle the fact of the producers' endeavour to work in harmony and co-operation with the Public Analysts.

If these conferences continue and spread, we may be within measurable distance of having, not, indeed, legal standards, but at least agreed standards of composition of many foodstuffs, which will acquire in the courts the force and authority of legal standards; and we shall no longer have the anomaly of the same substance being held in one place to be genuine and in another adulterated.

And, as all this tends in the direction of informing the public as to what it is actually buying, I have hopes that it may go further yet, and that we may agree more upon definitions and descriptions; so that we shall not have tapioca sold as sago; shall know when we have a custard that it is not made from cornflour; and that a lemonade syrup shall not be said to be "made from the finest Messina lemons," if it has no nearer connection with them than the fact o fits containing citric acid.

Our experience with the jam manufacturers showed that we can attain results in conference that no amount of written correspondence would reach. Each side was able to put before the other its point of view, of which previously the other side was quite oblivious, and which hence it was unable to appreciate. One, at least, of our proposals was met at first by direct and absolute refusal, and yet we were able to show that it was desirable, even in the jam manufacturers' own interest, and to carry their acceptance of it. And we learnt something of the difficulties of the manufacturers, when we found that things on which we had come to agreement with them raised strenuous opposition on the part of the retailers, to whom they sold their products. So, give and take were both necessary; and if the new situation is not quite ideal in the view of some of us, it is at least a working arrangement which marks a considerable advance on the state of things that preceded it, and gives us reasonable hope of further progress in the same direction.

The jam manufacturers and their research chemists found that in black-currant jam made from pulp it was very difficult, sometimes impossible, to get

down to the maximum permitted quantity of sulphur dioxide: 40 parts per million; and Professor Roberts, of Liverpool, and I, who had both had some experience, not only of samples taken under the Food and Drugs Acts, and containing more than the permitted quantity, but also of the honest but unsuccessful efforts of local jam manufacturers to comply with the regulations, had also come to the conclusion that the requirement was too stringent. Along with Mr. Macara and two of the jam manufacturers' chemists, therefore, we had an interview with Dr. Hamill at the Ministry of Health, and put before him facts and figures on the question. So far we have heard of no result of the interview, but I mention it as another illustration of friendly co-operation where formerly there was antagonism and distrust.

Co-operation in Forensic Work.—Speaking of co-operation between manufacturers or their associations and analysts leads me to mention another aspect of work in which co-operation is extremely desirable, and, in my experience at least, frequently lacking. I refer to chemico-legal or toxicological cases, involving the examination for poisons of the organs of animals or of human beings. Here you have the veterinary surgeon or the medical practitioner and the analyst both involved, and it would seem eminently desirable that they should examine the organs together in the first place. But this is seldom done, and the organs are handed over to the analyst, frequently in a way that should not happen. I have two extreme cases in mind, both of them recent. In one we received a stomach for examination, the whole contents of which had been removed and the stomach washed. In the other, the whole of the intestines, with their contents, of a person supposed to have been poisoned, were sent to us, apparently without any previous examination. On opening them we found growths in such numbers and of such a size as seemed to us to be in themselves sufficient to account for death. My partner and myself were of course not competent to say definitely whether or not that was so; but had the medical man been present with us at the examination he could have given an authoritative opinion, and the search for poisons might not have been necessary. On the other hand, in another recent case, where the organs first came into the possession, not of a medical practitioner, but of a professor of pathology, he brought them to me and we opened and examined them together, so that each was furnished at the outset with the observations and inferences or suggestions of the other. It seems a great pity that this is not the usual practice.

Sale of Milk Regulations.—We are every now and then brought up against efforts of one kind or other to minimise the effective application, in the Courts, of the Sale of Milk Regulations. Last year's report of Council referred to a pamphlet of the Board of Agriculture, "Variations in the Composition of Milk," which was so obviously one-sided and unfair in its presentation and interpretation of the figures it contained, that your Council found it necessary to send the letter of protest that was published in the Analyst of August, 1929. This year there was brought to the notice of Council, in the Journal of the Ministry of Agriculture, an account of a meeting in May of the "Council of Agriculture for England," at which, among other business, there was moved and adopted a report on the "Law

Relating to the Sale of Milk," which the mover said would be "brought to the notice of the Ministry of Health, with a view to seeing how far action could be taken in accordance with the spirit of the representation."

The report proposes to remove milk from the operation of the Food and Drugs Act, and to place it under a special Act. Under this Act the presumption of adulteration, which the vendor must rebut, when the milk falls below specified minimal contents of fat or of non-fatty solids, would disappear, and punishment under the criminal law would only be inflicted if wilful adulteration were actually proved by the prosecution (no specified penalties are suggested); but there would be an implied warranty that all milk sold should contain at least 3 per cent. of fat and 8·5 per cent. of non-fatty solids (though sellers would be at liberty to issue specific warranties guaranteeing either more or less than these quantities, presumably obtaining correspondingly higher or lower prices), and failure to maintain the standard guaranteed by the warranty would be a civil offence, punishable by the award of five shillings damages, if the case were proved in a County Court.

There is something to be said for the infliction of civil penalties only in certain Food and Drugs cases; and occasions do arise where a tradesman is branded with the stigma of criminal conviction under circumstances that do not warrant so severe and far-reaching a penalty. But the probable effect on the general milk-supply of the country of such an alteration in the law as is suggested in this report, where the maximum penalty, for what would always be a breach of contract and might frequently be deliberate fraud, would be "the award of five shillings damages," is very obvious. It is so obvious, I think, as to make the likelihood of the alteration ever coming into operation negligibly small; but I mention it because at the meeting in question the Minister of Agriculture was present, and in his speech said he "would like to emphasise how closely he and the whole Department attended to the recommendations and reports of the Council." Your Council is giving a watchful attention to the matter.

It is, perhaps, of interest to say that during last year I received a pamphlet from a large firm of food distributors on "Proposals for legislation amending the law as to the Sale of Milk." This made reference, apparently, to a former pamphlet which I had not seen, and went on to say: "We now put forward somewhat detailed proposals for milk legislation, which have been evolved. . . ." I read the pamphlet through, but took no further note of the proposals, in such a matter, of a private firm; but perusal of the report of the Council of Agriculture stirred up some dim remembrance in my brain, and I looked up the pamphlet again. I found, curious to say, that nearly the whole of the pamphlet is a verbatim transcription of the Council of Agriculture's report. I make no comment on the coincidence.

STANDARD METHODS OF ANALYSIS.—A year ago your President devoted a large part of his very interesting address to the question of standard methods of analysis. This is a question that from time to time has occupied the attention of the Society. I find that Mr. Rudd Thompson, in addressing you in 1925, referred

to standard methods that had then been adopted, here and abroad, and discussed the question generally; and Mr. Bolton, three years later, speaking of the same matter, said: "It will be clear to everybody that conditions have arisen requiring the institution of standard methods for certain purposes, and these standard methods must be provided," and criticised the Society for neglecting this work, and leaving it to be done, as in certain directions it was then being done, by the British Engineering Standards Association, an engineering body.

Last year your Society was asked by the Association of British Chemical Manufacturers to appoint delegates to a conference to consider the question of standards generally as applied to chemical products and to analytical methods, and Mr. Bolton and I duly attended on June 4th. The Conference agreed that the formation of a chemical standardising body should be attempted, and that it was desirable that such a body should form a part of a larger organisation dealing with all forms of standardisation rather than act independently; and a small committee was formed to explore the situation in conjunction with the B.E.S.A., and report to the Conference.

The Committee has just made its report, and recommends that there should be a general organisation to be called the "British Industrial Standards Association," and that it should consist of four divisions—Building, Chemical, Engineering, and Textile. Each division will be under the government of a representative council chosen from the bodies interested in its particular work, and, as far as that particular work is concerned, it will be autonomous, though it will be subject, in matters of general policy, to a general council, largely elected by the four divisions themselves. The constitution of the councils, and the relations between the divisional councils and the general council are still under discussion, and it would not be right for me to indicate the proposals which have so far been made in regard to them. I will only point out that, when the scheme is carried through, the chemical division, besides undertaking the standardising of substances or methods not yet dealt with, will take over for adoption or revision those parts of the work hitherto carried out by the B.E.S.A. which are chemical in their nature.

Whatever our views on standardised methods of analysis may be, we are forced to concede, I think, that under the present conditions of industry and commerce they are very frequently necessary, and that they are, in fact, insisted on by those who have the power to insist; and if we are to have them, it is all to the good that they should be elaborated by a general chemical body, on which this Society will always be able to make its voice heard; and which, as it prescribed the methods, will also be able to modify and improve them, as that may become necessary.

In parenthesis, I may perhaps express the pious hope that, when standardising is undertaken by a general chemical body, rather than by the particular industry concerned, more care will be exercised, especially where the methods of analysis, or of testing, involve the measurement of physical characters of one kind or another, to make sure, before insisting on a standardised instrument or form of apparatus,

that such a standardised instrument or form of apparatus is really necessary to obtain consistent results. Our friends, the Institution of Petroleum Technologists, for example, dealing with the question of the effect of cold on the viscosity of oils, prescribed for us a standard method of determining the "pour-point" of an oil, which involved a special apparatus. But in a second edition of their book they tell us that the "pour-point" is of no value; we must determine the "setting-point," which has no relation to, and gives results in no way comparable with those of the "pour-point" determination, but which requires another and different special apparatus. And they give us two alternative methods of inquiry into the carbonising qualities of an oil, embodying the names of Conradson and Ramsbottom, respectively. Some users specify Conradson; others, Ramsbottom. No doubt each gives results by which different oils can be usefully compared; but Conradson's results have no relation to Ramsbottom's, and the only feature common to both methods is that for each there is prescribed a special and expensive apparatus.

Now, to the firm or specialist who is doing a particular kind of work all day and every day, the purchase of a costly instrument to carry out the work is not very serious; but when the general practitioner is asked to carry out an operation which may not recur more than half-a-dozen times in a year, and finds that his results will not be accepted unless he has used Brown-Jones's automatic electropolar antiviscosimeter, or other prescribed instrument, which Messrs. Thermo and Muffle's latest gorgeous catalogue tells him will cost £45, and that there will be another £35 for the "accessories" that are necessary to make the instrument work, he is placed in a not very happy position, having to decide whether to refuse the work and perhaps lose a client who might give him other work besides, or to cut the loss, and risk getting repaid in other ways. It is quite possible for such a man to spend as much on apparatus as he makes in fees, and be left with nothing but the melancholy satisfaction of reflecting that he is serving his country, when he pays income tax on the income that he would have had, if it had not all gone in capital expenditure. That is perhaps an extreme instance; but I do seriously think that there is a strong tendency nowadays to rely too much on the instrument-maker, and too little on our own powers of manipulation, which tend, as a result, to become atrophied.

When you honoured me last year by offering me the presidency of the Society, I accepted it, though with great pleasure and pride at the thought that I was considered worthy of it, yet with some diffidence and trepidation; for, obviously, a President living so far away from London, and debarred from frequent personal consultation with the Secretary or the other members of Council, works under conditions of some difficulty; but I have had the loyal help of all the members of Council, whom I wish to thank for their uniform kindness, and among them perhaps I may without offence name especially my immediate predecessor, Mr. Hinks, for whose advice and assistance I am most deeply grateful. Mr. Arnaud, too, was most considerate, and spared himself no trouble in keeping me abreast of successive situations that arose, until he was laid off by that very severe illness, his recovery from which is matter for such sincere gratification to us all; and during

his enforced absence Dr. Mitchell took over his work and relieved me of the anxiety I might otherwise have felt at being bereft of a guide whom I felt I greatly needed. All this has made my year of office a time of most pleasurable memories. You have entrusted me with the office for a second year; I hope to carry out its duties acceptably; and should I fail, it will certainly not be for lack of either effort or inclination to fulfil them.

The Determination of the Milk Proteins.

By GEORGE M. MOIR, M.Sc., Ph.D., A.I.C.,
Pedler Research Scholar of the Institute of Chemistry, 1928–1930.

(*Read at the Meeting, November* 5, 1930.)

IV. A. THE COMBINED DETERMINATION OF ALBUMIN AND GLOBULIN.

Owing to the small proportion of globulin in milk most investigators have been content to precipitate the albumin and globulin together from the filtrate remaining after the removal of the casein. For very many years this separation has been effected by one of two processes, (*a*) by heat coagulation,[1] (*b*) by the use of Almen's tannic acid reagent. The fact that the first method gave distinctly low results was pointed out nearly thirty years ago by Simon,[2] and more recently by others[3,4,5].

These investigators all recommended the use of Almen's reagent, but, unfortunately, tannic acid may precipitate other material besides the albumin and globulin. This possibility is evident from the reports of workers[6,7,8] whose experiments show that tannic acid may precipitate peptones and also certain amino-acids, traces of which may be present in fresh cows' milk. Directions[14] to report albumin and globulin as the difference between casein and total nitrogen, have probably led to even higher values being published. The tendency to high values has no doubt been accentuated also by the fact that casein cannot be completely removed by the use of acetic acid alone.

Other reagents which have been used for the removal of protein material are trichloroacetic, phosphotungstic and phosphomolybdic acids. Evidence is available[8,9,10] which favours the first of these.

Preliminary experiments were carried out in order to compare trichloroacetic and tannic acids as milk protein precipitants. For this purpose quantities of 20 ml. of the same milk were pipetted into beakers and treated in different ways. To A, 20 ml. of 10 per cent. trichloroacetic acid were added and the mixture filtered after standing about half-an-hour. To B, 20 ml. of 10 per cent. trichloroacetic acid were added, and the mixture maintained at a temperature of 60–65° C. for

half-an-hour before cooling and filtering. To C, 5 ml. of water, 5 ml. of 10 per cent. trichloroacetic acid and 20 ml. of absolute alcohol were added, and filtration commenced about half-an-hour later. To D, 20 ml. of Almen's tannic acid solution were added, the mixture filtered after standing about half-an-hour, and the precipitate washed with water.

In each case the complete filtrate and washings were collected for nitrogen determination by Kjeldahl's method. The duplicate non-protein-nitrogen results, which appear in Table I, are typical of others similarly obtained, and demonstrate

TABLE I.

Milligrams non-protein-nitrogen per 100 ml. milk.

A	B	C	D
45·2	36·3	36·8	30·8
41·0	36·3	37·8	31·6

the following points: Trichloroacetic acid in the cold gave distinctly high and irregular N.P.N. figures. The use of alcohol with more dilute acid gave rather lower but still irregular N.P.N. figures. In both these cases besides the irregular figures, the opalescent filtrates and washings were unsatisfactory. By warming the trichloroacetic acid mixture, clear filtrates and very good agreement of duplicates were always obtained. Still lower N.P.N. figures were regularly obtained by the use of tannic acid (see also Table II).

Further experiments were carried out upon the filtrate obtained after removing the casein from 250 ml. of milk by the isoelectric method previously described. In order to obtain a clear liquid, the mixture was filtered only after some hours, or next morning, so that the finest casein particles could accumulate upon the larger ones.

Portions of 50 ml. of the filtrate were carefully pipetted into small beakers for removal of the soluble protein by various treatments. Usually after precipitation of the protein, the filtrate therefrom was collected in a beaker in which it was boiled for a few seconds. The appearance of a further precipitate showed incomplete removal of protein, so that the necessity for completing the washing and nitrogen determination of the original precipitate could be eliminated. In using trichloroacetic acid, sufficient 20 per cent. or 50 per cent. solution was added to make the final concentration 4 per cent. Lower concentrations (2·5 per cent.) sometimes gave slightly low results, while higher concentrations were avoided because of the work of Hiller and Van Slyke,[9] which indicated that heating in the presence of strengths of 5 per cent. and over was liable to cause hydrolysis. Two tannic acid solutions were tried—A, Almen's, consisting of 240 ml. of 50 per cent. alcohol together with 10 ml. of 25 per cent. acetic acid and 5 grms. of nitrogen-free tannic acid; B, consisting of 10 grms. of tannic acid, 2·5 grms. of sodium acetate, 2·5 grms. of sodium chloride, and 5 ml. of glacial acetic acid, made up to 200 ml. with water. For the 50 ml. portions of filtrate, 10 ml. of A were used or 5 ml. of B.

Trials were made to see if the heat coagulation method could be improved by repeated heating of the protein solution at intervals of some hours, accompanied

by replacement of the acetic acid volatilised by the heat. In other trials, when the protein was precipitated by heating on the water-bath with sufficient magnesium sulphate or sodium sulphate to saturate the cold solution, washing with water was found to dissolve some of the precipitate. This occurred even after repeated heating of the saturated solutions in the presence of sufficient acetic acid; consequently, washing had to be carried out with saturated salt solution. Filtration and washing required to be completed as quickly as possible, a pleated filter being used to facilitate the operation. After rinsing out the beaker two or three times on to the filter, washing could be completed with small amounts of solution if this were allowed to drop from a pipette round the margin of the paper. Alternatively, by means of a small wash-bottle of saturated solution, the filter could be sprayed with a fine stream. After each application the paper should be allowed to drain well, but not for so long that evaporation causes crystallisation to occur. To avoid this difficulty, watch glasses may be used to cover the filter funnels.

Attempts were made to use trichloroacetic acid for the purpose of rendering less soluble the precipitate from saturated salt solutions. These attempts were unsuccessful, partly because in such solutions the protein floated on the top and could not be reached by the dissolved acid. Provided the solutions were not over-heated, the results differed little from those obtained similarly without the use of trichloroacetic acid.

TABLE II.

Expt. No.	Repeated heating with acetic acid only.	4 Per cent. CCl_3COOH. added before heating.	4 Per cent. CCl_3COOH. added after heating.	Saturated $MgSO_4$ and acetic acid.	Saturated Na_2SO_4 and acetic acid.	4 Per cent. CCl_3COOH. with saturated $MgSO_4$.	4 Per cent. CCl_3COOH. with saturated Na_2SO_4.	Cold 4 per cent. CCl_3COOH.	Tannic acid A	Tannic acid B
1	7·4	10·5	10·2*	10·7	9·8*	11·4	11·2	Precipitation incomplete*	11·5	11·2
	7·4	10·4	10·2*	10·7	10·0*	11·3	11·0		11·1	11·2
2		11·4	11·5	11·7	11·6	11·4	11·6		11·9	11·8
		11·5	11·4	11·5	11·5		11·7		11·9	11·8
3		8·2	8·1*	8·5	8·4	8·5	8·5	6·7*	8·7	8·5
		8·2	8·1*	8·4	8·3*	8·4	8·5		8·6	8·5

The figures are mg. of nitrogen precipitated from each 50 ml. portion of solution used.

* In these cases opalescence appeared either in the filtrate after boiling or in the washings.

The results of three separate comparative experiments are given in Table II, and these are typical of others similarly obtained. It is remarkable that, in the case of Experiment 1, repeated heating of the protein solution with acetic acid by itself should precipitate only about 70 per cent. of the protein and yet leave a filtrate which remained clear even on further boiling. Heating of the solution with trichloroacetic acid gives precipitates which filter rapidly, and can be easily washed (with about 1 per cent. acid). If this acid is added after heating, the results may sometimes be very slightly low; whilst if the heating is omitted, precipitation is incomplete at low concentrations.

The use of saturated salt solutions (with or without trichloroacetic acid) regularly gave results which were slightly higher than those obtained by using trichloroacetic acid alone. Various reasons can be suggested for this difference. First, the difficulty of washing the gelatinous precipitate with a solution which tends to crystallise on the filter paper may make it impossible to remove all the non-protein material. Second, the experiments of Wasteneys and Borsook[8] suggest that the saturated salt solution may precipitate any traces of "proteose" material which may be present in milk, but which may not be precipitated by trichloroacetic acid. Third, slight hydrolysis of the protein may be initiated during the heating with the trichloroacetic acid. The last possibility is suggested by some results of Hiller and Van Slyke;[9] but, on the other hand, the action of trichloroacetic acid in rendering proteins insoluble has been emphasised by the experiments described by Loeb.[11]

A number of reagents for the removal of the total protein from milk were studied by Simon,[2] who favoured especially tannic or phosphotungstic acid. A careful study of his figures leads to the conclusion that in the majority of cases tannic acid precipitated slightly more nitrogen than trichloroacetic acid, whilst phosphotungstic precipitated more than tannic acid. This comparison of tannic and phosphotungstic acids has been confirmed by the results (Tables I and VII) of Grimmer, Kurtenacker and Berg[12] upon milk sera, while my experiments have emphasised the differences in the amounts of material removed from milk serum by tannic and trichloroacetic acids. Since these experiments were carried out uranyl acetate (10 ml. of 1·57 per cent. per 10 ml. of milk, diluted to 50 ml.) has been recommended by Kopatschek[13] for the determination of the non-protein-nitrogen content of milk, but I have not yet been able to compare it with other reagents.

As a result of my experience I prefer trichloroacetic acid for the purpose of removing albumin and globulin from milk sera; but, before making a final decision, experiments ought to be carried out to determine the action of trichloroacetic acid upon pure solutions of the milk proteins. In the meantime, the following method is suggested :

PROPOSED TENTATIVE METHOD.—To the filtrate obtained after the iso-electric precipitation of the casein, sufficient trichloroacetic acid is added to make the final concentration approximately 4 per cent. The mixture is heated for half-an-hour on the boiling water-bath, and, after standing to cool, it is filtered and washed with a 1 per cent. solution of trichloroacetic acid. The nitrogen content of the precipitate is estimated by Kjeldahl's method.

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B. THE SEPARATE DETERMINATION OF ALBUMIN AND GLOBULIN.

The accurate analytical separation of the two soluble milk proteins is practically the same problem as the determination of the albumin and globulin of blood serum. The difficulty of this problem is obvious to anyone who reads the extensive literature thereon.

Howe[1] has critically reviewed the various methods which have been proposed for the estimation of serum proteins and has made the statement that "until independent procedures are devised for the identification of the proteins, 'salting out' must remain as the point of departure for future work." This agrees with statements made by Cohn[2] and Schryver.[3] Methods involving the use of the refractometer or viscometer seem to be less satisfactory than "salting out," associated with either gravimetric or Kjeldahl determinations.[4,5].

In deciding what procedure should be adopted for any particular investigation, consideration must be given to the object in view. For certain purposes the methods which Howe[6] has applied to colostrum would provide a series of comparative values of the same degree of accuracy throughout. Such methods are liable to a slight constant error, due to the volume of the precipitate, a fact which Howe apparently realised (p. 65, *loc. cit.*). He worked with colostrum from which the fat had been removed, so that the application of his methods to milk or colostrum containing the fat would magnify the error, since the fat would be precipitated with the protein. The error might be eliminated by washing the precipitate and determining the nitrogen content of the filtrate and washings, instead of using an aliquot part only of the filtrate. Alternatively, duplicates might be precipitated at different dilutions so that a calculation could be made to discover the error due to determinations on aliquot portions of the filtrates. Thus more accurate absolute values for the albumin and globulin content of milk ought to be obtainable. At the same time, one must remember the limitations of fractional precipitation, which are obvious from the statement of Schryver (*loc. cit.*) and the experiments of Woodman.[7]

In dealing with colostrum Howe[6] was able to sub-divide the globulin into no less than three fractions, but the amount of globulin in milk is rather small for this to be attempted, and the validity of such a sub-division has recently been questioned.

I have determined the globulin content of milk by different macro-methods. The filtrate from the isoelectric precipitation of casein is first neutralised to phenolphthalein with $N/10$ sodium hydroxide solution. It is then saturated with an approximately weighed amount of either magnesium or sodium sulphate (anhydrous), which must be stirred in while the temperature is maintained at about 20° C. by means of a water-bath. After standing some time, filtration and washing (with saturated salt solution) ought to be completed without interruption, hence it is preferable not to commence filtering the large bulk of solution until next morning, using a pleated filter. Mechanical impurities are sometimes present in the large amounts of salt required for saturation, so that after washing is completed it may be desirable to wash the globulin through the filter paper into the Kjeldahl flask, with the use of distilled water or a very dilute solution of sodium chloride.

Alternatively, casein and globulin may be precipitated together as in the difference method outlined below. Since considerable dilution of the protein solution is desirable, 10 ml. of milk (weighed as for casein) should be neutralised and mixed with at least 90 ml. of saturated sodium or magnesium sulphate solution, and sufficient extra salt added to saturate 10 ml. of water. The protein precipitate is filtered off, washed with saturated salt solution, and its nitrogen content determined by Kjeldahl's method. In each of the above cases the albumin remaining in the filtrate of saturated salt solution may be separated by acidifying with 3 ml. of 10 per cent. of acetic acid and heating on the boiling water-bath for at least half-an-hour. After cooling, the precipitate is filtered off and washed, preferably with saturated salt solution. The precipitation beaker is rinsed into the Kjeldahl flask with the aid of a little soda, as suggested for casein. Gentle heating during the early stages of digestion is essential to avoid frothing. In preliminary trials with the above methods, the writer obtained the following values from different samples of milk: 0·020, 0·015, 0·027, 0·030, 0·022, 0·0215 grm. of globulin nitrogen per 100 ml. of milk. The figures in each case are the average of several determinations, and, although the agreement between duplicates left a little to be desired, the results are worth recording for comparison with the very few others which have been published. Simon[9] found slightly higher values: 0·051, 0·049, 0·029, 0·043, 0·029, 0·030, 0·034 grm. of globulin nitrogen per 100 grms. of milk. During a study of colostrum, Mrozek and Schlag[10] determined globulin by two different methods, but the figures differ so widely that their reliability may be questioned. At the forty-second milking, when the colostral phase ought to have passed off, they found globulin nitrogen 0·12 per cent. by one method and 0·02 per cent. by another.

CONCLUSIONS.—On the basis of the foregoing investigations, the following scheme may be suggested for the purpose of obtaining values for casein, albumin and globulin in milk.

A. Casein by isoelectric precipitation at pH 4·6.

B. Casein and globulin by neutral saturated magnesium sulphate or sodium sulphate.

C. Total protein by warm 4 per cent. trichloroacetic acid.

From B—A globulin may be obtained; from C—B albumin may be obtained. Alternatively, albumin and globulin may be determined by treating the filtrate from A with trichloroacetic acid, and albumin obtained by subtracting the globulin value, B—A. In order to avoid the difficulties encountered in dealing with large volumes of saturated solutions, which so readily crystallise on the paper during filtration, a micro-procedure (similar to that of Howe), together with the micro-Kjeldahl method for nitrogen, may be found advantageous.

This paper represents part of the work carried out during my tenure of the Pedler Research Scholarship of the Institute of Chemistry, the assistance of which and the interest of the members of the Pedler Fund Committee are gratefully acknowledged. I wish to add my appreciation of the facilities made available to me at the National Institute for Research in Dairying, and especially of the valuable advice of Capt. J. Golding, Head of the Chemical Department.

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DISCUSSION.

The PRESIDENT said that the Society was glad to have the opportunity of publishing the work of the Pedler Scholar of the Institute of Chemistry, and was very grateful to Captain Golding for his synopsis of the four papers.

Mr. BACHARACH said that by a strange coincidence he had that morning, while anticipating the pleasure of hearing Dr. Moir's papers, been confronted with a paper, 56 pages long, by two German chemists whose work touched Dr. Moir's very closely. They were investigating the residual nitrogen of milk, and set out to revise the existing methods of separating casein, albumin and globulin. Although he had not yet been able to read this paper thoroughly, there were one or two very interesting points he had noticed. The first was that, although these authors did not use the sodium acetate buffer solution, they considered the only safe method of determining casein and nitrogen in milk to be iso-electric precipitation with acetic acid at pH 4·6; they had overlooked the advantage of using a buffer with sour samples. They tried other methods, using tannic acid and phosphotungstic acid. They stated that globulin was present in normal milks to a very small extent; this was in disagreement with Dr. Moir, who found quite appreciable quantities of globulin in normal milks. It was to be hoped that Dr. Moir would continue his investigations of the various nitrogenous constituents of

cow's milk; when he had done that, he might, perhaps, turn his attention to a problem even harder than this—the precise distribution of nitrogen in human milk.

Mr. J. H. BUSHILL referred to the two methods adopted to prove the similarity of the casein precipitated at *p*H 4·6 by an acetate buffer solution, and that precipitated at *p*H 4·2 by the usual acetic acid method. He doubted whether these methods would detect the presence of 1 to 2 per cent. of lactalbumin or of lactglobulin in the casein, and asked Captain Golding whether he had any similar curves showing the effect of the addition of such an amount of lactalbumin or of lactglobulin to a casein precipitate.

Captain GOLDING said that he was sure Dr. Moir would be glad to see the German paper referred to by Mr. Bacharach. There were very many contrary suggestions made on this subject. Dr. Moir determined the amount of non-protein nitrogen in milk; if the amount of globulin in milk was small the amount of non-protein nitrogen must be similar. Captain Golding pointed out that he had not been able to read the whole of the papers to the meeting, but with regard to the curves Dr. Moir had gone into the matter fully. Precipitation with acid only gave a figure about 2 per cent. too low, and so he took it that the precipitation with acetic acid by itself would give a substance which had been generally regarded as casein. He was then getting a substance which was, in fact, pure casein, or casein plus a little albumin and globulin, and went into that point and proved that this method would show contamination with 2 per cent. of albumin.

The following communication has been received from Dr. MOIR:

When my papers were discussed at the Society's meeting, on November 5th, 1930, a speaker enquired about the curves which albumin and globulin would give with the formol titration or the hypobromite oxidation methods. In the time at my disposal I was unable to investigate this matter. A study of the work I cited by Woodman, Abderhalden and Kroner, and by Goldschmidt and Steigerwald, would lead one to expect that, owing to their structural differences, albumin and globulin would give distinctly different curves from casein. Assuming, for example, that the higher nitrogen values obtained at *p*H 4·6 were due to a precipitate of (say) casein plus albumin, instead of (say) casein only at *p*H 4·2, the curves from the former ought to differ slightly, but definitely, in shape from those of the latter.

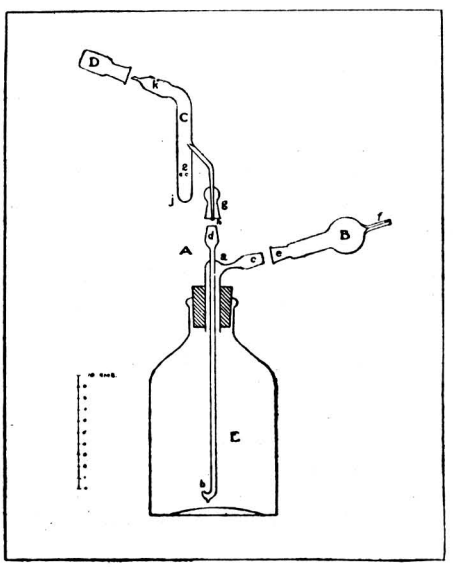
Another method which I tried was the determination of the ratio of nitrogen to sulphur in casein, which has a very low sulphur content compared with that of lactalbumin. After overcoming a number of difficulties, I obtained promising results, but unfortunately was unable to complete them to the stage required for publication. Apart from these experimental methods of investigating the casein precipitated at different *p*H values, I feel that the new method I have evolved derives very great support from the fundamental conceptions associated with the iso-electric point, which is one of the most definite physico-chemical properties of a protein.

A Storage and Delivery Apparatus for Antimony Chloride Solution and other Corrosive Reagents.

By G. MIDDLETON, B.Sc., A.I.C.

(*Read at the Meeting, December* 3, 1930.)

Of the various colour tests which have been proposed for cod-liver oil, that of Carr and Price (*Biochem. J.*, 1926, **20**, 497) has found most general adoption, on account of its quantitative character. The chromogen reaction, of which it is a measure, is employed as an indication of the vitamin *A* content of the oil. The reagent (a solution of antimony chloride in chloroform), is volatile, poisonous and corrosive, and, when handling it, special precautions have to be observed—in particular against contact with the fingers, and against unnecessary exposure to moist air.



In the routine examination of oils, the need for a convenient way of storing and measuring the reagent was keenly felt. To meet this need a special apparatus has been designed, and it is thought that other workers may find the following description useful.

The purpose of the special joints at *g* and *k* is to prevent the reagent from getting on to the ground-glass surfaces and cementing them together by the formation of antimony oxychloride.

DESCRIPTION OF APPARATUS.—The apparatus, which is shown in section in the figure, consists of six portions:

(1) An amber glass bottle, *E*, conveniently holding about 600 c.c.

(2) A glass portion, *A*, fitted to the bottle, *E*, by means of an ordinary cork, and consisting of an internal joint, *a*, with the inner tube, *ab*, extended downwards into the liquid at *b*, the end, *b*, being turned over through an angle of 180°, as shown, in order to draw up the reagent without disturbing the sediment. At the lowest point of this end there is made a very small hole, the object of which is to allow the escape of any of the heavy oily liquid which separates from the reagent on keeping, and which would otherwise accumulate in the lower bend of the tube and be carried up into the measuring pipette. At *c* and *d* are the inner portions of ground-glass joints.

(3) A calcium chloride tube, *B*, with ground joint, *e*, fitting on to *c*. The other end, *f*, is open but constricted.

(4) A rubber bulb fitting on to *f*.

(5) The measuring portion, *C*, with ground-glass joint, *g*, fitting outside the taper *d* of *A*, having an inner tube, *h*, the end of which is cut at an angle in such a way that when the joint is assembled, the lowest part nearly touches the wall of *A*, so that liquid drains straight down from *h* to *a*. The portion, *j*, is of such capacity that the measuring apparatus delivers exactly 2 c.c. Owing to the transient nature of the colour produced in the reaction, and the consequent necessity for quick working, not more than one or two seconds should be allowed in the calibration for drainage. At *k* there is a ground taper running off into the jet which is ground off at an angle.

(6) A glass cap, *D*, ground on to the taper *k* of *C* in such a way that it does not come into contact with the jet.

The scale represents divisions of cm. in a total of 10 cm.

USE OF THE APPARATUS.—The tube, *B*, is filled with calcium chloride and the bottle, *E*, with reagent.* When required for use, the cap, *D*, is removed, and the measuring portion, *C*, filled by blowing air (cautiously at first) through *B* by means of the rubber bulb. The excess of liquid in *C* runs back on releasing the pressure, but one or two minutes are allowed so that the tube, *h*, may drain thoroughly. *C* is then detached, care being taken that none of the liquid in it runs back through the tube, *h*, and the 2 c.c. of reagent are poured out. When not in use, the cap, *D*, should be replaced and the rubber bulb removed from *f*. The reagent should not be allowed to get on the ground parts of the joints, as it may cause them to stick together, in which case they should be freed by soaking in hydrochloric acid.

The special features claimed for this apparatus are as follows: (1) The reagent has a minimum amount of exposure to moist air. (2) There is no danger of accidental contact of the reagent with the hands of the operator. (3) The sediment in the bottle is not disturbed. (4) The measuring portion is always ready for use and does not need washing out and drying after each time it is used, as with an ordinary dipping pipette. (5) There are no ground-glass taps or joints in actual contact with the reagent.

The apparatus as described above has been in continuous use in the laboratories of The British Drug Houses, Ltd., for several months, and has been found most advantageous. It may be used also for other corrosive liquids, *e.g.* bromine, in which case the cork must be replaced by a ground joint fitting in the neck of the bottle; and especially for liquids, such as strongly alkaline solutions, which cannot be used with glass taps.

* It is recommended that the apparatus should first be given a trial with chloroform only.

Tests for Impurities in Ether.

Parts II. and III.

By G. MIDDLETON, B.Sc., A.I.C., and F. C. HYMAS, B.Sc., A.I.C.

(*Read at the Meeting, December* 3, 1930.)

PART II: TESTS FOR ACETALDEHYDE.

Acetaldehyde and peroxide form the two impurities of most significance in ether, not only because they are the cause of undesirable after-effects when the ether is used for the production of anaesthesia, but also because of the intimate relation between the two. On the one hand, the presence of acetaldehyde increases the rate of formation of peroxide in ether, and, on the other, the organic peroxide itself decomposes, forming acetaldehyde, so that the rate of oxidation increases progressively. The importance of a delicate test, so that a high standard of freedom from acetaldehyde may be maintained, is therefore evident.

The current pharmacopoeias all require anaesthetic ether to comply with tests for freedom from aldehyde; the French Codex also requires freedom from vinyl alcohol. If, as is often considered, the latter is a desmotropic form of acetaldehyde,[2] the two forms should be in equilibrium, and there would be no object in testing for each separately.

Vinyl alcohol has never been obtained in the pure state, and the evidence for its existence is based on Poleck and Thummel's[3] examination of the precipitate which they obtained on treating impure ether with a solution of mercuric chloride and potassium bicarbonate. This precipitate is not obtained with acetaldehyde, so that the two compounds cannot, without further evidence, be regarded as desmotropic. It is proposed for this reason to deal separately with tests for acetaldehyde and for the isomeric vinyl alcohol, the latter being considered together with the tests for other vinyl compounds.

Determination of Sensitiveness.—The sensitiveness of a test for acetaldehyde may be expressed in terms of the smallest proportion of acetaldehyde required to give a just distinctly perceptible reaction. Except where otherwise stated, all tests were carried out in colourless glass-stoppered bottles of about 30 c.c. capacity (1 ounce bottles), using 25 c.c. of ether and 6 to 8 c.c. of the reagent. A standard acetaldehyde solution was prepared by dissolving about 5 c.c. of freshly distilled acetaldehyde in 100 c.c. of pure aldehyde-free ether, the strength of this solution being determined by adding 5 c.c. to an excess of a neutral solution of hydroxylamine hydrochloride in a stoppered bottle, shaking well, and titrating the liberated acid with normal alkali, using methyl orange as indicator. The solution was then diluted with aldehyde-free ether to the required strength. Both the pure ether and the acetaldehyde solution were freshly prepared as required, and control experiments were carried out in all cases, in order to ensure that the pure ether gave no reaction in the test under investigation.

The detection of aldehyde is complicated by the fact that ether peroxide also gives positive reactions with reagents for acetaldehyde. For this reason the sensitiveness of tests towards ether peroxide was also determined, using the method given in Part I of this paper.[1] The results obtained are given below:

Test.	Sensitiveness to acetaldehyde (parts per million).	Sensitiveness to ether peroxide (calculated as H_2O_2, in parts per million).
Ammoniacal silver nitrate	1000	over 560
Solid potassium hydroxide (6 hours) ..	40	over 560
Diazobenzene sulphonic acid	3	—
Schiff's reagent, with pyrogallol	2	0·2
Tollens' reagent	1	9
Nessler's reagent	1	5
Nessler's reagent, diluted 1–4	1	2·5

THE CAUSTIC ALKALI TEST.—In all modern pharmacopoeias, with only one exception, either sodium or potassium hydroxide is used as a test for the presence of aldehyde in ether. The prescribed time of contact varies from one-half to six hours, and there is no uniformity in the instructions concerning exposure to light during this period.

This test has been fully investigated by Mallinckrodt,[4] who found that the official test of the United States Pharmacopoeia with solution of potassium hydroxide had a sensitiveness of 300 parts per million under optimum conditions. The official German test with solid caustic potash was found to be more sensitive, but not capable of detecting less than 100 parts per million with certainty.

Though this very low sensitiveness, compared with that of other tests available, is in itself sufficient to justify the abandonment of this test, the significance it has acquired on account of its almost universal official adoption calls for a more detailed examination.

When freshly broken potassium hydroxide is added to ether containing acetaldehyde, it is observed that, after standing for some time, a brown colour is produced at certain points on the surface of the potash. The colour appears on the original outer surfaces of the sticks of alkali, owing to the fact that a certain amount of moisture is necessary for the development of the colour. In the absence of moisture, ether containing as much as 750 parts of acetaldehyde per million gives no colour after three hours' exposure.

The presence of alcohol in the ether under examination often causes the formation of a turbidity, and, on long standing, a yellow colour. This interference is removed to some extent by the addition of water.

Our experience is in general agreement with the above results, taken direct from the paper of Mallinckrodt, who sums up the test in the following words: "Anyone using the test with solid potassium hydroxide day in and day out on ethers of varying composition will be struck by the variations in the phenomena

attending the test, and herein is its chief defect, for the observer must make allowances for the disturbing influences caused by the presence in the ether of such common impurities as alcohol and peroxides. The pure brown coloration on the lumps of potash normally produced by pure aldehyde in pure ether may be obscured or replaced more or less by the other appearances which, while difficult to describe in an adequate manner, are readily recognised after having once been seen."

SCHIFF'S REAGENT.—This is employed as an official test for aldehyde only in the French Codex (1908), which requires that ether shall not redden a solution of rosaniline decolorised with bisulphite. No time limit is given for the test, although this is very important, as with small quantities of aldehyde the colour develops very slowly, whilst the purest ether obtainable will give a colour with the reagent on long standing. Further, the colour is more or less intense according to the amount of shaking, and depends to some extent on the temperature. In spite of these objections, the test is a very useful one if employed under standard conditions, as it combines sensitiveness with specificity for aldehyde. Our procedure is to shake the ether thoroughly with the reagent, allow the mixture to stand for 10 minutes at room temperature, and note the colour of the lower layer. Only the purest samples of ether show no colour after this time.

All attempts to prepare ether that would give no colour on standing for a long time with Schiff's reagent proved unsuccessful, and it was finally found that the progressive development of colour was due to the formation of aldehyde from the ether under the conditions of the test, and that sulphur dioxide greatly increases the velocity of oxidation of ether in air. This effect may be inhibited by the addition of pyrogallol, which exerts an anti-oxidising action. By the addition of 0·1 per cent. of pyrogallol to Schiff's reagent the sensitiveness to aldehyde is not decreased, while at the same time no colour is obtained even after several hours' contact with pure ether. In the presence of aldehyde the maximum colour is produced in about 20 minutes.

The Schiff's reagent was prepared as described in the British Pharmacopoeia, with the use of fuchsin known to be sensitive. Samples of old and badly deteriorated ether, containing a large amount of peroxide, give a very weak reaction with Schiff's reagent; but, after dilution with 20 parts of pure ether, they then gave a strong reaction with the reagent. A strong reaction was also obtained after the ether had been washed with four successive small portions of water, in order to remove hydrogen peroxide; and it was found further that the presence of hydrogen peroxide prevented the development of colour with Schiff's reagent. It follows, therefore, that in an ether, giving a heavy reaction for peroxides, the absence of a colour with Schiff's reagent does not indicate the absence of aldehyde.

AMMONIACAL SILVER NITRATE.—In the French Codex, a solution of silver nitrate, to which ammonia is added until the precipitate has almost redissolved, is given as an additional test for aldehyde; but no statement of the exact time of

contact or of quantities of ether and solution to be taken is made. The figure of 1000 parts per million, given above as representing the sensitiveness, refers to a period of 20 minutes. With smaller proportions of aldehyde a positive reaction is obtained only after standing for several hours. It is, however, unnecessary to consider this test in further detail, as an ammoniacal silver solution in the form of Tollens' reagent is very much more sensitive and more rapid in its indications.

TOLLENS' REAGENT.—The reagent was prepared and employed as described in Part I of this paper.[1] The test mixtures were kept in the dark, and the full colour developed within 10 minutes. The test is not specific for aldehyde, as it is given also by peroxide.

p-DIAZOBENZENE-SULPHONIC ACID.—Though this reagent is frequently used for the detection of acetaldehyde, its employment for testing ether does not appear to have been suggested; a closer examination of the test, with this object in view, was, therefore, thought desirable. The test was applied as follows:—To 2 c.c. of a 1 per cent. solution of sulphanilic acid in 5 per cent. hydrochloric acid were added 2 c.c. of 0·38 per cent. sodium nitrite solution. After a few moments 25 c.c. of the ether and about 0·3 grm. of solid caustic soda were added, followed by a piece of semi-solid sodium amalgam about the size of a pea. In presence of aldehyde a red colour developed slowly in the aqueous layer. Though at first this test appeared promising, it was found that pure aldehyde-free ether gave a red colour with the reagent, even when the amount present was only sufficient to give a layer of ether 1 mm. deep above the reagent. Further investigation showed that alcohol also gave a red colour. One mgrm. of specially purified aldehyde-free alcohol gave a distinct red colour, possibly due to the rapid oxidation of alcohol to aldehyde, which occurs on exposure to air of strongly alkaline spirit solutions. Half a mgrm. of acetaldehyde gave a red colour with 4 c.c. of the reagent, but 0·1 mgrm. and smaller amounts gave a brown colour which was not so distinct, in view of the brownish-yellow colour obtained in the absence of aldehyde.

NESSLER'S REAGENT.—Most commercial samples of ether give at least a yellow turbidity with this reagent, and Baskerville and Hamer[5] state that it is impossible to obtain ether which gives an entirely negative reaction. This is not correct; ether giving no reaction with Nessler's reagent may be prepared experimentally.

The British Pharmacopoeia method of preparing Nessler's reagent was employed, and the solution was tested for sensitiveness before use. When 25 c.c. of ether containing a trace of acetaldehyde are treated with 5 c.c. of the reagent, a brownish-red colour develops in the aqueous layer in about half a minute, changing in a few minutes to grey. Though the latter tint is slightly less conspicuous, it has the advantage, over the transient brown colour, of greater permanence. If acetone is added to a sample of ether which gives no colour or precipitate with the reagent, a cream-coloured turbidity or precipitate is obtained, quite distinct from that due to acetaldehyde or ammonia, but liable, if sufficiently heavy, to obscure the reaction of the last two. The test is very sensitive to acetone.

The German and Belgian Pharmacopoeias allow narcotic ether to give an opalescence, but no turbidity, with Nessler's reagent. In the Dutch and Swedish official tests the reagent is diluted with water before use. Comparative tests showed that there was no difference in the sensitiveness of diluted and undiluted reagent towards either acetone or acetaldehyde, but that the diluted reagent was slightly more sensitive towards peroxide. The diluted reagent has the advantage that the yellow colour due to the reagent itself is greatly reduced. It is, therefore, recommended that, in employing this test, 25 c.c. of the ether should be shaken well with a mixture of 1 c.c. of Nessler's solution and 4 c.c. of water, and allowed to stand for 15 minutes.

PHARMACOPOEIAL TESTS.—A table of the official tests for acetaldehyde adopted in the various pharmacopoeias is given. It shows that chief reliance is placed on the caustic alkali test, which has been shown to be insensitive and unreliable.

Pharmacopoeia.	Ammoniacal silver nitrate.	Nessler's reagent.	Caustic alkali.	Schiff's reagent.
British, 1885	—	—	—	—
1898	—	—	+	—
1914	—	—	+	—
American, 10, 1926	—	—	+	—
French, 1908	+	—	+	+
German, 6, 1926	—	+	+	—
Belgian, 3, 1906	—	+	+	—
Swedish, 10, 1925	—	+	+	—
Norwegian, 4, 1913	—	—	+	—
Italian, 4, 1920..	—	—	+	—
Swiss, 5, 1926	—	—	+	—
Dutch, 5, 1926	—	+	—	—
Spanish, 7, 1915	—	—	+	—
Portuguese, 1876	—	—	—	—
Japanese, 4, 1922	—	—	+	—
Argentine, 1921	—	—	+	—
Danish, 1907	—	—	+	—
Roumanian, 1926	—	—	+	—

+ indicates that the test is adopted. — indicates that the test does not appear.

DISCUSSION.—It has been shown that the caustic alkali test, which is in general use, is not sufficiently delicate to ensure the freedom of anaesthetic ether from objectionable quantities of acetaldehyde. This test should, therefore, be superseded by a more sensitive test.

Of the three more sensitive reagents given in the table, both Tollens' and Nessler's reagents give positive reactions with other impurities which may be present in ether, so that the interpretation of the results obtained is uncertain.

The application of Schiff's reagent has been limited by the uncertainty as to its significance, owing to the colour showing a progressive increase with time. This objection has now been removed by the addition of pyrogallol to the reagent,

to inhibit the catalytic effect of the sulphur dioxide, and the test may be recommended for general and official adoption.

PART III: TESTS FOR ACETONE.

Only two pharmacopoeias employ tests specific for acetone, and ether containing it may fail to pass the tests of two others.

Baskerville and Hamer[5] state that they "cannot anticipate its presence in ether prepared from pure ethyl alcohol," and that "it is unnecessary to look for such an improbable contaminant at the present time." This view is by no means justified; many commercial samples of high grade narcotic ether contain traces of acetone, whether they have been made from denatured or from duty-paid spirit.

DETERMINATION OF SENSITIVENESS.—For determining the sensitiveness of the different tests, acetone prepared from the sodium iodide compound was used as a standard and dissolved in acetone-free ether. The results obtained are given below. In all cases 25 c.c. of ether were taken for the test.

Test.	Sensitiveness, etc., in parts per million.
Mercuric chloride and baryta	100
Hydroxylamine and hypochlorite[6]	100
Sodium nitroprusside (German Pharmacopoeia)	25
Sodium nitroprusside (B.P.C.)	20
Acid mercuric sulphate	5
Vanillin (Dutch Pharmacopoeia)	1
Nessler's reagent	0·5 (upper limit 20)

MERCURIC CHLORIDE AND BARYTA.—Twenty-five c.c. of ether are shaken with 5 c.c. of saturated barium hydroxide solution and 5 drops of mercuric chloride solution. The aqueous layer is run off, filtered, and the filtrate tested for mercury by the addition of several drops of ammonium sulphide solution. This test was employed in the Dutch Pharmacopoeia of 1905, but in a less sensitive form—only 2 c.c. of ether being taken.

SODIUM NITROPRUSSIDE.—There is little to choose between the two forms of the test which were examined, but that of the German Pharmacopoeia has the advantage that the colour due to the reagent itself is less than is the case with the test of the British Pharmaceutical Codex.

ACID MERCURIC SULPHATE.—Twenty-five c.c. of ether are shaken with 5 c.c. of water, the aqueous layer separated, mixed with 1 c.c. of Denigès' acid mercuric sulphate reagent, and heated for half-an-hour in the water bath. If acetone is present, a white precipitate is obtained.

VANILLIN.—The test of the 1926 Dutch Pharmacopoeia is as follows:—Twenty c.c. of ether are shaken with 5 c.c. of water, the water layer is separated, and to it are added 5 mgrms. of vanillin and a piece (about 0·75 grm.) of potassium hydroxide. The mixture is warmed (cautiously at first) for 15 minutes at 60 to 70° C., without shaking. The presence of acetone is indicated by an orange or red layer above

the potash. This is useful as a specific test for acetone when the presence of other impurities prohibits the use of Nessler's solution for this purpose.

NESSLER'S REAGENT.—Though Nessler's reagent was found to be a very sensitive test for the detection of small quantities of acetone, it was also found that a positive reaction was no longer obtained when the concentration of the acetone exceeded a certain limit, which, for the conditions employed, was 20 parts per million. When the concentration is higher than this a transient precipitate may be obtained on the addition of the ether to the reagent, and a precipitate may also be formed on long standing, but the cream-coloured turbidity or precipitate which is most characteristic of acetone does not appear.

No difference in sensitiveness could be observed between Nessler's solution (prepared according to the British Pharmacopoeia), and the same solution diluted with 5 parts of water, as used by the Dutch and Swedish Pharmacopoeias. The latter form has the advantage that the natural colour of the reagent is considerably reduced.

PHARMACOPOEIAL TESTS.—The only pharmacopoeias which employ tests which respond to acetone are the following:

Pharmacopoeia.	Nessler's reagent.	Vanillin.	Sodium nitroprusside.
German, **6**, 1926	+	—	+
Belgian, **3**, 1906	+	—	—
Dutch, **5**, 1926	+	+	—
Swedish, **10**, 1925	+	—	—

Owing to the fact that Nessler's reagent forms a satisfactory test for acetone only when the concentration of the acetone is below a certain limit, it cannot be recommended for general adoption. The Dutch vanillin test should, therefore, replace it.

SUMMARY.—(1) A comparison of the sensitiveness of tests for acetaldehyde and acetone is given.

(2) The colour developed with Schiff's reagent, even with the purest ether, is due to rapid oxidation of the ether during the period of the test—this reaction being catalysed by sulphur dioxide. The addition of 0·1 per cent. of pyrogallol to the reagent prevents this, while it does not interfere with the sensitiveness of the reagent towards acetaldehyde.

(3) The caustic alkali test should be superseded by a more delicate test.

(4) Nessler's reagent is a sensitive test for small quantities of acetone, but fails to indicate the presence of large quantities.

(5) The tests recommended for official adoption are: for acetaldehyde, the modified Schiff's reagent; and for acetone, the vanillin test of the Dutch Pharmacopoeia.

We wish to express our thanks to the Directors of The British Drug Houses, Ltd., for permission to publish this paper.

CORRECTION.—In Part I of this communication[1], page 205, line 19: *For* "100 c.c. of 3·3 per cent. (w/v) sulphuric acid" *read* "300 c.c. of 3·3 per cent. (w/v) sulphuric acid."

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Nickel Uranyl Acetate as a Qualitative Reagent for Sodium.

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DURING the last few years several papers have been published dealing with the use of certain double uranyl acetates proposed by Streng (*Z. wissensch. Mikrosk.*, 1886, **3**, 129; *Chem. Zentrbl.*, 1886, **17**, 488) as microchemical reagents for the sodium ion. Thus a solution containing magnesium uranyl acetate has been investigated by Miholić (*Bull. Acad. Sci. Zagrab.*, 1920, 16), Blanchetière (*Bull. Soc. Chim.*, 1923, **33**, 807), Kolthoff (*Pharm. Weekblad*, 1923, **60**, 1251), Crepaz (*Ann. chim. applic.*, 1926, **16**, 219), Caley and Foulk (*J. Amer. Chem. Soc.*, 1929, **51**, 1664), Caley (*ibid.*, 1930, **52**, 1349), and Kahane (*Bull. Soc. Chim.*, 1930, [IV], **47**, 382; *J. Pharm. Chim.*, 1930, **11**, 425). Zinc uranyl acetate has been investigated by Kolthoff (*Z. anal. Chem.*, 1927, **70**, 397), Barber and Kolthoff (*J. Amer. Chem. Soc.*, 1928, **50**, 1625; 1929, **51**, 3233), Malitzky and Tubakaiev (*Mikrochem.*, 1929, **7**, 334), Blenkinsop (*J. Agric. Sci.*, 1930, **20**, 51), and cobalt uranyl acetate by Caley (*J. Amer. Chem. Soc.*, 1929, **51**, 1965).

In our experience, zinc uranyl acetate, as proposed by Barber and Kolthoff, is more sensitive than a solution of magnesium uranyl acetate (containing a concentration of uranyl acetate equal to that used in the zinc salt), and gives a more granular precipitate than that obtained with the magnesium salt. The cobalt reagent is less sensitive than either of these, and the large volumes required make it impracticable as an ordinary reagent. Streng also mentioned that other double uranyl acetates give sparingly soluble precipitates of triple acetates containing sodium. We have tried these, and have found that nickel uranyl acetate compares very favourably with the reagents already proposed. It is quite as sensitive

as the zinc reagent. The light green precipitate which separates from the dark green solution of nickel uranyl acetate is rather more granular, and settles more rapidly than the corresponding zinc salt. The crystals, when viewed under the microscope, resemble those of the zinc salt, and appear to be octahedra.

The nickel reagent used was prepared as follows: A mixture of 70 grms. of uranium acetate, 200 grms. of nickel acetate, 60 c.c. of glacial acetic acid, and 940 c.c. of water was warmed and stirred until the solids had dissolved. The solution was allowed to stand at room temperature for several hours, with occasional shaking, and was then filtered through a dry filter into a dry bottle.

DETECTION OF SODIUM.—The test was carried out by adding 2 c.c. of the reagent to 0·5 c.c. of sodium chloride solutions of known strengths. With 0·1 per cent. solution of sodium chloride a precipitate forms immediately; with 0·02 per cent. a precipitate is formed, and settles after a few minutes. Potassium chloride gives no precipitates at corresponding concentrations; from solutions of 5 per cent. strength and above, a precipitate rapidly forms (gentle agitation assists the formation of the precipitate in a well-crystallised condition); below these strengths, precipitation becomes much slower. With 2 per cent. potassium chloride solution no precipitate forms at once, but a slight precipitate separates after a few hours. The behaviour with zinc uranyl acetate is analogous, and the precipitate which separates from the solution is described by Barber and Kolthoff (*J. Amer. Chem. Soc.*, 1928, **50**, 1630) as zinc potassium uranyl acetate, but Kolthoff (*Z. anal. Chem.*, 1927, **70**, 490) refers to it as potassium uranyl acetate. The precipitate obtained from the nickel uranyl acetate, viewed under the microscope, is seen to consist of long fine needles, and is identical in appearance with that obtained from the zinc uranyl acetate solutions. Further, the precipitate obtained from nickel uranyl acetate is yellow, and does not contain nickel; there can be no doubt that it is potassium uranyl acetate, which can be precipitated from moderately concentrated solutions of potassium chloride and uranyl acetate (*e.g.* 7 per cent. uranyl acetate solution immediately gives a heavy precipitate with 10 per cent. potassium chloride, and this appears identical under the microscope with that obtained in the foregoing cases). The contrast between potassium uranyl acetate and sodium nickel uranyl acetate under the microscope is most striking, and provides a ready means of distinguishing between sodium and potassium. If, however, a microscope is not available, and the presence of potassium has been determined, a rougher test for the presence of sodium can be made as follows: Filter off the precipitate, wash twice with 2 to 3 c.c. of cold methylated spirit, dissolve the residual precipitate in water, add ammonium chloride and ammonium hydroxide, and, without filtering the precipitated ammonium uranate, add dimethylglyoxime. A positive test for nickel shows the presence of sodium.

INFLUENCE OF POTASSIUM.—Barber and Kolthoff (*loc. cit.*, p. 1630) have recorded that if potassium ions are present in sufficient amount to cause a precipitate to form, zinc uranyl acetate solution is less sensitive to sodium ions. The following observations on nickel uranyl acetate illustrate this point. A solution

(0·5 c.c.) containing potassium chloride (2·5 per cent.) and sodium chloride (0·02 per cent.) was examined in the usual way. A definite small precipitate was present after 5 minutes. This was examined under the microscope and found to consist of crystals of sodium nickel uranyl acetate only; the amount was insufficient to apply the precipitation reaction for nickel already described. When, however, the concentration of potassium chloride was increased to 7·5 per cent., whereas 0·04 per cent. of sodium chloride gave a precipitate immediately, in which microscopic examination showed sodium nickel uranium acetate definitely to be present, lower concentrations of sodium chloride did not give a conclusive reaction for sodium.

If sodium is present in the solution in relatively high concentration, sodium uranyl acetate may be precipitated as well as the triple salt, for 5 per cent. solutions and stronger of sodium chloride give an immediate precipitate with uranyl acetate (7 per cent.). With a 2 per cent. solution of sodium chloride a precipitate slowly forms; whilst a 1 per cent. solution gives no precipitate during 24 hours.

Nickel uranyl acetate is thus seen to be a very suitable reagent for sodium in the ordinary scheme of qualitative analysis. Usually the only cations present in the solution would be ammonium, magnesium, potassium, and sodium; ammonium and magnesium are without effect on the reagent, and the influence of potassium has been considered. Lithium causes precipitation of a salt closely resembling the sodium triple salt in appearance, and containing lithium, nickel and uranium.

Influence of Anions.—The only common anion likely to be present at this stage which would cause complications is phosphate. The appearance of the very finely divided precipitate of uranyl phosphate is quite different from that of the sodium precipitate. It can be at once distinguished under the microscope from sodium nickel uranyl acetate, or the less sensitive test for sodium may be carried out by filtering off the precipitate, washing twice with 2 to 3 c.c. of methylated spirit, shaking the precipitate with water (uranyl phosphate will remain undissolved), and then adding ammonium chloride and ammonium hydroxide, followed by dimethylglyoxime; the formation of nickel dimethylglyoxime shows the presence of sodium.

We have not attempted to use the reagent for the determination of sodium, as has been done in the cases of zinc and magnesium uranyl acetates. The method, which is rapid, has yielded results of moderate accuracy in the hands of the investigators cited. We hope next to investigate the application of nickel uranyl acetate to this.

Analysis of Sodium Nickel Uranyl Acetate.—The salt (0·60 grm.) was dissolved in water (200 c.c.), and the solution treated with 20 grms. of ammonium chloride dissolved in a small amount of water, and then with ammonia in slight excess. The precipitate of ammonium uranate was allowed to settle, washed twice by decantation with ammonium chloride solution, and then dissolved in dilute hydrochloric acid, and precipitation repeated as above. The precipitate was filtered off, well washed with ammonium chloride solution, and ignited in a platinum

crucible to U_3O_8. Nickel was precipitated from the filtrate by dimethylglyoxime, and the combined filtrates were evaporated to dryness. A small amount of nickel was found to be present in the residue obtained. Most of the ammonium salts were expelled, and the nickel determined by precipitation with dimethylglyoxime. Sodium was determined in the filtrate by evaporation and weighing as sodium chloride. Acetate was determined in a separate sample by distillation with phosphoric acid. Carbon and hydrogen were determined by combustion. The figure for carbon was obtained on the assumption that the material remaining after the combustion of the triple acetate was sodium carbonate, uranium oxide and nickel oxide.

		Calculated for $NaNi(UO_2)_3(CH_3CO_2)_9xH_2O$.		
	Found. Per Cent.	($x=6$). Per Cent.	($x=6{\cdot}5$). Per Cent.	($x=9$). Per Cent.
UO_2	52·80	52·90	52·60	51·10
Ni	3·81	3·85	3·83	3·72
Na	1·49	1·50	1·495	1·45
CH_3CO_2	34·3	34·7	34·5	33·5
H	2·55	2·55	2·59	2·84
C	13·98	13·72	13·64	13·25

The formulae given in the papers cited for the compositions of zinc or magnesium sodium uranyl acetates generally contain 6 or 9 molecules of water. Often experimental figures are not given in support. Caley and Foulk (*loc. cit.*) consider, from their results, that the magnesium salt contains 6·5 H_2O. Our experimental results are given, together with the calculated values for 6, 6·5, and 9 of H_2O. They are not in agreement with the 9 H_2O formula, but we do not regard them as of sufficient accuracy to decide between the formulae containing 6 and 6·5 H_2O.

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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.

THE DIPHENYLAMINE TEST FOR NITRATES IN MILK AS A MEANS OF DETECTING ADDED WATER, AND THE EFFECT OF DRENCHING COWS WITH "NITRE."

THE method of applying this test, described by Lerrigo (ANALYST, **1930, 55, 433**), has proved very satisfactory in our hands. So far, no samples of genuine milk have given a positive reaction for nitrates, whilst samples containing added water have given distinct indication of its presence. We find no difficulty in detecting 5 per cent. of added water containing 0·5 part per 100,000 of nitrogen as nitrates.

The test affords very useful confirmatory evidence of added water, and, in common with the freezing-point test, distinguishes actually added water from water normal to the milk, in contrast with the indirect evidence adduced or "presumed" from the figure for solids-not-fat, with or without comparison with the results given by an appeal sample. One is sometimes asked in Court if one can distinguish between actually added water and water normal to the milk, and it is satisfactory to be able to affirm that one can, and has done so.

We thought it desirable to determine, upon an experimental basis, whether or not cows drenched with nitrates secrete it in their milk. Accordingly, with the kind assistance of the County Agricultural Organiser, we arranged to have two cows put on the maximum dose of "nitre" for seven days and to test their milk day by day. The dose of "nitre" given was 1/3rd oz. twice daily. With this heavy dose of nitrates we anticipated that the appearance of, at least, some trace would be evident in the milk after several days, and were prepared to note how long it persisted, but no trace of nitrates was found in any of the fourteen samples from the two cows.

With regard to the precautions to be observed in order to eliminate fictitious results we would emphasise the importance of ensuring that the filter paper used is free from nitrates. In the early days of our experiments we were greatly puzzled by obtaining positive tests with the large majority of samples. Investigations showed that these fictitious results were due to nitrates contained in the filter paper.

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ON THE TESTING OF DISINFECTANTS.

In a recent communication (Analyst, 1931, 93), Patterson and Frederick give a reasoned and detailed description and discussion on the most recent Admiralty method for the testing of disinfectants, and at the same time criticise the Rideal–Walker method for a supposed lack of accuracy. It is not our desire in any way to enter into a discussion on the relative advantages and disadvantages of either method of testing; but, since we have had frequent occasion to use these and other methods for the determination of relative germicidal powers, a few points in the paper of Patterson and Frederick appear to us to be worthy of some comment. In the first place they rightly emphasise the necessity of rigorous standardisation of materials and technique; the variable coefficients cited by these authors, as obtained by the Rideal–Walker method, were, in fact, from a series of tests which emphasised the necessity for such rigorous standardisation in the case of this method. For uniform samples of disinfectant which do not change or separate with age, it is, we think, fairly definitely established, that, with the modern Standardised Rideal–Walker technique, an error of ± 5 per cent. in the Rideal–Walker figure is the usual tolerance, whilst the frequent occurrence of ± 10 per cent. would indicate some departure in detail from the test. We should be surprised if any other test, including the Admiralty method, could be claimed, at the present time, to be so standardised as to bring the range of accuracy down to these limits.

Since the paper of Patterson and Frederick is published by permission of the Director of the Medical Department, Royal Navy, it is presumably an authoritative account of the correct details of the Admiralty Method, information on which, as printed on contract forms, has hitherto been somewhat meagre; and,

indeed, there are still some points on which further information would be desirable. Some authorities, believing that the disadvantages of comparison of disinfectants by a method which gives coefficients confined to a small numerical scale, are outweighed by advantages in attempting to reproduce the form of medium in which the actual operation of disinfection on a large scale is frequently carried out, add organic matter to the disinfectant dilutions. The Admiralty employs a gelatin, rice starch, salt mixture. It would be desirable to have more information on the colloid reactions occurring on the addition of the rice starch to the gelatin dispersion, as, presumably, the gelatin is partly adsorbed by the starch. In the light of Nugent's work (*Trans. Faraday Soc.*, 1922, **17**, 703), the question of the time of contact may not prove immaterial, whilst the purity and effect of the rate of hydrolysis of the gelatin, and the *p*H of the artificial sea-water, are all variables which may have an important bearing on the possible limits of accuracy of the test. We would certainly agree that the gelatin, rice-starch suspension is superior to urine, faeces or pus, one of which is employed in the Martin–Chick test, and is, at the moment, adopted by another Government Department.

The authors, on pp. 98, 99, lay stress on the need of accurate dosage of culture to diluted disinfectant, and suggest that the dosage employed in the Admiralty Test, *viz.* 0·25 c.c., works satisfactorily. It may, however, be observed, on p. 102, that the actual dosage varies from 0·25 c.c. of culture to 4·5 c.c. of disinfectant, to 0·25 c.c. of culture to 5·4 c.c. of disinfectant dilution, a difference of nearly 2 per cent. This introduces a grave source of error, which should be eliminated in the final draft of the test.

There is also another point in the description of the test on which more clarity would be desirable so as to ensure more uniformity in the results obtained by different observers, namely, the material for making the broth. In the Rideal–Walker method, Lemco is specified. Whilst it is generally agreed that this material is not perfectly uniform in respect of the salt, creatine and creatinine, it is at least more uniform than a bullock's heart or a beef-steak. A meat medium produced on a large scale must always be more uniform than single ingredients bought casually from time to time. Among the users of the Admiralty tests great uncertainty still exists as to whether bullock's heart is employed or beef-steak, as used by McIntosh and Smart. One manufacturer actually possesses a letter from the Admiralty stating that bullock's heart is used, the variability of which has long been established.

Finally, two minor points might be noted. On p. 100, in a discussion on the time given for the test, the reader would gather that time intervals up to $12\frac{1}{2}$ and 15 minutes are necessary in the Rideal–Walker method, and that some latitude is permissible in the choice of the time periods found necessary for killing, from which the coefficient is obtained. Such extended periods have not been used in the Rideal–Walker technique for many years, and it is incorrect to state that a choice of the coefficient is permissible, for a definite statement is made in respect of the technique of the Rideal–Walker method to guard against the possibility of such action by the tester, namely, that the concentration of disinfectant and carbolic acid which gives life in $2\frac{1}{2}$ and 5 minutes and death in $7\frac{1}{2}$ and 10 minutes shall be chosen.

It is to be hoped that the publication of further details of the Admiralty method will remove some of the uncertainties which, at present, confront those who wish to use it.

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THE VALUATION OF CARBOLIC POWDER.

WITH reference to the note by the Borough Analyst for Stepney (ANALYST, 1931, 31), there are one or two points upon which I should like to comment. Among the unsatisfactory powders condemned was one stated to consist of spent gas-lime containing 9 per cent. of naphthalene. Such a powder would not, of necessity, be unsatisfactory bacteriologically, since it might have a Rideal–Walker coefficient at least twice that of a powder complying with the specification.

Some time ago we carried out a long series of investigations on the bactericidal value of various coal-tar products, and found that powders of similar composition to that quoted gave Rideal–Walker coefficients up to 4, whereas a powder containing 15 per cent. of cresylic acid gave average results of 0·75. The character of the tar-acids in these powders has, of course, a very great influence on the Rideal–Walker coefficient; the tar acids having a boiling point range from 200° to 300° C. showed a Rideal–Walker coefficient as high as 20.

In our Rideal–Walker coefficient tests we followed the technique laid down in the booklet "Approved Technique of the Rideal-Walker Test" (H. K. Lewis). This does not give any special directions for powders; in our tests a 1 per cent. suspension of the powder was prepared, and further dilutions were made from this, the 1 per cent. dilution being carefully shaken to ensure fair sampling of soluble and suspended material.

Analysts and others who are responsible for the control of disinfectants supplied to local authorities would be well advised to insist that a Rideal–Walker coefficient be included in the tender, and the chemical specification be so modified that a content of 15 per cent. "cresylic acid" be no longer regarded as the criterion of quality. Such an amended specification would prevent the condemnation of powders which, in actual use, are more efficient than those complying with the rather antiquated specification that "Carbolic Powder must contain not less than 15 per cent. of tar-acids calculated as cresylic acid."

C. E. COULTHARD.

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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.

COUNTY OF ESSEX.

REPORT OF THE COUNTY ANALYSTS FOR THE FOURTH QUARTER, 1930.

TIN IN CHEESE.—Among the samples of cheese examined during the quarter were 35 samples of what is known as "packet" or "processed" cheese, about which a good deal of alarm was recently raised by some observations in a report issued by the Ministry of Health in regard to metallic contamination. These cheeses, of which there are very many varieties, prepared at home and abroad, are wrapped

in tin-foil which is a potential source of metallic contamination, though not necessarily to a serious extent. Much no doubt depends first upon the actual composition of the foil itself, and, secondly, upon the way in which it is prepared. Some packers, we understand, take care to have the foil treated by an invisible process which is akin to the process of lacquering, in order to inhibit as far as possible actual contact between the metal and the cheese. Cheese is a substance which necessarily contains free acid (lactic acid), which must have some tendency to attack metal. The extent to which this may take place will depend a good deal upon care in selection of the foil used, as already indicated, and in cases in which the nature or condition of the foil may invite attack, the extent to which this takes place will be dependent largely upon the age of the cheese, that is to say, the time which has elapsed between its being wrapped in the foil and delivered on to the plate of the consumer. We think that some of the more sensational cases that have been referred to must have related to cheese that had been packed for a long time before being examined.

Of the 35 samples submitted from the various county inspectors during the quarter, no sample was entirely free from tin. Expressing the quantities found in the terms in which tin contamination in preserved foods is recorded, namely, the number of grains or fraction of a grain per pound, we have found in the samples submitted to us a variation from 0·3 grain per pound as a minimum up to 5·5 grains per pound as a (quite exceptional) maximum.

It is difficult to draw a logical line between what might be regarded as passable and what might be regarded as excessive in contamination of this kind. As long ago as 1908 the Ministry of Health undertook an extensive investigation into the question of the contamination with tin in what are ordinarily described as "tinned" or "canned" goods, that is, preserved meat products, soups, fish, vegetables, fruit, etc., put up in tin, or rather tin-plated containers; and, as an outcome of that investigation, it was suggested that when there was an approach to 2 grains per pound in such articles as were under survey, further examination and consideration might be called for on the ground of potential deleteriousness to health. A good deal of discussion was contained in the Ministry's report on the question of the influence on health of small quantities of tin, but the evidence cited was not very definite, and, in some cases, conflicting.

Although no definite regulation has been made in limitation of such contamination, it has, since the report referred to, been generally accepted that any contamination up to 2 grains per pound need not be taken too seriously, while, at the same time, anything above this arbitrary figure of 2 grains per pound should be regarded as excessive. As this generally received limitation applies to articles which are usually consumed at any given meal in far larger quantities than cheese, it would seem that, in the case of cheese, the presence of tin, at any rate up to 2 grains per pound, need not be taken as being a grave contamination, but, having regard to the fact that, in this recent investigation of our own, 28 samples out of 35—or 80 per cent. of the whole—showed a contamination which did not exceed 2 grains per pound, anything above this figure may reasonably be looked upon as unnecessarily and undesirably excessive.

The 28 samples in which the contamination did not exceed two grains per pound included samples prepared from many varieties of cheese, *viz.* many cheeses of the Cheddar variety, both home-made and "Colonial," and a large proportion prepared from Gruyère cheese in Switzerland, France and elsewhere. The seven samples which contained more than two grains per pound, and which we have recorded as unsatisfactory, were all samples of Gruyère cheese (either Swiss or

French), the contamination, expressed as grains of tin per pound of cheese, being 2·2, 3·0, 3·0, 3·0, 4·0, 4·5, 5·5 grains, respectively.

It seems probable that the wide attention which has lately been directed to this question will stimulate any packers who may have been lax in the matter to pay increased attention to the preparation of the foil used, and also, as far as possible, to influence those concerned in the channels of distribution with a view to shortening, as far as possible, the interval between packing and the delivery of the cheese to its final customer.

BERNARD DYER.
GEORGE TAYLOR.

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.

THE ARTIFICIAL CREAM ACT, 1929. AN APPEAL CASE.

KEATING *v.* LYONS.

ON 6th February, an Appeal was heard in the King's Bench Division (before the Lord Chief Justice, Mr. Justice Avory, and Mr. Justice Macnaghten) from a conviction by the Swindon magistrates of a firm for selling Swiss rolls, chocolate cream sandwiches, and vanilla cream sandwiches containing a substance purporting to be cream, but which was alleged not to be cream as defined by the Act (ANALYST, 1930, **55**, 689).

Mr. Roland Oliver, K.C., for the appellants, contended that the Act did not apply to a composite article, such as had been sold, but only to cream sold separately as cream. In his submission, the Act was not meant to apply to compound articles such as Swiss roll or chocolate cream, which had long been sold without complaint.

The Lord Chief Justice (Lord Hewart) gave the decision of the Court, which was that the case should be sent back to the Swindon justices for them to consider whether the articles in question were sold as substances purporting to be cream as defined in the Act.

MILK: THE FREEZING-POINT TEST.

ON February 12th, the South Staffordshire Stipendiary (Mr. B. G. Grimley) delivered his reserved judgment in a case in which a dairy farmer was summoned for selling adulterated milk.

The solicitor for the defence had endeavoured to establish, through the evidence of employees, that no water had in fact been added; and, secondly, by expert evidence, that the legal presumption, based upon the analyst's certificate, was not justifiable, since the conventional tests at present employed were in themselves inconclusive.

The Stipendiary referred to the report of the Standing Committee on the Law relating to Milk (1930) (*J. Ministry Agric.*, 1930, p. 386), which said that the result of an analysis of a single sample was never proof of adulteration, and that the only proof

was the evidence of an eye-witness (which one never had), or by circumstantial evidence based on the analysis. It was his duty, he said, to take the law, not as the circular suggested it should be, but as it was at present. It was certainly significant that when no one watched the milking, the milk was presumably adulterated; whereas, when the milking was watched, the milk was all right. After an "appeal to the cows" on the day after the summons, the milk was found to be of good quality, and there must have been some unnatural cause for this. It has been laid down that, in order to succeed, the defence must satisfy the Court beyond doubt that no water had been added to the milk, and the evidence must be positive, clear and complete, leaving no gap from the moment at which the milk was taken from the cows to the moment at which it was handed to the purchaser; and where a doubt remained, it was quite clear that the burden of proof had not been discharged, and the defence had failed. In this case a wagoner about the farm had not been called as a witness, and the evidence of one brother was contrary to that of the other. He was bound by the decision of the Lord Chief Justice; the defence did not leave his mind free from doubt, and, therefore, failed.

Dealing with the scientific evidence, the Stipendiary said that it had complicated the case, and was outside his experience in such cases. "What I certainly do hope," he said, "is that, in subsequent cases, I shall have evidence about the freezing-point test; and, in the meantime, if Mr. Hanley (who gave evidence for the defence) meets with any herd of cows similar to the one he mentioned, which gave an exceptionally low freezing point, I hope he will communicate with Mr. Jones (the Staffordshire County Analyst), and that Mr. Jones will join him in an investigation." In view of the fact that the defendant knew nothing about the matter, he would deal with him under the First Offenders' Act, not recording a conviction, and reducing the costs to £25 4s.

Department of Scientific and Industrial Research.

THE INVESTIGATION OF ATMOSPHERIC POLLUTION.

REPORT ON OBSERVATIONS IN THE YEAR ENDING 31ST MARCH, 1930. (SIXTEENTH REPORT.)*

THE general arrangement of the Report follows that of 1929 (ANALYST, 1930, **55**, 755). A map is included to show the positions of the contributing stations, and data have been collected as to steps taken by co-operating authorities to abate the smoke nuisance, and these are discussed.

Report of the Atmospheric Pollution Research Committee.

In addition to the usual investigations, the Atmospheric Pollution Committee have initiated an enquiry into the spread of pollution, and measurements are being made at Norwich, as a centre isolated enough to prevent interference from other sources of pollution. Daily observations on wind direction and velocity are taken, followed by pollution measurements at selected spots downwind, made by means

* Published 27th February, 1931, pp. 74. Obtainable from H.M. Stationery Office, Adastral House, Kingsway, W.C.2. Price 4s. net.

of an Owens Jet Dust Counter. The data obtained will be analysed when a year's cycle is completed. Suggestions as to the best method of protecting the deposit gauge bottles from being burst by frost are being tested.

Superintendent of Observations.—During the year there were set up 8 new deposit gauges, making a total of 84 maintained by 38 authorities, and 11 automatic filters maintained by 6 authorities. The maximum and minimum monthly deposits in metric tons per 100 sq. km. were:—*Tar*: Salford, Regent Road, 60; Glasgow, Bellahouston Park, 2; *Other insoluble carbonaceous matter*: Rochdale, St. Mary's Church, 386; Birmingham, West Heath, 39; *Ash of insoluble matter*: Rotherham, Town Hall, 819; Garston, Watford, 61; *Ash of soluble matter*: London, Kew, 481; Leicester, Western Park, 85; *Total solids*: Liverpool, Netherfield Road, 1840; Leicester, Western Park, 304. The total solids comprised:—*Sulphates*: Huddersfield, Cooper Bridge, 300; Leicester, Western Park, 36; *Chlorine*: Salford, Regent Road, 119, Edinburgh, Princes St. Gardens, 15; and *Ammonia*: Huddersfield, Cooper Bridge, 61; Bournville, 1; *Rainfall*: Rochdale, 101 mm.; London, South Kensington, 40 mm.

The different observation stations have been classified A—D on the basis adopted in previous reports; and in Table II tar, other carbonaceous matter, insoluble ash, loss on ignition and soluble ash are classified under the different letters. Tar shows a reduction in 21, an increase in 13 out of 38 stations; other carbonaceous matter, a reduction in 23, an increase in 16 of 39 stations; insoluble ash, a reduction in 20, an increase in 17; loss on ignition, a reduction in 28, an increase in 11; soluble ash, a reduction in 28, an increase in 7; and total solids, a reduction in 24, an increase in 14 stations all of 39 stations.

On the whole, the current year shows a general improvement in the purity of the air, but tar and sulphates, regarded specifically as combustion products, increased in 1 Edinburgh and 2 London stations.

Automatic Filter Results.—The fact that the maximum concentration of sooty impurity for Westminster Bridge station, although occurring at 9 a.m. last year, was this year maintained on to 10 a.m., is regarded as probably due to incidence of foggy weather. The graph of the 3 days of London fog of January 7th–9th, 1929, shows the maximum at 10 a.m. for the 7th and 8th, and 4 to 5 p.m. for the 9th, owing to the banking up of the smoke, due to insufficient ventilation, with consequent delay of the maximum. The maximum at Kew for "Z" days* only was at 5 p.m., and at South Kensington the second p.m. maximum for ordinary days was higher than the 9 a.m. maximum, which was markedly different from what was observed in 1929.

Measurements of Daylight.—The measurements carried on at Leeds are set out as daily average figures for each month at 2 stations, and it was found that the measurements at the centre of the city were consistently lower than those about 2½ miles away.

Ultra-Violet Rays.—The measurements at Sheffield and Rotherham show that in the case of Surrey Street, Sheffield, no ultra-violet rays were present in November and December.

Appendix.—This discusses observations on ultra-violet light in Rochdale and other places. There is evidence that the great loss of ultra-violet light in the middle of the week is mainly due to factory smoke, for non-factory towns have most light in the middle of the week and least on Sundays. The power of the

* "Z" days are any which show, at any time of the day or night, a concentration of 4 or over on the scale of shades, or 128 mgrms. per 100 cb. metres.

ultra-violet rays, measured by their fading action per hour, is not uniform, but depends on the season in such a way that it is proportional to the sine of the altitude of the sun.

DEPOSIT OF MICRO-ORGANISMS AT SOUTHPORT.—In March, 1930, a deposit fell over everything in the neighbourhood of Southport, and for an area of about 10 square miles the deposit is described as "remarkably thick." The dust, examined immediately after falling, was found to consist of algae, which are common epiphytes on tropical leaves, but are only found in this country on the windward side of rocks in mountain districts, and they have been noticed in the Peak district. At the time of the deposit the wind was from that direction, and it is possible that the algae, which are propagated by yellowish brown spores, which under suitable conditions grow as filaments, encountered soot particles which adhered to them, as the microscopical appearance of the dried deposit showed adhering bodies.

The usual detailed General Deposit tables are included in the Report.

D. G. H.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Physico-Chemical Constitution of Spray-dried Milk Powder. Fat in Spray-dried Milk Powder. L. H. Lampitt and J. H. Bushill. (*J. Soc. Chem. Ind.*, 1931, **50**, 45–54T.)—The "free" fat of a milk powder is taken to be the fat extractable under the authors' standard conditions by organic solvents, and on spray-process milk, calculated on total fat, it varied between 3·33 and 14·22 per cent., and on roller-process powder, between 91·6 and 95·8 per cent. To 2 grms. of powder in a glass-stoppered 250 c.c. bottle are added 100 c.c. of carbon disulphide or other solvent, and, after standing for 18 hours with gentle shaking during the first hour, the contents of the bottle are filtered through a Green's fluted filter paper (591½, 19·5 cm. diameter) into a 50 c.c. graduated flask, the first few c.c. being disregarded and the level adjusted by a capillary tube. The contents and washings from the flask are then poured into a weighed Soxhlet flask, dried and weighed. The residue is the "free" fat from 1 grm. of powder, and in the case of substances all the fat of which is normally extractable, the results agreed well with those obtained for the same substance by the Soxhlet process. If the surface of the spray-process particles is increased by grinding (3 hours), the "free" fat may be increased up to 83 per cent. of the total fat, but the fat is practically all "free" in the roller-process powder. The reasons for these differences are discussed. When milk powder absorbs moisture it first becomes clammy, and at the next stage is distinctly dry, hard and powdery to the touch, and this corresponds with the "freeing" of the fat. The definite range of moisture content (about 8·6 to 9·2 per cent.) at which the fat is freed is called the "critical moisture" content,

and is accompanied by the crystallisation of the amorphous lactose in the spray-process milk, but, apparently, a freshly-made powder reacts more slowly than an older one. On drying out such absorbed moisture the powder does not revert to its original state; and the critical moisture range, while independent of the fat content as such, was found to be proportional to the amount of solids-not-fat in the powder. While no theory is put forward as to the structure of the milk powder particle, it is clear that the lactose, in its amorphous state, has a marked effect on the availability of the fat to solvents, and that crystallisation of the lactose appears to result in freeing the fat. D. G. H.

Determination of the Ash of Bread. F. Bernardini and E. A. Gauthier. (*Giorn. Chim. Ind. Appl.*, 1931, **13**, 7–9.)—In the ordinary method used for determining the ash of bread the preliminary digestion with water does not extract all the sodium chloride, and the subsequent incineration of the residue for 15 minutes at a dull red heat results in a black ash, and, if the heating is prolonged, loss of chlorine occurs. Satisfactory results are obtained by Cutolo's method (*Boll. Soc. Naturalisti Napoli*, 1914, **26**, 69), in which the ash is determined on the bread alone, and also on a second sample after treatment with alcoholic potassium hydroxide; the chlorine in each residue is determined by Volhard's method, and the difference between the two, calculated as sodium chloride, added to the weight of the first ash. This procedure is, however, laborious if many samples have to be tested, and equally good results may be obtained as follows:—Five grms. of the finely powdered dry bread, weighed in a platinum dish, are transferred to a beaker, treated with 3·2 c.c. of water, and, after about 10 minutes, when the bread has swollen, with 50 c.c. of 99·5 per cent. methyl alcohol. The mass is stirred occasionally during an hour, after which the liquid is decanted on to a filter, and the residue twice treated similarly with one-half of the above quantities of water and methyl alcohol. The residue in the beaker is dried in an oven and then restored, with the filter, to the platinum dish, in which it is charred over a naked flame, incinerated in a muffle furnace at a low red heat, and weighed. This procedure gives the total ash, less sodium chloride, and thus furnishes information of use in judging the character of the original flour. T. H. P.

Essential Oils from Grain Germs: Improver for Bread and Foodstuffs. (*Perf. Essent. Oil Record*, 1931, **22**, 38.)—According to the process here described, which is the subject of a British patent application by M. Baumann, wheat germs contain, in addition to the oily and fatty constituents which rapidly produce rancidity in flour, etheric oils capable of imparting a pleasant flavour to bread. Such ethereal oils, which may be obtained, not only from wheat, but also from the germs of rye, barley, oats, maize, buckwheat, etc., and from such fruits as nuts, almonds, and coconuts, may be separated either by extraction and distillation processes, or exhaustion or solution processes with the aid of various organic solvents, or "enfleurage" processes. The oils may be incorporated directly in the flour, or may be mixed with the yeast or other products used in the manufacture of bread. T. H. P.

Ursolic Acid. C. E. Sando. (*J. Biol. Chem.*, 1931, **90**, 477–495.)—Sando (*J. Biol. Chem.*, 1923, **56**, 457; ANALYST, 1923, **48**, 496) reported the results of an investigation of the constituents of the wax-like coating on the surface of the apple, from which there was isolated, among other substances, a compound insoluble in petroleum spirit to which the name malol was given. It possessed the same formula, $C_{30}H_{48}O_3$, and characteristics as urson, originally isolated by Trommsdorff (*Arch. Pharm.*, 1854, **80**, 273), from bearberry leaves, *Arctostaphylos uva ursi*, and exhibited the same general chemical properties as prunol, isolated from the leaves of the wild black cherry, *Prunus serotina*, by Power and Moore (*J. Chem. Soc.*, 1910, **97**, 1099). In 1924, van der Haar (*Rec. trav. chim. Pays-Bas*, 1924, **43**, 367) reported a re-investigation of urson, suggested the name ursolic acid as being in accord with its chemical character, and claimed for the substance the formula $C_{31}H_{50}O_3$. On the basis of this work he concluded (*Rec. trav. chim. Pays-Bas.*, 1924, **43**, 542; 548) that malol, prunol and ursolic acid were identical. The formation of the acetylation products was also brought into question. Because of these discrepancies a more exhaustive chemical study of the three substances has been made, the results of which are given, and may be summarised as follows: Malol, prunol and urson are identical; van der Haar's conclusions in this respect are thus confirmed. The name, ursolic acid, has been adopted for the substance. The formula for ursolic acid appears to be $C_{30}H_{48}O_3$, and not $C_{31}H_{50}O_3$, as claimed by van der Haar. This conclusion is based on the results of 92 combustions, including those of the parent substance and many of its derivatives. The following compounds have been prepared and analysed: ursolic acid from three sources, the diacetyl derivative of ursolic acid, monoacetyl-ursolic acid, regenerated ursolic acid, methylursolate, monoacetylmethyl ursolate, regenerated methyl ursolate, phthalylursolic acid, phthalylmethyl ursolate and phenacyl ursolate. The average values of numerous combustions of these substances agree more closely with the C_{30} formula than with the C_{31} formula. The preparation of monoacetylmethyl ursolate, by treating monoacetylursolic acid with thionyl chloride and boiling the product with methyl alcohol, constitutes further confirmatory evidence of the hydroxy-acid structure of ursolic acid. P. H. P.

Origin, Occurrence and Detection of 2.3-Butyleneglycol in Wine and Fruit Wine. J. Pritzker and R. Jungkunz. (*Z. Unters. Lebensm.*, 1930, **60**, 484–488.)—The sample (25 c.c.) is evaporated in a dish on the water-bath to 5 c.c., which is transferred to a 50 c.c. Erlenmeyer flask, and 4 grms. of crystalline sodium carbonate added and dissolved by warming. The mixture is then cooled in ice, 0·5 c.c. of bromine water added, and, after 30 minutes at room-temperature, during which time it is shaken to remove carbon dioxide, the mixture is neutralised (to litmus) with 10 per cent. hydrochloric acid. Any remaining excess of bromine should be removed by a drop of concentrated sodium thiosulphate solution, and the potassium iodide and starch test applied. The diacetyl, produced by oxidation of the 2.3-butyleneglycol, is then detected by distillation with 50 c.c. of 30 per cent. ferric chloride solution as oxidising agent in a Reichert–Meissl–Polenske apparatus,

the distillate (20 c.c.) being collected in a mixture of 2 c.c. of 20 per cent. hydroxylamine hydrochloride, 3 c.c. of 20 per cent. sodium acetate and 1 c.c. of 10 per cent. nickel chloride solutions. Dimethylglyoxime is produced, and when the distillate separates into 2 layers, the characteristic crystals of nickel dimethylglyoxime are formed as a ring at the junction. The distillate may then be warmed in a water-bath at 80° C. for an hour, cooled, and the precipitate separated by filtration, washed with hot water and dried in the water oven till constant in weight. The factor 0·624 gives the weight of 2.3-butyleneglycol. This procedure gives higher results than other methods, a value of about 0·6 per cent. being found for wine and fruit wine (Spanish Montagner), though theoretical considerations indicate the possible presence of 0·2 per cent. None was found in bottom-fermentation beer, but it is uncertain whether this is due to the difference in the nature of the sugars or of the yeast, or to the working temperature involved. The method serves as a specific distinction between fermented and unfermented wines, *e.g.* between true sweet wine (such as Malaga), from which alcohol has been removed, and wine prepared from unfermented grape or fruit must. The German official method for the determination of glycerin (*id.*, 1921, **13**, 93) requires no correction for the presence of 2.3-butyleneglycol. J. G.

Oleic Acid Rancidity of Fats. II. Measurement of Rancidity. K. Taüfel and J. Müller. (*Z. Unters. Lebensm.*, 1930, **60**, 473–484.)—The literature dealing with qualitative tests for rancidity is discussed fully and critically, with particular reference to the effects of light and of interfering substances, and to the possible modification of such methods in order to obtain approximately quantitative results which will enable the evolution of rancidity to be followed. The Kreis test was found to give reproducible results if measurements were made by comparison of the resulting colour with the colours of a series of 12 photographic filters, ranging from the colour obtained from freshly-prepared ethyl oleate to the maximum colour obtainable from the strongly rancid ester. The colour match was made 2 minutes after mixing the reagents. The rate of production of rancidity, according to this method, attains a constant maximum (colour No. 12) after 14 days in the light, but increases very slowly and linearly in the dark, reaching only colour No. 1 after 25 days. The acid value increases almost linearly between the 8th and 25th day at a rate intermediate between those shown by the above Kreis tests. No relation between the 3 curves was detectable. Comparison was then made with the rate of absorption of oxygen (Genthe). The volumetric method, in which the change in pressure of oxygen in contact with oil was measured manometrically, gave unreliable results, and the increase in weight of about 2 grms. of oil (oleic acid and its ethyl ester) exposed to the air in a glass dish was preferred. Both compounds showed approximately the same rates of increase in weight, these being approximately constant (70·0 and 76·4 mgrms., respectively), after 70 (and up to 90) days. Although the method allows a wide range of time to be studied, unreliable results are obtained over the first 2 days. Comparison with the Kreis test for 1 to 20 days showed a much

more rapid increase in rancidity in this test, though both curves tend to approach the same value at the end of this period. It is probable that the different methods correspond with changes of different character. J. G.

Oil of *Wrightia annamensis, Dubard and Eberhardt*; an Oil resembling Castor Oil. M. L. Margaillan. (*Compt. rend.*, 1931, **192**, 373–374.)—The seeds of *Wrightia annamensis*, gathered in Tonkin as "cay thu muc" N.O. *Apocyanaceae*, had the following composition:—Moisture, 5·8; fat (to petroleum spirit), 36·1; protein, 29·1; cellulose, 5·1; ash, 4·3 and non-nitrogenous residue, 19·6 per cent. The ash contained alumina, 50·6; silica, 19·1; potash, 10·7; and phosphoric acid, 11·2 per cent. The oil is of a dark pomegranate-red colour; it is soluble in alcohol in all proportions and has, $d^{25°}$ C. 0·966, viscosity coefficient C.G.S. at 20° C., 6·76, $n_D^{20°}$ C., 1·480; flash point (open-dish method), 279° C.; ignition point, 320° C.; calorific value (bomb), 8720 grms.-cal.; saponification value, 184; iodine value (Hübl), 85; acetyl value, 127; unsaponifiable matter, 1·0 per cent.; free fatty acids (as oleic acid), 2·7 per cent. The oil showed no trace of drying in one month. The viscosity curve for varying temperatures was analogous to that of castor oil; the acetyl values of the two oils are similar; their combinations with sulphur, their polymerisation powers, and their soaps are closely alike, but *Wrightia* oil is more soluble than castor oil in petroleum spirit. A hydroxy-oleic acid, probably identical with ricinoleic acid, is the principal constituent of the fatty acids of *Wrightia* oil, and the presence of a small proportion of a hydroxy-linoleic acid is considered probable from the results obtained by fractional crystallisation of the barium salts and by fractionating the cooled fatty acids.

D. G. H.

Nicotine Contents of Dutch Cigars. A. Van Druten. (*Z. Unters. Lebensm.*, 1930, **60**, 501–518.)—The methods and results of previous workers are discussed, and the highest, lowest and mean values of the nicotine contents found by them for ordinary, and partly and "wholly" denicotinised cigarette and cigar tobaccos are tabulated. Examinations of Bodnár, Straub and Nagy's micro-titrimetric method (*Biochem, Z.*, 1928, **195**, 103), of Rasmussen's silicotungstic acid method (ANALYST, 1916, **41**, 208) and of Pfyl and Schmitt's method (*id.*, 1927, **52**, 728), showed that the second is usually reliable under the conditions specified (*loc. cit*), but requires 10 grms. of tobacco, whilst the first gives high results, and the third, modified as follows, is to be preferred. The sample is ground in a mortar, 3·5 grms. mixed in a 200 c.c. distillation flask with 35 c.c. of water, and 25 grms. of sodium chloride and an aqueous paste containing 1 grm. of magnesium oxide added. The mixture is steam-distilled, 150 c.c. of distillate collected and neutralised to methyl red with 0·1 *N* hydrochloric acid, and the indicator removed by a drop of bromine water. Not less than 75 c.c. of 0·05 *M* picric acid solution are then added, the mixture cooled in water for 2 hours, and the precipitated nicotine dipicrate filtered on a platinum cone-filter (5·5 cm. in diameter) and washed with 4 c.c. of 10-fold diluted reagent, and twice with water. The filter and contents are then transferred to a 100 c.c. flask, 10 c.c. of water and 4 drops

of phenolphthalein (1 : 100) added, and the mixture titrated, with shaking, with 0·1 *N* sodium hydroxide solution, 25 c.c. of toluene being added when the red colour first appears just before the end-point. The factor 0·243 gives the percentage of nicotine. The results for 104 "normal" Dutch cigars varied from 0·5 to 2·7 (mean 1·42) per cent. on the dry tobacco, and were independent of size and price, whilst 0·57 to 1·53 per cent. was found in six cigars sold as containing "minimum" and "absolutely harmless" amounts of nicotine. The opinion is expressed that "nicotine-free" cigars should contain only traces of nicotine, and that 0·5 per cent. is a suitable upper limit for "denicotinised" cigars. J. G.

Importance of the Acidity of Tobacco for its Hygienic Evaluation. A. Faitelowitz. (*Z. Unters. Lebensm.*, 1930, **60**, 518–523.)—The acidity of cigarette tobacco (*A*) is determined by mixing 10 grms. and 100 c.c. of water for 2 hours and titrating 100 c.c. of the strained liquor with 0·1 *N* sodium hydroxide solution. The basicity of the un-neutralised smoke (*B*) is given by the volume of 0·1 *N* sulphuric acid neutralised when the smoke from 10 cigarettes is drawn through the acid. The ratio *A*/*B* is an indication of the degree of mildness of the tobacco. By "smoking" cigarettes in a glass tube by means of an aspirated current of air, and analysis of the smoke before and after neutralisation, the following data were obtained for 6 tobaccos:—Nicotine in tobacco, 0·4 to 3·03 per cent.; nicotine in neutralised smoke (100 cigarettes), 0·2 to 1·66 per cent.; *A* 22 to 27·5; *B* 50 to 83; free bases in neutralised smoke (100 cigarettes), 0 to 30 c.c. of 0·1 *N* sulphuric acid. Similar values are also given for the tobacco after making alkaline, the nicotine and free bases in the neutralised smoke then being 0·3 per cent. and 51 to 85 c.c. higher, respectively. Attempts to correlate these data with the tastes of the corresponding tobaccos indicate that amino-bases are liberated during the smoking process and irritate the throat, but that their effect depends on the degree of acidity of the tobacco, *i.e.*, on the extent to which they are neutralised as the smoke traverses the cigarette to the mouth. Cigar tobacco has a lower acidity than cigarette tobacco, and is sometimes alkaline. J. G.

Analysis of Hops. W. Wöllmer. (*Allgem. Brauer- und Hopfen-Z.*, 1930, **70**, 1531–33; *J. Inst. Brew.*, 1931, **37**, 81–83.)—The following scheme, proposed by the author, has been adopted by the Analysis Commission of the German Brewing Institutes: *Moisture.*—Three grms. of finely ground sample are dried at 104° to 105° C. for 3 hours, as in the German method of malt analysis. *Extraction for Resins.*—Ten grms. of the sample are shaken for 2 hours in a bottle (with a stopper, if of rubber, of diameter less than $\frac{2}{3}$ inch) with 100 c.c. of ether, and, after a period of rest of 30 to 60 minutes, according to the age of the hops, 50 c.c. of the top liquor are pipetted into a 200 c.c. conical flask and evaporated at 70° C. The last traces of ether are removed in a current of air free from carbon dioxide, contact of air with the hot resins being avoided as far as possible. The cold residue is then re-extracted with 20 c.c. of methyl alcohol, the mixture filtered through a paper (3 inches in diameter), and the filtrate and methyl alcohol washings

made up to 50 c.c. *Total Resins.*—Ten c.c. of this extract are pipetted into a weighed flask, the alcohol removed on the water bath, and the residue dried by passage of a current of dry carbon dioxide, rapidly at first, and then at 2 bubbles per second under a 20-inch vacuum (to remove hop-oil). This is followed by a current of air, and the flask is weighed, when the weight of extract (grms.) × 102 gives the percentage of total resins in the air-dry hops. *Soft (Hexane-Soluble) Resins.*—Fifteen c.c. of methyl alcohol extract are shaken by hand with 50 c.c. of hexane and 25 c.c. of water in a 150 c.c. stoppered cylinder for 5 minutes, and after separation, 40 c.c. of the upper (hexane) layer are pipetted into a weighed flask, and the same procedure followed as for the total resins (factor 85). *Humulone.*—If 1·07 grms. of pure crystalline lead acetate are rinsed into a 100 c.c. flask with methyl alcohol, followed immediately by 0·05 grm. of acetic acid, then 1 c.c. of the solution obtained after dilution to 100 c.c. with methyl alcohol will precipitate 1 per cent. of humulone in the sample under the following conditions:—The resin extract (10 c.c.) is warmed quickly at 50 to 60° C. in a 50 c.c. conical flask, and the above solution added till a slight excess of lead is present. This is shown by a brown stain at the junction on a piece of filter-paper of adjacent drops of supernatant liquid and 5 per cent. sodium sulphide solution, and, in the case of new hops, 4 to 5 c.c. may be added at once, followed by additions of 0·5 c.c. The precipitate (which dissolves in a large excess of reagent) is collected after 10 minutes on a Schott glass filter-crucible (No. 1 G4, emptying, if filled with water, in 20 to 25 seconds at a pressure of 1 atmos.), washed with 20 c.c. of methyl alcohol and once with ether, and weighed, after 30 minutes, at 105° C., followed by cooling in a desiccator (factor 65·2). The determination of the end-point of the precipitation, which depends on the age of the hops, is facilitated by centrifuging or by allowing the liquid to stand overnight; but in all cases the filtrate should be tested with 0·5 c.c. of reagent. *Hard Resins.*—The soft resins are subtracted from the total resins. The limit of error is about 0·3 per cent. in each case. Extraction of hops in a Soxhlet apparatus gives high values for the total resins. J. G.

Determination of Arsenic and Mercury in Donovan's Solution. T. T. Cocking. (*Quart. J. Pharm.*, 1930, **3**, 575–577.)—*Arsenic.*—The determination of arsenic in Donovan's Solution should involve the determination of the total arsenic in addition to the determination of the arsenious iodide, because some of the latter becomes oxidised on keeping the solution. The following method is suggested: To 50 c.c. of Donovan's Solution is added 1 grm. of sodium bicarbonate; the solution is titrated with $N/10$ iodine and the result calculated to AsI_3. Starch is not used to indicate the end-point, as this is quite sharp to one drop of the volumetric solution. Forty-five c.c. of hydrochloric acid (31 to 32 per cent. HCl) are now added, and the mixture is kept at the laboratory temperature for 10 minutes and then titrated with $N/10$ thiosulphate, without using starch. The total arsenic thus indicated is calculated to the equivalent of AsI_3. The solution *must not* be diluted with water before the titration. It is pointed out that deviation from this method for the determination of total arsenic may lead to the formation

of yellow arsenic iodide which would obscure the end-point. The author notes that the formation of a brilliant yellow precipitate of arsenious iodide, when an excess of conc. hydrochloric acid and potassium iodide solution are added to a solution of arsenious acid, is a sensitive reaction for arsenic.*

Mercury.—Fifty c.c. of Donovan's Solution are rendered strongly ammoniacal and the mercury precipitated by hydrogen sulphide; the mercuric sulphide is filtered off on a Gooch crucible, washed with ammonia, dried at 120° C., and the weight calculated to the equivalent of mercuric iodide. The method of the United States Pharmacopoeia is stated to give low and variable results. S. G. C.

Abstractor's note* :—Papers by G. Bressanin on the qualitative and quantitative aspects of this arsenious iodide reaction appeared in *Boll. Chim. Farm.*, 1911, **50, 691, 727 ; and *Gazz. Chim. Ital.*, 1912, **42**, 456.

Biochemical.

Phosphorus Distribution, Sugar and Haemoglobin in the Blood of Fish, Eels and Turtles. C. M. McCay. (*J. Biol. Chem.*, 1931, **90**, 497–505.)—Little information is available concerning the chemical composition of the blood of the lower vertebrates, owing, probably, to the general difficulty of obtaining unclotted samples. The blood of all species that have nucleated erythrocytes has the common property of very rapid clotting after it is removed from the body. Among these, the blood of the turtle is probably the slowest to clot, whilst fish and eel blood are at the other extreme. A study has now been made to determine the phosphorus distribution in the blood of some of the lower cold-blooded vertebrates. A few other data, such as those upon the phosphorus distribution in the blood of the cow, have been incorporated in order to show the contrast in composition of two very different vertebrates. Values upon the sugar and haemoglobin of blood of these lower vertebrates have also been included in the cases in which they may throw some light upon the variations in phosphorus that have been discovered. The results show that the total phosphorus in the blood of animals with nucleated erythrocytes is higher than that in blood with non-nucleated cells. The blood of carp and pike has approximately four times as much phosphorus per unit volume as beef blood. The plasma phosphorus of pike blood is higher than that of carp; the blood sugar is also higher in pike blood. This may be a species characteristic related to activity, or may be associated with the differences of the two species in their resistance to asphyxiation. The phosphorus distribution in turtle blood differs from that of fish blood in the low values for the plasma constituents. The phospholipids of turtle blood are especially low. These are associated with the low cholesterol values that have been shown previously in turtle blood. The blood of eels is lower in phosphorus than that of fish blood, but higher than that of cattle. The blood changes that accompany the death of the lamprey-eels after spawning show some lowering of the phosphorus values, but the changes are chiefly concerned with glucose and haemoglobin decreases. Blood with large nucleated cells contains the same amount of total phosphorus as that with small cells. The plasma of both fish and turtle blood is slightly yellow, like that from the cow. P. H. P.

Colorimetric Determination of the Tyrosine and Tryptophan Content of Various Crude Protein Concentrates. W. D. Mcfarlane and H. L. Fulmer. (*Biochem. J.*, 1930, **24**, 1601–1610.)—In the course of investigations on poultry nutrition it was desired to know the tyrosine and tryptophan content of various crude protein concentrates. Though the colorimetric methods have sometimes given low values, and are open to the usual objections to colorimetric methods in general, they were the only methods which could be satisfactorily applied to the determination of tyrosine and tryptophan in a routine way, and it is doubtful whether any of them are absolutely reliable for determinations on purified proteins. The colorimetric method of Folin and Ciocalten (*J. Biol. Chem.*, 1927, **73**, 627) and other methods were tried. The tyrosine and tryptophan content of the proteins of buttermilk powder has been found to be much higher than that of the other crude protein materials investigated. No very appreciable difference in the tyrosine content of fish meal, cod-liver meal, meat meal and tankage was found. Conflicting results as to the tryptophan content of the proteins of fish meal and meat meal, depending upon the method of determination, have been found. In general, the tryptophan content of fish meal was found to be higher than that of meat meal. The tryptophan content of tankage has invariably been found to be much lower than that of fish meal and meat meal. The limitations in the methods described for the determination of tryptophan leave some doubt as to the actual tryptophan content of any of these materials, particularly in the case of the cod-liver meal proteins. The alkali digest of these crude protein materials contains some substance, or substances, precipitable by mercuric sulphate and giving a blue colour with the phenol reagent, which, unlike tryptophan, is soluble in toluene. This unknown chromogenic substance does not appear to be indole, as Kraus (*J. Biol. Chem.*, 1925, **63**, 157; ANALYST, 1925, **50**, 246) has concluded. The results obtained after extraction of the alkali hydrolysates with toluene would appear to represent the true tyrosine and tryptophan content of these protein concentrates. P. H. P.

Presence of Allantoinase in Fungi. [Test for Glyoxylic Acid.] A. Brunel. (*Compt. rend.*, 1931, **192**, 442–444.)—To detect allantoinase in fungi, a weighed quantity of the material is treated with an equal weight of anhydrous glycerin for 12 hrs. in an ice-chest, then with an equal weight of water for 6 hrs., and centrifuged. To 100 c.c. of 0·2 per cent. allantoin solution are added 25 c.c. of the centrifuged liquid, 0·125 grm. of ammonium bicarbonate and 1 c.c. of chloroform; a control, prepared similarly, but after heating the centrifuged liquid at 100° C. for 30 mins., is treated in the same way. Both preparations are kept at 40° C. for 12 hrs., and are then rendered distinctly acid with 5*N* hydrochloric acid and heated at 100° C. for one minute. To detect the glyoxylic acid formed, together with urea, by hydrolysis of the allantoic acid, 2 c.c. of each of the two liquids are treated with 4 drops of 1 per cent. phenylhydroxylamine hydrochloride and, after cooling, with 2 drops of 5 per cent. potassium ferricyanide solution and 3 c.c. of concentrated hydrochloric acid; an intense red coloration indicates the presence of

glyoxylic acid, the control remaining colourless. To detect the urea, 3 c.c. of each of the two liquids are neutralised with potassium hydroxide and treated with double the volume of acetic acid and with 5 per cent. (of the total volume) of a 10 per cent. solution of xanthydrol in methyl alcohol; the characteristic precipitate of the xanthydrol derivative indicates the presence of urea, the control remaining clear. In addition, the silver salt of allantoic acid may be prepared from the allantoin solution after fermentation for 48 hours at 40° C. This is defecated with 1 grm. of solid silver nitrate and filtered, the cold filtrate being treated with solid mercuric acetate and the precipitate formed separated by centrifuging, washed, and decomposed by hydrogen sulphide in presence of water (15 c.c.). After expulsion of the excess of hydrogen sulphide by means of a stream of air, the liquid is filtered, neutralised, and treated with silver nitrate. The silver allantoate precipitated, when crystallised from boiling water, forms groups of microscopic needles; it contains 38·12 per cent. of silver. By the above means the presence of allantoinase in 67 different fungi has been ascertained. T. H. P.

Reaction of Antimony Trichloride with Cod-Liver Oil and its Unsaponifiable Fraction. E. L. Smith and V. Hazley. (*Biochem. J.*, 1930, **24**, 1942–1951.)—The authors have found that the unsaponifiable fraction of cod-liver oil gives with antimony trichloride in chloroform a blue colour proportional to its concentration. The line representing the dilution effect for the total unsaponifiable fraction is tangential at the origin to the dilution curve for the corresponding cod-liver oil. The chromogen is more stable than has sometimes been supposed, and there is no particular difficulty in obtaining practically complete extraction of the total unsaponifiable matter from cod-liver oil. The unsaponifiable fraction can be extracted almost without loss with ether, ethyl acetate, chloroform or petroleum spirit under suitable conditions. Norris and Church (*J. Biol. Chem.*, 1930, **85**, 477; **87**, 139; ANALYST, 1930, **55**, 204, 458) did not extract the whole of the chromogen from their saponified oil. A method is described in detail for carrying out the colour test on the unsaponifiable fraction extracted with chloroform. The colour developed is somewhat difficult to match against Lovibond glasses. Instead of the purplish blue given by cod-liver oil, the colour is almost invariably greenish blue, and is matched by a combination of blue and yellow glasses in the ratio of 10 to 4, very approximately. P. H. P.

Anti-scorbutic Fraction of Lemon Juice. IX. S. S. Zilva. (*Biochem. J.*, 1930, **24**, 1687–1698.)—This paper deals in the main with two aspects of the antiscorbutic factor, namely, its fractionation and the mechanism which controls its spontaneous inactivation. Several years' work on the fractionation of the antiscorbutic factor is reviewed, and it is shown that variable activity is obtained under, presumably, the same conditions of fractionation. It is necessary to exercise caution in order to interpret correctly the available evidence on the subject. Batches of lemon juice sometimes yield fractions of low or no antiscorbutic activity when equal volumes of a saturated neutral lead acetate solution are used in the manipulation. It is possible, however, by adjustment of the quantity of the

precipitating agent to obtain from the same juice preparations of high activity. Therefore, in each case the conditions must be adjusted in order to obtain uniform high activity. Fractions, of which the phenolindophenol decolorising capacity falls below a certain limit, are found antiscorbutically inactive. This reducing property is not an index of antiscorbutic activity itself, but serves rather as an indication that the vitamin has been protected from inactivation. The re-precipitation of an antiscorbutic fraction from lemon juice or from cabbage juice with lead acetate in the neutral or slightly alkaline zone (pH 8–9) yields an inactive preparation. The addition of an ethereal extract from autoclaved lemon juice, which decolorises iodine, but not phenolindophenol, of quinhydrone or of benzo-quinone to unheated lemon juice accelerates markedly the destruction of the reducing principle and of the antiscorbutic factor in a neutral medium in the presence of air. Possibly a substance of phenolic character, oxidisable in the air, is formed in the process of autoclaving. This compound in its oxidised form destroys, in conjunction with the peroxidase present in the juice, the reducing principle that acts as an agent for the protection of the antiscorbutic factor. Decitrated lemon juice kept aerobically at pH 0·6–0·8 for 7 days does not lose its reducing capacity for phenolindophenol to a greater extent than when kept at pH 7, and shows little loss of its antiscorbutic activity. A discussion of the results obtained is given. P. H. P.

Toxicity of Vitamin *D*. **J. B. Duguid.** (*Lancet*, 1930, **219**, 983–985.)—Although there is a wide margin of safety between the therapeutic dose of vitamin *D* and the dose which may prove toxic, the drug is remarkably potent, so that both care and knowledge must be exercised in deciding suitable dosage. The amount of ergosterol used gives no indication of the amount of vitamin *D* included, uniformity of production being obtainable only by a careful control of technique such as has not been observed in the past. As regards rats, the most useful information is furnished by experiments in which the substance is administered in quantities representing multiples of the antirachitic or curative dose—the rat unit of vitamin *D*. This dose is determined biologically by feeding young standard rats on a standard rachitogenetic diet for 3–4 weeks and then adding the vitamin in measured doses so as to ascertain the minimum daily dose which will effect healing in 10 days. Healing is estimated by calcification of the bones, which may be detected by radiological examination or by the "line test." Deficiency in other vitamins appears to contribute in some measure to the toxic effects of vitamin *D*, but as yet the evidence on this point is not quite clear.

The Council of Pharmacy and Chemistry of the American Medical Association have adopted the name "viosterol" for a preparation of irradiated ergosterol standardised in its antirachitic properties by comparison with a certain standard, potent cod-liver oil, this preparation (in oil 100 D) containing about 1333 curative rat units per grm. Hess and his co-workers find that 10 drops daily of this viosterol represent the minimum prophylactic dose for infants, and that healing invariably results when from 30 to 60 drops daily are given.

Until recently there has been, in this country, no officially recognised standardisation of the various preparations of irradiated ergosterol obtainable, and the adjustment of the strength of the preparations has been at the discretion of the manufacturers, on whose directions the physician has mainly depended. The British Pharmaceutical Society has now, however, suggested the use of the Coward unit, based on a certain preparation of irradiated ergosterol, 0·0001 mgrm. of which, in daily doses, brings about healing in standard rachitic rats in 10 days. The general adoption of this rat unit is highly desirable, and there is a pressing need for the official standardisation of all preparations on the market and for an authoritative pronouncement on the proper dosage for therapeutic purposes.

T. H. P.

Bacteriological.

Selective Fermentation. II. Fermentation of Sugar Mixtures by Sauterne Yeast. H. Sobotka and M. Reiner. (*Biochem. J.*, 1930, **24**, 1783–1786.)—Sobotka and Reiner (*Biochem. J.*, 1930, **24**, 926; ANALYST, 1930, **55**, 712) reported that two strains of "Sauterne" yeast preferred glucose to fructose in the fermentation of a glucose-fructose mixture. The value of $K_{G/F}$ was similar to that of all other strains of *Saccharomyces* investigated. This was at variance with the findings of several French workers. The authors have now been able to secure a pure culture of authentic Sauterne yeast, and have repeated the work. The results show that genuine Sauterne yeast ferments fructose preferably to glucose in a mixture. $K_{G/F}$ varies from 1·2 to values above 10 according to adaptation phenomena. This yeast ferments fructose alone 50 to 100 per cent. faster than glucose alone. The mechanism of this anomalous behaviour is discussed. It is possible that the capacity of this yeast to initiate the alcoholic fermentation of glucose is so much reduced that the cells in a pure glucose solution cannot utilise to their full extent the enzymes involved in the liberation of carbon dioxide and ethyl alcohol. By other yeasts, both glucose and fructose are converted at such a rate into a three-carbon intermediate compound that the latter is supplied in excess to the enzymic apparatus governing the last stages of alcoholic glycolysis. The bearing of the anomalies in alcoholic fermentation by Sauterne yeast upon other peculiarities in the metabolism of this micro-organism is being investigated.

P. H. P.

Behaviour of Moulds on Expanded Corks. J. Greger. (*Z. Unters. Lebensm.*, 1930, **60**, 532–536.)—The outer bark of the cork tree and the waste material from the manufacture of cork stoppers are used for the preparation of heat insulators, but may be infected with bacteria or moulds which, under favourable conditions, will develop subsequently and derive their sustenance from the nutrient matter carried in the cork. This is known as "primary infection," and treatment for 2 hours in air at 100° C. (or for 30 minutes at 150° C.) destroys the spores. Secondary infection may, however, occur subsequently, but a temperature of 260° C. for 10 minutes destroys the nutrient substances in the cork and ensures

complete sterilisation. Observations with *Mucor, Penicillium, Aspergillus* and *Dematium* are recorded. Sterilised cork soaked in water containing nutrient substances may become a source of secondary infection. J. G.

Identification of Aspergilli in Cotton Goods. G. Smith. (*J. Text. Inst.*, 1931, **22**, T 110).—There are two main groups of *Aspergillus*, the *A. glaucus* series and the *A. penicilloides* (a series intermediate between *A. glaucus* and *A. fumigatus*, most of the strains resembling certain *Penicillia*.) A study has been made of 34 forms of mould of the group *A. glaucus*. They were grown on wort-agar at 25° C., the dimensions of the conidial apparatus were taken, the time required for the ascospores to ripen and the dimensions, markings, and colour of the latter observed. From these data, the forms were divided into four sub-groups: I, smooth without crests, ridges or furrow, green and yellow conidiophores (*A. repens* (*Corda*) Saccardo). II, smooth shallow furrow, no crests, intense red colour (*A. ruber*, Spieckermann and Bremer). III with furrow and small rounded crests (*A. amstelodami*, Mangin). IV with deep furrow and well-marked crests (*A. chevalieri*). The three species of the *A. penicilloides* series, recognised by Thom and Church, are diagnosed more fully and somewhat differently from the conceptions of the original authors. Eight plates are given, representing drawings and photomicrographs. R. F. I.

Toxicological.

Toxicology of Thallium. G. Roche Lynch and J. M. S. Scovell. (*Lancet*, 1930, **219**, 1340.)—The authors give details of three fatal cases of poisoning by thallium acetate, an accidental overdose of which was given for the treatment of ringworm. Other cases are recorded, in some of which the thallium salt had been used in form of a cream, to remove hair. Zelio corn and zelio paste, containing 2·1 to 2·8 per cent. of thallium, are extensively used in Germany as rat poisons, and have been responsible not only for accidental poisoning, but also for a number of criminal cases.

In the three fatal cases, referred to above, the thallium in the viscera and urine was determined as follows: A weighed quantity of the material was broken up with hydrochloric acid and potassium chlorate by the method of Fresenius and von Babo, and, when solution of all the material except the fat had been achieved, the liquid was filtered, the precipitate well washed, and the filtrate boiled until practically all the excess of chlorine had been driven off, or it was removed by the sulphur dioxide method. If necessary, it was filtered again, and the precipitate discarded. To the filtrate, ammonium chloride and ammonia were added, until it was distinctly alkaline, and it was then again boiled. The precipitate, consisting of iron, calcium, and magnesium, chiefly in the form of phosphates, was filtered off and discarded. The filtrate was then saturated with hydrogen sulphide, or an excess of freshly prepared ammonium sulphide was added; a black precipitate was formed. This was filtered off, well washed with dilute ammonium sulphide, and, finally, with distilled water. It consisted of

thallium sulphide (Tl_2S), together with traces of other metals (copper) which are precipitated with hydrogen sulphide in alkaline solution. The precipitate, when completely washed, was dissolved off the paper with hot dilute hydrochloric acid. The thallium was converted into thallous chloride, any trace of copper which was present as the sulphide remaining behind. To the solution, ammonia was added in slight excess, and the liquid was boiled. Any precipitate was filtered off and discarded. The filtrate was then made very faintly acid with hydrochloric acid, and excess of potassium iodide solution was added. An immediate yellow precipitate of thallous iodide formed. As there is some tendency for thallous iodide to come down in colloidal form, the liquid was boiled and allowed to stand for 12 hours. The precipitate was then collected in a weighed Gooch crucible and well washed, first with potassium iodide solution, and subsequently with alcohol, until the washings gave no reaction for an iodide. The crucible and its contents were then dried at 120° C. until constant weight was obtained.

Although thallous iodide is very slightly soluble in water (1–17,000), it is almost completely insoluble in potassium iodide solution and in alcohol, so that filtration in the cold and washing with these reagents will give an accurate estimate of the amount present. The iodide, after weighing, may be confirmed as thallium by dissolving it in a solution of sodium thiosulphate, in which it is dissolved only with difficulty, whereas lead iodide is readily soluble. Finally, some of the iodide may be heated in a Bunsen flame on a platinum wire, and the characteristic green line in the spectrum obtained.

AMOUNTS FOUND IN THE VISCERA, EXPRESSED AS THALLIUM ACETATE.

	Case A.		Case B.		Case C.	
	Grains.	Mgrms.	Grains.	Mgrms.	Grains.	Mgrms.
Stomach	0·24	15·6	0·11	7·4	—	—
Liver	0·9	58·1	1·33	86·2	0·74	48·2
Intestines	1·4	90·8	1·73	112·8	0·23	15·0
Kidneys	—	—	0·12	7·9	0·09	6·0
Urine	—	—	—	—	0·28	18·2
Total	2·54	164·5	3·29	214·3	1·34	87·4
Amount given	39	2527	36	2333	28	1814
Amount intended to be given	3·9	253	3·6	233	2·8	181

B, who died first, had eliminated less of the drug than A, who was given a larger quantity.

From their experience and consideration of the literature (a full bibliography is given) the authors conclude that thallium acetate should not be used as an ordinary routine treatment for ringworm of the scalp, for the following reasons:—(1) Thallium in itself is a highly toxic substance, showing a marked similarity to lead, both in its chemistry and in its toxic symptoms, and the far-reaching effects of the poison are much greater than is generally supposed. (2) It has a definitely selective action on all forms of nervous tissue, and it has been

demonstrated that, even in infinitesimal doses, it causes slight degenerative changes in the brain cells of rats. It is, therefore, most unlikely to leave the human brain entirely unchanged, and it seems impossible to be certain that it does not hinder further brain development. (3) The margin between an epilating and a toxic dose is extremely small, and allows for no idiosyncrasies, whereas with *X*-rays the dosage is very accurate, trouble is rare, and if a mishap occurs it is at least local. (4) Ringworm of the scalp is not in itself a fatal disease, and, though often troublesome, can usually be cured by other means. It, therefore, does not seem justifiable to use such a powerful poison in an attempt to cure it a little more quickly. (5) Toxic phenomena do not appear to be due to the use of old solutions.

Organic Analysis.

Determination of Water in Glycerin. L. F. Hoyt and P. C. Clark. (*Oil and Fat. Ind.*, 1931, **8**, 59–61.)—The glycerin containing less than 10 per cent. of moisture was determined by a modification of the Bidwell and Sterling distillation method (*Ind. Eng. Chem.*, 1927, **17**, 243), wherein toluene is used as the boiling liquid. It was found advisable to reverse the 45° slope of the tube leading from the boiling flask, to give a better flow back into the flask and more even distillation. For dynamite glycerin, 50 grms. or more, and for C.P. and crude glycerins 25 to 50 grms. are weighed to within 0·01 grm. into a 1 litre Pyrex flask, and 300 c.c. of toluene added. The graduated stem of the Bidwell–Sterling tube is filled with water-saturated toluene, and the flask is electrically heated until no more droplets of water come over. The bulk of the water comes over in the first half hour, and the last traces require 1½ to 2 hours. A small proportion of glycerin is carried over with the water, but the correction in no case exceeded 0·15 per cent. Commercial xylene, isopropyl ether and benzene were found unsuitable as boiling liquids. A dynamite glycerin, to which 10·37 per cent. of water had been added, gave by this method a recovery of 10·39 per cent. of water. D. G. H.

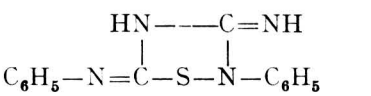
Catechin of the Cacao Bean. W. B. Adam, F. Hardy and M. Nierenstein. (*J. Amer. Chem. Soc.*, 1931, **53**, 727–728.)—The authors have shown that the catechin isolated from cacao beans grown in Java is *l*-acacatechin, $C_{15}H_{14}O_6$, m.pt. 229° (which yields a penta-acetyl derivative, m.pt. 151°) and not a methyl derivative of catechin, as reported by previous workers. The optical rotatory power of this catechin is $[\alpha]_D - 69°$ (in water), and that of its penta-acetyl derivative, $[\alpha]_D - 12°$ (in tetrachloroethane). The same catechin was also isolated from cacao beans from West Africa and Trinidad. It is concluded that the catechin content of the cacao bean is about 0·8 per cent. Details of the method of isolation of the catechin are given. S. G. C.

Chemical Properties of some Commercial Rayon Yarns. B. P. Ridge, H. L. Parsons and M. Corner. (*J. Text. Inst.*, 1931, **22**, T117.)—The materials investigated (first grade only) have included viscose, Lilienfeld, cuprammonium,

acetate and nitrocellulose rayons, and also purified chemically unchanged cotton, the following determinations being made: Copper number, loss of weight on boiling with alkali, methylene blue absorption, fluidity in cuprammonium hydroxide solution, ash content and ash alkalinity, material extracted by solvents, sulphur, copper and iron content, and (for acetate rayons) the acetic acid content. The methods of carrying out these determinations are given in full. Methylene blue absorption was carried out on the solution buffered to *p*H 7. The test for fluidity in cuprammonium hydroxide solution was the same as described by Clibbens and Geake, except that the cellulose in the solutions was 2 per cent., instead of 0·5 per cent. in concentration. For the acetic acid content of acetate rayons the methods of Ost and of Barnett were used. In extracting with solvents, the acid-washed, rinsed and dried material was extracted with ether in one series, and with chloroform in another. The sulphur was determined by ashing the material with hydrochloric and nitric acids, the sulphate in the residue being precipitated and weighed as barium sulphate. The copper was determined by three methods: (*a*) direct precipitation as thiocyanate from a solution of the ash; (*b*) indirectly by measuring the catalytic power of a solution of the ash in accelerating the reduction of ferric salts by sodium thiosulphate; (*c*) colorimetrically, by means of diethyl-dithio-carbamate (Callan and Henderson). Iron was titrated with potassium dichromate solution, using diphenylamine as the indicator. The results are tabulated and very fully discussed. R. F. I.

Inorganic Analysis.

Detection of Carbon Disulphide. F. Feigl and K. Weisselberg. (*Z. anal. Chem.*, 1931, **83**, 93–104.)—The methods for the detection of carbon disulphide were re-investigated and compared. The most sensitive of the published tests is the precipitation of cuprous xanthate. Two new tests are described: (I) Hector's base,



is derived from phenylthiourea by oxidation with hydrogen peroxide. When a solution containing carbon disulphide is treated with the base and a few crystals of nickel acetate and gently warmed, a violet-red precipitate is produced, or a pink coloration, according to quantity. If the nickel salt is insoluble in the liquid to be tested, 5 to 10 drops of water are also added. The reaction is exceedingly sensitive, still more so if volatilisation is prevented by working in a closed tube. The precipitate has the composition $C_{30}H_{22}N_8S_6Ni$. (II) The formation of lead sulphide in an alkaline lead solution is considerably hastened by the presence of formaldehyde: one drop of the test solution, 2 to 3 of formalin, and one of plumbite solution are stirred together on a spot plate. A darkening occurs in a few seconds. If hydrogen sulphide is thought to be present, the drop to be tested is first treated with bromine water till yellow, after which a tiny crystal of sodium sulphite is

stirred in to effect decolorisation. Working with larger quantities in a test tube allows of the detection of smaller quantities of carbon disulphide. If the liquid is immiscible with water, a ring of lead sulphide will be formed at the zone of contact.

W. R. S.

Cobaltic Sulphate as an Oxidising Agent. S. Swann and T. S. Xanthakos. (*J. Amer. Chem. Soc.*, 1931, **53**, 400–404.)—A study has been made of the preparation of cobaltic sulphate by electrolytic oxidation of cobaltous sulphate, and its use as an analytical reagent for the wet oxidation of certain organic compounds to carbon dioxide. For the preparation of cobaltic sulphate a saturated solution of cobaltous sulphate in 10 *N* sulphuric acid, contained in a porous cell which dipped into a beaker containing the same liquid, was oxidised anodically, using an anode of platinum sheet, a sheet of copper surrounding the porous cell serving as the cathode. A good yield of cobaltic sulphate was obtained by electrolysing for about 4 hours with an anode current density of 0·01–0·2 amp. per sq. cm., provided that the temperature was kept below 10° C. by cooling the electrolyte vessel in ice. The cobaltic sulphate was not isolated in the tests of its oxidising power on organic compounds; the suspension of the solid in sulphuric acid, which was the product of the electrolytic preparation, was used. The organic compound was stirred mechanically with an excess of this product for an hour at the ordinary temperature in a three-necked flask, which was fitted with an absorption train for carbon dioxide; air was then aspirated for half an hour through the apparatus, while the flask was heated on a water-bath. By this means it was found that, whereas amylenes, benzene and glycerin gave no carbon dioxide, and acetic acid, ethyl alcohol and acetone gave only a little, formic acid, tartaric acid, citric acid and malic acid gave the theoretical yield. Ethylene glycol gave a 96·05 per cent. yield of carbon dioxide. Formic acid remained unoxidised in the presence of amylene and glycerin.

S. G. C.

Study of Spacu's Reaction. Volumetric Determination of Copper. J. Golse. (*Bull. Soc. Chim.*, 1931, **49**, 85–100.)—The author has studied Spacu's reaction (the precipitation of copper as cupric pyridine thiocyanate by the addition of pyridine and an alkali thiocyanate) with a view to making it the basis of a volumetric method of determination of copper. The methods proposed involve the precipitation of the copper by the addition of a regulated excess of the reagents, filtering off the precipitate, and determining the uncombined thiocyanate ions remaining in the solution, either (*a*) by precipitation by a known quantity of silver nitrate, the excess of which is determined by titration with standard ammonium thiocyanate, using iron alum as indicator; or (*b*) by oxidation with a known quantity of sodium hypochlorite, the unused excess of which is determined by addition of potassium iodide and titration of the liberated iodine with sodium thiosulphate. Even under the most favourable conditions which could be found, a certain proportion of the copper—about 2 per cent.—remained unprecipitated, and this necessitates the application of empirical corrections in the calculation of the amount of copper from the quantity of volumetric solution employed. The

methods, as so far worked out, cover the determination of copper in pure copper sulphate solutions only over a limited range of concentration, and they break down in presence of zinc, nickel, etc., owing to the pyridine thiocyanates of these metals being insoluble. On account of these limitations the details of the methods are not abstracted. S. G. C.

Separation of Beryllium from Aluminium. A. Travers and Schnoutka. (*Compt. rend.*, 1931, **192**, 285–287.)—The following modification of the method of Berthier was used for the separation of the two metals and the extraction of beryllia from beryl: the freshly-precipitated hydroxides are dissolved in excess of alkali, the solution saturated with sulphur dioxide and boiled for 10 minutes, and left in the cold for some hours. The alumina is quantitatively precipitated, occluding a little beryllia. The precipitate is, therefore, re-treated, when it yields nearly all the remaining (*"la presque totalité"*) beryllia. The advantages claimed are, that the alumina precipitation is quantitative, and that a high yield is secured in the extraction of beryllia from the mineral. No numerical data establishing the completeness of the separation are given. W. R. S.

Rapid Colorimetric Estimation of Potassium. E. R. Caley. (*J. Amer. Chem. Soc.*, 1931, **53**, 539–545).—The following method is proposed for the determination of amounts of potassium between 0·01 and 0·001 grm.; it depends on the precipitation of potassium as picrate, which is separated, dissolved in water, and the yellow solution thus obtained matched colorimetrically: The solution containing the potassium as chloride (sulphates must be absent) is evaporated to dryness in a 25 c.c. beaker, the residue is dissolved in 1 c.c. of water, and 7·5 c.c. of a saturated solution of picric acid in 95 per cent. alcohol are added. The liquid is stirred until a precipitate forms, and kept for 40 minutes, with stirring at 5-minute intervals, the beaker being placed in a dish containing water at 20° C., to maintain a definite temperature during the precipitation. The potassium picrate is filtered off on a sintered glass filtering funnel having a medium porosity (filter paper will not do), and the precipitate is washed with successive 1–2 c.c. portions of ether until the washings are colourless. The ether having been removed by drawing air through the funnel, the precipitate is dissolved by pouring several successive quantities of water through the funnel, the solution being received in a 50 c.c. volumetric flask. The intensity of colour of the solution is compared in a plunger-type colorimeter with that of a standard solution of potassium picrate, containing practically the same amount of potassium as is being determined, prepared by submitting a measured volume of standard potassium chloride solution (1·907 grm. KCl per litre; 1 c.c. = 0·0010 grm. K) to the same procedure. This condition can be fulfilled only by preparing a range of standards, and choosing the one which is nearest to the unknown in colour for the final matching in the colorimeter; the standards keep well if protected from evaporation. The range of standards is called for to compensate for the solubility of potassium picrate. The method breaks down for amounts of potassium below 0·001 grm. Ammonium,

caesium and rubidium give insoluble picrates just like that of potassium. More than 0·007 grm. of sodium gave high results in the determination of 0·001 to 0·005 grm. of potassium. The presence of 0·005 grm. of lithium, magnesium, calcium, barium, strontium, aluminium or ferric iron caused no interference.

S. G. C.

Decomposition of Refractory Silicates by Fused Ammonium Fluoride and its Application to the Determination of Silica in Glass Sands. A. C. Shead and G. F. Smith. (*J. Amer. Chem. Soc.*, 1931, **53**, 483–486.)—Berzelius noted the fusibility of ammonium fluoride, and Rose and Jannasch had independently employed ammonium fluoride for decomposing silicates, but, it is here suggested in such a way as to obscure the peculiar advantages of the reagent. The following process was tested with good results on two samples of glass sand, the silica content of which was known: The sample is thoroughly mixed with 5 grms. of crystalline ammonium fluoride in a weighed 25 ml. platinum crucible provided with a well-fitting lid. The crucible is placed in a hole through a heavy asbestos board so that about one-third of the surface of the crucible projects underneath. This part is maintained at a dull red heat for about 10 minutes. Experience showed that about 1 grm. of quartz (passing one hundred mesh sieve) should be volatilised within this time, but that a repetition of the process is necessary to complete the decomposition. Towards the end of the fume evolution, the burner is held in the hand with the flame impinging on the lid; this causes the detachment of the deposit of ammonium salt, which falls into the crucible, carrying with it any residue which may have been projected upwards during the decomposition. Before the crucible is allowed finally to come to redness, after the fluoride has been volatilised, it is recommended to add a drop or two of concentrated sulphuric acid, or a few fragments of ammonium persulphate, to prevent the escape of traces of aluminium or iron as halide. The residue is weighed, the loss in weight being due to silica.

S. G. C.

Gas Analysis.

Analysis of Mixtures of Hydrogen, Methane and Ethane. O. J. Walker and S. N. Shukla. (*J. Chem. Soc.*, 1931, 368–370.)—The method depends on the removal of ethane by condensation (cf. *Trans. Faraday Soc.*, 1931, **27**, 35), and of hydrogen by absorption on palladium. The apparatus consists of a Töpler pump connected through a 3-way tap to (1) a U-tube which may be cooled in a vacuum vessel with liquid air, and (2) a steam-jacketed palladium absorption-tube containing 3 grms. of palladium-sponge. Each of these 2 tubes is connected also to a train consisting of a mercury-trap, a transferring-pipette, and a bent capillary tube dipping into a mercury trough. The mixture (5 to 10 c.c.) is measured in a Bone and Wheeler burette and transferred, by means of a small tube inverted over mercury in a crucible, to the capillary tube at the end of the apparatus nearest the U-tube (1), and thence to the latter by manipulating the pump and the transferring pipette. After 5 minutes at the temperature of

liquid air the residual hydrogen and methane are removed and measured (the absorption giving the ethane), and passed to the palladium-tube, where they are heated for 20 minutes at 100° C. The steam is then replaced by cold water, and the residual gas measured to determine the hydrogen. A complete analysis takes $1\frac{1}{2}$ hours, and the mean errors for mixtures containing ethane (10 to 45), hydrogen (31 to 64), and methane (16 to 50 per cent.) were $\pm$ 0·8, 1·1 and 2·6 per cent., respectively. The percentage by volume found for any constituent seldom differs by more than 1 per cent. from the amount taken. Owing to the small volume of sample, no error arises from solution of methane in the condensed ethane (cf. Mulders and Scheffer, *Rec. trav. Chim. Pays Bas*, 1930, **49**, 1057), but a check is provided if the methane and ethane are freed from hydrogen and exploded separately, and the ratio *contraction after explosion/contraction after absorption* in potassium hydroxide solution determined. This is essential if nitrogen is present. J. G.

Microchemical.

Micro-Chemical Tests for Benzoic Acid, Salicylic Acid and Esters of *p*-Hydroxybenzoic Acid in Food and Drugs. R. Fischer and F. Stauder. (*Mikrochem.*, 1930, **7**, 330–338.)—The analysis is carried out on 1 to 2 grms. of material, such as jam, fruit juice, preserved vegetable, or syrup, which may contain 0·05–0·1 per cent. of the preservative to be identified. The material is acidified with hydrochloric acid, and, if the consistence is too thick, diluted with hot water. Fats are melted and digested with 2 to 3 c.c. of dilute sodium bicarbonate solution, and then the aqueous solution is poured off and acidified. The acid solution is extracted with a few c.c. of ether, and the ethereal solution is transferred, drop by drop, to a sublimation tube in a paraffin bath at a temperature not higher than 50° C. The different compounds are then separated by fractional sublimation, and the sublimates are identified by their crystalline form, their reactions, or melting points. J. W. B.

Reviews.

QUANTUM CHEMISTRY. By Prof. A. HAAS, Ph.D. Translated from the German by L. W. CODD, M.A. Pp. 75. London: Constable & Co., Ltd. 1930. Price 6s.

This book contains, in a somewhat extended form, the subject-matter of a series of four lectures that were delivered in 1929 before an audience which lacked both the time and opportunity for a comprehensive study of modern theoretical physics. In these lectures the recent views on atomic structure, based on the

principles of the quantum theory and wave mechanics, are given, and the scope of the book will be evident from the titles of the lectures, *viz.* (1) The Arithmetic of Chemical Periodicity, (2) The Quantum Theory of Valency and Chemical Forces, (3) Electron Grouping and the Periodic System, (4) Quantum Problems of Molecular and Nuclear Structure. Despite the highly mathematical nature of the subject, Professor Haas has succeeded in giving the gist of it without introducing mathematics. The book certainly achieves its object in providing a simple and lucid outline, and will, therefore, appeal to readers who desire to become acquainted with the modern trends of this branch of scientific knowledge. It also supplies an excellent introduction to the subject for more serious students.

As points in the book of special interest, reference may be made to the forecast and discovery of parahydrogen, and also to the calculations from theoretical considerations made by Gurney and Condon of the actual rate of radio-active disintegration.

In conclusion, it must be mentioned that the translation has been admirably done.

H. T. S. BRITTON.

PRACTICAL PHYSICAL CHEMISTRY. By ALEXANDER FINDLAY. Fifth Edition. Pp. xii+312. London: Longmans, Green & Co., Ltd. 1930. Price 7s. 6d.

This well-known work has undergone a real revision, and, owing to judicious excision, much new matter has been added without materially increasing its bulk. Contracted arithmetic has now been left for the school to teach before the student attempts physical chemistry. It is rather a pity that Prof. Findlay has not taken the opportunity afforded by the usual discussion of errors to explain what the "mean error," "mean square residual," or "standard deviation," as Pearson and Udney Yule (who prefer it to the "probable error") call it, really means. This is seldom made clear in works on probability; in fact, one sympathises with Lippmann's remark to Poincaré, apropos the fundamental theorem of the theory of errors, "Tout le monde y croit, parce que les expérimentateurs y voient un théorèm de mathématiques, tandis que les mathématiciens le considèrent comme un fait d'expérience." A clear exposition of the meaning of the conventional expressions of this theory, so far as they apply to experimental work, would be really useful. As to the actual example, given on pp. 9–10, the subtraction of the mean 27·828 from its constituent 27·30 does not necessarily yield 0·003, although, as the third figure of that constituent is unknown, this is not impossible. A little algebra, applied to the formula given, will show how much more easily the precision of an average can be increased by greater accuracy of individual measurements than by the laborious method of repetition.

The author ignores the excellent work done in this country by the National Physical Laboratory on graduated apparatus; he adheres to the cubic centimetre, as against the millilitre, and does not mention the use of burettes with long graduations, which can be seen back and front, and so eliminate errors of parallax.

These are, of course, rather expensive for students' use; but, surely, were worth mentioning. He has abandoned the blowing-out method of delivery from pipettes and the use of the Shellbach burette.

The chapter on The Density of Gases and Vapours has been enlarged and is good, but there is too much rubber tubing in Fig. 21, where the two screw clips should be replaced by a two-way tap.

In the chapter on Viscosity and Surface Tension, the viscometer of Gibson and Jacobs, based on Stokes's law, is described. This good and simple instrument can be made and used by anyone who has a fairly exact centimetre scale, a thermometer and a few steel bicycle bearing balls, besides a stop-watch and the usual apparatus of the laboratory. The section on surface tension has been improved, but only the capillary tube and the drop method, both of which possess the merit of convenience, are described. The parachor is mentioned, and experiments on it are given. The rôle of the burette in the preparation of standard baryta solution (pp. 149–150) is not clear; a sentence seems to have dropped out.

It is now unusual to use the Lippmann electrometer in potentiometric work, but this is more or less a matter of taste. In this section on measurements of electromotive force, besides an adequate discussion of the electrometric method of determining hydrion concentration, including Biilmann's quinhydrone electrode, there is some description of buffer solutions and their use in determining the *p*H value. The glass electrode, which is not very suitable for students' use, is not described.

The section on calorimetry is good, but it is a matter of taste whether a Beckmann thermometer, one of the German enclosed pattern, or one of the excellent solid-stem instruments which are made in this country, is used; in fact, the two latter are simpler to use, as the degree value does not need correction for varying temperature. It is unnecessary to give more than one standard substance for determining the water equivalent of a calorimeter, as benzoic acid is, by international agreement, used for this purpose, and its heat of combustion (based on Dickinson's work, which has been confirmed) is 6324 calories (15° C.) per grm. weighed in air. Naphthalene is a very unsuitable substance, and is not always easy to burn. The use of iron wire for firing a charge *seems* economical, but platinum with a cotton thread is better, as it does not injure the bomb lining, and there is no loss of platinum. The Regnault–Pfaundler correction formula is as simple as the graphic method given in this and other works, and is generally used by those who do much bomb calorimetry.

The reviewer has called attention to the above points, where, in his opinion, the book could be improved. He has been encouraged to do this by noting that some matters to which he called attention in the fourth edition have been altered in the fifth. The merits of the work are so outstanding in clarity of description, in the introduction of sufficient explanations of theory, and in copious references to literature, giving the student a chance of carrying his studies further, if an overcrowded curriculum allows him time to do this, that captious criticism, always

regrettable, is here impossible. It is a very useful book for the analytical chemist to have available for reference when some recondite problem seems a little outside the range of the usual analytical methods. It is to be hoped that before another eight years the author may give us another edition.

J. H. COSTE.

THE CHEMICAL ANALYSIS OF ROCKS. By HENRY S. WASHINGTON, Ph.D. Fourth Edition, re-written and enlarged. Pp. xi+296. New York: John Wiley & Sons, Inc. 1930. Price 20s.

This well-known hand-book has now reached its fourth edition, which may be taken as a measure of the well-deserved esteem in which it is held by the analytical fraternity. Its salient features are, or should be, by now so familiar that the reviewer need no longer give a sketch of the work. Suffice it to say that the operations of silicate rock analysis are described with a wealth of detail sufficient to enable a student or beginner, working by himself, to become competent in this class of work. The principal changes in the new edition refer to up-to-date appliances (such as Munroe and sintered glass crucibles), and special reagents (cupferron for titania, *o*-hydroxyquinoline for magnesia, and periodate for manganese); new text-matter on lithium and beryllium has been included.

The statement, on p. 274, that ignited beryllia "should always be dissolved in dilute hydrochloric acid and reprecipitated," is at variance with the general experience that the oxide is rendered insoluble in acids by strong ignition.

The author's technique in the bisulphate fusion of the ignited ammonia precipitate is very different from my own practice; a brief discussion of the operation may be of some practical interest. Pulped filter fibre is added to the precipitate (p. 173), so that a porous oxide results on ignition. This is subjected to fusion with 5 to 7 grms. of pyrosulphate (p. 180), which operation "can often be accomplished in three or four hours, especially if macerated paper has been used" (p. 182). The fusion is carried out in a platinum crucible.

Unless I am quite mistaken, the long time taken by the author over the fusion is due to the low temperature at which he conducts the fusion of a large quantity of pyrosulphate in platinum; raising the temperature in an attempt to hasten solution would be attended by great risk of loss due to foaming. This risk does not arise when a silica crucible is used.

The author, however, in common with Hillebrand and Lundell, rejects a silica crucible for bisulphate fusions, on the ground that "some silica may be taken up from it" (p. 178). If so, the amount is almost negligible, and can always be allowed for by a determination of the weight before and after fusion. The contamination with platinum is, in my opinion, a much more serious matter, the dissolved metal being more difficult to account for than silica; approximately, 0·0025 grm. of platinum is introduced into the analysis, "of which about one-half should be subtracted from the alumina" (p. 188).

A few unimportant misprints were noticed. There is one, however (p. 178, line 23), to which attention should be called: the loss in weight of a vitreosil crucible is not "0·02 to 0·04 gram" for a bisulphate fusion, but 0·0002 to 0·0004 grm. The author, whose attention I called to the statement as it appears in print, has requested me to rectify it in the sense indicated.

W. R. SCHOELLER.

RECENT ADVANCES IN ANALYTICAL CHEMISTRY. Vol. I: ORGANIC CHEMISTRY. Editor: C. AINSWORTH MITCHELL. Pp. 421. London: J. & A. Churchill. 1930. Price 12s. 6d.

From time to time there arises the necessity for some considered statement of the advances made in chemical subjects. To some extent annual reports published by various bodies meet the need, but more than this is required by those not actually engaged in investigating processes for themselves. The volume now considered is an attempt to supply critical summaries of organic analytical chemistry. There are eleven chapters, each written by well-known workers on the subject under discussion. It can be stated at once that the objects aimed at, namely, to enable the reader to ascertain how far he can displace his present methods for new ones with safety, is in the main achieved.

The chapter on sugar analysis by Mr. Hinton is a very full discussion of analytical methods. He calls attention to the utility of invertase in place of acid hydrolysis of sucrose, and deals adequately with the ratio of ash and conductivity of sugar solutions. He states—rightly, in the reviewer's opinion—that the conductivity should be accepted on its own merits, for it is not safe to assume that the soluble inorganic impurities are always the same in sugar solutions. The various copper methods for determining reducing sugars receive full treatment, and the value of Lane and Eynon's use of methylene blue as an internal indicator is stressed. McLachlan's method of examining malt extract and glucose by the use of various types of yeast is likewise discussed in some detail.

Chapter II, by Dr. Ainsworth Mitchell, is a careful discussion of present-day methods of oil analysis. In view of the fact that there is a tendency to discount the utility of "iodine" values in investigating the chemistry of fats and oils, it is satisfactory to note that the use of halogen absorption methods still holds the field. Bolton and Williams's classification of oils into groups by means of iodine values of the unsaponifiable matter is here given suitable prominence. Kaufmann's thiocyanogen absorption method receives due attention, and its importance for determining the character of unsaturated bonds is indicated briefly. The work of Armstrong and Hilditch on the oxidation of unsaturated fatty acids provided a new mode of attack of glycerides in two directions: (1) Determination of the proportion of saturated and unsaturated glycerides, and (2) the nature of the unsaturated fatty acids in the glycerides. This enabled Hilditch to deal with the question of the distribution of fatty acids in the glycerides of seed fats.

Dr. Mitchell also contributes a chapter on tannins, which deals especially with recent methods for the analysis of tanning materials, galls, and tannin-bearing materials of all kinds. Mitchell's quantitative method for determining pyrogallol tannins by means of ferrous tartrate, first described in 1923, and his separation of gallic acid from gallotannin by means of alkaloids, are discussed in detail. The method described here is convenient and rapid for the purpose. Recent gravimetric processes for tannin estimations receive adequate treatment, and the utility of precipitation by alkaloids is critically examined in this chapter.

Mr. Parry contributes the chapter on essential oils, and he calls attention to the two valuable reports issued by the Committee of the Society. The more recent work on determinations of ionone, carvone, citral, alcohols and phenols in essential oils, is described with clearness. The utilisation of magnesium methyl iodide in evaluating essential oils for their content of hydroxy compounds is stated to be complicated for ordinary use, but it does not appear so to the reviewer, for the technique of the preparation of the reagents is easy to work with the materials available to-day. A useful account of the methods of detecting addition of esters is given as fully as the subject calls for, particular attention being devoted to ethyl laurate, an adulterant which is sometimes used. Parry gives an account of some tests carried out by himself on the determination of ascaridole, the main constituent of American wormseed oil, the results of the tests leading to the adoption of an iodometric method based on a factor developed from determinations with as pure ascaridole as was available.

Dr. Dorothy Jordan Lloyd provides a chapter on the proteins, giving in detail the carbamate method for the analysis of gelatin and describing some of the new units obtained by hydrolysis and carbamation. Methods for the determination of tyrosine, tryptophan and cystine are dealt with in the light of recent work, and a short account of enzyme action as applied to protein analysis is included in the chapter. Methods of electro-dialysis, with illustrations of apparatus, find a place in this section. Dr. Lloyd's conclusion is sufficiently significant to be quoted:

> "In conclusion, it is important to notice that in considering the value of proteins as food materials for the living (and particularly the growing) animal, the knowledge obtained by a chemical analysis does not carry the matter very far forward. While certain units must be present in a "good" food protein, the availability of any unit for absorption and synthesis is not indicated as yet by any known method of analysis. For the assay of the value of a protein from the biological standpoint, recourse must still be had to biological methods."

This leads naturally to the account of the biological analysis of proteins by Dr. Harriette Chick, who supplies a brief description of the methods for ascertaining the biological value of a protein.

The section on Cereals by Dr. D. W. Kent-Jones should be studied, if only for

its introductory paragraph and penetrating enquiry into the utility and scope of the various determinations suggested or made on cereals from time to time. Typical sections illustrating the method adopted by Dr. Kent-Jones in his discussion are those on moisture, protein distribution in flour, gluten (see particularly the first three lines on page 226), sugar and colour. One cannot but feel that the kind of criticism applied in this chapter is desirable for methods of analysis in many other fields of work. "Milk and Milk Products" are dealt with by Mr. Elsdon, who considers critically the determinations usually carried out on milk, and also sets out the methods of the Committee of the Society on condensed milk. Hortvet's modification of Monier-Williams's method for determining the depression of the freezing point of milk for detection of added water is examined and recommended. It is well to emphasise the fact that sour milk is not suitable for testing by the method (page 252). If any comment is called for on this chapter, it is solely on the ground of its being too short.

Mr. Dickson's chapter on paper analysis is full, and calls for little comment. The special section on the use of polarised light in the examination of fibres is largely the outcome of Mr. Dickson's own experience, and is a valuable aid to the chemist who has to examine paper critically. Recent methods for determining the nature and proportions of fillers and sizing are furnished for general use, and a number of machines for physical testing is described. A simple readily constructed apparatus for air porosity is depicted (page 300), which appears to be quite suitable for practical purposes. The method for determining tar acids in special antiseptic wrapping papers (page 291) is likely to give seriously low results with phenol and the cresols, for it is known that the rejected aqueous layer contains appreciable quantities of these substances.

Two chapters are assigned to petroleum and coal, both written by Dr. King and Mr. Acton-Taylor, jointly. A general description of elementary analysis, as applied to oils, forms the first part of these chapters, with a useful commentary on the value of the methods of ascertaining moisture and aromatic hydrocarbons. A specially useful section is that dealing with viscosity, some of the more novel methods or applications of older ones receiving attention. It is satisfactory to note that the tendency to use absolute methods for determining viscosity is a feature of the recent technical investigations here discussed. In the chapter on coal the statement that some methods still remain which are not fully approved (page 350) is one with which the reviewer is in entire agreement. Even such determinations as that of phosphorus in coal are not yet completely worked out in all cases. For arsenic the most serious source of error is the method of preparing the solution for estimating the element, and not so much the actual separation of the arsenic recovered.

Although the volume is entitled "Organic Analysis," a very valuable chapter on Gas Analysis is included, written by Mr. Ambler. The methods for the detection and determination of the constituents of gases in large or small proportions are, as would be anticipated, given with due regard to their importance or probable

utility in practical testing. Thus the sections on carbon monoxide, hydrogen and methane, and on fractional combustion processes are important critical compilations on these subjects. The sole remark called for on this chapter is that Mr. Ambler has probably been somewhat reticent in stating his opinion on some of the methods proposed in the literature, particularly on some of the "rapid" methods.

In the opinion of the reviewer this volume is a valuable work of reference on recent analytical methods. It furnishes an illustration of the fact that analytical chemistry offers a field for research of increasing importance in its application to pure chemistry and to technological practice.

J. J. Fox.

Publications Received.

BIOASSAYS. A HANDBOOK OF QUANTITATIVE PHARMACOLOGY. By JAMES C. MUNCH. London: Baillière, Tindall & Cox. Price 45s. net.

INDUSTRIAL MICROBIOLOGY. By H. F. SMITH and W. L. OBOLD. London: Baillière, Tindall & Cox. Price 27s. net.

A MONOGRAPH OF VISCOMETRY. By GUY BARR. Oxford University Press. Price 30s. net.

TECHNICAL METHODS OF CHEMICAL ANALYSIS. 2nd Ed. Vol. III. By G. LUNGE and C. A. KEANE. London: Gurney & Jackson. Price £3 3s. net.

A LABORATORY MANUAL OF ELECTROCHEMISTRY. By E. MÜLLER. Translated from the 4th Edn. by H. J. T. ELLINGHAM. London: Routledge. Price 15s. net.

RECENT ADVANCES IN PHYSICAL AND INORGANIC CHEMISTRY. By A. W. STEWART. 6th Edn. London: Longmans, Green & Co. Price 18s. net.

CHEMICAL ENGINEERING AND CHEMICAL CATALOGUE, 1931. London: Leonard Hill. Price 15s.

BRITISH CHEMICALS AND THEIR MANUFACTURERS, 1931. Association of British Chemical Manufacturers. Gratis to genuine users of chemicals.

LES EAUX D'EGYPTE. Vol. I. By A. AZADIAN. Cairo: Imprimerie Nationale. Price (for 3 vols.) P.T.200.