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 6th, The President, Mr. Edward Hinks, was in the Chair.

The Hon. Treasurer presented the accounts of the Society for 1928, and votes of thanks were passed to the Hon. Treasurer and the Hon. Secretary.

Messrs. Marreco, Houseman & Brandon, Chartered Accountants, were appointed auditors of the Society's accounts for 1929.

The President delivered his Annual Address. Mr. E. R. Bolton moved that a hearty vote of thanks be accorded to the President for his address, and that his permission be asked to print the address in THE ANALYST. This was seconded by Dr. G. W. Monier-Williams, and the motion was carried.

The following were elected as Officers and Council for the year 1929:

President.—Edward Hinks.

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

Vice-Presidents.—John Evans, J. T. Hewitt, T. Macara.

Hon. Treasurer.—E. B. Hughes.

Hon. Secretary.—F. W. F. Arnaud.

Members of Council.—A. P. Davson, E. V. Jones, R. Lessing, A. More, W. Partridge, C. A. Seyler, J. T. Dunn, N. Evers, G. Roche Lynch, C. J. H. Stock, J. R. Stubbs, Geo. Taylor.

An Ordinary Meeting of the Society then followed, the President, Mr. Edward Hinks, being in the chair.

Certificates were read for the first time in favour of:—Peter Trevisa Clarke, B.A., Alfred Clive James, B.Sc., A.I.C., Herman Lee, B.Sc., A.I.C., James Frederick Morse, Lawrence John Odling, Willie Horner Wilkinson.

Certificates were read for the second time in favour of Frank Atkins, Edmund Baron Bennion, M.Sc., A.I.C., John Haslam, M.Sc., A.I.C., Stanley Gordon Kenrick, B.Sc., A.I.C., Bryn Jones, B.Sc., A.I.C., John Upton Lewin, B.Sc., A.I.C., and Leslie John Walker.

The following were elected Members of the Society:—William Bennett Adam, M.A., A.I.C., Alfred Louis Bacharach, B.A., F.I.C., Andrew Dargie, B.Sc., A.I.C., and Wadie J. Itayim.

The following papers were read and discussed:—"The Alkaloid Test for Tannin," by Christina Mary Fear, B.Sc. (work done under the Analytical Investigation Scheme); "The Cryoscopic Method for the Detection of Added Water in Milk," by A. L. Andrew; and "Investigations on the Relations between the Acidity and Freezing Point of Milk," by Alfred J. Parker and L. S. Spackman.

NORTH OF ENGLAND SECTION OF THE SOCIETY OF PUBLIC ANALYSTS.

THE Fourth Annual General Meeting was held on March 1st in Manchester. In the absence of Dr. Dunn, Mr. J. Wood presided, and fourteen members were present. The following committee was elected for the coming year:—Mr. S. E. Melling (Chairman), Mr. G. D. Elsdon (Vice-Chairman), Messrs. R. F. Easton, H. T. Lea, H. M. Mason, J. Miller, Prof. Roberts, J. P. Shenton.

Mr. J. R. Stubbs was elected Hon. Secretary and Treasurer, in place of Mr. H. T. Lea, who resigned. Messrs. Marshall and Coates were re-elected Hon. Auditors.

A paper was then read on "The Examination of Further Samples of Milk by the Refractometer," by G. D. Elsdon, B.Sc., F.I.C., and J. R. Stubbs, M.Sc., F.I.C.

Annual Report of Council

March, 1929.

THE Roll of the Society stands at 600, the Society having a larger membership than at any previous time.

During the past year the Council has had to report, with regret, the deaths of the following members:—

Benedict Kitto (Obituary, THE ANALYST, 1928, **53**, 314).
W. P. L. Hope (Obituary, THE ANALYST, 1928, **53**, 567).
M. S. Salamon (Obituary, THE ANALYST, 1928, **53**, 568).
A. Smetham (Obituary, THE ANALYST, 1928, **53**, 566).
J. H. B. Jenkins (Obituary, THE ANALYST, 1929, **54**, 73).
J. West Knights (Obituary, THE ANALYST, 1929, **54**, 132).
Frankland Dent.*
T. P. Blunt (Obituary, THE ANALYST, 1929, **54**, 133).
G. Watson Gray.*

The above names include that of Alfred Smetham, who was President of the Society during the years 1920 and 1921.

* Obituary notices will be published later.—EDITOR.

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

"Composition of the Fatty Acids present as Glycerides in Elasmobranch Oils." By Professor T. P. Hilditch, D.Sc., F.I.C., and A. Houlbrooke, M.Sc.
"Behaviour of Indicators in the Titration of Ammonia, Sodium and Calcium Phosphates, the Methylamines, Pyridine Bases and Boric Acid." By R. T. Thomson, F.I.C.
"Cacao Tannin." By H. R. Jensen, M.Sc., F.I.C.
"Coffee Parchment as an Adulterant of Bran and Sharps." By John Evans, F.I.C., and T. E. Wallis, B.Sc., F.I.C.
"Determination of the Colour-producing Constituents of the Cacao Bean." By W. B. Adam, M.A., A.I.C.
"Determination of Vanadium in Steel." By A. T. Etheridge, Ph.D., F.I.C.
"Colorimetric Determination of Antimony and its Separation from Tin." By S. G. Clarke, B.Sc., A.I.C.
"Determination of Carbon Dioxide in Soils." By A. Riad, B.Sc., Ph.D.
"Locust Kernel Gum and Oil." By A. L. Williams, A.I.C.
"Investigations into the Analytical Chemistry of Tantalum, Niobium and their Mineral Associates. XII, Observations on the Pyrosulphate Hydrolysis Method." By W. R. Schoeller, Ph.D., and E. F. Waterhouse.
"The Separation of Lead Tetra-Ethyl from Solution in Petroleum Spirit." By F. W. Toms, F.I.C., and C. P. Money, B.Sc., A.I.C.
"A New Precipitation Method for the Determination of Vanadium, and its Application to Steel Analysis." By B. S. Evans, Ph.D., F.I.C., and S. G. Clarke, B.Sc., A.I.C.
"Method for the Analysis of Liquorice Mass." By P. Houseman, Ph.D., F.I.C.
"Polarimetric Determination of Sucrose in Milk and Sucrose Mixtures." By G. W. Monier-Williams, O.B.E., Ph.D., F.I.C.
"The Analysis of Starch Sugar Degradation Products by Selective Fermentation." By T. McLachlan, F.I.C.
"Investigations into the Analytical Chemistry of Tantalum, Niobium, and their Mineral Associates. XIII, A New Method for the Separation of Zirconium and Hafnium from Tantalum and Niobium." By W. R. Schoeller, Ph.D., and E. F. Waterhouse.
"Improved Method for the Determination of Small Quantities of Antimony in the Form of Stibine." By Julius Grant, M.Sc., A.I.C.
"Determination of Unsaponifiable Matter in Oils and Fats." By E. Lester Smith, M.Sc., A.I.C.
"Composition of Irish Butter." By Paul Arup, M.Sc., F.I.C.
"Volumetric Determination of Mercury." By H. B. Dunnicliff, M.A., Sc.D., F.I.C., and H. D. Suri, M.Sc.
"The Occurrence and Determination of Boron Compounds in Vegetable Products." By A. Scott Dodd, B.Sc., F.I.C.
"The Determination of Small Quantities of Alcohol in the Human Subject." By John Evans, F.I.C., and A. O. Jones, M.A., F.I.C.
"The Analysis of Mixtures containing Acetone, Ethyl Alcohol and Iso-propyl Alcohol." By C. A. Adams, B.Sc., F.I.C., and J. R. Nicholls, B.Sc., F.I.C.
"The Specific Gravities and Immersion Refractometer Readings of Dilute Mixtures of Acetone and Water. By J. R. Nicholls, B.Sc., F.I.C.
"The Wijs Method as the Standard for Iodine Absorption." By J. J. A. Wijs.
"The Fatty Acids and Component Glycerides of some New Zealand Butters." By T. P. Hilditch, D.Sc., F.I.C., and Eveline E. Jones, M.Sc.

"A New Test for Boric Acid and Borates." By A. Scott Dodd, B.Sc., F.I.C., F.R.S.E.

"The Determination of Beryllium in Rocks." By B. E. Dixon, M.Sc., A.I.C.

The sales of THE ANALYST have continued to increase, indicating the useful character of the matter published.

Reference to the Treasurer's Statement, published separately, shows that the continually increasing cost of our publication and other expenses have again been successfully met.

The Council has continued to act with the Institute of Chemistry with regard to the terms of appointments of Public Analysts. A statement relative to the conditions of appointment, etc., of Public Analysts and of Official Agricultural Analysts was prepared by a Sub-Committee of the Public Appointments Committee of the Institute of Chemistry. The statement was forwarded to the Royal Commission on Local Government as representing the views of the Institute of Chemistry and of the Society.

A Committee consisting of representatives of the Society and of the Association of British Chemical Manufacturers issued its report concerning the maximum permissible amounts of arsenic in colouring matters used in foodstuffs. (See THE ANALYST, 1928, **53**, 217.)

The Society was approached by the Metropolitan Branch of the Society of Medical Officers of Health with regard to a suggested legal definition of ice-cream which had been submitted to them by a trade association. The Council did not agree with the definition and suggested the lines upon which legislation on this matter was desirable.

Consideration was given to the Food and Drugs (Adulteration) Bill which subsequently became law. Representations were made to the Joint Committee of both Houses of Parliament, with the result that certain clauses were amended.

The Society has now retained the services of Parliamentary Agents to advise them of the introduction of all Bills which might be likely to be of interest to the Society.

The four Sub-Committees constituted by the Standing Committee on Uniformity of Analytical Methods are at work on the problems submitted to them, namely:

(1) Essential oils.
(2) Milk products.
(3) Dirt in milk.
(4) Metallic contamination of food colours.

Although these Sub-Committees have issued no report during the year under review, it is expected that further reports will be forthcoming at an early date.

Work under the Analytical Investigation Scheme has been continued. The reports of three investigations have been received, six problems are still being investigated, and two grants from the Fund have been made.

The Council considered the Reconstituted and Synthetic Cream Bill, 1928, introduced into the House of Commons, and communicated with the promoters of the Bill expressing disagreement with some clauses. The Bill was withdrawn.

EDWARD HINKS, *President.*
F. W. F. ARNAUD, *Honorary Secretary.*

Annual Address of the President.

(Mr. E. Hinks, M.B.E., B.Sc., F.I.C.)

Delivered at the Annual General Meeting held on March 6th, 1929.

Ladies and Gentlemen,

The past year has been one of great legislative interest to this Society, and it seems to me fitting that I should devote to this subject the remarks which it is my privilege to address to you this evening.

The legislation to which I shall mainly refer deals with food for man or beast and with fertilisers for the soil. These matters are not of immediate concern to many of our members in their professional capacity, but they are of moment, directly or indirectly, to a larger number than those who are themselves engaged directly in the work of the Public Analyst or the Official Agricultural Analyst. I would ask the indulgence of those who are not so concerned. Their time will come. It may not be long before we have a Metals and Ores (Sophistication) Act; perhaps we may soon have the Rare Earths (Tantalum, &c., in Titanium) Regulations. In fact, our metal specialists may even now be called upon to determine whether, within the meaning of a Merchandise Marks (Imported Goods) Order, a safety razor blade, or a ball bearing, or a zinc sheet has been made in Great Britain or has been imported, or whether the indication of origin given is a correct one or not—a problem which is of the same order of difficulty as that of determining whether an egg was produced on a farm in Denmark or a farm in Devonshire. This is not entirely a fanciful picture: Orders dealing with these articles, and scores of others, are in force, though to what extent Analysts will be called upon in the execution of them is uncertain.

It is remarkable that within the space of one year the two main Acts of Parliament with which we are officially concerned have been repealed and new Acts have come into force, the Fertilisers and Feeding Stuffs Act of 1926, and the Food and Drugs (Adulteration) Act of 1928, whilst it was within the same period that the Public Health (Preservatives, &c., in Food) Regulations, in their present form, gradually introduced, came into the plenitude of their power.

With regard to the Adulteration Act, 1928, piety should compel us to remember that the Sale of Food and Drugs Act, which it replaces, was the direct cause of the foundation of this Society. The Society's activities now cover a much wider field, and its membership embraces chemists engaged in every branch of analytical chemistry, and even wider still, chemists who, though not practising as analytical chemists themselves, realise the fundamental importance of analytical chemistry. But, looking back, we see that the old Sale of Food and Drugs Act of 1875, just repealed, was the spark that brought this Society to life. Were this a dinner instead of an Annual General Meeting we should be drinking in silence the toast "In piam memoriam fundatoris nostri."

There is other legislation, to which I shall refer later, but for the moment may I confine myself to a consideration of these two Acts—the Fertilisers and Feeding Stuffs, and the Food and Drugs (Adulteration) Act.

It is of interest to note the different manner in which these two Acts have been treated. The Fertilisers and Feeding Stuffs Act was repealed and an entirely new Act, based to a large extent upon a new principle, was put in its place. It is not my intention to discuss this Act in detail—such a discussion would indeed occupy the whole of the time at my disposal. It is a complicated Act, and an inspector appointed under it has my sympathy. We wish it, I am sure, success. Time will show whether it will prove to be successful in having removed what were considered to be faults in the old Act and in ensuring to the farmer and his farm that protection which it is the object of the Act to ensure. I will refer only to matters of principle, and the main change of principle involved is the complete divorce of the civil and criminal procedure under the Act. Any sample taken at the farm can be the basis of civil claims only, whilst criminal proceedings can follow only upon the taking of a sample at the premises of the seller, before delivery to the ultimate purchaser takes place.

The Food and Drugs Acts, and some of the kindred Acts, were not altered in principle or amended in any way, but were submitted to a process of consolidation. Consequently, if they had faults and deficiencies, those faults and deficiencies remain. I have not been able to see myself, neither have I found anyone who has discovered, the merits of this consolidation. Moreover, consolidation is a difficult process, and there is in my mind the horrid fear that in this difficult process some wording has been altered, or a word omitted or inserted, which will give the ingenious legal mind, which, we may be sure, is being directed to the matter, an opportunity of making the new Act inoperative. The future will show whether or not this fear is ungrounded—I trust it is. Certain it is that the alteration in form of a few words has reintroduced an old difficulty which, in its day, required an amending Act for its removal. History may have to be repeated: I will not prophesy, especially with regard to a legal point.

Why have these Acts had such different histories?

It is plain that, though in essence the relations between the seller and purchaser of a fertiliser and feeding stuff or of food are the same in either case, the method of treatment of these relations must be different in the two cases. The vast number and the small bulk of most purchases of foods, as compared with those of feeding stuffs and fertilisers, alone is responsible for differing treatments of the relation. I do not know that this alone is responsible for such widely differing treatments as are to be observed. Incidentally, a curious difference in viewpoint is that, under the Food and Drugs Act, the requirement is that the article should be of the nature, substance and quality demanded, whereas, in the case of the Fertilisers and Feeding Stuffs Act, the requirement may be that the guarantee shall be in accordance with the article sold, a difference which, to my mind, makes it very difficult to frame a certificate strictly in accordance with the latter requirement.

This, however, is rather a matter of detail. Of greater significance is the fact that, under the Fertilisers and Feeding Stuffs Act, there is established an Advisory Committee, carrying on the work of the previous Committee which advised on technical matters before the Act was drafted. This Committee advises the Minister on the question of Regulations made under the Act. Further, there are established under the new Act, as under the old, definitions, statutory guarantees of important constituents, and methods of analysis. Moreover, and it is most important to note this, the Advisory Committee continues in being, and provision is made for alteration of the schedules, definitions and methods of analysis as occasion may require. It is, I think, true that the definitions are meagre and, if I may criticise them, not very illuminating, but for the moment I concentrate upon the principle. Why should not these principles be adopted, and widely adopted, in Food and Drugs legislation? Admitted that in the case of foods the variety of article is greater, and admitted also that in a piece-meal manner some definitions and standards have been established, one or two by the Minister of Agriculture under the Food and Drugs Act, a few more by the Minister of Health under the Public Health Acts. Whilst acknowledging the help that these have been, I feel that only the fringe, or not much more than the fringe, has been dealt with. An Advisory Committee or Committee of Reference for Food Legislation has been advocated by Departmental Committees, by a Royal Commission, and by this Society.

Is it faulty reasoning to argue that, if such principles can be incorporated in one Act, so can they be, if desirable, in the other? Perhaps the dimension of the problem with regard to food for human consumption is a reason for the difference in treatment of the two questions: it can hardly be considered a sufficient reason for leaving the matter alone.

Presumably the Fertilisers and Feeding Stuffs Act, as an Act, will stand as it is for some time. Can the same be said of the Food and Drugs (Adulteration) Act? And, if it can, is it desirable that the legislation should stand as it is?

And here I would refer to an address, given in June of last year by Mr. Haygarth Brown, of the Ministry of Agriculture, to the Incorporated Society of Inspectors of Weights and Measures, entitled "On Quasi-criminal Offences." This is of much importance, as it was an expression of the views of a high official in the Ministry of Agriculture: perhaps, I do not know, similar views are held in other Ministries, and Ministries are very powerful in the shaping of legislation. Mr. Haygarth Brown's main thesis is, that there is a growing revolt against the "quasi-criminal offence"; by which I understand him to mean a revolt against the principle of offences under Public Control laws, such as the Food and Drugs Acts, being criminal offences. I think we shall all agree that there are occasionally offences against the Food and Drugs Act which should hardly be stigmatised as criminal offences—I wish it to be understood that I use the word "criminal" in a popular sense and not the legal sense—but, on the other hand, I think we shall all agree that offences which involve fraud are criminal offences. There is, I admit, a distinct difference between selling an article which has fallen naturally, and perhaps

quite unsuspectedly, below some legal limit, and the action of deliberately adulterating. But who is to decide which of these two things has happened except the Courts, even if they can?

Mr. Haygarth Brown founds many arguments on what he calls the "tinned food offence," when a retailer gets into trouble for an offence which really is committed by a manufacturer; he does not mention warranty defence, but it will be allowed, I think, by everyone that, granted an offence, the real offender is the person upon whom should fall the opprobrium of meeting the charge.

Mr. Haygarth Brown seems to blame the Acts of Parliament for creating offences and thereby creating offenders. In a way, of course, it may be argued that the law creates an offence, but, if we look deeper, is not the offence already there? Take the case of the Preservatives Regulations. The assumption underlying these Regulations is that chemical preservation of food may be, and if practised to an excessive extent, is, injurious to the health of the consumer. Or take the case of Butter Regulations. Selling butter containing an excessive proportion of water is prejudicial to the purchaser's pocket and, to the extent to which the excessive addition is made, is prejudicial to his health. Can we allow that it is the Regulations that create offences and thus create offenders, who, if it were not for the Regulations, would be doing nothing wrong? Was short weight always wrong, or did it only become wrong—and did having unjust scales and measures only become wrong—when Acts of Parliament made such things offences?

It is clear, on the other hand, that some legislation does create offences in matters in which there was no suggestion of offence or wrong before. The Merchandise Marks Act, 1926, does this. No one can suggest that before the Regulations were made under this Act there was anything morally or legally wrong in selling an imported article without declaring it to be such, and marking it as such. With certain specified articles this Act has now created this offence.

However, in a complicated civilisation, public control legislation would appear to be necessary, and, within reason, beneficial. It may be carried to excess, but it would be the greatest mistake to undermine such important and essential legislation as the Food and Drugs, and kindred Acts, because there may be excesses in legislative control of the citizens' and the traders' activities—and a revolt against such excesses.

Mr. Haygarth Brown discusses methods of procedure, alternative to the present "quasi-criminal" procedure, but it is difficult to see in them any hopeful programme which would placate those, if there be any, who object to the procedure of the Food and Drugs Act, and, at the same time, afford adequate protection to the purchaser, worthy, however, of study as is the paper to which I have referred. It must not be forgotten, and I think it is sometimes forgotten, that it is the purchaser's interests which are primarily to be protected.

In parenthesis, I would mention the question of drugs. We are all looking forward to a new edition of the British Pharmacopoeia. If this is delayed, I, for one, would not complain. We can all appreciate, from a distance, the amount of

work that is necessary for revision and amendment. But what of its position in relation to the Food and Drugs Act?

In many of the Dominions the British Pharmacopoeia is the statutory standard for drugs under the Food and Drugs legislation: in this country, the country of its origin, it is not: perhaps this is another case of a prophet not being without honour save in his own country. The Committee of Civil Research (Sub-Committee on the British Pharmacopoeia) has considered this matter. The fact that the Pharmacopoeia has been accorded a definite statutory position overseas, the Committee considered to be "a tribute to the authority of the work which we recognise with satisfaction." The Committee, however, reports definitely against giving it a similar position here. To quote from this Report: "The topic is one which is scarcely within our terms of reference, but as we have considered it we may be permitted to express our opinion, which is that no legislative action is called for in this country. . . . Indeed we are apprehensive that an attempt to give a specific legal sanction to the Pharmacopoeia might do more harm than good. We see practical difficulties in making the Pharmacopoeia an absolute legal standard for the articles mentioned in it, and indeed the work is not designed to serve this object." I suggest that the point is rather, could it not, and should it not, be so designed? However, what I have quoted is the authoritative opinion of the Committee of Civil Research, so, presumably, the somewhat equivocal position of the Pharmacopoeia in relation to Food and Drugs legislation will remain.

I have referred to these legal, and ethical, matters for the purpose of my argument, which is, that with regard to future legislation, in particular Food and Drugs legislation, this Society should have, and should have ready, a policy.

In the year 1894 the Council of this Society produced a complete draft of a Sale of Food and Drugs Act. I was much surprised to find it, as I did, one day when I was glancing through old volumes of the ANALYST. Some of our senior members here this evening must have taken part in framing that draft Act.

Personally, I do not think that the Council should attempt to repeat that effort. If we have any new principles which we think should be adopted, if there are changes in procedure which our experience shows to be advisable, let us urge these and leave to others their incorporation in a Bill. But let us beware of decrying the existing Act, or Acts, and then being found wanting when we are appealed to for concrete suggestions for amendment. I am fairly familiar with the proceedings of this Society for a number of years now, but, if I were appealed to at this moment to say what is the policy of the Society, I should find it difficult to give an answer.

There are the big questions of definitions and standards. What is the Society's policy in regard to these? As I have mentioned, there are many definitions under the Fertilisers and Feeding Stuffs Act. How many are there, under the Food and Drugs Act? A cotton cake for cattle must contain, within one-tenth, the amount of oil which it is declared to contain, and its oil content must be declared: a cream for human consumption must contain, how much oil or fat? I do not know: no one knows. All we know is that it does contain anything, say, from eighteen to eighty

per cent., or nearly those figures. Standards and definitions—if we had more definitions we should hear less of the innocent manufacturer being pilloried in the Police Court, an infliction with the contemplation of which you were harrowed from this chair two years ago. I have every sympathy with the manufacturer who is trying to produce at a competitive price a sound, wholesome article: but, at the same time, I have equal sympathy with the Public Analyst who, seeing what he thinks at any rate to be wrong, after making such enquiries, investigations and efforts in other directions as he can, has to put the matter to the test, or let things slide. The fault lies, I submit, with the vagueness of the legislation under which the Public Analyst has to work.

For our consideration there are the questions of the pre-packed or tinned article—the responsibility of the retailer and the manufacturer—and of labelling, which is of great importance, though one wonders to what extent the public reads labels. Does the Society still consider that a body such as a court of reference or advisory committee should be formed, and what form should it take? There is the much-debated question of prescribed methods of analysis. Then, again, there is the control of the purity of substances entering into the composition of food. Cases must arise in which such control can hardly be exercised by analysis merely of the finished article. The purity of food colours is such a case. A similar problem arises under the Preservatives Regulations—it is not possible, by analysis of the finished article, to determine whether a preservative has been added to a non-scheduled compounded article, which would be a breach of the Regulations, or whether it has been introduced through the use of an ingredient which is itself a scheduled article in which the preservative is permitted. Hence arises the necessity for right of entry, conceded in certain circumstances by the Preservatives Regulations and by the provisions in the Act relating to butter factories.

There is the very difficult question of the control of the vitamin activity of foods, or the checking of statements made about that activity. How is such control to be exercised? Some may think that, in this rapidly developing and changing realm of enquiry, it is too early at present to attempt to establish legal control. I am afraid that it is the difficulty of the question, rather than any lack of need for its consideration, that is a determining factor. There has, as you know, been one case under the Food and Drugs Act where vitamin activity was the issue; it would appear to be certain that before long it will become a very big issue.

It is on these matters that the Society should attempt to have a policy. I say "attempt," because it is possible that on some of them agreement could not be reached. It might be that Public Analysts on the one hand, and other analytical chemists on the other, might think differently. Towards the end of last year there occurred such an event, in which the Society, as such, was not at first officially concerned, but in which a number of its members were. Agreement has not yet been reached.

I do not mean that there is antagonism between those whom I may term the official chemists and the manufacturers' chemists. Two years ago Mr. Bolton referred to the way in which this Society had brought the two bodies of chemists

together to live and work in harmony. I fully endorse what he said on that point. Nevertheless it is possible that there may be some divergence of views. This Society, for the very reason that its members are working in very different fields, should, at any rate, be able to view all sides of these questions. Seeing all sides of a question has, however, its drawbacks as well as its advantages.

This matter of having a policy is one which I commend to the new Council elected this evening, for their immediate and earnest attention.

There are now some other Acts of which mention must be made. The Sale of Food (Weights and Measures) Act, 1926, which is, I believe, now fully in force, concerns sale by net weight. There are three schedules, enumerating some 30 articles of food to which the Act applies. Analysts will, no doubt, be consulted as to whether the article sold is or is not suet, is or is not bean flour, and so forth.

The Merchandise Marks Act, 1926, I referred to incidentally earlier; it relates to the marking of the country of origin of imported goods. Eight articles of food are at present affected and scores of other articles. I do not know how these Orders are going to be enforced—possibly it will all be done at the port of entry, but it is significant that, with regard to articles of food, Food and Drugs Authorities are empowered to take samples—what is to be done with the samples is obscure. The problem raised here is the differentiation of home, foreign and Empire apples, honey, eggs, dried eggs, oat products, etc., mowing machines, gloves, glue, artificial teeth (artificial eyes are especially excluded), pumps, briar tobacco pipes, ladies' handbags, carbon paper, and so on, a regular stores catalogue.

The Agriculture Produce (Grading and Marking) Act raises similar problems, except that, in some respects, they are explicitly chemical—the differentiation of fresh, preserved, cold-stored and chemical-stored eggs. A respite is granted by an Order of the Minister of Agriculture exempting from the operation of Section 3 of the Act eggs preserved by cold storage or chemical storage because, the Minister says, "it is not possible to ascertain by analysis whether eggs have, in fact, been kept in cold storage or chemical storage." Eggs preserved by immersion in lime water, water-glass or oil do not get exemption.

The Safeguarding of Industries Act, though now many years old, has, no doubt, produced its annual crop of problems for the Government Chemist.

One further small point. By Statutory Rules and Orders No. 975, 1928, under the Silicosis scheme, Workmen's Compensation Act, silica rock by definition does not include any rock containing less than 50 per cent. of free silica, whilst for other purposes under the scheme the limit is 80 per cent. of total silica.

It is more than probable that I have mentioned only a proportion of these modern legislative enactments and regulations—and the flood of orders may continue.

Now what is the lesson to be learnt from all this? It is, surely, that the services of the analytical chemist are to be called upon to an ever-increasing extent. I have referred to the ethical side of some of the legislation—that is not particularly the business of the Society; then I spoke of the necessity of having a policy,

especially with regard to Food and Drugs legislation—that is more our business; but our main function is the advancement of analytical chemistry, and it is in this direction that our main efforts should be directed. The efficacy of much of the legislation I have spoken of depends upon our ability as analysts.

As you know, certain problems are in the hands of Sub-Committees convened by the Society. An enormous amount of work has been done by the Sub-Committees, but the rate of progress is disappointingly slow. I am not, I think, betraying any confidences when I say that one cause of the slow progress is the surprising extent of the experimental differences or errors disclosed when a number of analysts employ the same analytical process, often a process that has been intensively studied. As these Sub-Committees are working under a scheme for the uniformity of methods of analysis, uniformity of results is one of the essentials.

But whatever problems are being investigated by these or other Committees, by official or semi-official bodies which are or may be formed for specific purposes, individual members of this Society are not relieved of the responsibility of individually contributing to the solution of the many problems that confront us. Mr. Chaston Chapman, in his admirable address to you in 1915, quotes an eminent chemist, whom he kindly omits to name, as saying "Analytical chemistry presents no further problems"—I quote Mr. Chapman's refutation, "A statement more wide of the mark could scarcely be imagined." The popular view of analysis, and apparently the view of that "eminent chemist," would seem to be that by analysis a material is infallibly separated out into neat little heaps of the various constituents, which heaps can then be conveniently weighed or measured; this view must, I think, account for the existing marked disinclination to disclose to the analyst anything about the material to be analysed. How different the reality —how often is an analysis, with all the knowledge that we have, and it is not inconsiderable, yet an adventure through unexplored or, at any rate, very imperfectly mapped, territory. Even the well-trodden paths have a disconcerting tendency to become slippery and to afford an insecure foothold, or they become entangled with undergrowth and obscure; often, by degrees or abruptly, the path is no more a path, it is lost in the jungle. Is it an exaggeration to say that even butter is on a slippery part of the path and, with many other things, jam is in the jungle? Of course, thanks to the labours of those who have gone before and those who are here now, we can do some things, and do them very well. But the labours must be continued, and every branch of chemical science must be impressed into the service of analytical chemistry.

We can fulfil what I have described as our main function in relation to legislation only by research; the abrogating order of the Ministry of Agriculture referred to just previously comes aptly to point the moral. There is need for imaginative research of a very high order; there is room also for research of a humbler nature. The President of one of the Sections of the British Association last year, in speaking of research, coined what is, I think, a very arresting phrase, "If there is one thing worse," he said, "than a mediocrity who does no research, it is a mediocrity who does." By that I do not think that he meant to decry honest,

useful research of a humble character; it was, I should think, rather a protest against the person who labels himself as a research worker because he has neither the ability, nor the skill, nor the character to do anything else.

When one looks at the number of communications to this Society, one cannot be satisfied that nearly all that should, and could be done is being done. The Society, and Analytical Chemistry, is indebted to private members, to the Government Chemist and his department, to the Chemist at the Ministry of Health, to chemists in other Government departments, and to a number of chemists at Research Association laboratories, for many very valuable contributions.

I should like to take this opportunity, too, of expressing appreciation of certain enlightened commercial firms who are liberally giving the services of their chemists and the resources of their laboratories for the purpose of investigations, in which we, and they, are interested.

There is hardly a single substance with which we have to deal which does not require investigation; there is hardly a process which by investigation cannot be improved; many substances and many processes cry aloud for this investigation. It is pleasant to be able to record that, under our Analytical Chemistry Research Scheme, three reports have been received during the year, and that six problems are in hand. I suggest, however, that amongst our members there are many who could contribute to the resolving of some of our uncertainties but do not do so.

I have directed your attention this evening to legislation and our relation to it. Quite apart from other fields in which chemical analysis is of fundamental importance, it is clear that in the fields covered by legislation more and more will be demanded of the analytical chemist. It is for us to see that, up to the limits of possibility, that demand is met; and for the future it can be met only by investigation and research.

The Cryoscopic Method for the Detection of Added Water in Milk.

By R. L. ANDREW.

(*Read at the Meeting, March* 6, 1929.)

During the last twenty years numerous papers on this subject have been published. So far as the writer is aware, none of these deals with the use of the method, over a period of years, as a routine test in the examination of milk samples. It was therefore thought that a description of the test, as employed in the Dominion Laboratory, New Zealand, would be of value.

Interest in the method was aroused by a paper entitled "The Freezing Point of Milk, its use in the Detection of Added Water," by J. B. Henderson, Government Analyst, Brisbane, Queensland, which was published in the Proceedings of the Australasian Association for the Advancement of Science, 1909, p. 159. In this paper Henderson stated that after experimenting with milks of known purity, and with milks with known amounts of added water, he adopted the method for samples obtained by inspectors under the Health Act.

A careful investigation seemed warranted, and a number of genuine milks from various farms were examined in this laboratory. The samples were taken throughout the year, the greater number being obtained during winter and spring. Both morning and evening milks were sampled. Good and poor farms were visited. In some cases samples were obtained from individual cows; in others the sample was from the mixed milk of two to twelve cows. The inspector in every instance watched the milking of the cows, and satisfied himself that no adulteration was possible. In all, 270 samples were obtained. The usual analysis of each for fat and non-fatty solids was made. The zero point of the thermometer was checked both before and after each set of observations. The acidity of the milk was also determined, to ensure that no appreciable souring (which would result in a lowering of the freezing point) had taken place.

As was to be expected, analysis disclosed wide variations in the fat and solids-not-fat. The fat ranged from 2·35 per cent. to 5·9 per cent. (though only ten samples were below 3·25 per cent., the legal standard for New Zealand). The solids-not-fat were from 8·06 to 9·43; eighteen samples were below the legal minimum (8·5 per cent.). The maximum variation of the freezing point was from —0·545° C. to —0·565° C. One sample only was above —0·550° C., and five only below —0·560° C. With the other 264 the freezing point ranged from —0·550° to —0·560° C.

It would serve no useful purpose to publish the whole of the results, but a few which show gradations in fat and in non-fatty solids have been selected, and arranged in the following tables.

In Table I it is seen that large variations in the fat make no difference in the freezing point. This, of course, might be expected from theoretical considerations.

Table II shows a series of milks, varying somewhat uniformly in solids-not-fat from 8·06 to 9·43. The freezing points are all practically identical.

TABLE I.

Fat.	Solids-not-fat.	Freezing point.
5·90	8·59 per cent.	—0·550° C.
5·70	8·99 ,, ,,	—0·560° C.
5·17	8·95 ,, ,,	—0·560° C.
4·40	8·93 ,, ,,	—0·560° C.
3·70	9·15 ,, ,,	—0·550° C.
3·20	8·83 ,, ,,	—0·550° C.
2·90	8·32 ,, ,,	—0·557° C.
2·65	8·95 ,, ,,	—0·550° C.
2·35	9·15 ,, ,,	—0·550° C.

TABLE II.

Fat.	Solids-not-fat.	Freezing point.
3·65	9·43 per cent.	—0·550° C.
3·95	9·29 ,, ,,	—0·555° C.
4·20	9·12 ,, ,,	—0·555° C.
3·80	8·88 ,, ,,	—0·550° C.
3·80	8·67 ,, ,,	—0·550° C.
3·15	8·50 ,, ,,	—0·560° C.
3·20	8·33 ,, ,,	—0·555° C.
5·20	8·26 ,, ,,	—0·550° C.
4·00	8·16 ,, ,,	—0·550° C.
4·70	8·09 ,, ,,	—0·550° C.
3·45	8·06 ,, ,,	—0·550° C.

These results were so favourable, that the method was adopted and has now been in use for fifteen years as a routine test, in the examination of milk samples submitted by inspectors under the Sale of Food and Drugs Act.

A description of the apparatus employed, the procedure in carrying out the test, and an account of its general use are given below.

APPARATUS. –The cryoscope employed is the simple form of Beckmann's Freezing Point Apparatus. The thermometer is of the usual Beckmann type, and is used for the one purpose only, thus avoiding the necessity of making frequent adjustments. The stirrer is made of fairly heavy brass wire, which is more suitable than the platinum stirrer usually supplied with the apparatus.

METHOD OF CARRYING OUT THE TEST.—Although the determination of a freezing point is a simple operation, it was found that much practice and strict attention to details were required if dependable results were quickly to be obtained. At first an attempt was made to carry out the test as recommended by Barthel in *Milk and Dairy Products*, p. 106 (1910), but difficulties were met with, and the procedure finally adopted was as follows:—The apparatus is filled with crushed

ice mixed with a small quantity of common salt, and in a separate vessel is placed a strong freezing mixture of ice and salt. The zero point of the thermometer is first ascertained by observing the freezing point of water. Sufficient distilled water to cover the bulb of the thermometer completely is placed in the freezing tube and the thermometer inserted. The tube is then placed directly into the strong freezing mixture, so that the surface of the water is just above the surface of the freezing mixture. The water is stirred slowly until a skin of ice is formed on the inside of the tube. The tube is then removed from the mixture, wiped with a dry cloth and warmed in the hand until the ice can be detached and broken up by means of the stirrer. The tube is then placed in the large tube of the apparatus and stirring continued. The mercury column falls slowly and finally remains stationary. The reading is now taken. In determining the freezing point of water some experience is necessary in order to arrive at the correct proportion of ice required in the freezing tube, and the degree to which it must be broken up.

To obtain the freezing point of milk the same quantity of milk is cooled in the strong freezing mixture. Super-cooling usually takes place, and immediately the mercury column begins to rise, the tube is quickly removed, wiped dry and placed in the large tube of the apparatus. Stirring is then continued until the mercury reaches its highest point and remains stationary. The reading is now taken, and is the freezing point of milk. With milk about one degree of super-cooling most usually takes place, and in these cases no correction is made. Occasionally there is greater super-cooling, and it has been found that the reading is then 0·01° C. too low for every extra degree of super-cooling. Sometimes it is difficult to obtain super-cooling. In such cases the mercury column becomes almost stationary at a point somewhat above the freezing point of the milk. At this stage the tube is removed from the freezing mixture, wiped, placed in the apparatus, and stirring continued. The mercury falls slowly, and finally remains constant. The reading is then taken, and agrees closely with that obtained after one degree of super-cooling.

I am aware that certain corrections should be made to obtain the true freezing point, but these have been intentionally omitted for the sake of simplicity and ease of working, and, as similar conditions are observed in each determination, the results obtained are strictly comparable. The procedure outlined above has the great advantage of simplicity both in apparatus and materials employed, while the manipulation required is reduced to a minimum. In practised hands twelve determinations per hour can be made, which compares very favourably in speed with other routine determinations, such as for fat and solids-not-fat.

At first readings to 0·001° C. were attempted, but experience showed that readings to 0·005° C. were sufficiently exact for practical purposes.

Samples giving any evidence of souring are rejected.

As far as possible all abnormal or apparently abnormal samples have been followed up and investigated. This has resulted in the accumulation of a series of most interesting results. As they afford particularly strong evidence as to the reliability of the test, a number of these investigations is given in detail.

1. A sample received from Napier contained:—Fat, 3·40; solids-not-fat, 8·50; and ash, 0·68 per cent. Freezing point, —0·500° C.

At one time this milk would have been passed as complying with the standard, but the freezing point showed that it contained nine per cent. of added water. The inspector was advised as follows:—"The analysis of the sample showed that sufficient water had been added to bring the solids-not-fat just down to the standard (8·5 per cent.). It is very important that systematic adulteration of this kind be stopped, and samples from the farm would make our case quite conclusive." A sample of the mixed milk (evenings) was accordingly obtained at the farm and contained:—Fat, 5·20; solids-not-fat, 8·92; and ash, 0·75 per cent. Freezing point, 0·550° C.

If allowance is made for the additional fat in the farm sample, the solids-not-fat would be 9·1 per cent., which agrees closely with the percentage found in the original sample, when corrected for the added water.

2. A milk forwarded by the Health Department's inspector at Wanganui gave:—Fat, 4·50; solids-not-fat, 8·46 per cent.

As with the previous example, this milk would formerly have been passed as complying with the standard. The freezing point was, however, found to be —0·510° C., showing that the sample contained 7·2 per cent. of added water. Prosecution was advised, and it is interesting to note, that it was then found, that the milkman had admitted adding water to the milk.

3. A sample received from Taranaki was found to be of the following composition:—Fat, 5·00; solids-not-fat, 8·85 per cent.; freezing point, —0·505° C.

This was a milk of good quality, the proportion of fat being above the average, but the freezing point indicated that it contained 8·2 per cent. of added water. It was decided to investigate, and an inspector obtained at the farm samples representing both morning and evening milkings. Analyses resulted as follows:

	Fat. Per Cent.	Solids-not-fat. Per Cent.	Freezing point. °C.
Evening milk ..	4·50	8·63	—0·520
Morning milk ..	4·85	8·81	—0·510

These results appeared to show that the milk from this herd had an abnormal freezing point. The inspector was instructed to obtain further samples, and as it was found that on his first visit, he had not been able to watch all the cans, he was told to take an assistant with him. All the cans, as well as the milking were closely watched, and the analyses of the samples then obtained were:

	Fat. Per Cent.	Solids-not-fat. Per Cent.	Freezing point. °C.
Evening milk ..	5·80	9·16	—0·555
Morning milk ..	4·85	9·55	—0·560

The freezing point of the pure milk was thus proved to be normal. The composition of the original sample (when corrected for the 8·2 per cent. of added

water indicated by the freezing point) would be:—Fat, 5·45; and solids-not-fat, 9·64 per cent.

This agrees closely in solids-not-fat with the second sample of morning milk. Although the cows on the farm were all Jersey, it was thought that the milk was exceptionally rich. Subsequent experience has, however, shown that such milk is by no means unusual in districts where Jerseys predominate.

4. The analysis of a milk received from Palmerston North was:—Fat, 3·10; solids-not-fat (per cent.), 7·46 per cent.; freezing point, —0·510° C.; added water (calculated from the freezing point), 7·2 per cent.

Allowing for the added water, the unadulterated milk would be of the following composition:—Fat, 3·34; solids-not-fat, 8·04 per cent.

If this deduction were correct, the sample came from a herd yielding milk containing a percentage of solids other than fat below the legal standard (8·5 per cent. of solids-not-fat). Samples were obtained from the farm and examined, with the following results:—Fat, 3·70 and 3·15; solids-not-fat, 7·82 and 8·09; freezing point, —0·550 and 0·555° C.

These figures show that the deduction drawn from the freezing point of the original sample was correct. The milk came from a herd of Friesian cows, a breed which is known often to give milk of poor quality. The farmer was notified that he must not sell the milk for town supply, and he accordingly diverted it to a butter-factory.

5. Two samples obtained in Wellington were analysed, with the following results:—

	Per Cent.	Per Cent.
Fat	3·90	3·60
Solids-not-fat	8·26	7·98]

The freezing points were normal, indicating that the milks did not contain added water. Samples were obtained at the farm. The herd was a small one, and a sample of each cow's milk was obtained, as well of the mixed milk. Analyses resulted as follows:

	Cow No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	Mixed milk.
Fat, per cent.	4·00	3·90	3·65	3·40	4·20	3·80
Solids-not-fat, per cent.	8·12	8·22	7·12	7·98	7·88	7·90

The freezing points were all normal. These results confirmed the previous analyses, and showed that the freezing points correctly indicated that the milk was naturally poor and not rendered so by the addition of water.

6. A milk traced to a farm in the Hutt Valley gave the following results:—Fat, 4·20; solids-not-fat, 8·20 per cent.; freezing point, —0·550° C.

Although the percentage of solids-not-fat was below the standard, the freezing-point showed that the milk contained no added water. An inspector visited the farm and obtained single samples from the twenty-two cows comprising the herd. The herd was a mixed one, and the cows were in poor condition.

Most of the samples were below the average in quality, while four of them were very poor. The analyses of these were:

	Cow No. 6.	Cow No. 8.	Cow No. 10.	Cow No. 13.
Fat, per cent.	4·00	2·40	4·00	3·70
Solids-not-fat, per cent.	7·80	5·76	7·54	8·00
Freezing point	—0·555° C.	—0·550° C.	—0·560° C.	—0·560° C.

No. 8 is an abnormally poor milk. Examination by a veterinary surgeon showed that the four cows were all suffering from mammitis.

7. Three samples were obtained from a farmer at Otaki and analysed, with the following results:

	(1)	(2)	(3)
Fat, per cent.	3·70	4·40	4·30
Solids-not-fat, per cent. ..	7·08	8·22	8·62
Freezing point	—0·410° C.	—0·450° C.	—0·480° C.
Added water (calculated from the freezing point), per cent.	25·4	18·1	12·7

Allowing for the added water, the original milks would have the following composition:

	(1)	(2)	(3)
Fat, per cent.	4·96	5·37	4·92
Solids-not-fat, per cent. ..	9·49	10·03	9·87

These figures would indicate that the samples came from a herd giving exceptionally rich milk. Samples of the milk from each of the twenty cows in the herd were obtained at the farm, and the analyses fully bore out this supposition. The milk from individual cows contained abnormally high percentages of fat and of solids-not-fat, and the mixed milk of the herd was exceptionally rich. The results were:

Single cows.	Fat.	Solids-not-fat.	Freezing-point.
1	7·20	10·50	—0·560
2	7·40	10·70	—0·560
3	7·35	10·75	—0·565
4	7·00	10·02	—0·555
5	6·75	9·95	—0·565
6	5·60	9·90	—0·560
7	5·85	10·29	—0·555
8	5·70	10·28	—0·555
9	7·60	10·50	—0·560
10	8·40	11·40	—0·555
11	6·60	10·64	—0·560
12	7·05	10·13	—0·555
Mixed milk of 20 cows	5·40	9·70	—0·555

No. 10 contains a higher percentage (11·4) of solids-not-fat, than I have seen recorded for cows' milk. This herd was partly Jersey and Jersey cross, and several of the cows were being dried off.

The real value of the test is strikingly shown in the improvement brought about in the milk supply of Wellington City. Prior to the adoption of the test the sale of watered milk was a very common practice, and the milkmen knew that about seven per cent. of water could be added to average milk, without the risk of prosecution.

After its adoption the milkmen concerned very soon realised that the hands of the analyst had been greatly strengthened. Numerous cases were taken, and instead of offenders being charged with a deficiency, calculated on a standard of 8·5 per cent., solids-not-fat, the amount of added water was stated, usually seven per cent. more than that which would have been calculated from the standard. Many of the cases were keenly fought, but in no instance was the reliability of the test disproved or weakened.

The effect of such action was quickly reflected in the average solids-not-fat content of the milk sold in the city. The average solids-not-fat in 880 samples taken during 1910–1913 was 8·50 per cent. For the year 1916 (two years after the adoption of the test) the average had risen to 9·01 per cent. This average has since been maintained, being 9·02 per cent. over that of the ten years 1917–1926.

The account of the method of using the test has been given at some length, but that is necessary to illustrate fully all its advantages, which may be summed up as follows:

It provides a simple and reliable means of detecting added water in milk. It makes possible the prevention of the practice of adding sufficient water to rich milk to bring the solids-not-fat down to the legal standard.

It provides a means of distinguishing between naturally poor milk and milk to which water has been added.

From an experience extending over seventeen years, and as a result of thousands of determinations, and also from the investigation of apparently abnormal cases, I have concluded that the freezing point of genuine milk, determined in the manner described above, may be taken as not higher than —0·550° C.

If the freezing point of a sample rises to —0·530° C. watering may be suspected, and if to —0·520° C., the milk has certainly been adulterated with approximately five per cent. of added water.

I should like to acknowledge the helpful advice given by Dr. J. S. Maclaurin (Dominion Analyst) in the early stages of the work, and the continued interest shown by him over the whole period.

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Investigations on the Relations Between the Acidity and Freezing Point of Milk.

By ALFRED J. PARKER and L. S. SPACKMAN.

(*Read at the Meeting, March* 6, 1929.)

In the determination of added water in milk by the cryoscopic method, it is necessary to use a correction for the disturbing effect of acidity when testing "sour" milk (Monier-Williams, *Food Report*, No. 22, p. 3). The correction in use in this laboratory for some time had been that given by the " Connecticut Agricultural Experimental Station, 27th Report on Food Products (1922) (*cf.* Analyst, 1924, **49**, 280), namely, 0·003° C. for each 0·01 per cent. of acidity as lactic acid in excess of 0·20 per cent. Experiments made in this laboratory caused us to doubt the validity of the above correction, and the present investigation was therefore undertaken.

A sample of freshly drawn milk is taken, and its freezing point and acidity determined. The sample is then allowed to stand and become progressively more acid. The freezing point and acidity are now determined conjointly at intervals, and the results plotted on rectangular co-ordinate paper, with freezing point as ordinates and acidity as abscissae; then, if the above-mentioned correction be valid, the points will all lie on a straight line, the equation for this being:

$$T = [C(A - A')] + t \qquad (1)$$

where T is the observed temperature in degrees centrigrade below zero; t, the normal freezing point of milk; C, a constant (in this case 0·003); A, the acidity (expressed as degrees of acidity); and A', acidity of fresh milk (also expressed as degrees of acidity).

To simplify matters we used the Dairy Chemists' "Degrees" of acidity, according to which one "degree" corresponds to 0·01 per cent. of acidity as lactic acid.

The term "fresh milk" used hereafter, refers to milk, cooled at the farm and delivered to the city depot; our samples were taken on arrival, which, in the case of morning's milk, represented an interval between milking and sampling of about six hours, and the first tests were made as soon as possible after sampling. In the case of evening's milking an interval of about 4 hours elapsed between milking and sampling, and, after sampling, the samples were kept in a cool chamber (50° F.) overnight, and the first tests were made next morning.

At no time during our experiments did we have a *fresh* milk with an acidity as high as 20 degrees, practically every sample falling within the limits of 14 and 17 degrees; 31 per cent. of those examined had an acidity of 14, and 52 per cent an acidity of 15.

Generally speaking, the samples did not develop acidities near 20 at room temperatures (60° F.) until they had been kept for a day, and sometimes longer.

Our method of testing was as follows:—The freezing point and acidity were taken as soon as possible after drawing the sample. The remainder of the milk was loosely covered with a watch glass or clean piece of paraffined cardboard (milk bottle tops) and allowed to stand on the laboratory bench. At varying intervals afterwards, the bottles were shaken, a portion withdrawn, and the freezing point and acidity determined as before.

A Beckmann freezing-point apparatus was used, cooled by an ice and salt mixture, and fitted with a stirrer made from a piece of 12-gauge copper wire. The thermometer was graduated in 100ths of a degree Centigrade and could be read to half a graduation by inspection. The thermometer was checked repeatedly both with distilled water and cane sugar solutions.

The temperature of the ice and salt mixture was chosen to give a super-cooling of about $-1{\cdot}5°$ C., as, contrary to the experience of other observers, we found that the best agreement between readings was obtained at this point. The mercury column would fall steadily to about $-2°$ C., then rise rapidly to a maximum, and then fall again slowly, the variation between readings seldom being more than approximately $\pm 0{\cdot}002°$ C. When the super-cooling was regulated to a lower degree, as recommended by Monier-Williams (*Food Report* No. 22), the results obtained were by no means so consistent. The portion to be tested was cooled in crushed ice, and when at a temperature of approximately 0° C., it was poured into the containing vessel, and stirred steadily by hand, with an up-and-down movement, at the rate of about twice per second (four movements) during the whole time of the test.

The acidity was determined by titrating 17·6 c.c. of milk with $N/50$ sodium hydroxide solution, with phenolphthalein as indicator, when each c.c. of standard solution corresponds to 1 degree acidity; readings were taken to 0·5 degree.

To save space, the results are not tabulated here in detail, but in Fig. 1 are shown graphically the mean freezing points for different acidities (in circles), while for purposes of comparison the graph of $T = [C(A - A')] + t$ (with $C = 0{\cdot}003$ and A prime $= 20$ degrees) is shown in dotted lines. The points are not plotted beyond an acidity of 65 degrees, as it was found that at acidities in excess of this figure the results were too erratic to have any practical value.

It will be noted that between 17 and 60 degrees acidity, a straight line could be drawn through the points, which would have a slope virtually the same as the dotted straight line of equation 1. Beyond 60 degrees the graph curves down rather steeply. Between 14 and 70 degrees, a straight line could also be drawn through the points without serious error, and by substituting 14 degrees instead of 20 for A prime in equation 1, and giving t a value of 0·545° C. and solving for C, a value of 0·010 is obtained.

With but one or two exceptions, all the above results were obtained from samples drawn from the cows received from farmers milking mixed herds, selected

after the usual analytical tests indicated that they were normal unadulterated milks. For purposes of comparison, several samples were taken from a herd of pedigree Jersey cows, and were found to behave in the same way as the other samples. Although it is an undoubted fact that some milks give abnormal results

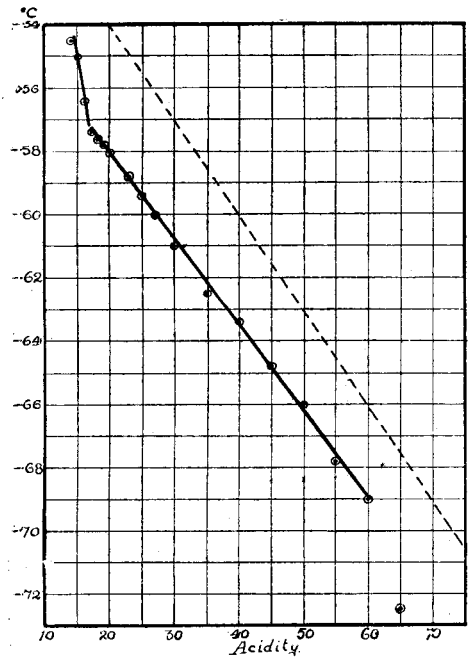

Fig. 1.

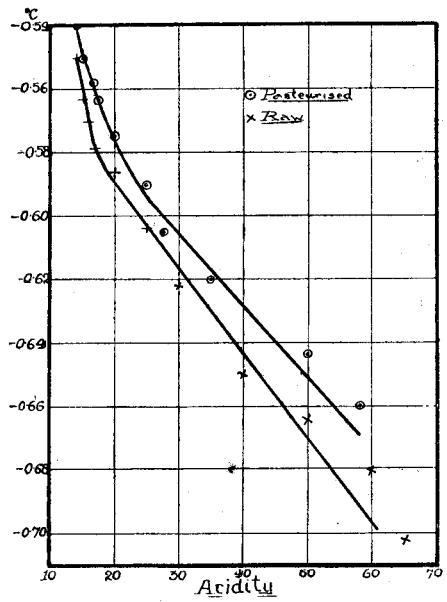

Fig. 2.

when subjected to the cryoscopic test, no such milks were encountered during the course of this investigation (this being due in all probability to the fact that the bulk of the work was on the mixed milks from herds of cows), although abnormal milks have occasionally been found in routine testing in this laboratory.

Having found in previous experiments that pasteurisation affected the freezing point, a series of experiments was made on milks before and after pasteurisation. A sample was drawn from the receiving vat of a local dairy factory, and a further sample was taken after pasteurisation, both samples being tested as previously described. The mean results of several determinations are shown graphically in Fig. 2. As a check on the results, a sample was drawn from the vat, taken to the laboratory, divided, and one portion pasteurised in a model pasteuriser made in the laboratory to imitate as closely as possible a "Cherry" pasteuriser, the heating coil being a glass spiral. The sample was heated rapidly by steam to 145° F., maintained at this temperature for 30 minutes, and cooled rapidly with cold brine to a temperature of 50° F. The raw and pasteurised milks were then tested. The results confirmed our previous experiments.

The effect of pasteurisation was to raise the freezing point by 0·01° C., which in some cases would indicate "added water" in pure milk. The *A.O.A.C. Official Methods* (2nd Edition, p. 269), states that a tolerance of 3 per cent. of added water may be allowed on milk showing not more than 3 per cent. of added water, which is equivalent to a depression of 0·017° C.

Experiments were then made on milks to which distilled water was added, so as to give final products containing percentages of added water ranging from 2·5 per cent. to 40 per cent., and the same series of acidity and cryoscopic tests were repeated. In Fig. 3 the mean results are shown graphically for milks containing 5, 10, 20, and 40 per cent. of added water. It will be noticed that the

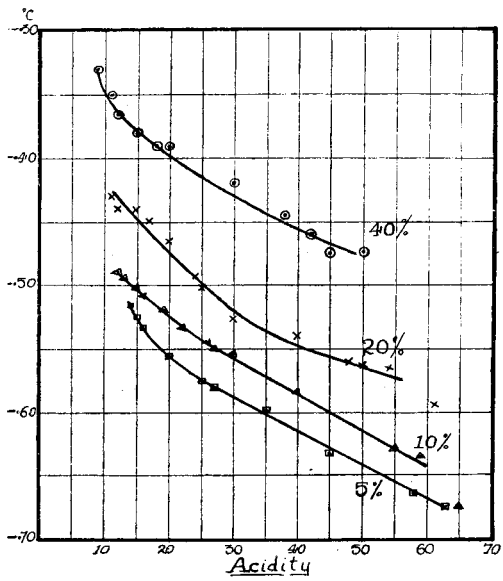

Fig. 3.

addition of the water lowers the original acidity of the fresh milk to a degree agreeing with calculated values. This is a factor which must be considered when applying a "correction" for acidity to samples which contain added water. Taking the 40 per cent. of added water curve as an example, it can be seen that the acidity of the first milk at a value of, say, 14 degrees, had been reduced by the dilution with water to an acidity of 9 degrees, and accordingly, any correction, to be of value, must be led back to this point. The error caused by calculating back to an acidity of 20 degrees would amount to approximately 11 per cent. of added water less than that which is actually present, which is a considerable error. Moreover, in our experiments, we failed to get results as consistent and reproducible with the high dilutions as with the low dilutions and with normal undiluted milks. Again, taking the 40 per cent., we found that results became most erratic when an acidity in excess of 50 degrees was reached, and this same feature was noted in all the diluted milks, becoming progressively less as the degree of dilution diminished.

The variations in the shapes of the different curves are to be noticed, the 10 per cent. curve being almost a straight line. The 5 per cent. curve shows some resemblance to the curve for normal milk, and could probably be drawn as two straight lines crossing at a point with co-ordinates approximately at 23 : 0·57°.

Auckland, New Zealand, being situated in a sub-tropical zone, the various experiments were carried out at times when the laboratory temperature was approximately 60° F., thus eliminating changes due to abnormal temperatures.

It might be mentioned that in the Auckland Province, dairy cattle remain outdoors in the grass pastures all the year round. During the winter, some of the cattle are provided with a wool-lined, waterproof rug.

SUMMARY.—Determinations of the variations of the freezing points of milk with increasing acidities have been made on a number of samples, both unadulterated and containing definite amounts of added water.

The value of 0·20 per cent. of acidity, given by the "Connecticut Agricultural Experiment Station, 27th Report on Food Products (1922)," as the normal acidity of fresh milk is criticised, and a value of 0·14 per cent. is suggested as being nearer the truth.

The correction factor of 0·003° C. for each 0·01 per cent. excess acidity is shown to hold between acidities of 0·17 per cent. and 0·60 per cent., and a value of 0·010° C. has been suggested for acidities ranging from 0·14 to 0·17 per cent. of lactic acid.

Results with milks containing added water are tabulated, which tend to show that when the cryoscopic method is used for the determination of added water in milk, it can be applied with accuracy only when the samples are quite fresh.

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DISCUSSION.

The PRESIDENT stated that this paper had come at a very opportune moment, when there was renewed interest in the analysis of milk and in methods of analysis which were not ordinarily applied, and there were signs that these would shortly be of increased importance. As far as this paper was concerned, he could not help regretting that fuller chemical analyses had not been made of some of the peculiar milks; it would be of importance and extreme interest to know the protein, lactose and ash contents, so that the relation of these factors to the freezing point factors could be studied. It would greatly increase the value of the paper if these details were given. He was surprised by the extraordinary constancy of the freezing point. So far as he had studied it, it always seemed that the freezing point, although the most constant property, was not nearly so constant as these results showed.

Dr. MONIER-WILLIAMS said that this was a most valuable paper. He could confirm the constancy of the freezing point of milk by work which he did in 1912 on a large number of samples, some of which were supplied by Capt. Golding, although his freezing points were rather higher than those given, *i.e.* —0·530° instead of

$-0{\cdot}550°$. However, he noticed that the author's remarks as to the relative and not the absolute accuracy of the figures given rather disarmed criticism on this point. The real question was whether this method could be used in this country under the present system of administration. It appeared to be necessary that the milk should be "fresh," and generally it was not at all fresh when it arrived in the hands of the Public Analyst. This difficulty would be accentuated in the case of the third sample retained for eventual examination by the Government Chemist.

On the other hand, the constancy of the freezing point of milk appeared to him to open up considerable possibilities in the chemical analysis of milk. The freezing point was a measure of the osmotic pressure, and this depended on the relative proportions of the soluble constituents, chiefly lactose, chlorides, phosphates and citrates. In 1914 Mathieu and Ferré suggested a lactose-chlorine number for milk which they calculated from the percentages of lactose and chlorine, and which, they claimed, never fell below 74 for genuine milk. It might be possible by taking into consideration not only lactose and chlorine, but other soluble constituents, such as phosphates and citrates, to arrive at an expression which might show a degree of constancy of much the same order as that of the freezing point. The President, in his address to the Annual General Meeting, had made some very stimulating references to the need for research. Was not this problem an eminently suitable one for research? We knew really very little about the composition of milk. The analytical basis of the legal limit, depending upon the proportion of non-fatty solids, was admittedly crude. This figure represented the sum of a number of constituents which showed wide variation among themselves. The mutual variation of these different constituents must, however, be controlled by some natural law, or the osmotic pressure would not remain constant. It was for milk chemists to ascertain whether the results of chemical analysis could not be handled in such a way as to bring them more into line with the data afforded by the freezing point. The freezing point had been used on the Continent for the detection of added water for over thirty years, and the only attempt to apply the knowledge thus gained to chemical analysis was that of Mathieu and Ferré. He thought this was a matter which might well engage the attention of chemists in the future, and that such work might be of great value administratively. Later in the discussion Dr. Monier-Williams suggested that it might be worth while to ascertain the effect on the freezing point of adding thymol to milk, with the object of preventing the development of acidity.

Mr. H. T. CRANFIELD thought that this was certainly one of the most important papers concerning milk which the Society had received, in view of the fact that the problem of distinguishing between abnormal samples of milk and those containing added water was one which, in the near future, would demand very serious consideration. Dairy chemists, particularly those in direct touch with farmers, were brought right up against this problem repeatedly, and he thought that the method under discussion was one which showed promise of getting to the bottom of this difficulty. He agreed with the points that Dr. Monier Williams had brought forward, and, relative to these, he wished to state that in recent years determinations of the soluble ash in a number of these abnormal milks had been made in his laboratory. In the majority of cases, he had found that there was a strong negative correlation between the lactose and soluble ash percentages, but isolated samples gave figures which fell away considerably from the normal curve. One case under observation, that of an abnormal cow which produced milk of exceedingly poor quality, low in solids-not-fat, had yielded many interesting data. The results of the analysis of 250 samples of milk from this cow indicated that this correlation of lactose and soluble ash was, on the whole, quite good, but here again several samples did not follow the normal correlation curve. It was the presence of such samples which constituted the problem requiring solution.

He would like to suggest that if the Society were of the opinion that the "depression of freezing point" method was a promising one relative to our present difficulties, there was room for a very thorough investigation of the method in this country, but such an investigation would have to be on a large scale, dealing with thousands of samples. Personally he would warmly welcome the initiation of such a scheme.

The PRESIDENT here stated that with regard to his reference to abnormal samples he referred to those low in solids-not-fat content rather than those high in this respect.

Captain GOLDING said that he felt very strongly on this subject, and was greatly indebted to the Editor for asking him to read the paper. Since he had seen the paper he had made some tests by the method, and had compared it with the Hortvet test, which was official in America, and he was much impressed by the ease of the test. He suggested that instead of a Beckmann thermometer (with which, as the author stated, the zero point needed checking before and after each set of determinations) a Hortvet thermometer should be used. This was a very fine thermometer on which each degree was twice the length of those on the Beckmann thermometer, and there was no trouble in adjusting it. With regard to the point Dr. Monier- Williams had raised as to determining the other constituents of milk, it seemed to him that it would be comparatively laborious for the Public Analyst to make all those determinations, and, even then, there would be the question of decomposition of lactose, etc., whereas the freezing point was a fundamental and constant property of the milk. He had been very much impressed on reading the new book by Rogers to note the stress laid on the importance of this constant in fresh milk. Accurate determination of the freezing point, he said, would show whether there was added water, and surely if our methods for testing milk did not fit in with the present existing organisation, could not we alter the organisation? It seemed to him that it would be a very great help if there could be mobile laboratories where samples could be received, tested for freezing point, and fat determined by the Gerber method, and then the doubtful samples could be sent to the laboratory, where they could be fully analysed. This would mean that there would be no further cases of prosecutions of farmers who were unfortunate enough to possess cows which gave milk containing solids-not-fat below the average, and it would also detect the adding of water to rich milk. With reference to Dr. Monier-Williams' suggestion regarding thymol, Captain Golding thought it would be very interesting to investigate this point.

Mr. JEPHCOTT stated that he had the good fortune, when in New Zealand, to discuss this test with the author, and although he could see objections to the test from the point of view of the Public Analyst, it was of very great use in testing supplies of milk brought into the factories (where it was received approximately 2 hours after milking). He had never known it to fail, and in his own experience it had proved invaluable for detecting added water, often proving that quite a considerable amount of added water was occurring in milk which, considered from the point of view of fat and solids-not-fat, was satisfactory. Presence of added water was in every case confirmed at the farm.

The Use of Mixed Bromides in Place of Chlorides in the Determination of Alkalis.

By E. SPENCER, Ph.D., F.I.C., A.R.S.M., and
K. B. SEN, M.Sc., A.I.C.

Introduction.—It must have occurred to many analysts accustomed to routine determinations of potassium and sodium in rocks and refractory substances by the method of Lawrence Smith (*Amer. J. Sci.*, 1871, **50**, 269; *Chem. News*, 1871, **23**, 222) and that of Berzelius (*Pogg. Ann.*, 1824, **1**, 169), that the weighing of the two metals in the form of chlorides and the subsequent determination of sodium by difference, represents a weak link in these two methods. An error in the weight of the mixed chlorides, or in the potassium determination, involves an error in the sodium figure.

Some time ago, while carrying out a number of alkali determinations on felspar by the two methods above mentioned, it occurred to one of the authors that the substitution of ammonium bromide for ammonium chloride, and of hydrobromic acid for hydrochloric acid, in these determinations might lead to greater accuracy in the method, through yielding an increased weight of mixed halides.

Experiments were therefore made with the Lawrence Smith method thus modified. It was found that the rock decomposition could be effected at least as thoroughly with the bromide as with the chloride, and the resulting mixed alkali bromides were also found to be purer, less fusible, and less volatile than the corresponding chlorides. The weight of mixed alkali bromides is, of course, greater than that of the chlorides, by an amount depending on the proportion of potassium to sodium present, the increase varying from about 50 to 100 per cent.

It might be mentioned here that experiments were also carried out on the use of ammonium iodide and hydriodic acid as alternative halides, but, owing to the relative instability of hydriodic acid and its salts, consistent results could not be obtained, and these attempts were abandoned.

Modified Lawrence Smith Method.—A pure aqueous solution of hydrobromic acid was first prepared by distillation from potassium bromide and sulphuric acid. Similarly, pure ammonium bromide was prepared from hydrobromic acid and ammonia, the product being recrystallised. For the Lawrence Smith method 0·5 grm. of the dry ammonium bromide was intimately mixed with 0·5 grm. of the finely powdered mineral (as in the chloride method), and the mixture was then thoroughly incorporated with 3 grms. of precipitated calcium carbonate. About 0·5 grm. of calcium carbonate was placed in the platinum thimble, the assay mixture then transferred to the thimble, and another 0·5 grm. of carbonate placed on the top. The mixture was then heated in the usual way, after which the mass was lixiviated, as in the ordinary method, and the residue was treated with hydrobromic acid and found to dissolve completely.

The combined aqueous extracts were evaporated to a convenient volume, the excess of calcium precipitated with ammonia and ammonium carbonate, the filtrate evaporated to dryness, and ammonium salts removed by gentle heating. The residue was taken up in a little water, the last traces of calcium precipitated with a few drops of ammonium oxalate solution, and the precipitate filtered off. The filtrate was collected in a platinum dish and evaporated to dryness, the residue moistened with a little hydrobromic acid and again evaporated to dryness, and then heated to below a dull red heat for some time to drive off traces of ammonium bromide.

The resulting mixed alkali bromides were found to be whiter than the corresponding chlorides, owing, apparently, to the oxidising action of the hydrobromic acid or the ammonium bromide on the traces of organic matter usually present at this stage. The mixed bromides also appear to be less fusible than the chlorides.

After the mixed bromides had been weighed they were taken up in a little water and treated with 10 c.c. of perchloric acid, and the liquid was evaporated on the water bath until it fumed, diluted, and, after the addition of a further 2 c.c. of perchloric acid, again evaporated until fumes appeared. During the first evaporation the mass turned yellowish-brown, owing to the evolution of hydrobromic acid and its interaction with the perchloric acid. The second evaporation left the residue practically colourless. It was then cooled and taken up with about 30 c.c. of 97 per cent. ethyl alcohol, and the insoluble potassium perchlorate filtered off and weighed as in the ordinary perchlorate method.

Modified Berzelius Method.—The success of this modification of the Lawrence Smith method suggested the application of the mixed bromide modification to the Berzelius method. In this method the alkalis are dissolved by means of sulphuric acid and hydrofluoric acid. After complete solution the liquid is heated to fuming to drive off free hydrofluoric acid, then cooled, the residue taken up in about 150 c.c. of hot water, and a slight excess of ammonia added to the solution to precipitate iron oxide and alumina. The precipitate is filtered off and well washed (if at all bulky it is reprecipitated). The combined filtrates are evaporated to dryness, and the residue heated below a dull red heat for some time to drive off the excess of ammonium sulphate.

The residue consists mainly of alkali sulphates, which in the Berzelius method are converted into the chlorides by adding a slight excess of barium chloride.

At this stage barium bromide was substituted for barium chloride, and, after the addition of a slight excess, the resulting barium sulphate was filtered off and well washed. The filtrates and washings were concentrated by evaporation, and the excess of barium (together with any calcium) precipitated with ammonia and ammonium carbonate. The filtrate and washings were evaporated to dryness in a platinum dish, the residue heated below a dull red heat to drive off the excess of ammonium bromide, cooled, again taken up in a little water, and any traces of barium or calcium precipitated with a little ammonia and ammonium oxalate and filtered off. The filtrate was finally evaporated to dryness in the platinum dish,

the residue moistened with a little hydrobromic acid, again evaporated to dryness, and heated to remove the last traces of ammonium salts. The mixed sodium and potassium bromides were then weighed and treated with perchloric acid, as in the Lawrence Smith method.*

DETERMINATION OF POTASSIUM AS PLATINIBROMIDE.—In using the above alternative method of weighing the alkalis as mixed bromides, one sacrifices the possibility of determining the potassium by the platinic chloride method. If, however, the alkali platinibromides show the same relative difference of solubilities that the platinichlorides do, it should be possible to determine the potassium in the mixed alkali bromides by the use of platinic bromide.

In order to examine this possibility platinic bromide was prepared (with difficulty) by dissolving precipitated platinum in hydrobromic acid and evaporating the solution to a syrup. The solubilities of sodium and potassium platinibromides were then determined in alcoholic solutions of various strengths, and it was found that ethyl alcohol of 90 per cent. strength gave a complete separation of the potassium and sodium salts. The following experimental tests with known weights of mixed alkali bromides illustrate the efficacy of the separation. The potassium platinibromide precipitate contains about 10 per cent. of potassium, as against about 16 per cent. in the platinichloride.

	Potassium bromide taken. Grm.	Sodium bromide taken. Grm.	Potassium oxide (calc.). Grm.	Potassium oxide found. Grm.
1.	0·005	0·0049	0·002	0·0029
2.	0·010	0·0049	0·0039	0·0039
3.	0·0250	0·0243	0·0099	0·0099
4.	0·0300	0·0292	0·0118	0·0120
5.	0·0350	0·0340	0·0138	0·0139
6.	0·0400	0·0389	0·0158	0·0159

CONCORDANCE OF RESULTS.—The following results of potassium and sodium determinations on rock substances of varying alkali content indicate the degree of concordance obtainable in the modified processes described in this paper:

METHOD.

Material.	Lawrence Smith method with chlorides and potassium platinic chloride.		Lawrence Smith method with bromides and potassium perchlorate.	Berzelius method with bromides and potassium platinic bromide.
	Sodium oxide. Per Cent.	Potassium oxide. Per Cent.	Potassium oxide. Per Cent.	Potassium oxide. Per Cent.
Moonstone felspar ..	4·75	9·59	9·61	9·68
Clinker	0·62	0·49	0·48	0·49
Fireclay	0·40	1·61	1·58	1·65

* The barium bromide for this experiment was prepared by dissolving pure baryta in a slight excess of hydrobromic acid solution.

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.

SANIO'S POTASSIUM DICHROMATE TEST FOR TANNINS.

O. HENRY (*J. prakt. Chem.*, 1834, **3**, 7) was the first to suggest the use of potassium dichromate as a precipitant for tannins, especially gallotannin. His observations were confirmed by Wackenroder (quoted in Dekker, *Die Gerbstoffe*, 1913, p. 263), with the result that this reagent was accepted by Sanio (*Botan. Ztg.*, 1863, p. 17) as a specific test for tannins, and has since been extensively used in plant physiology, although it has been pointed out by Schroeder (*Jahr. Wiss. Bot.*, 1868, **7**, 261), and by Drabble and Nierenstein (*Biochem. J.*, 1906, **2**, 97), that gallic acid is also precipitated by potassium dichromate. In view of this, I have, at the suggestion of Dr. Nierenstein, investigated the matter, and have obtained the following results:

In accordance with Sanio's directions, saturated potassium dichromate was used. One c.c. of this solution, added to twenty-four different substances, in approximately 1 per cent. aqueous solution, precipitated gallic acid, gallotannin, pyrogallol, phloroglucinol, maclurin, cinchonine sulphate, and the hydrochlorides of berberine, quinine, strychnine, papaverine, narcotine and narceine. The remaining twelve substances, namely, β-resorcylic acid, veratric acid, salicylic acid, vanillic acid, oxalic acid, phenol, quercetin, rhamnetin, and the hydrochlorides of betaine, caffeine and pilocarpine, gave no precipitate.

In the case of the alkaloids, the precipitates obtained might possibly have been unchanged alkaloid hydrochlorides. The precipitate from berberine hydrochloride was therefore dried and ashed, when a distinct chromium ash was obtained.

It is necessary to point out that, although relatively concentrated solutions, compared with those existing in the plant, were used in this investigation, this is experimentally compensated for by the facts that precipitation occurs very much more easily on the cell wall than *in vitro*, and that plant precipitates are microscopical, compared with those obtained under laboratory conditions.

From these results, therefore, it is evident that no reliance whatever can be placed on the Sanio test for tannins.

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THE SOLUBILITY OF ANTIMONY IN WATER.

THE recent paper by S. G. Clarke (ANALYST, 1929, 99) sheds useful light on a subject in which I have been interested for some time past. The object of my experiments was to prepare a number of cathodic deposits by the electrolysis of certain neutral and alkaline solutions with an antimony cathode and platinum anode under varying conditions of electrolysis, and to compare their properties with those of the supposed hydride of antimony (Sb_2H_2) described by Weeks and

Druce (*J. Chem. Soc.*, 1925, **127**, 1069). Such deposits are obtained under certain conditions as a finely divided black granular powder, adhering to the cathode, floating in the solution or settled on the bottom of the electrolysis vessel. As my experiments have shown (*ibid.*, 1928, 1987), the deposits obtained were in all cases metallic antimony containing a trace (about 0·1 per cent.) of hydrogen, and not antimony hydride, but the interesting point is that the principal property by which they could be distinguished from the hydride was their solubility in distilled water.

For example, when it was necessary to wash the deposits free from electrolyte with hot distilled water,for the purposes of analysis, a diminution in bulk of the deposit was visible to the naked eye. Even when the deposit was washed rapidly on a small Buchner funnel with cold distilled water, the washings were found to give a substantial precipitate with hydrogen sulphide.

As a result of further experiments it was found that solution of the antimony took place to a far less extent in the absence of air. Thus, when the electrolysis was carried out with a closed porous pot for cathode compartment in an atmosphere of hydrogen, the amount of deposit obtained was greatly increased.

Powdered antimony was found to have the same properties in this respect, though if the powder was freshly prepared the solubility in water was less marked but increased after exposure to air.

It appears therefore, that metallic antimony in a finely divided state is soluble in distilled water in the presence of oxygen owing, probably, to oxidation. Under certain conditions this may prove an appreciable source of error not only in Clarke's method, but also in other analytical operations where deposits of antimony (produced, for example, electrolytically) have to be washed. (*Cf.* Schoeller, *J. Soc. Chem. Ind.*, 1913, **32**, 260.)

JULIUS GRANT.

THE DETECTION, DETERMINATION AND OXIDATION OF SULPHUR DIOXIDE.

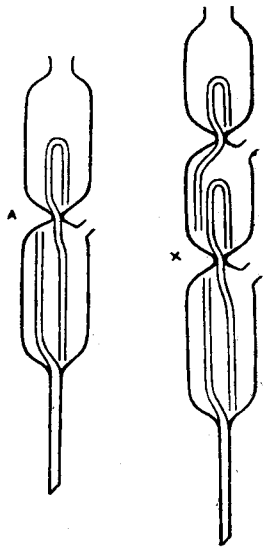
THE apparatus shown in the accompanying diagrams has been designed with the intention of combining the rapidity of the method of the Manufacturing Confectioners' Alliance and of the Food Manufacturers' Federation (ANALYST, 1928, **53**, 118) with the accuracy of the Monier-Williams method (ANALYST, 1927, **52**, 343, 515).*

Qualitative Test for Sulphur Dioxide by means of Apparatus A.—It will be seen that the apparatus consists of two bulbs, the lower one of which prevents any liquid being drawn back into the flask, and also acts as an absorption bulb, while the upper bulb contains the bulk of the liquid by which the sulphur dioxide is absorbed.

Ten ml. of hydrogen peroxide are placed in the lower bulb, two drops of bromphenol blue added, followed by $N/10$ sodium hydroxide solution until the liquid is just blue, and the vent is closed with a small rubber stopper. The apparatus is then fitted by means of a rubber stopper into the top of a reflux condenser, and a 500 ml.-flask, containing 150 ml. of air-free water, is fitted to the bottom of the condenser by means of a rubber stopper, through which passes a glass tube connected with a cylinder of carbon dioxide; the flask is supported by wire gauze. Carbon dioxide is then passed through the apparatus to expel air, and

* The apparatus was made by the Scientific Glass-blowing Co., 95, Gray's Inn Road, London, W.C.1.

most of the hydrogen peroxide is brought into the top bulb, leaving one or two ml. in the lower bulb. A weighed quantity of the sample is placed in the flask, followed by 50 ml. of a 16 per cent. (by volume) solution of hydrochloric acid, and the flask is then heated over a Bunsen flame. If a considerable quantity of sulphur dioxide is present, the indicator in the lower bulb will change to yellow before the liquid in the flask has reached the boiling point. (The indicator is not, by itself, reduced by sulphur dioxide.) In the presence of traces of sulphur dioxide the liquid in the lower bulb will show the colour change within 5 minutes from the commencement of boiling



A series of qualitative tests for the presence of sulphur dioxide may be made in a very short time by this method. If a positive result is obtained, the amount is determined by continuing as follows:—

Quantitative Test for Sulphur Dioxide by means of Apparatus A.—It was found that the sulphur dioxide was entirely absorbed by the liquid in the two bulbs.

The change of colour of the indicator in the lower bulb is followed by a colour change in the upper bulb. The liquid in the upper bulb is then titrated with $N/10$ sodium hydroxide solution as the test proceeds. When the neutral point is reached the liquids in the bulbs are mixed by manipulating the supply of carbon dioxide, and again titrated, etc., until the final neutral point is reached. The flow of water through the condenser is now stopped, and and heating continued until the neck of the condenser is hot. The liquids are then finally titrated.

To guard against any sudden evolution of gas, the neck of the bulb may be fitted with a splash-tube, which consists of a short piece of wide-bore tubing containing a small bulb and inclined at an angle. In order that the tube may be as wide as possible, a small piece of rubber tubing is used as a stopper, but with a steady flow of gas this fitting is not necessary.

The bulbs of the inner jacket of the condenser should not touch the outer jacket, as condensed water collects at these points, and the sulphur dioxide retained may not be entirely driven out when the condenser water is heated at the end of the test. Moreover, a condenser which allows a wide passage from the flask to the absorption bulbs may cause traces of chlorides to be carried through; for this reason diluted hydrochloric acid is added in the proportions previously given, to avoid any possibility of fumes being carried up to the bulbs, as might occur if strong acid were added just before distillation.

A blank test is advisable, and for this purpose distilled water containing two drops of the indicator is placed in the bulbs. (The indicator turns blue with distilled water, as the P_H figures for bromphenol blue range from 3·0 to 4·7.) Carbon dioxide is then passed through the boiling hydrochloric acid solution, and no change should take place in the indicator.

The following comparative results were obtained in test experiments, in which 10 ml. of a freshly made aqueous solution of sulphur dioxide were taken in every case, the strength of the solution being determined by titration with $N/10$ sodium hydroxide solution after oxidation with neutral peroxide. The time taken for

each test varied from about 20 to 30 minutes, including the time taken to heat the liquid to the boiling point.

	Sulphur dioxide added.		Sulphur dioxide found.
	By titration after oxidation with H_2O_2 $N/10$ NaOH. Ml.	By titration $N/10$ Iodine. Ml.	By titration $N/10$ NaOH. Ml.
Apparatus *A*	10·40	10·45	10·35
	10·20	10·15	10·20
	15·0	14·95	14·80
	13·05	13·05	12·90
	12·95	12·85	12·70
Apparatus *X*	13·05	—	12·6
	13·3	—	12·9
Method of *Monier-Williams**	31·9	31·7	31·2
	14·8	14·4	14·3
	10·3	10·4	9·9
	6·3	6·4	6·1

* These figures are calculated from mgrms. of sulphur dioxide shown in the table on page 44 of his Report.

It will be seen that the use of the apparatus *A* affords a rapid qualitative and quantitative method of determining sulphur dioxide.

Quantitative Determination by means of Apparatus X.—This apparatus was designed with an extra bulb, so as to make certain that no sulphur dioxide could escape.

Fifteen ml. of neutralised hydrogen peroxide are placed in the lower bulb, and the vents in the two bulbs closed with rubber stoppers. The length of the tube in the centre bulb is such that when carbon dioxide is passed through it and the peroxide is raised into the bulbs 10 ml. will remain in the centre bulb and the remainder will pass into the upper bulb. The peroxide may be titrated during or at the end of the determination.

The Oxidation of Sulphur Dioxide.—As mentioned by Monier-Williams in his Report, Cazenave and Claassen considered the use of carbon dioxide unnecessary. Froboese stated that an atmosphere of carbon dioxide is not so important as the use of air-free water in the distilling flask, and that the carbon dioxide does not prevent oxidation, but merely assists in carrying over the sulphur dioxide. Raschig states that when titrating by running iodine into sulphurous acid the errors observed are due solely to the escape of sulphur dioxide from the liquid during titration. This was confirmed by Mason and Walsh (ANALYST, 1928, **53**, 144) using sulphite solutions, and in this case the loss, though mainly due to volatilisation, is partly due to oxidation of the *sulphite*. The use of glycerin by Brown to prevent oxidation resulted probably in checking, to a great extent, the loss by volatilisation. The loss of sulphur dioxide obtained by many chemists has been shown to be mainly due to volatilisation.

In the early experiments with the apparatus which has been described sulphite solutions were used, but the oxidation was so rapid that these were discarded and solutions of sulphur dioxide in air-free water were used. Although these solutions lost strength slowly, the loss was due to volatilisation and not to oxidation. This

was proved by means of iodine titrations made at the same time as the titration with alkali after oxidation.

A series of experimental determinations with tap water and air has shown that sulphur dioxide itself is not appreciably oxidised, at any rate during the period of the test, the determinations being quite as accurate as when air-free water and carbon dioxide were used. In a test with jam, 50 grms. of the sample (from the same quantity of which 0·5 ml. $N/10$ sodium hydroxide had been required by the Monier-Williams method 5 days previously) were placed in tap water, diluted acid added, and air blown through. 0·4 ml. of $N/10$ sodium hydroxide solution were required. Sulphur dioxide in combination with aldehydes and sugars is not readily oxidised, which fact may explain this result.

These, and other experiments, proved that the loss of sulphur dioxide is not due to oxidation but to volatilisation, and that more sulphur dioxide is lost during the mixture of the sample, weighing out, and introduction into the flask than in the actual determination. Inaccuracies in the determination of sulphur dioxide in past experiments have been due, firstly, to the interference of volatile acids, sulphur-containing substances, etc. (this interference is now prevented in the Monier-Williams method); and secondly, to the difficulties in sampling, with loss of sulphur dioxide by volatilisation and of sulphur dioxide as sulphites, etc., by oxidation.

Mr. E. Hinks (ANALYST, 1928, **53**, 128) expressed the opinion " that oxidation really took place in solution rather than in the gaseous state."

The experiments which have been described show that neither in solution nor in the gaseous state, during the time of the experiment, is sulphur dioxide (*as distinct from sulphites, etc.*) appreciably oxidised.

DOUGLAS HENVILLE.

STEPNEY BOROUGH LABORATORY,
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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 SOMERSET.

ANNUAL REPORT OF THE COUNTY ANALYST AND BACTERIOLOGIST FOR 1928.

THE total number of samples examined was 11,652, of which 1072 were taken under the Sale of Food and Drugs Acts. Of the 1043 samples submitted by the police, 1010 were genuine, 8 suspicious, and 25 adulterated.

MILK SAMPLES EXAMINED FOR TUBERCLE BACILLI.—Of 391 samples examined, 26 were found to contain tubercle bacilli. Systematic examination of the milk from the herds of the County have been made, and, as before, about 2 per cent have been found to contain tubercle bacilli. When a tuberculous herd has been found, the infecting cow or cows are tracked down, veterinary surgeons send samples from suspected cows, and the herd is sometimes sampled in small groups.

During the year six herds, A, B, C, D, E, and F were found in this laboratory to give tuberculous milk, and in the Bristol Laboratory one herd, G. Five cows, and a small group of 4 cows (under investigation), giving tuberculous milk, were discovered—one in herd A, one in herd B, two in herd C, and two in the herd G supplying Bristol. One infecting cow, which had gone dry, was discovered by the veterinary surgeon in herd D. In herds E and F no infecting cow was found, but further investigations are being made. In five out of the six herds veterinary inspection was insufficient to discover the infecting cow. Apart from the examination of milk from herds, animals are inspected by veterinary surgeons and samples of milk are sent under the provisions of the Diseases of Animals Act.

DENYS R. WOOD.

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.

STANDARD FOR WATER IN MARGARINE.

ON January 31st a grocer was summoned at Southampton for selling, to the prejudice of the purchaser, margarine containing an excess of moisture.

On analysis, the sample was found to contain: Water, 17·43; fat, 80·10; salt, 2·34; and curd, 0·13 per cent.

The solicitor for the prosecution explained that there was no legal standard for water in margarine when sold, although there was a standard under the Butter and Margarine Act, 1907. Sec. 4 of the Sale of Food and Drugs Act stated that if any margarine prepared for sale or consignment contained more than 16 per cent. of water, and was present in any margarine factory, it should be an offence, but there was no offence under the Act for selling the article, so that the prosecution had to fall back upon the Act of 1875, which made it an offence to sell any article to the prejudice of the purchaser. The authority was the case of *Burton* v. *Mattison*, in which it was held that the sale of margarine containing 21 per cent. of moisture was an offence under Sec. 6 of the Food and Drugs Act.

For the defence it was urged that the excess of moisture was due to the margarine being beaten with wet beaters, but that, as the beating took place after the margarine was weighed, the customer obtained full weight. That, however, was not the point, for the question was, what amount of moisture was present. Although there was no fixed standard, this was not relied upon by the defence, because prior to the Butter and Margarine Act of 1907 the Magistrates had made their own standard. The defendant had bought the margarine under a warranty, which required him to sell the article as received, but as it was sliced off the bulk and beaten on the block, it was not sold as received, and therefore defendant did not rely on the warranty.

A fine of 10s. was imposed.

Department of Scientific and Industrial Research.

FUEL RESEARCH. Technical Paper No. 21.

THE ASSAY OF COAL FOR CARBONISATION PURPOSES (PART II).*

CONTINUED experience with the Gray–King apparatus, designed to obtain reliable data as to the suitability of coal for carbonisation, has shown its value, but for strongly swelling coals it is necessary to mix the air-dried coal with air-dried coke or electrode carbon, so that the swelling of 20 grms. of the mixture does not fill the cross-section of the retort tube, and the coal is thus not projected beyond the zone of uniform heating. Correlation of the assay method with low temperature carbonisation on a larger scale may be made to give information as to the yield of products (tar, condensible spirit and gas) and caking power. Many experimental data are given, and two plates showing different assay cokes produced from various types of coals.

Appendix I is reprinted from Methods of Analysis of Coal. Physical and Chemical Survey of the National Coal Resources No. 7 (ANALYST, 1927, **52**, 594), and describes the Gray–King Assay of Coal.

Appendix II comprises notes on manipulation. The furnace should be so wound and lagged that the temperature gradient towards the end is not too great, and so that the centre 6 in. (at least) of the tube is at uniform temperature. It is most important that the layer of coal should be of uniform depth and 6 in. in length. Acetone is suitable for removing the film of tar from the end of the retort tube, but the tube should be so clamped that no solvent reaches the coke, and vapour should be gently blown out. A small wad of asbestos wool should be used with coals showing a tendency to form a tar fog. In order to determine the composition and density of the gas, duplicate experiments may be run and the gases from the second swept backwards through the absorption train to remove air, or, alternatively, the amount of oxygen may be determined and a correction made. To obviate the difficulty of gas of different composition in the train and holder, a gas reservoir may be attached and the gas mixed by circulation. The effect of excluding the gas in the train is an increase of 0·02 on a density of 0·64, *i.e.* 0·25 per cent. of the coal. A distillation apparatus for determining the water of distillation is illustrated; it gives results accurate to within 0·02 c.c. In the standard assay, coal dried at 105° C. is used, and the water is therefore water of distillation, less that carried forward in the gas, and the latter is negligible with condenser water at 15° C.; but when air-dried coal is used the water collected is the sum of water of distillation, and the moisture, as determined by drying at 105° C. Uncondensed liquid hydrocarbons may be determined by absorption in activated charcoal or condensation in liquid air after passing the gases through a drying tube and removing carbon dioxide.

Appendix III deals with large scale Low Temperature Assay. A diagram is given of the apparatus. A cylindrical retort (15 in. long by 4 in. internal diameter) with an inset thermometer, has a cover with a baffle plate, 8 in. within

* By J. G. King, C. Tasker, and L. G. Edgcombe. H.M. Stationery Office, Adastral House, Kingsway, W.C.2. Price 1s. net.

the retort, with a 1 in. off-take pipe inclined at 45° and fitted with an iron condenser 3 in. long, cooling sufficiently to allow of a rubber joint from the off-take pipe to the first condenser. This is connected with a second condenser by a side tube, and at its lower end with a seal pot. The second condenser is packed with contact rings and has a side manometer and loose plugs of asbestos. Ammonia is removed by 2 wash bottles charged with 40 per cent. sulphuric acid, and a glass scrubber removes light spirit from the gas. The coal is crushed to pass a 10-mesh I.M.M. sieve and air dried, and the condensing system with seal pot are weighed, and about 50 grm. of water weighed into the seal pot. Five hundred grms. of coal are weighed, and the retort placed in the furnace previously heated to 500° C. The temperature of the coal is raised to 300° C. in 20 minutes, and from 300°–600° in 1 hour, and kept at this temperature for 40 minutes. The pressure in the manometer is maintained at level gauge by adjusting the weights in the gas holder. Preliminary separation of the tar and liquor can be effected during carbonisation by changing the receiver to the overflow of the seal pot. After draining for 30 minutes, the condensing system is weighed and the increase counted as tar. The tar and liquor are weighed, separated, and the liquor in the wet tar obtained by distillation. When cold, the coke is removed and weighed. Typical results are given for Dalton Main Coal.

Appendix IV is a reprint of Gray–King assays of a range of coals.

D. G. H.

MANUFACTURE, USE AND STORAGE OF CELLULOSE SOLUTIONS.*

THE principal liquids used in the manufacture of cellulose solutions are: (I) Fatty esters, such as amyl, butyl, propyl and ethyl acetates; (II) Higher esters of the corresponding alcohols, such as amyl tartrate and ethyl lactate; (III) True ketones, particularly acetone, and mixed ketones of which methyl ethyl ketone is typical; (IV) Methylated spirit, wood spirit, variable mixtures of the higher homologues of ethyl alcohol known as fusel oil, and butyl alcohol; (V) Coal tar hydrocarbons, principally benzene (90 per cent. benzol) and its homologues; (VI) Dibutyl phthalate, diamyl phthalate and tricresyl phosphate; (VII) Flexible oils, principally castor, rape-seed and linseed oils.

The most valuable solvents are amyl, butyl and ethyl acetate, and particularly ethyl lactate, but owing to high price these are diluted with cheaper ones. Usually the makers supply thick solutions and a stock of mixed solvents as thinnings. As the solvent is the vehicle for conveyance of the solids to the surfaces to be treated, drying takes place by evaporation of those solvents, which gives rise to special dangers.

The recommendations of the Report are mainly concerned with the importance of ventilation and avoidance of fire. It is suggested that all articles should be treated in cabinets or enclosures of fire-resisting construction, enclosed on 3 sides, with properly designed mechanical ventilating appliances, such that the inward air velocity through the working opening of the cabinet should be at least 75 linear feet per minute, or in a room the air should be renewed at least 30 times an hour. Direct fan discharge should be used and ducts avoided. All parts where residues may accumulate should be frequently cleaned with non-ferrous implements. No flame or other agency capable of igniting the mixtures of air and vapour should be

* Factory Dept., Home Office. Form 826. Obtainable at H.M. Stationery Office, Adastral House, Kingsway, W.C.2. Price 3d. net.

permitted in or near the workrooms, particular care being taken of all electric installations, with bonding to earth of all metal pipe lines, metal parts of mixers, etc. Storing of bulk solutions needs great care, and where the flash point is below 73° F. a license under the Petroleum Act is necessary. No stocks beyond the day's requirements should be kept in the work rooms, and these, as far as possible, in metal cupboards. Floors should be impermeable to vapours. Adequate means of escape from the buildings is required, with appliances for fighting fires, and in all cases of making and adapting premises the advice of the District Factory Inspector should be sought.

D. G. H.

Ministry of Health.

BACTERIOLOGICAL TESTS FOR GRADED MILK.*

STANDARDS.

1. The following bacteriological standards for the various classes of graded milk are prescribed by the Milk (Special Designations) Order, 1923:—

CERTIFIED MILK AND GRADE A. MILK PASTEURISED.	The milk must not contain more than 30,000 organisms per c.c. and must not contain coliform bacillus in 1/10 c.c.
GRADE A. (TUBERCULIN TESTED) MILK AND GRADE A. MILK.	The milk must not contain more than 200,000 organisms per c.c. and must not contain coliform bacillus in 1/100 c.c.
PASTEURISED MILK.	The milk must not contain more than 100,000 organisms per c.c.

SAMPLING.

2. Where the milk to be sampled is contained in bottles each sample should consist of one bottle (with seal unbroken) taken anywhere between the place of bottling and the consumer. Where the milk to be sampled is not contained in bottles, samples should be taken and despatched in specially sterilised four-ounce or six-ounce bottles, each bottle being properly fastened and sealed.

3. On collection the bottles must be transferred forthwith to a carrying-case and well packed in ice, and must be kept in this condition until plated at the laboratory. (This precaution may be dispensed with only if the bacteriologist considers it unnecessary on account of the proximity of the laboratory to the place in which the samples are collected.)

4. If the plates are not made within 30 hours of the time of milking, or in the case of pasteurised milk, of the time of pasteurisation, the additional time must be stated on the report, and in any case must not exceed 12 hours.

LABORATORY TECHNIQUE.

5. In order that the results obtained by different bacteriologists engaged in the examination of official samples of graded milk may be comparable, the adoption of a strictly uniform technique is highly desirable. The technique described below has been found to be satisfactory, and should for this reason be universally adopted.

MEDIUM FOR PLATES.

6. To prepare the medium take—

1,000 c.c.	Tap water.
5 grms.	Peptone.
3 grms.	Lemco.

* Memo. 139/Foods. H.M. Stationery Office, Kingsway, W.C.2. Price 1d. net.

7. Dissolve by heat and filter hot through paper, add 15 grms. agar (best quality, clean); dissolve by heat, titrate with phenolphthalein. The reaction will usually fall between +5 and +10 on Eyre's scale, and the medium may then be used without any further adjustment of titre. If a batch does not fall within these limits, it should be brought within them by adding the minimum amount of acid or alkali.

8. Cool to 45° C., then bring to boiling point and filter through paper or absorbent cotton until clear. Eggs must not be used for clearing.

9. Distribute in flasks and sterilise for 30 minutes in 15 lb. pressure, or for 20 minutes on three successive days in the Koch steriliser.

DILUTIONS.

10. Dilutions of (*a*) $\frac{1}{10}$, (*b*) $\frac{1}{100}$ and (*c*) $\frac{1}{1000}$ should be made in bottles containing accurately measured quantities of sterile water and fitted with glass stoppers; or by some other means which makes shaking possible. The dilution should be:—

(*a*) 90 c.c. water plus 10 c.c. milk;
(*b*) 90 c.c. water plus 10 c.c. of the (*a*) dilution;
(*c*) 90 c.c. water plus 10 c.c. of the (*b*) dilution.

11. At least two pipettes are required for each sample, one for dilution (*a*), another for dilutions (*b*) and (*c*); the latter pipette should be washed out ten times in each dilution as it is made. Alternatively, a separate pipette may be used for each dilution. Straight-sided pipettes (not bulbed) should be used.

12. In making dilutions the original sample and each dilution bottle must be shaken 25 times, each shake being an up-and-down motion, with an excursion of about one foot.

In making the plate, put the required quantity of diluted milk into a sterile tube (5 in. by 1 in.) and add about 15 c.c. of melted agar cooled to 45° C.; then pour the mixture into a Petri dish (3½ ins. internal diameter). The depth of the agar in each Petri dish should be uniform.

13. Not more than half an hour should elapse between the dilution of the milk and the pouring of the plate.

14. After the agar has thoroughly hardened, incubate for 48 hours at 37° C.

COUNTING OF COLONIES.

15. If among the different dilutions there are plates containing from 30 to 300 colonies, these should all be counted, and the number, multiplied by the dilution, reported as the final count. If there are no plates within these limits, that which comes nearest to 300 should be counted. No plate that contains less than 20 colonies should be counted, unless there are no plates with a larger number. If the number of colonies on a plate is over 300, a part of the plate may be counted and the whole plate averaged.

"COLI" TESTS.

16. For Certified milk and Grade A. milk Pasteurised, three tubes, each containing 10 c.c. of bile-salt lactose peptone water,* and a Durham's fermentation tube, should be inoculated each with 1/10 c.c. of the sample under examination, and incubated at 37° C. For Grade A. (Tuberculin Tested) and Grade A. milk, three tubes should each be inoculated with 1/100 c.c. of the milk.

17. An uninoculated control tube should also be incubated.

18. The tubes should be examined for acid and gas production at the end of 48 hours. The milk is regarded as satisfactory in respect of this test if two out of the three tubes are found to be free from acid plus gas after 48 hours' incubation.

REPORTS.

19. The results of both bacteriological examinations should be recorded on a form similar to that contained in the Appendix to this Memorandum, and the report should be sent to the Ministry or the Licensing Authority immediately on the completion of the examination.

MINISTRY OF HEATLH,
WHITEHALL, S.W.1.
February, 1929.

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* This should be prepared as follows:—Five grammes each of sodium taurocholate and lactose, 20 grammes of peptone, and 1 litre of water are heated together until the solids are dissolved. The mixture is filtered and sufficient strong neutral litmus solution is added to give a distinct colour. The medium is then distributed into fermentation tubes, and sterilised by steaming for 20 minutes on three successive days.

APPENDIX.

MILK (SPECIAL DESIGNATIONS) ORDER, 1923.

REPORT OF BACTERIOLOGICAL EXAMINATION OF.....*MILK.

Name and Address of Producer:
Date and Time of Production:
Name and Address of Dealer:
Date and Time of arrival at Dealer's Premises:
Place where sample taken:
Date and Time sample taken:
Quantity of sample.
Date and Time of { delivery at / dispatch to } Laboratory:
Signature of Inspector obtaining sample:

BACTERIOLOGIST'S REPORT.

No. of sample:
Age of sample when received.....hours.
Temperature on arrival:
Number of Bacteria per 1 c.c.:
Presence or absence of Coliform Bacillus in .....† c.c. (in each of three tubes) after 48 hours' incubation:
Tube 1.
Tube 2.
Tube 3.
REMARKS.
CONCLUSION.—I am of opinion that the sample { complied / did not comply } with the prescribed conditions.

Signature.
Name and address of Laboratory.
Date.

* Insert designation, *i.e.* "Certified," &c.
† Insert 1/10 or 1/100 according to grade of milk (*see* instructions).

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Tests for the Degree of Heating of Milk. P. Weinstein. (*Z. Unters. Lebensm.*, 1928, **56**, 457–467.)—The following are the author's methods of applying the more important tests to obtain an indication of the extent to which milk has been heated. *Schardinger's test.*—Ten c.c. of milk and 1 c.c. of a mixture of 5 c.c. of a saturated alcoholic solution of methylene blue, 5 c.c. of 40 per cent. formaldehyde solution and 190 c.c. of water, are maintained at 40 to 45° C., and the time to produce complete decolorisation noted. Raw milk requires 5 to 10 minutes.

Catalase test.—The oxygen liberated from 15 c.c. of milk and 5 c.c. of a 1 per cent. solution of hydrogen peroxide after 2 hours at 22 to 25° C. is determined, a normal value being 30 to 50 c.c. per 100 c.c. of milk. *Amylase test.*—A series of test tubes containing 10 c.c. of milk and 0·1 to 0·9 c.c. of a 1 per cent. solution of starch is heated at 37° C. for 50 to 60 minutes, cooled, and 3 c.c. of a solution of 1 grm. of iodine and 2 grms. of potassium iodide in 300 c.c. of water added. The mixtures are coagulated with 5 c.c. of 5 per cent. acetic acid, filtered, and the colours of the filtrates, which vary from yellow to violet-blue, according to the amount of unchanged starch, are compared after dilution to 100 c.c. *Skim test.*—A mixture of 48 c.c. of milk and 0·15 c.c. of a saturated alcoholic solution of alkali blue-6B is heated for 90 minutes at 45 to 50° C. in a graduated cylinder, and the height and colour of the fat layer noted after 30 minutes, at 15-minute intervals. The colour, which depends on the lactic acid present, is pale blue for raw milk. The Rothenfusser–Storch oxydase test was also used. Experiments with raw milks and milks heated under varying conditions of time and temperature showed that milk pasteurised at 85° C. for 1 minute gave no Storch reaction. After 30 minutes at 70° C., however, the reaction was positive, though no decolorisation was produced in the Schardinger test within 22 minutes. Well-sterilised milk gave a positive Storch reaction, decolorised Schardinger's reagent within 10 minutes, and had the low catalase value of 9 c.c. of oxygen per 100 c.c. Insufficiently sterilised milk had a catalase value of more than 10, and a positive amylase reaction. Milk heated at 55° C. or less had an amylase value of 0·1 to 0·5 c.c. of starch, and the catalase value of normal milk, and gave a light blue fat layer in the skim test. Mixtures of raw and heated milks gave a strongly positive amylase reaction, and a bright blue fat layer, the other properties being dependent on the proportions of the mixture. J. G.

Albuminous Compounds from the Meat of Different Animals. K. Beck and E. Casper. (*Z. Unters. Lebensm.*, 1928, **56**, 437–457.)—Striegel's method (*Chem. Ztg.*, 1917, **41**, 313) was used for the separation of glutose, a 2 per cent. solution of the sample being coagulated by heating under a reflux condenser for 5 hours, and then for a further 30 minutes after the addition of 1 per cent. of tartaric acid. The clear solution was neutralised with sodium hydroxide solution, the albumoses removed by precipitation with 10 per cent. of a saturated solution of zinc sulphate, and the glutose finally obtained by the addition of a solution of 22·5 grms. of nitrogen-free tannin and 9 grms. of acetic acid in 100 c.c. of water. Van Slyke's phosphotungstic acid method, as described by Abderhalden (*Handbuch der Biochemischen Arbeits-methoden*, 1912, p. 1011), was used for the determination of the various nitrogen compounds obtained after hydrolysis of the sample or of the glutin-tannin precipitate for 5 hours at 135° C. with 6 times the amount of 20 per cent. hydrochloric acid, the excess of acid being finally removed *in vacuo* and the solution diluted with water. The distribution of the nitrogen was then calculated from Van Slyke's formulae (*loc. cit.*), non-amino $N = (\frac{3}{4}$ arginine $N + 2/3$ histidine $N)$, and lysine $N =$ total N of the bases (arginine $N +$ histidine N). The total nitrogen

was determined by Kjeldahl's method. The results lead to the main conclusion that there is a close relationship between the distribution of nitrogen in the glutose obtained from edible gelatin and from a Liebig's meat extract, and a common origin is suggested. Examination by similar methods of the hydrolysis products of muscular fibres of the ox, calf, pig, sheep, horse, goose and cod previously freed from fat, washed, dried and powdered, gave figures which were similar in all cases, and therefore cannot be used as a guide to the origin of a particular product. Extracts of the meat of the above animals were also examined, the total nitrogen, albumoses and glutose being obtained by the above methods, while, in addition, amino-acid nitrogen was determined gasometrically by means of nitrosyl chloride, the creatinine by Folin's colorimetric method, and the total phosphorus by titration of a solution of the ash. The results, which are tabulated, show wide variations, the extract from the cod, in particular, being high in coagulable nitrogen, albumoses and glutose, but low in creatinine, phosphorus and amino-acid nitrogen. Horse and pig's flesh gave extracts low in glutose. J. G.

Determination of Traces of Iodine in Vegetables. J. F. McClendon and R. E. Remington. (*J. Amer. Chem. Soc.*, 1929, **51**, 394–399.)—Five kilos. of the fresh vegetables are ground, dried, and formed into rods about 50 mm. long and 24 mm. in diameter. These rods are placed in a steel tube fitted with a screw piston by which they are forced slowly into a combustion tube where they are burned in a current of oxygen. The other end of the combustion tube connects with a series of absorption vessels containing sodium hydroxide solution, and these in turn connect with a Cottrell precipitator and an air pump. When the combustion is completed, the ash in the combustion tube is removed, ground, extracted with water, and the solution added to the evaporated contents of the absorption vessels and precipitation vessel. The whole is then evaporated, the residue heated in a nickel boat in a Pyrex combustion tube, the ash is dissolved in sodium hydroxide solution, acidified with a mixture of phosphoric acid and sulphurous acid, boiled to expel sulphur dioxide, and rendered acid to bromphenol blue paper by the addition of a few drops of sulphuric acid. The solution is treated with a small crystal of sodium nitrite, carbon tetrachloride is added, and the liberated iodine is determined colorimetrically. An alternative method consists in igniting the sample, previously moistened with calcium lactate and sodium carbonate solution, at a temperature not exceeding 450° C. until the ash is light grey in colour, and then proceeding as described. W. P. S.

Isolation of Mesaconic Acid from Cabbage Leaves. H. W. Buston. (*Biochem. J.*, 1928, **22**, 1523–1525.)—The isolation of mesaconic acid from the products extracted from green leaves (cabbage) was mentioned by Buston and Schryver (*Biochem. J.*, 1923, **17**, 470), and a more detailed account of its discovery is now given. From 90 kilos. of fresh leaves 6·0 grms. of acid were obtained (5·0 grms. of recrystallised product from the "dicarboxylate" fraction and 1·0 grm. from the "organic phosphate" fraction). Mesaconic acid, $C_3H_4(COOH)_2$, is an unsaturated dicarboxylic acid, with a molecular weight of 130, and m.pt. of 202° C.

The 6 grms. obtained must not be regarded as the total amount present, as the methods of separation were by no means quantitative. Mesaconic acid has not previously been met with as a naturally occurring product. It may be connected with citric acid, which with loss of water gives aconitic acid, and this with loss of carbon dioxide gives citraconic acid, the optical isomer of mesaconic acid. The change from citraconic acid to mesaconic acid is not easily explained; however, mesaconic acid appears to be present as such in the leaf. P. H. P.

Isolation of Protocatechuic Acid from Pigmented Onion Scales. K. P. Link, H. R. Angell and J. C. Walker. (*J. Biol. Chem.*, 1929, **81**, 369–375.)—Protocatechuic acid (3, 4·dihydroxybenzoic acid) has been isolated from pigmented onion scales. This phenolic acid appears to be one of a group of toxic substances that enable the pigmented onions to resist the inroads of the fungus, *Colletotrichum circinans*, the organism responsible for the disease commonly known as onion smudge. It was stated by Walker (*J. Agric. Research*, 1923, **24**, 1036) that the chief factor which imparted the resistant property to the pigmented onion scales was a substance, or group of substances, either closely associated or identical with the red and yellow pigments present. Protocatechuic acid, the toxic entity which has now been isolated, is not present in the white scales. Although protocatechuic acid is widely distributed in plants as a constituent of many aromatic compounds, its occurrence in the free state has been reported only in a few cases. Its isolation from pigmented onion scales represents the third instance in which the acid has been found associated with the flavonol quercetin. The isolation of quercetin from pigmented onion scales was reported by Perkin and Hummel (*J. Chem. Soc.*, 1896, **69**, 1295). Upon alkaline fusion the pigment quercetin breaks up into phloroglucinol, oxalic acid and protocatechuic acid. The toxic action of the pure protocatechuic acid isolated, in dilutions of 1 part to 3000 parts of water, is identical with the toxic activity of the crude active aqueous extract from which it can be isolated. The toxicity of the crude aqueous extracts is, however, greater than the toxic effects that could be ascribed to the amount of protocatechuic acid isolated from a given unit of toxic extract. From the aqueous extract of 100 grms. of the dry pigmented scales approximately 0·1 grm. of the pure acid was isolated. It appears, therefore, that the quantity of protocatechuic acid isolated either represents only a fraction of the total present or suggests the alternate contingency that there are present still other phenolic substances or groups of substances to which some of the toxicity of the aqueous extract can be ascribed. A quercetin-free extract was still toxic to the fungus *Colletotrichum circinans*. P. H. P.

Fluorescence of Honey in Ultra-Violet Light. G. Orbán and J. Stitz. (*Z. Unters. Lebensm.*, 1928, **56**, 467–471.)—The 28 samples of honey examined in ultra-violet light (3,000 to 4,000 Å.) all showed luminescence, the intensity of which was dependent on the ultra-violet absorption, the colour and the thickness of the layer of honey used, and was unchanged when the honey was heated to 100° C., and then cooled to 30° C. Removal of water by evaporation produced an increase

in luminosity proportional to the increase in viscosity, and weakly caramelised honey showed a luminescence which was stronger for thin layers, but weaker for thick layers, than that of the unchanged sample. The method is of little value for distinguishing real and artificial honey, but the relation of the absorption to the powers of luminescence may sometimes serve as a guide to the origin of the sample (*cf.* Popp, ANALYST, 1926, **51**, 540). J. G.

Quantitative Determination of Oxymethylfurfural in Honey and Artificial Honey. J. Fiehe and W. Kordatzki. (*Z. Unters. Lebensm.*, 1928, **56**, 490–492.)—Fiehe's criticism of Troje's method (ANALYST, 1929, 108) is justified by comparative experiments on genuine and artificial honeys by means of (1) Troje's method of titration with an alkaline solution of iodine; (2) quantitative precipitation by phloroglucinol; (3) Lenk's method (*Z. angew. Chem.*, 1917, **30**, 49). Methods (2) and (3), only, gave reliable results, a heavy precipitate and marked reduction of the dilute alkaline copper solution being obtained, respectively, with artificial, but not with genuine honey. A solution of 100 grms. of sample was precipitated with zinc acetate and potassium ferrocyanide, and the filtered liquid extracted with ether thrice in 12 hours. The ethereal extract was well shaken with anhydrous sodium sulphate and an equal volume of petroleum spirit, and after 24 hours filtered and evaporated at a low temperature. The residue was then extracted with 20 c.c. of water, and 5 c.c. of the filtered extract used for each determination. J. G.

Composition of Californian Walnut Oil. G. S. Jamieson and R. S. McKinney. (*Oil and Fat Ind.*, 1929, **6**, 21–23.)—The chemical and physical characteristics of a sample of walnut oil, expressed in California, were found to conform to those generally accepted for European oils, with the exception of the iodine value, which was 161·7 (Wijs) and 158·5 (Hanus), whereas the usually accepted limits are 138 and 148 (Wijs). The oil contained 89·7 per cent. of unsaturated and 5·3 per cent. of saturated acids, and the composition is given as: Oleic acid, 17·6; linolic acid, 72·8; linolenic acid, 3·2; myristic acid, trace; palmitic acid, 4·6; stearic acid, 0·9; arachidic acid, trace; and unsaponifiable matter, 0·5 per cent. It should be noted that only a trace of myristic acid was found. The tables show: (1) The chemical and physical characteristics of the oil, (2) the fractional distillation of the methyl esters of the saturated acids; (3) analyses of the fractions from distillation of the methyl esters, and (4) figures for the saturated acids. D. G. H.

Reactions of Soya Bean Oil. A. Richard. (*Ann. Falsif.*, 1929, **21** (240), 579–582.)—The presence of soya bean oil may be detected in olive or arachis oils by adding 10 c.c. of the sample to 1 c.c. of nitric acid and comparing the result with those produced on adding the same amount of acid to 10 c.c. of each of the pure oils. With arachis and olive oils more or less complete solidification occurs, increasing with time, and being practically complete in 24 hours. Soya oil remains liquid and also assumes a reddish brown colour which is absent with the

other oils. The coloration deepens in proportion with the percentage of soya oil, and 10 per cent. of soya bean oil may thus be detected in arachis or olive oils.

D. G. H.

Detection of Coconut Oil and Palm Kernel Oil by means of a Test for Lauric Acid. J. Grossfeld and A. Miermeister. (*Z. Unters. Lebensm.*, 1928, **56**, 423–437.)—The test for lauric acid previously described (ANALYST, 1929, 108) is modified as follows:—The fat (100 mgrms.) is saponified with 2·5 c.c. of 0·5 *N* alcoholic potassium hydroxide solution, the alcohol removed by evaporation, and an aqueous solution of the residue heated for 5 minutes on the water-bath in the presence of 2 c.c. of a 30 per cent. aqueous solution of glycerin, and precipitated with 2 c.c. of magnesium sulphate solution (150 grms. per litre). The liquid is filtered clear while hot, and in the presence of 10 per cent. or more of lauric acid (*i.e.* 20 per cent. of coconut oil) white flocks of precipitate form overnight. In negative or doubtful cases 1 grm. of oil is saponified with 0·4 c.c. of an aqueous 50 per cent. solution of potassium hydroxide and 2 c.c. of glycerin, 200 c.c. of water and 50 c.c. of the glycerin solution added, and the boiling mixture precipitated with 10 c.c. of reagent and filtered as before. A white opalescence is produced by 2·5 per cent. of lauric acid. Finally, to detect 0·5 per cent. of acid, the filtrate is shaken with 5 c.c. of dilute hydrochloric acid and 60 c.c. of ether, and the fatty acids removed and dissolved in 2·5 c.c. of 0·5 *N* alcoholic potassium hydroxide solution. The alcohol is removed, the residue dissolved in 2 c.c. of water, and the usual procedure followed. The method is not quantitative, but gave positive results in the presence of 0·3 mgrm. of coconut oil, and is unaffected by the presence of myristic, 4 mgrms. of capric, or 30 mgrms. of nonylic acid. By fractional steam distillation and fractional crystallisation of the magnesium salt from 1 grm. of oil it was possible to detect 10 and 1 per cent. of oil, respectively, and Don's distillation method (*Z. Unters. Lebensm.*, 1908, **16**, 705) is criticised on the ground that for small concentrations of lauric or myristic acids these acids are not completely removed. Dilution, or the addition of ammonium salts or methyl alcohol assists the separation of the magnesium laurate, but glycerin is preferable. The method was also applied to palm kernel, babassu, cotton-seed and ground nut oils (including the hardened oils) and to butter fat. Two rancid cotton-seed oils and one rancid oleostearin were found to be free from lauric acid, whilst hardened arachis and cotton-seed oils were found to contain it, and a sample of the former oil free from lauric acid gave a faint positive reaction after oxidation with potassium permanganate.

J. G.

Component Glycerides of Cacao Butter. C. H. Lea. (*J. Soc. Chem. Ind.*, 1929, **48**, 41–46T.)—The molecular proportions of the component acids of cacao butter are not widely variable, and this fact, coupled with the phenomenon of even distribution of fatty acids amongst the glycerides of a seed fat, is probably the cause of the fat possessing its peculiarly useful technical qualities. Further, the greater part of the mono-oleo-glycerides consists of oleo-palmito-stearin, and this class of glyceride is probably mainly responsible for the characteristic texture

and related properties of the fat. Oxidation of the fat by potassium permanganate in acetone shows the presence of only 2·5 per cent. of fully saturated glycerides, probably mixed palmito-stearic compounds. The content of mono-oleo-di-saturated glycerides lies between 73 and 85 per cent., and that of dioleo-mono-saturated glycerides cannot exceed 24·5, or that of triolein 12·5 per cent. The molecular ratio of saturated acids linked with unsaturated in the mixed glyceride is 1·4:1, the same as in coconut and palm kernel oils, and these results are supported by a study of the mono-azelao-glycerides in the oxidation products. Probably dioleostearin is present, and in greater amounts than dioleopalmitin in the dioleo-glycerides. The amount of triolein can hardly exceed 4 per cent. The following general estimate of the composition of cacao butter is suggested: Fully-saturated glycerides (mixed palmito-stearins), 2·5; mono-oleo-disaturated glycerides, 77; dioleo-mono-saturated glycerides, 16; triolein, 4 per cent.

D. G. H.

Component Glycerides of a Mutton Tallow. G. Collin, T. P. Hilditch and C. H. Lea. (*J. Soc. Chem. Ind.*, 1929, **48**, 46–50T.)—The mixed fatty acids of the original tallow consisted of myristic 4·6; palmitic, 24·6; stearic, 30·5; oleic, 36·0; and linolic, 4·3 per cent. Complete oxidation was carried out by means of potassium permanganate in acetone; and 26 per cent. of fully saturated glycerides were found, of which the mixed fatty acids comprised: Myristic, 6·1; palmitic, 50·2, and stearic acid, 43·7 per cent., so that palmitic acid tends to accumulate in the fully saturated glycerides and stearic acid more in the mixed saturated-unsaturated part of the fat. Fractional crystallisation of the fully saturated glycerides indicated the presence of tristearin, palmitodistearin and dipalmito-stearin, but individual glycerides were not isolated in the pure condition. The mixed saturated-unsaturated glycerides were found to contain 0·9 equivalent of saturated fatty acids per equivalent of unsaturated acids, and this was arrived at (i) from the percentage of fully saturated glycerides, the mean equivalents of the latter and of the original tallow, and (ii) from the composition of the fatty acids present in the mixed saturated-unsaturated glycerides (determined by difference between that of the fully-saturated glycerides and that of the whole fat). The limiting values for the classes of glycerides present are then: Fully saturated (chiefly mixed glycerides), 26; mono-unsaturated-di-saturated, 30–52; di-unsaturated-mono-unsaturated, 44–0; and tri-unsaturated, 0–22 per cent. Examination of the acidic products of oxidation suggests that the amount of mono-unsaturated glycerides tends towards the lower figure, *i.e.* there is probably a fairly large amount of di-unsaturated glycerides and a correspondingly small percentage of triolein.

D. G. H.

Colour Reaction of Diphenylamine. L. Desvergnes. (*Ann. Chim. anal.*, 1929, **11**, 1–4.)—A solution of diphenylamine in alcohol gives the best results in producing a violet colour on addition of chlorine water, and the limit of sensibility is about 1 in 65,000. The presence of diethyldiphenylurea does not affect the colour. When testing for diphenylamine in "poudre B" it is best to extract with

ether, adding water to the filtered solution, to evaporate the ether slowly and, after cooling in ice, to add 10 c.c. of 95 per cent. ethyl alcohol, and to filter. It is necessary to add a sufficiency of chlorine water, as the violet coloration does not appear until the yellow colour has been destroyed. From 15 minutes to about 3 hours, according to the quantity of diphenylamine present, is required for the development of the colour. D. G. H.

Analysis of Spirit of Nitre. L. Van Italie, A. J. Steenhauer and A. Harmsma. (*Pharm. Weekblad*, 1929, **66**, 15–22.)—Ten c.c. of the sample, 10 c.c. of 2 *N* potassium chlorate solution, and 5 c.c. of dilute sulphuric acid are shaken for 5 minutes in a stoppered flask, and diluted to 100 c.c. Ten c.c. of this are boiled with 3 c.c. of water and 2 c.c. of ammonia till 10 c.c. of liquid remain. To the cooled solution in a stoppered flask are added 1 grm. of potassium bromide and 15 c.c. of concentrated hydrochloric acid, and after 5 minutes, 10 c.c. of a 10 per cent. solution of potassium iodide. The mixture is titrated with a 0·1 *N* solution of sodium thiosulphate, and each c.c. of *N* potassium chlorate solution, reduced according to the equation $3C_2H_5NO_2+KClO_3=KCl+3C_2H_5NO_3$, corresponds with 37·5 mgrms. of ethyl nitrite. The method gives satisfactory results when compared with the gasometric and other suggested methods. J. G.

Determination of Chloral in Syrup of Chloral. Ch. Lormand. (*J. Pharm. Chim.*, 1929, **121**, 151–153.)—Chloral may be determined in its syrup by means of an ammoniacal and alkaline solution of silver nitrate, which is reduced by the syrup, with formation of silver, and silver chloride which remains in the ammoniacal solution. For 10 grms. of syrup containing 0·5 grm. of chloral, 50 c.c. of ammonia, 4 grm. of silver nitrate and 5 grm. of potassium hydroxide are used, and after 24 hours' contact the solution is heated to drive off excess of ammonia, slightly acidified with nitric acid, diluted to about 100 c.c. and again warmed, when the silver chloride dissolved in the concentrated salt solution is precipitated. After cooling, the silver chloride is filtered off, dried and weighed, and multiplied by 0·3844, to give chloral hydrate. A sample of syrup was found to give 4·76 per cent. of chloral by the alkalimetric, and 5·0 per cent. by the gravimetric method. D. G. H.

Microchemical Reactions of Theobromine. M. Wagenaar. (*Pharm. Weekblad*, 1929, **66**, 1–5.)—Theobromine crystallises in rhombic, *d*-rotatory needles, m.pt. 329–330° C. (sublimes at 300° C.), refractive indices 1·51 (α) and 1·74 (β). It is soluble in cold water (1 in 1600), hot water (1 in 48), alcohol (1 in 1460), ether (1 in 17000), and in hot chloroform (1 in 105). Directions are given to obtain the best results with the following reagents, and the figures in brackets show limiting concentrations and smallest amounts detectable (in μ grm.), respectively, in each case:—Precipitation of theobromine (1:100 and 10), mercuric chloride (1:1000 and 5), gold chloride (1:100 and 10), silver nitrate (1:100 and 10), iodine in potassium iodide solution (1:1000 and 2), bromine in potassium bromide solution (1:1000 and 1), potassium bismuth iodide in the presence of a mineral acid (1:1000 and 2), and potasssum antimony iodide (1:1000 and 1). J. G.

Determination of Pilocarpine. P. Bourcet. (*Ann. Falsificat.*, 1929, **241**, 23–24.)—Jaborandi leaves may contain a satisfactory proportion of total alkaloids and yet only very little pilocarpine. This may be determined as follows: 25 grms. of the leaves, ground to pass a No. 30 brass sieve, are moistened with 200 c.c. of 10 per cent. sodium carbonate solution and extracted with hot benzene in a Soxhlet extractor for three hours. The cooled benzene solution is shaken immediately with successive quantities of 30, 20, 20, and 10 c.c. of 1 per cent. sulphuric acid solution, the alkaloids passing into solution as sulphates. The green solution is filtered, neutralised to Congo red by means of ammonia, and oxidised with 1 per cent. potassium permanganate solution (a stronger solution may precipitate pilocarpine permanganate) until a drop of the permanganate solution gives a pink coloration persisting for a moment. The oxidised solution, rendered alkaline by addition of excess of ammonia, is extracted with twelve quantities of chloroform, the total chloroform solution (50 to 60 c.c.) being filtered, treated with fused and powdered sodium carbonate, and exactly neutralised by dropwise addition of 1:50 nitric acid solution. The neutral solution is evaporated to dryness in a small glass basin on a water-bath, and the cold residue is treated with a slight excess of acetone, which dissolves the impurities without dissolving an appreciable amount of the pilocarpine nitrate. This is collected on a Gooch crucible, dried below 100° C. and weighed. It forms a white, crystalline powder, and should melt at 174–175° C.; a melting point below 165° C. indicates unsuitability of the jaborandi for the preparation of pilocarpine.

It has been noticed, especially since the war, that certain samples of jaborandi show a satisfactory pilocarpine content if analysed as described above, whereas, if the benzene solution of the alkaloids is left for 24–48 hours, particularly in the light, less than one-half of the proportion of pilocarpine is obtained, and a slight deposit forms on the walls of the vessel. If, on the other hand, the powdered leaves are first extracted with a volatile solvent, such as benzene or petroleum spirit, and are treated with sodium carbonate and benzene only after this volatile solvent has been expelled, such retrogradation of the pilocarpine is not observed. No explanation is advanced for this behaviour. T. H. P.

Biochemical.

Determination of Copper in Biological Materials. C. A. Elvehjem and C. W. Lindow. (*J. Biol. Chem.*, 1929, **81**, 435–443.)—The wide distribution of copper in minute quantities in biological materials has been regarded, until lately, as accidental. The recent discovery by Hart, Steenbock, Waddell and Elvehjem (*J. Biol. Chem.*, 1928, **77**, 797) of the importance of copper as a supplement to iron for haemoglobin building in the rat has shown the necessity for an intensive study of the distribution of copper in nature, and for a suitable method for the quantitative determination of this element. All the known methods were considered, and an accurate and rapid method has been outlined for the determination of copper in biological material which is a modification of the colorimetric method of Biazzo (*Ann. chim. appl.*, 1926, **16**, 2), which was found to be the

most satisfactory. It is based on the fact that the neutral solution of a copper salt, when treated with a few drops of concentrated potassium thiocyanate solution and a few drops of pyridine, gives a green precipitate of the composition $Cu(C_5H_5N)_2(CNS)_2$, which is soluble in chloroform, and this is quantitatively removed by the chloroform from the solution in which it was formed. The green colour of the chloroform layer varies in intensity according to its copper content. This method is rapid, simple and accurate, and requires only a small weight of sample. Samples which contain 0·02 mgrm. of copper can be analysed with a high degree of accuracy. Care must be observed to prevent the introduction of any copper from the use of new porcelain dishes. A method is given for the removal of copper from the glaze of a porcelain dish, since one 3-inch evaporating dish may contain as much as 0·033 mgrm. of copper. The results of the Biazzo method are shown to agree closely with those from the xanthate method. Procedures are given for the analysis of substances rich in iron, for iron salts, and for milk and bones. Of ten samples of iron salts, only 2 were found to be copper-free. These figures point to the possibility of copper playing a rôle in many cases when beneficial effects of iron salts in the treatment of anaemia have been noted.

P. H. P.

Note on the New Ferricyanide Method for Blood Sugar. O. Folin. (*J. Biol. Chem.*, 1929, **81**, 231–236.)—Many workers have experienced difficulties with the new colorimetric ferricyanide method for the determination of blood sugar devised by Folin (*J. Biol. Chem.*, 1928, **77**, 421; ANALYST, 1928, **53**, 392–393), and, as little was said in the original paper about the keeping quality of the different reagents used in the method, such information as has since been obtained on these points is now given. One investigator stated that the dilute tungstic acid solution used for the protein precipitation must be fresh in order to give the low sugar values reported by Folin. Research has shown that the determining factors in that case were sunlight and toluene. Tungstic acid solutions kept in a dark cupboard without toluene gave perfectly water-clear extracts, and correct blood sugar values for at least 5 months, but in sunlight, with toluene added to preserve them, they deteriorated, and in a week gave 15 to 18 per cent. higher sugar values. Toluene in the presence of tungstic acid is decomposed by light, partly into reducing products, and partly into products which are oxidised in the presence of some other easily oxidisable substance (glucose). In the absence of tungstic acid toluene is not similarly decomposed. This destructive effect of light in the presence of tungstic acid may not be specific for toluene. Therefore the dilute tungstic acid solution need not be freshly prepared, provided that no preservative is used and that the reagent is not exposed to too much light. The sodium cyanide and carbonate solution keeps satisfactorily for several months, and so does the potassium ferricyanide solution if completely protected from light (in a brown glass bottle in a dark cupboard). The ferric iron solution has given most trouble. The author now uses gum ghatti in place of gum arabic, for it does not hydrolyse so easily, and is at least 5 times as effective as gum arabic as a protective colloid for Prussian blue. The preparation of this reagent is described in

detail. Oxidation with potassium permanganate is now included, even when gum arabic is used. The acid iron phosphate solutions containing gum ghatti have shown no signs of deterioration at the end of 2 months. The Prussian blue, by which the sugar reduction is measured, will remain in a clear uniform dispersion practically indefinitely, but the colour comparison must be finished promptly, as originally directed. P. H. P.

Determination of the Digestibility of Protein by Bergeim's Method. W. D. Gallup. (*J. Biol. Chem.*, 1929, **81**, 321–324.)—The method of Bergeim (*J. Biol. Chem.*, 1926, **70**, 29) for the determination of the digestibility of food consists in "the addition to the food of small amounts of iron oxide (or other suitable substance)," followed by determination of the ratio of the amount of iron to the amount of any food substance in the diet and in the faeces, and calculation from this of the percentage of utilisation. Results by this method closely agree with those obtained by the usual procedure, *i.e.* measurement of the intake and output of nitrogen over a given period. The method, however, was found unsuitable for some diets which contain as much as 1 per cent. of soluble iron salts, and silica, which may be regarded as an insoluble compound and one that can be excreted with practical completeness in the faeces, has been used in place of iron oxide. A table shows a comparison of the results of the silica method, the iron oxide method and the usual method, when applied to the determination of the digestibility of the protein in a diet made up of natural foods. Albino rats were used for the study. The silica determinations gave results even more closely in agreement with those obtained by the usual method, than the iron determinations. Therefore, in carrying out digestibility studies by Bergeim's method with certain diets or under conditions which do not permit the use of iron oxide, silica in sufficient quantities can be used as a suitable substitute. When such a substitution is made, certain alterations in the procedure become necessary, but the method still retains most of its desirable features. P. H. P.

Micro Method for the Determination of Total Creatinine in Muscle. S. Ochoa and J. G. Valdecasas. (*J. Biol. Chem.*, 1929, **81**, 351–357.)—A method is given for the determination of total creatinine in as small amounts of muscle as 5 to 100 mgrms., which is a slightly modified version of the original technique described by the authors (*Bol. Soc. Españ. Biol.*, 1927, **13**, 17). The muscle creatine is extracted and converted into creatinine by hydrochloric acid in the autoclave, then the proteins are precipitated by picric acid, and the creatinine is colorimetrically determined in the filtrate by the Folin technique. For the method from 5 to 100 mgrms. (preferably 20 to 50 mgrms.) of muscle are removed promptly from living animals under anaesthesia, and dropped into previously weighed Erlenmeyer flasks of about 50 c.c. capacity, which are kept tightly closed until they have been weighed again. A torsion balance, accurate to 0·1 mgrm., should be used, and the muscle samples should not be too much impregnated with blood. The weighed muscle is treated with 0·2 c.c. of 0·2 *N* hydrochloric acid for each mgrm. in weight (introduced by means of a standardised pipette graduated

in 0·01 c.c.), and the flasks are covered with tin-foil and heated in the autoclave to 120° C. for 25 minutes, cooled, 0·2 c.c. of pure 1·2 per cent. picric acid added for each mgrm. of muscle, and the contents then mixed and allowed to stand for 5 minutes. The abundant protein precipitate is separated by filtration. A fixed volume of fluid is pipetted from the clear filtrate, transferred to a flask, 0·5 volume of 5 per cent. sodium hydroxide solution is added, and the contents mixed. After standing for 5 to 8 minutes the colour comparison is made against a standard prepared as follows:—To 5 c.c. of a 0·002 per cent. creatinine solution in 0·2 *N* hydrochloric acid, 5 c.c. of pure 1·2 per cent. picric acid, and 5 c.c. of 5 per cent. sodium hydroxide solution are added, mixed, and left for 5 to 8 minutes when the standard is ready for use. The height of the standard (10 or 20 mm.), divided by the reading of the unknown and multiplied by 400, gives the total creatinine in mgrms. per 100 grms. of muscle. The mean error, as experimentally determined, can be given as 0·5 to 2 per cent. when all the operations are carried out with accuracy. P. H. P.

Antineuritic Vitamin. II. Properties of the "Curative" Substance. J. L. Rosedale and C. J. Oliveiro. (*Biochem. J.*, 1928, **22**, 1362–1367.)—During the preparation of extracts of the water-soluble vitamin from rice polishings it was noticed that, unless precautions were taken, alcoholic fermentation readily occurred at laboratory temperature, due to the presence of yeasts. Experiments on pigeons have been carried out to determine whether fermentation has any deleterious effect upon the extracts; results show that the antineuritic vitamin of an extract of rice polishings is destroyed by fermentation, and by sterilisation by filtration and by heat. The distribution of enzymes in the alimentary canal of the normal pigeon has been investigated, and the study extended to human cases. The potent curative extract of rice polishings contains sucroclastic and lipoclastic enzymes, but it has not been possible to show the presence of proteoclastic enzymes. Results indicate that during polyneuritis, metabolic disturbbances occur which involve a certain amount of inactivation of the pancreas. In cases of "dry" beriberi, the pancreas has been found incapable of lipoclastic and tryptic digestion, but no deterioration of sucroclastic enzymes has been shown. The authors cannot conclude with Kon and Drummond (*Biochem. J.*, 1927, **21**, 632) that vitamin *B* bears relationship only to protein metabolism. Plimmer, Rosedale and Raymond (*Biochem. J.*, 1927, **21**, 913) found that the balance between vitamin *B* and protein was more difficult to demonstrate than that between carbohydrate or fat and vitamin *B*, but the experiments of Reader and Drummond (*Biochem. J.*, 1926, **20**, 1256) leave no doubt that protein is similarly affected. It is considered from the general results that the curative substance has at least some control over the action of pancreatic enzymes. P. H. P.

Biological Inertness of Irradiated Mycosterols other than Ergosterol. O. Rosenheim and T. A. Webster. (*Biochem. J.*, 1928, **22**, 1426–1428.)—The authors have worked up the mother-liquors resulting from the recrystallisation of large amounts of ergosterol from ergot, and have isolated a mycosterol which has

been proved to be identical with one prepared from ergot supplied by Messrs. Burroughs, Wellcome & Co., and for which the name "fungisterol," given by Tanret (*Ann. Chim. Phys.*, 1908, **15**, 313), is retained. Biological and spectroscopic examination showed the presence of less than 5 per cent. of ergosterol still in the specimens. The physical constants of the two specimens agreed with each other, but not with those of fungisterol, as described by Tanret. Apparently the latter product was still a mixture of several sterols, of which two further constituents have been isolated in the laboratories of Messrs. Burroughs, Welcome & Co. These sterols, as well as the specimens of fungisterol, were found by the authors to be biologically inactive after irradiation, except for a slight action definitely due to the contaminating ergosterol. The results lend further confirmation to the view expressed previously by Rosenheim and Webster (*Biochem. J.*, 1928, **22**, 762; ANALYST, 1928, **53**, 551), that ergosterol is the only substance which can be converted into vitamin *D* by irradiation. It is interesting to note that zymosterol, which occurs together with ergosterol in yeast, is evidently not identical with fungisterol. P. H. P.

"Hypervitaminosis" and "Vitamin Balance." L. J. Harris and T. Moore. (*Biochem. J.*, 1928, **22**, 1461–1477.)—Many writers have assumed that vitamins have no injurious effect when consumed in abnormally large amounts, and that an increased or diminished consumption of one vitamin does not affect the body's requirements for the others. With regard to the water-soluble vitamins no evidence is as yet available to suggest that any ill-effects result from overdosing, but in the case of fat-soluble vitamins, instances of supposed hypervitaminosis have been recorded, and attempts have been made to show that the fat-soluble and water-soluble requirements of the animal are, to some extent, inter-related. A thorough survey of the subject is now in progress, but results so far obtained are recorded, which, in a general way, definitely confirm the idea of a harmful effect resulting from excessive intake of certain materials rich in fat-soluble vitamins. Results show that young rats lost weight rapidly and died when receiving synthetic diets containing 0·1 per cent. of an irradiated (but not non-irradiated, over-irradiated, or heated) ergosterol (*i.e.* about 100,000 times a minimal protective dose). There was loss of appetite, ill condition of coats, etc., diarrhoea and inanition. No appreciable alleviation of these symptoms resulted when the vitamin *B* (marmite) allowance was increased to only 4 times the normally adequate level; in 2 cases where still further vitamin *B* (and *C*) (wheat-germ extract plus orange juice) was given, loss of weight was prevented. Results due to "toxicity" are contrasted with those due to mere loss of appetite. Apparently the rat is able to discriminate in its choice of diets; in a quantitative study rats refused food overloaded with irradiated ergosterol (5 per cent.). They showed lower growth rates compared with litter mates, and had rough coats, when cod-liver oil was substituted for 15 per cent. of arachis oil (inactive) in a ration which contained restricted allowances of vitamin *B* complex. Normal gestation always failed in rats receiving a diet containing 15 per cent. of cod-liver oil. Rats receiving

excessive doses of vitamins *A* and *D* concentrate from cod-liver oil, in conjunction with a diet deficient in the vitamin *B* complex, developed loss of hair and severe skin lesions. A close parallelism is suggested between the development of anti-rachitic and of toxic properties. It is obvious that any possibility of simple arithmetical equivalence in balance between vitamin *D* and the *B* vitamins is out of the question, yet, whereas the conception of a strictly quantitative balance cannot be tenable in any general sense, the possibility of a large excess of one vitamin emphasising the effects of deficiency of another cannot, as yet, be ruled out. Toxic effects at such enormous dosages should not in any way discourage the rational use of the properly standardised materials at the ascertainable correct physiological levels. P. H. P.

Fluorescence of Some Vitamin *A*-containing Fats. R. S. Morgan and K. MacLennan. (*Biochem. J.*, 1928, **22**, 1514–1522.)—A method has been devised by which the actual brightness of the fluorescence of a solid fat, illuminated by ultra-violet light filtered practically free from visible light, may be determined, and the colour expressed in terms of three additive primaries: red, green and blue. The apparatus for the measurement of fluorescence is described in detail, and diagrams are given. Actual colour readings are measured when a standard lamp giving white light replaces the filtered ultra-violet light. The unsaponifiable matter from cod-liver oil contains a brightly fluorescent substance. Curves and tables show the effect of the addition of unsaponifiable matter from cod-liver oil on the fluorescence of two fats, one already slightly fluorescent (jus), and the other brightly fluorescent (hardened coconut oil). Although there is a close association between vitamin *A* and a characteristic fluorescence, yet discrepancies occur. No fat containing vitamin *A* has yet been observed that does not also show the fluorescence associated with it, and an explanation of the discrepancies may be that, whereas vitamin *A* is actually a brightly fluorescent substance, in certain circumstances other substances may be formed in oils, giving a fluorescence somewhat similar to that due to the vitamin. No connection seems to be apparent between fluorescence and vitamin *D*. The fluorescence of butter or butter fat is yellow in colour, but the normal fluorescence of margarine is blue. This difference cannot be accounted for solely by the known differences in vitamin content. The blue fluorescence of margarine can be modified as follows: (*a*) By variation of the fat mixture; certain vegetable fats fluoresce bright blue, whilst the fluorescence of oleo and jus is pale greenish; (*b*) by the addition of unsaponifiable matter from cod-liver oil; the first small additions markedly increase the brightness and diminish the blueness of the fluorescence; (*c*) by variation of the nature of the pigment present; some pigments depress the fluorescence more markedly than others. The following table shows that samples that respectively match Danish butter-fat and New Zealand or Irish butter-fat in the quality of their fluorescence, also match them in the actual colour by ordinary illumination. Here, as would be expected, the greater opacity of the jus increases the brightness by reflected light.

The actual colour by reflected light of samples matching in fluorescence was as follows:—

	Shade.	Quality. Red.	Green.	Blue.
Danish butter fat I	58	47	40	13
Oleo+0·8 per cent. red palm oil+unsap. to vitamin potency of butter fat	57	48	41	11
Jus+0·8 per cent. red palm oil+unsap. ..	69	47	39	14
New Zealand butter-fat	45	51	40	9
Jus+1·6 per cent. red palm oil+unsap. ..	53	52	39	9

A sample of oleo coloured with sufficient red palm oil to match it with butter-fat, and with sufficient unsaponifiable matter from cod-liver oil to bring it up to butter-fat in vitamin *A* potency, exactly matches butter-fat in fluorescence. P. H. P.

Bacteriological.

Quantitative Determination of Indole in Bacterial Cultures. H. B. Pierce and R. B. Kilborn. (*J. Biol. Chem.*, 1929, **81**, 381–387.)—The method of Bergeim (*J. Biol. Chem.*, 1917, **32**, 17) for the determination of faecal indole has been adapted to the quantitative determination of indole in bacterial cultures. Bergeim's method consists in a steam distillation of a faeces suspension in an alkaline medium to remove phenols. The distillate, which contains indole and ammonia, is redistilled from an acid solution or treated with permutit to remove ammonia. A portion of the ammonia-free distillate is treated with β-naphthoquinone sodium monosulphonate, and the blue indole compound thus formed is extracted with chloroform, and determined colorimetrically. For the bacterial cultures the distillation was carried out at a P_H of 8·5 to 10·0, and permutit proved to be entirely satisfactory for the removal of ammonia from the distillate. Steam should be passed through the contents of the distilling flask during the entire period of distillation, and the solution remaining in the flask at the end of the period should be approximately 150 c.c. An average of 91 per cent. of the indole added to peptone water or to bacterial cultures in peptone water has been recovered by use of this method. The average percentage of indole recovered from aqueous solutions ranged between 94 and 97. Fellers and Clough (*J. Bact.*, 1925, **10**, 105) and Zoller (*J. Biol. Chem.*, 1920, **41**, 25; ANALYST, 1920, **45**, 177) have criticised Bergeim's method on the grounds that the procedure is involved, and the reagent very difficult to obtain. This method for bacterial cultures is not quite quantitative, but it is more nearly so than that of Fellers and Clough, and once the technique is standardised, the procedure is not involved. The reagents required are comparatively inexpensive. If the solution containing this reagent (a 2 per cent. solution) is kept in an ice box, it does not deteriorate during a period of 3 days. When a precipitate forms the reagent should be discarded. P. H. P.

Toxicological and Forensic.

Distribution of Bismuth in the Organs after Injection of Aqueous Solutions. R. Fabre and M. Picon. (*J. Pharm. Chim.*, 1929, **121**, 97–112.)—Aqueous solutions of ammoniacal bismuth citrate and bismuth cacodylate were used for injecting into rabbits. In the former case the kidneys were particularly affected, and the hair helped to excrete the bismuth, probably supplementing the insufficiency of the renal separation. With the cacodylate the toxicity was much less, and the liver retained more bismuth than the kidneys. It is concluded generally that the cacodylate and campho-carbonates of bismuth are less toxic than the ammoniacal citrate, and tables are given showing the proportions of bismuth found in the various organs after injection. D. G. H.

Organic Analysis.

Application of the Hydrogen Value to Unsaturated Fatty Acids. H. J. Waterman, S. H. Bertram and H. A. Van Westen. (*J. Soc. Chem., Ind.*, 1929, **48**, 50–51T.)—The hydrogen value (ANALYST, 1929, **54**, 119) has now been determined for elaidic, linolic and stearolic acids, and in each case pure stearic acid was the product of hydrogenation. The results with elaidic acid are considered as standard measurements. The results of the hydrogenation of $\Delta^{9:12}$-linolic acid showed that two double linkings are saturated (not 1 double and one triple), 2 molecules of hydrogen being consumed per molecule of linolic acid. Stearolic acid behaved towards the thiocyanogen solution as though almost completely saturated, and towards iodine solution in accordance with one double linking, whereas treatment with hydrogen with palladium catalyst proved the presence of a triple linking. D. G. H.

Conversion of Higher Fatty Acids into their Barium Salts. H. H. Escher. (*Helv. Chim. Acta*, 1929, **12**, 103–105.)—Owing to the readiness with which both the acids and their barium salts separate and include one another, quantitative conversion of the higher fatty acids into their barium soaps by means of barium salts or aqueous barium hydroxide is difficult. The titration of solutions of fatty acids in concentrated or dilute alcohol or in methanol with aqueous 0·1 *N* barium hydroxide solution gives low results, which are improved but not rendered quite satisfactory by adding a large excess of alcohol, by heating, and by adding the baryta very slowly and with vigorous swirling of the liquid.

Conversion of the fatty acids into their barium salts proceeds quantitatively and more easily if a methanol solution of barium hydroxide is used. In this way solutions of stearic, palmitic and oleic acids, and also those of oleic acid dibromide and linolic acid tetrabromide in methanol, ethanol, ether, chloroform, carbon tetrachloride, dichloroethane, etc., may be titrated sharply. Crystallised baryta ($+8H_2O$) dissolves to the extent of about 30 per cent. in commercial pure methanol and, although the latter contains about 0·1 per cent. of acetone, the solutions

maintain their titre and keep clear and colourless for a year. The solution is kept in a vessel closed by a soda-lime U-tube, 40 cm. long, drawn out at the end to 1 mm.; the rubber of the syphon tubes does not swell and is only slightly attacked. The "sticking" of the glass-rod cock when only occasionally used may be obviated by frequent loosening or by using rubber tubing which has been artificially aged in a hot solution of barium hydroxide and methanol. T. H. P.

Preparation of Styrolenes. Detection and Identification of β-Phenylethyl Alcohol. S. Sabetay. (*Bull. Soc. Chim.*, 1929, (iv), **45**, 69–75.)—Distillation of β-phenylethyl alcohol over anhydrous potassium hydroxide furnishes an almost quantitative yield of styrolene, other analogous alcohols behaving similarly. The reaction is characteristic of the group $\cdot C_6H_4\cdot CH_2\cdot CH_2\cdot OH$, and is explainable by the facility with which a double linking is formed adjacent to the benzene nucleus owing to the influence of the phenyl radical on the mobility of the neighbouring hydrogen atom.

The identification of β-phenylethyl alcohol by means of such crystalline derivatives as its phenylurethane, diphenylurethane, acid phthalate, monobenzylphthalate, etc., is difficult in presence of geraniol, rhodinol, phenylpropyl alcohol, etc., and such mixtures are not easily resolved by simple distillation. In these cases, it is necessary only to distil the mixed alcohols over anhydrous potassium hydroxide, to collect the first few c.c. of distillate, and to test this for styrolene by its odour and by means of the dibromide, which crystallises readily from 80 per cent. alcohol and melts at 72° C. Although rhodinol, geraniol, etc., also form brominated derivatives, these are not solid under the conditions employed. The minimum amount of the dibromide detectable in this way varies, but the quantity formed gives an approximate indication of the quantity of β-phenylethyl alcohol present. T. H. P.

Quantitative Separation of Dextrins and Gum Arabic. A. Hamy. (*Ann. Falsificat.*, 1929, **241**, 24–26.)—The methods used for the determination of gum arabic, such as precipitation by alcohol or the formation of gum-iron compound, give erroneous results in presence of highly condensed dextrins, owing to simultaneous precipitation of these. The method now suggested for use in such cases depends on the fact that treatment of an aqueous gum solution with basic lead acetate yields an abundant precipitate which, in presence of a sufficient quantity of glucose or sucrose, dissolves completely; this solvent action may, however, be annulled by addition of alcohol. The procedure is as follows: Twenty c.c. of the syrup of half-concentration and 23 c.c. of 95 per cent. alcohol are made up to volume with water in a 55 c.c. flask, and the liquid left for some hours to deposit the dextrins and then filtered through a close filter-paper into a 50 c.c. flask. The 50 c.c. of filtrate are treated with 90 c.c. of water containing glucose and sucrose in such quantities that the final mixture contains 5 and 8 grms., respectively, of these sugars, and then with 42 c.c. of 95 per cent. alcohol and 15 c.c. of basic lead acetate. After being shaken three or four times, the liquid is left until the next morning, when the supernatant liquid is either siphoned off

or, if there is gum in suspension, decanted on to a Gooch crucible. The deposit is centrifuged in two 45 c.c. tubes, vessel and precipitate being washed with 50 c.c. of 5 per cent. basic lead acetate solution to remove excess of dextrins and alcohol. After centrifuging, the precipitate in each tube is mixed with 4·3 c.c. of a 75 per cent. sugar solution, which dissolves it partially, and is then mixed well with 35 c.c. of a mixture of 120 c.c. of 95 per cent. alcohol, 27 c.c. of basic lead acetate solution, and 160 c.c. of water. The covered tubes are left to deposit and afterwards centrifuged, the precipitate being dissolved in 4 per cent. acetic acid (turbidity of the solution is of no account) and the gum precipitated by about 10 times the volume of alcohol. On the following day the precipitate is collected on a Gooch crucible, dried at 110° C., weighed, calcined and weighed again. The difference in weight represents the anhydrous gum contained in the 50 c.c. of filtrate taken. Trial determinations gave good results. T. H. P.

Study of the Digitonin Ergosterol Complex. M. H. Pénau and Z. Hardy. (*J. Pharm. Chim.*, 1929, **121**, 145–151.)—Digitonin forms a complex with ergosterol, which under certain conditions is practically insoluble. This complex may be resolved into its components again by prolonged treatment with boiling toluene. The conditions for determination of ergosterol by means of the complex are as follows. The ergosterol (175 mgrms.) is dissolved on a water-bath in 99 per cent. alcohol, cooled, made up to 100 c.c., and 10 c.c. put into a weighed centrifuge tube, 9 c.c. of the digitonin solution (1 per cent. by volume in 99 per cent. alcohol) added (measured at the same temperature, 15° C.), and 2 c.c. of water. After standing for 18 hours the mixture is centrifuged for 15 minutes, the supernatant liquid decanted, and the precipitate washed with 4 c.c. of Caminade's solution (water-alcohol-acetone), again centrifuged, the washing solution decanted, and the tube dried and weighed. One grm. of the complex contains 250 mgrms. of ergosterol. D. G. H.

Universal Indicator which gives the Colours of the Spectrum over a P_H Range of 3 to 11.5. H. W. Van Urk. (*Pharm. Weekblad*, 1928, **65**, 1246–1249.)—The indicator contains 0·1 grm. of methyl orange, 0·04 grm. of methyl red, 0·4 grm. of bromthymol blue, 0·32 grm. of naphtholphthalein, 0·5 grm. of phenolphthalein, and 1·6 grms. of cresolphthalein in 100 c.c. of 70 per cent. alcohol. One drop is added to 10 c.c. of the solution to be tested, and the colour changes from red-orange (P_H 3) to yellow-orange (P_H 5), yellow (P_H 6·5), green (P_H 8), green-blue (P_H 9), violet (P_H 11), and red-violet (P_H 12). J. G.

Inorganic Analysis.

Reaction of Cupric Salts with Thiosulphate. J. Hanus and V. Hovorka. (*Trav. Chim. Czechoslovak*, 1929, **1**, 65–82.)—Published investigations of this reaction show discordant results. The authors find that the precipitates deposited, when cupric salt solutions are boiled with a thiosulphate, consist of

mixtures of cuprous and cupric sulphides and free sulphur in proportions varying with the duration of the boiling, the amount of thiosulphate added, and the degree of acidity of the solution. Prolonged boiling results in a greater proportion of cupric than cuprous sulphide, and the percentage of the latter in the precipitates from neutral solutions reaches a maximum when the initial solution contains copper and thiosulphate in the molecular ratio 1:2·5–3. With a large excess of thiosulphate almost pure cupric sulphide mixed with sulphur is obtained. In acid solution decomposition of the thiosulphate itself alters the composition of the precipitates. The order of the operations also varies the result, since less cupric sulphide is formed when the whole amount of thiosulphate is added to the boiling acidified solution of cupric salt than when the cold mixture is subsequently heated to boiling.

For analysis, the precipitate was collected on a Gooch crucible with a filter-paper (Adams' filter for extracting fats), and washed with hot water, alcohol, and ether, the free sulphur being then extracted by means of nitrobenzene at 100° C. The residual precipitate was treated in a beaker with two successive quantities (15 to 25 c.c.) of 10–15 per cent. silver perchlorate solution, and the resulting mixture of silver sulphide and silver washed until free from silver ion, and afterwards treated twice or thrice with 40–50 c.c. of 6 per cent. ferric nitrate solution, which brings the metallic silver into solution but leaves the silver sulphide unchanged. The filtrate, acidified with nitric acid, was concentrated to 100–120 c.c., allowed to cool, and titrated with 0·1 *N* potassium thiocyanate solution. This gives the silver formed from the cuprous sulphide in accordance with the equation, $Cu_2S+4AgNO_3 \longrightarrow 2Cu(NO_3)_2+Ag_2S+2Ag$ (cupric sulphide giving only silver sulphide), and hence the cuprous sulphide. The initial amount of cupric salt being known, the cupric sulphide was readily determined by calculation. When the copper was not totally precipitated, the residual amount in the first filtrate was determined electrolytically. T. H. P.

Titration of Thallous Salts with Permanganate in Hydrochloric Acid Solution. A. Jílek and J. Lukas. (*Trav. Chim. Czechoslovak.*, 1929, **1**, 83–94.)—The incompleteness of the oxidation of thallous to thallic salts by permanganate in presence of hydrochloric acid, and the consequent necessity of using an empirical factor in such determinations, may be obviated by the addition of an alkali chloride, which forms a double salt with the thallic chloride formed and thus suppresses hydrolysis. The solution to be titrated is treated with 2 grms. of potassium chloride, repeatedly evaporated with hydrochloric acid to remove other acids, diluted and twice treated with 2–5 c.c. of sulphurous acid solution, the excess of sulphur dioxide being expelled by boiling after each addition. After a further addition of 10 c.c. of concentrated hydrochloric acid, the liquid is made up to 150 c.c. and titrated with 0·02 *N* permanganate solution. The excess of permanganate required to produce the pink end-point is determined by a blank experiment, and the result corrected accordingly; 1 c.c. of 0·02 *N* permanganate corresponds with 0·0020439 grm. of thallium. T. H. P.

Iodimetric Determination of Iron. E. C. Grey. (*J. Chem. Soc.*, **1929, 135,** 35–39.)—The iodimetric method for the determination of iron, first proposed by Mohr (*Ann.*, 1858, **105**, 53) yields trustworthy results under the following conditions: The ash of the sample is evaporated repeatedly with hydrochloric acid until any insoluble residue is colourless, then dissolved in water and the hydrochloric acid content of the solution adjusted so that there is not less than 0·4 c.c. of the concentrated acid in 10 c.c. (The volume of the acid used should be noted, for a control may be necessary to correct for the iron which it may contain.) If copper is present, the solution is treated with an excess of ammonia, the precipitate collected on a filter, washed, and re-dissolved in hydrochloric acid. To each 10 c.c. of the solution is then added 0·33 grm. of potassium iodide dissolved in 5 c.c. of water. The liberation of iodine is complete in three minutes at 15° C. The solution is next diluted, sodium acetate is added if desired, and the iodine is titrated with very dilute standardised thiosulphate solution. W. P. S.

Gravimetric Method for the Micro Determination of Molybdenum. J. B. Niederl and E. P. Silbert. (*J. Amer. Chem. Soc.*, 1929, **51**, 376–377.)—From 3 to 5 mgrms. of the sample, which should not contain non-combustible or non-volatile substances other than molybdenum, are placed in a micro porcelain combustion boat, a drop of nitric acid is added, the boat is inserted in a combustion tube and heated gradually. When all fumes have disappeared the boat and its contents are heated for a further five minutes, cooled, and the molybdenum trioxide is weighed. The heating employed must not be excessive since molybdenum trioxide is volatile at temperatures above 450° C. W. P. S.

The Phosphoric Ion as a Sensitive Reagent. Differentiation of Antimony and Tin. T. G. Y. Arnal. (*Chim. et Industrie*, 1928, Oct.; *Ann. Chim. anal.*, 1929, **11**, 11–12.)—On mixing sodium molybdate and antimony trichloride solution there is formed a yellowish precipitate soluble in excess of antimony trichloride, and the reagent thus produced is very sensitive to the phosphoric ion. If an ortho-phosphate is then added a blue coloration or precipitate results. If a solution of orthophosphate is added to stannous chloride followed by sodium molybdate, a blue coloration forms changing to rose. In the unknown solution tin is first sought with sodium molybdate, and to another portion an orthophosphate solution is added followed by sodium molybdate. If a blue coloration persists antimony is present. A table of reactions of the reagent is given. D. G. H.

Physical Methods, Apparatus, etc.

The Ultraviolet-Detector as an Aid in distinguishing Real Amber from its Imitations. G. Kostka. (*Chem. Ztg.*, 1929, **53**, 117–118.)—When exposed to the light from a Hanau quartz lamp filtered so that only rays of λ 440 to 280 $\mu\mu$ pass, natural amber exhibits intense fluorescence which varies from yellowish-green or greenish to bluish-white. With pressed amber (ambroid), the

fluorescence is weaker and yellowish-green. The phenol-formaldehyde resins show no fluorescence, and the urea-formaldehyde resins, and the casein preparations, and the plastic cellulose derivatives pale bluish or bluish-white fluorescence.

T. H. P.

Reviews.

DIE MASSANALYSE. ZWEITER TEIL: DIE PRAXIS DER MASSANALYSE. (THE PRACTICE OF VOLUMETRIC ANALYSIS.) By I. M. KOLTHOFF, in collaboration with H. MENZEL. Pp. 512. Berlin: Julius Springer.

In spite of the large volume of Kolthoff's publications in recent years, in this book the high standard of his work is maintained. The author does not claim the work to be exhaustive, nor does he expect it to replace the standard works on volumetric analysis; he hopes rather that it will be complementary to them. The book contains a critical survey of a number of typical volumetric processes, most of which have been studied by the author. The "personal" nature of the book will be realised when it is stated that there are nearly 150 references to Kolthoff's published researches, as well as frequent mention of his unpublished work; a considerable number of references to other authors are, however, also given, and the whole subject appears to be treated in an impartial manner. The titles of the chapters are: Measuring Apparatus for Volumetric Analysis; Practical Foundations; Alkalimetry and Acidimetry; Neutralisation Reactions; "Depression" Reactions; Hydrolytic Precipitation and Complex-forming Reactions; Special Methods in Alkalimetry and Acidimetry; Titrations with Silver Nitrate (Argentimetry); Formation of Complexes (Mercurimetry); Indicators in Oxidation and Reduction Processes; Permanganate (Oxidimetry); Iodimetry; Practical Methods of Iodimetry; Titrations with Potassium Iodate; Titrations with Potassium Bromate; Titrations with Dichromate; Other Volumetric Reagents. There is an appendix containing atomic and equivalent weights useful in volumetric work, and also an author index and a very complete subject index.

There are two special features in this interesting book; one is the use of the "rational" atomic weights recommended by Schoorl (*Z. anal. Chem.*, 1918, **57**, 209), which allow for the fact that weighings in analytical work are invariably made in air and not corrected for the buoyancy effect. The other is the special attention paid to the question of purifying and testing the standard materials, *e.g.* sodium thiosulphate, sodium carbonate, silver nitrate, and oxalic acid, from which volumetric solutions are frequently prepared by direct weighing. The work is quite up to date; it contains examples of the application of the adsorption indicators recently developed by Fajans, and of the use of 8-hydroxyquinoline as a precipitation reagent in the volumetric estimation of zinc, magnesium,

calcium, and aluminium (*cf.* Berg, *ibid.*, 1927, **71**, 23 and 171; Hahn and Vieweg, *ibid.*, p. 122).

Although this is not a book for the beginner in volumetric analysis, yet an analyst with some experience will find it an invaluable guide, to be used alongside his standard practical works, in the choice of accurate methods. Part of the work has now been published as an English translation (*cf.* ANALYST, 1929, 194).

S. GLASSTONE.

ANALYTICAL CHEMISTRY. Vol. II. QUANTITATIVE. BASED ON THE TEXT OF F. P. TREADWELL. W. T. HALL. Seventh Edition. Pp. xiii+848. New York: John Wiley & Sons, Inc.; London: Chapman & Hall. 1928. Price 30s. net.

A novel feature of the seventh edition of Professor Hall's adaptation from Treadwell's text is an Appendix outlining a course of instruction at the Massachusetts Institute of Technology. This outline accounts almost entirely for the additional 40 pages and, unfortunately for the general reader, its appearance coincides with an increase of 5s. in the price of the book. A number of methods published since the appearance of the sixth edition (ANALYST, 1924, **49**, 608) have been incorporated in the text, including Moser and Niessner's recent separation of aluminium from beryllium (ANALYST, 1928, **53**, 401).

It need not be repeated here that the volume under review is one of the indispensable reference-books for the laboratory, and it will doubtless continue to occupy that enviable position owing to its wide circulation, which ensures frequent opportunities for revision. If that is well done—both by addition of new important matter and excision of all that is becoming obsolete—the author will have deserved the gratitude of the analytical fraternity.

It is in an endeavour to help towards more uniform reliability, not in a spirit of adverse criticism, that the writer has selected the following points for discussion, as he believes them to constitute minor flaws in an otherwise excellent piece of work.

The student confronted with a pyrosulphate fusion must imagine it a formidable undertaking. He is told that "this is a difficult fusion to make satisfactorily and requires time and patience" (p. 115), and that the attack of *precipitated* iron-aluminium oxide is "usually complete in 2–4 hours" (p. 110). Were this so, the researches of the reviewer and his co-workers on the earth acids and other refractory oxides—largely a spare-time pursuit which so far has necessitated several thousand bisulphate fusions—could never have been carried out. As a matter of fact, the fusion of an ignited oxide precipitate is a matter of only a few minutes, provided the oxide is in a state of fine subdivision; this is ensured by the incorporation of pulped filter fibre before or after precipitation. The addition of filter pulp not only imparts porosity to the ignited precipitate, but accelerates the filtration, washing, and ignition to constant weight; it is, in the writer's opinion,

one of the most valuable expedients ever introduced into analytical practice, and should be inculcated as part of general analytical technique.

The text-matter dealing with the determination of bismuth (p. 180–183) could, no doubt, be made to gain by a process of revision. As many as six methods are given: (1) Precipitation with ammonium carbonate and ignition to oxide; (2) determination as sulphide, involving extraction with carbon disulphide; (3) reduction to metal by cyanide fusion; (4) reduction by alkaline formaldehyde; (5) electrolysis; and (6) determination as phosphate. Of these methods, (2) to (4) are, to say the least, given undue prominence, as they are of doubtful practical value, whilst the useful oxychloride method—certainly superior to the three methods cited—is not mentioned at all. The directions for the method *par excellence* (determination as phosphate) are inadequately given in five lines, followed by this comment: "Sometimes, when the bismuth solution is not sufficiently dilute or too much free acid is present, the filtrate comes through turbid. In such cases, wash the filter with hot water and return the filtrate to the original beaker." All will agree that this is not quite good enough for "Treadwell"; "too much free acid" is almost synonymous with incomplete bismuth precipitation, and if the exact conditions for the operation were specified (*cf.* ANALYST, 1920, **45**, 435), the possibility of such faulty work would be excluded.

For the determination of lead in ores containing barium sulphate (p. 178), the treatment prescribed is acid attack, followed by evaporation with sulphuric acid until fumes appear, dilution with water, and collection of the mixed sulphate precipitate, followed by the familiar ammonium acetate treatment; according to the text, the lead dissolves, whilst the barium sulphate remains insoluble. If, as is more usually the case, the baryta content of the ore is small and the lead content high, the results obtained by this method may go unchallenged; but its principle is faulty, for the sulphates re-precipitated by the dilution of the sulphuric acid contain more or less lead in an acetate-insoluble form, probably as mixed crystals of $(Ba,Pb)SO_4$.

An important separation in the hydrogen sulphide group of metals is that of the sulpho-acids from the sulpho-bases by means of alkali sulphide. This separation of the two sub-groups from one another is neither easy nor always perfect; it may necessitate a repetition of the treatment according to the nature and relative proportions of the elements to be separated, whilst the presence or absence of mercury, tin, or cadmium has a very important bearing on the success of the operation. Yet the directions are condensed into a few lines (p. 219), not even the concentration of the reagent or the time of digestion being given. Considering that, on the other hand, the determination of, *e.g.* carbon dioxide occupies some 18 pages, it must be admitted that the treatment of the subject is not uniformly thorough.

The systematic weeding-out from the literature of antiquated methods is a cause which all scientific authors, especially those engaged in teaching, should have at heart. A widely-circulated book like the present might well contain an *Index*

Expurgatorius of methods that have been definitely proved to be untrustworthy. The separation of selenium from tellurium by cyanide fusion (p. 262) is one of the processes that have outlived their usefulness. The description of the procedure is followed by a note to the effect that it gives "slightly low results for tellurium and high values for selenium"; but, since the two elements can be separated more accurately by other methods, there seems to be no valid reason why this process should still be given. It is far better to err on the side of incompleteness while selecting only the very best from the mass of available material, than to retain methods that have been condemned by more than one investigator.

W. R. SCHOELLER.

ARTIFICIAL SILK. By Dr. FRANZ REINTHALER. Enlarged and Revised Edition. Translated from the German by F. M. ROWE, D.Sc. Pp. xii+276. London: Chapman & Hall, Ltd. 1928. Price 21s. net.

This book is not merely a translation of Dr. Reinthaler's well known monograph, but is a revised and enlarged English edition, in the preparation of which both author and translator have collaborated. The result is a most useful and practical book on Artificial Silk in every aspect of the subject, and we are indebted to Professor Rowe for making this work available to English readers. A considerable deal of new matter has been introduced, together with a large number of illustrations, so that the book is somewhat longer and more up-to-date than the original. It is now a very complete summary of the manufacture, properties and uses of all types of artificial silk, including the ether silk of Lilienfeld. The object has been to give some account, at least, of all phases of the subject, and in this the authors have been very successful. The viscose process, exceptionally, has been treated in much greater detail. The illustrations of machinery represent almost exclusively those of German manufacture, as it is felt that the corresponding British products are sufficiently illustrated in E. Wheeler's "The Manufacture of Artificial Silk, 1928." A number of British and foreign companies have co-operated by supplying information and specimens for illustration.

The treatment of each process is systematic, beginning with the raw material, preparation of the cellulose, dissolving, spinning, finishing, etc. The "viscose process" occupies 50 pp., "artificial silk from cellulose compounds," in which are included acetate silk and ether silks, 23 pp. Chapters follow on the properties of artificial silks, on testing and on dyeing artificial silks and staple fibre, with four final chapters dealing with applications and uses of artificial silks and the economic position of the industry. The chapters dealing with manufacture are especially well done, and they include a brief account of Dr. Lilienfeld's cold viscose process (Eng. Pat. 212865), by which artificial silk can be produced of a tenacity exceeding that of natural silk, the strength being greater than 5 grm. per denier. The Lilienfeld ether process is also given with some detail and, throughout, the properties of this silk are included among those of the other types.

The chapter on dyeing does not attempt to detail processes, but gives a very useful account of the different problems and difficulties that arise in dyeing, and indicates the most satisfactory methods to employ in order to avoid faults. The chapters other than those dealing with manufacture are a little more open to criticism for two reasons. In the first place the authors' desire is obviously to make the book available to the non-scientific reader, for which purpose matter is introduced which is often the reverse of explanatory: secondly, the translator often follows the German idiom so closely that the style becomes confused. Two examples of the former may be quoted—"Now that the action of acids and alkalis on cellulose has been considered, the behaviour of salts formed by the action of acids on metals, oxides and hydroxides, is of interest" (p. 10). And again—"The copper number represents the number of grms. of copper absorbed from Fehling's solution by 100 grms. of dry commercial cellulose" (p. 34). The use of the words "absorbed" and "commercial" is certainly misleading.

The book contains about 30 per cent. more matter than the German edition, but, speaking from memory, we should say that it is more than twice as thick and three times as heavy. In these days of many books and limited accommodation it is a great pity that so much unnecessary bulk and weight should be put into a volume of this sort. The paper employed is needlessly thick, and the case in which it is bound is far too light, a combination which will certainly not make for durability if the volume is much used. On taking down, at random, a book of the same thickness, Mellor's "Modern Inorganic Chemistry," it was found to contain three times as many pages and to be far more comfortable to handle.

These, however, are minor criticisms. The book is an admirable, up-to-date treatise, excellent both in its completeness and in the terseness with which each section is handled.

C. DORÉE.

MOLECULAR REARRANGEMENTS. By C. W. PORTER, Professor of Chemistry in the University of California. 167 pp. New York: The Chemical Catalog Company, Inc. 1928. Price 4 dollars.

Organic Chemistry has just celebrated its hundredth birthday. In 1828 Wöhler described the first synthesis of an organic compound from an inorganic source, and observed the first molecular rearrangement namely, that of ammonium cyanate into urea:

$$NH_4CNO \rightleftharpoons CO\begin{matrix} NH_2 \\ NH_2 \end{matrix}$$

Since then it has been shown by Walker, in 1895, that the reaction is reversible, but the question how and why ammonium cyanate rearranges itself into urea is still unanswered. To Wöhler's molecular rearrangement numerous other cases have been added during the last hundred years, involving migration from carbon

to nitrogen, from nitrogen to carbon, from carbon to carbon, from oxygen to carbon, etc. Each individual case has been studied with care and skill involving the work of such masters of organic chemistry as Kolbe, Hantzsch, Meerwein, and others, but the mystery still remains unsolved; we have still no real explanation of the why and wherefore of these molecular rearrangements. A very suggestive generalisation which seemed to account for the mechanism of these reactions was put forward by Robinson in 1920, but Professor Porter dismisses it as being unacceptable, since it involves "the formation of intermediates that cannot be isolated" (p. 96).

Professor Porter reviews the main phenomena of molecular rearrangements in the first five chapters, and then deals in the remaining two chapters with the problems of mutarotation, racemisation, and that greatest of all mysteries the Walden inversion. The facts are clearly mustered, and the material presented in a very attractive way. Professor Porter is to be congratulated on the lucid manner in which he has dealt with his subject matter. It is noteworthy that he has not included Fischer's acyl migration in a book which will certainly become a standard work dealing with molecular rearrangements. The writer of the review has always looked with doubt on the Fischer acyl migration and its extension by Perkin, and has given voice to his objections. The fact that Professor Porter has omitted this phenomenon tacitly supports this opinion.

M. NIERENSTEIN.

CONTEMPORARY DEVELOPMENTS IN CHEMISTRY. New York: Columbia University Press; London: Oxford University Press. Price 55s.

The volume under review is a collection of lectures given at Columbia University during the summer session of 1926, on the occasion of the Chandler Chemical Laboratories being opened. These lectures were delivered by some of the most famous American and European chemists, including Sir James Irvine, of St. Andrews. Each lecture is in itself a summary of highly specialised work, and a variety of subjects treated includes the Chemistry of Odorous Compounds by M. T. Bogert, Chemical Reactivity by T. F. Norris, the Rare Gases by R. B. Moore, Radicals as Chemical Individuals by C. A. Kraus, and the Periodic Table by B. S. Hopkins. Altogether twenty-five lectures are embodied in this remarkable collection.

Out of patriotism, special reference must be made to the two lectures by Sir James Irvine, the only representative of Great Britain. In these Sir James gives a summary of his classical researches on the carbohydrates, to which he has devoted a life time. Many other workers have followed in his footsteps, but it must always be remembered that the elucidation of sugar chemistry is due to the two outstanding leaders in this field of research, Emil Fischer and J. C. Irvine. Their names will remain as permanent landmarks in the history of Chemistry.

In addition to the two lectures dealing with carbohydrates, the book contains much material which can strongly be recommended to the student of chemistry.

M. NIERENSTEIN.

COLLOID CHEMISTRY—THEORETICAL AND APPLIED. By Selected International Contributors. Collected and Edited by JEROME ALEXANDER. Volume II. BIOLOGY AND MEDICINE. Pp. 1029. New York: The Chemical Catalog Company, Inc. 1928. Price $15.50.

This is the second volume of Alexander's remarkable compilation of papers by international authorities on pure and applied Colloid Chemistry. Fifty-seven chapters deal with a wide range of material bearing on biochemistry, physiology and medicine. The status of the volume is evidenced by the contributions of such scientists as Sir William Bragg, Wolfgang Pauli, G. Bredig, Andor Fodor, H. Schade, Du Noüy, Jacques Loeb—to name but a few.

The editor (with C. B. Bridges) opens with a long paper on "Some Physico-Chemical Aspects of Life, Mutation and Evolution." This is as full of interest to the colloid chemist as to the biologist. Related papers are "The Colloidal Systems of the Living Organisms" (Bottazzi), "The Physical Properties of Protoplasm" (Seifriz), "Protoplasm" (Heilbrunn), "The Colloidal Structure of Protoplasm and Protoplasmic Action" (Lillie), "The Physical Basis of Life" (Wilson). The botanist and bacteriologist, too, are catered for in contributions of a specialised character.

The magnitude of the volume forbids detailed notice of all the papers. Several, however, sum up the present position of research and knowledge in readable and authoritative fashion. Bragg briefly surveys the task of applying the new X-rays analysis to colloidal systems. "The X-rays can find something to measure even in the substances that most seem to deserve the title of amorphous." The problem of the ageing of colloids is discussed by Rocasolano, who briefly recapitulates his research on the variations in viscosity, surface tension, electric charge and conductance in colloid systems. Dhar and Chakravarti have an important communication on "Hydration and Viscosity of Sols in the Presence of Electrolytes." Surface tension measurements and magnitudes are treated in several papers, Bottazzi, in particular, giving very clear data regarding the isoelectric point in relation to surface tension. His paper is critical of others and well supported by his own data.

Yoe has two useful articles: "Nephelometry" and "Colorimetry," both summarising present technique and knowledge in these fields.

"Proteins as Colloids," by W. Pauli, is a masterly contribution, and is followed by a lucid summary by Fischer of his well-known work, in his paper "Lyophilic Colloids and Protoplasmic Behaviour." Brailsford Robertson's familiar views regarding the combination of proteins with acids and bases are given in a long paper which should be read in relation to the appendix to the book: Loeb's Pasteur Lecture of 1922 on "The Explanation of the Colloidal Behaviour of Proteins." Spiegel-Adolf discusses the physical chemistry of the heat-denaturisation of proteins, a subject of profound importance in colloid-bio-chemistry.

Enzymes are treated by several authors. Medicine receives very full discussion, and the outstanding contribution is Schade's "Colloid Chemistry and

Internal Medicine," a subject already associated with the author's name. The medical papers are by specialists world-famous in their fields, as, for example, Kahn on "Serum Diagnosis of Syphilis." Lobar pneumonia, cancer, acute inflammatory process, external superficial burns, concretions, tuberculosis, dust hazard, and the therapeutics of colloids, are dealt with in valuable papers. The colloid chemist even proposes a new theory of vitamins, von Hahn again presenting his views on the vitamin-containing substances, that these act not because of a chemically definable constituent, but because of their inherent surface or capillary activity.

Alexander has done a great service to all interested in Colloid Chemistry. Every field of modern scientific research reacts to progress in the study of colloidal systems, and in the present volume it becomes abundantly evident that the organic chemist, biochemist, physiologist and medical practitioner, must, if he keeps up-to-date, follow the new implications and applications of colloid studies in his own subject.

The book is printed, illustrated and bound in commendable fashion, and printing errors are few. As a store of information and as a summary of modern work it will take its place in all laboratories where colloid study has any part in teaching or research.

WILLIAM CLAYTON.

Publications Received.

INDUSTRIAL CARBON. By C. L. MANTELL. London: Chapman & Hall. Price 21s. net.

BACTERIOLOGY. By F. W. TANNER. London: Chapman & Hall. Price 22s. 6d. net.

RECENT ADVANCES IN HAEMATOLOGY. By A. PINEY. London: J. & A. Churchill. Price 12s. 6d.

DIZIONARIO DI MERCEOLOGIA E DI CHIMICA APPLICATA. 5 Ed. Vol. I. (A to C). Price Lire 60.

A POCKET BOOK FOR CHEMISTS. By T. BAYLEY. 9th Edition. E. & F. N. Spon, Ltd. Price 8s. 6d. net.

TREATISE OF INORGANIC AND THEORETICAL CHEMISTRY. Vol. IX. By J. W. MELLOR. Longmans. Price 63s. net.