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 2nd. The President, Mr. E. Richards Bolton, F.I.C., was in the chair.

The Hon. Treasurer presented the accounts of the Society for 1926, and votes of thanks were passed to the Hon. Treasurer and the Hon. Secretary.

Messrs. Marreco, Houseman and Brandon, Chartered Accountants, were appointed auditors of the Society's accounts for 1927.

The President delivered his Annual Address. Mr. A. Chaston Chapman proposed 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 Mr. E. M. Hawkins, and the motion was carried.

The following were elected as Officers and Council for the year 1927:—

President.—E. Richards Bolton.

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

Vice-Presidents.—R. L. Collett, C. H. Cribb, John White.

Hon. Treasurer.—Edward Hinks.

Hon. Secretary.—F. W. F. Arnaud.

Other Members of Council.—L. K. Boseley, H. E. Cox, John Evans, J. Golding, J. T. Hewitt, H. T. Lea, E. K. Rideal, W. H. Roberts, E. H. Merritt, W. H. Simmons, M. S. Salamon and James Wood.

An Ordinary Meeting of the Society followed the Annual Meeting, the re-elected President, Mr. E. R. Bolton, F.I.C., being in the chair.

Certificates were read for the first time in favour of: Messrs. Alfred George James Lipscomb, B.Sc. (Lond.), A.I.C., William L. Matthews, Sydney John Rogers, B.Sc., F.I.C., Ernest Fred Waterhouse, Harold William Webb, Arthur Samuel Wood, M.Sc., Ph.D., A.M.I.Chem.E., F.I.C.

Certificates were read for the second time in favour of: Messrs. William Gordon Carey, F.I.C., William Farrand Elvidge, B.Sc., A.I.C., Lewis Sidney Fraser, B.Sc., A.R.C.Sc., A.I.C., Frederick Percival Hornby, B.Sc., A.I.C., Gerald Roche Lynch, O.B.E., M.B., B.S., D.P.H., Eric C. Martin, George Gilmour Philip.

The following were elected Members of the Society: Messrs. Solomon Greenberg, F.I.C., Frank Crafer Ray, M.A., F.I.C., and Geoffrey Charles Matthews, B.Sc., A.I.C.

The following papers were read: "Cacao Butter Substitutes and their Detection," by A. W. Knapp, B.Sc., F.I.C., J. E. Moss, M.Sc., A.I.C., and A. Melley; "The Determination of Illipé Butter in Chocolate," by H. W. Bywaters, D.Sc., F.I.C., F. T. Maggs, M.Sc., A.I.C., and C. J. Pool; and "A Study of the Determination of Saccharin, Colorimetrically and by the Ammonia Process" (work done under the Analytical Investigation Scheme), by A. F. Lerrigo, B.Sc., F.I.C., and A. L. Williams, A.I.C.

NORTH OF ENGLAND SECTION.

At the Second Annual General Meeting of the North of England Section of the Society of Public Analysts, held in Leeds on February 26, the following were elected members of the committee for the current year: Dr. J. T. Dunn (Chairman), Messrs. J. Evans, C. J. H. Stock, H. Lowe, J. R. Walmsley, W. M. Mackey, A. R. Tankard, and J. R. Stubbs. *Hon. Sec.:* H. T. Lea.

Mr. Coates and Mr. Marshall were re-elected Hon. Auditors.

At the close of the General Meeting the following papers were read: (1) "The Effect of common Salt on Lime Water used for Egg Preserving," by James Miller, F.I.C.; and (2) "A Preliminary Note on the Determination of Sulphur Dioxide in Food," by James Miller, F.I.C.

Death.

We deeply regret to record the death of Dr. Arthur Crossley, on March 5, 1927. An obituary will be published in a subsequent issue.

Annual Address of the President.

(MR. E. R. BOLTON, F.I.C.)

Delivered at the Annual General Meeting on March 2nd, 1927.

LADIES AND GENTLEMEN,

In accordance with time-honoured custom, it is my pleasant duty, as your President, to make a report to you upon such matters as I may consider to be of special interest to the Society which have occurred during the past year.

In former years the annual general meeting has been held on the first Wednesday in February—a date which caused certain inconveniences, particularly with regard to the preparation of the accounts. To overcome such difficulties your Council has decided to advance the date for the future to the first Wednesday in March, thereby giving great relief to the officers of the Society, though arousing in my breast some feeling of superstitious apprehension as to what fate may await a President who has held office for *thirteen* months without giving the members an opportunity of expressing their wishes through the ballot box.

I am glad to report that the Society enjoys the most cordial relationship with the kindred scientific bodies, both at home and abroad; and that, notwithstanding the great multiplicity of Societies which call for the support of the analytical chemist, our membership continues steadily to increase, 39 new members having been elected to the Society during the past year. On the other hand, we have lost 6 members through death, and 7 through resignation, making the net increase 26. This brings our total membership up to 541, of which 53 are members of the North of England Section.

The North of England Section, under the Chairmanship of Professor W. H. Roberts, with Mr. H. T. Lea as Honorary Secretary, represents a very active branch of the Society, formed, as you know, two years ago. Meetings are held at different towns in the area, and in January last the Honorary Treasurer and I attended a meeting at Leeds, when a most interesting discussion took place on the Preservative Regulations.

Those whose death we regret to record are: Sir John Burchmore Harrison (Obituary, ANALYST, 1926, **51**, 223); Richard Bodmer (Obituary, ANALYST, 1926, **51**, 381); Lester Reed (Obituary, ANALYST, 1926, **51**, 435); John J. Broadbent; Sir William Augustus Tilden, and John Webster.

Concerning the first three, you will find in our Journal, under "Obituary Notices," words of appreciation written by those who knew them; while of John J. Broadbent we have no information; so that it only remains for me once more to deplore their loss.

Sir William Tilden, who was one of our honorary members, passed peacefully away on the 11th December at the ripe age of 84. The positions he held and the honours he received were many and well-deserved. He was a student of Hofmann,

and from his early days was associated with many famous chemists, so that it is not surprising to find among the most interesting of his works those entitled "*Sir William Ramsay: Memorials of his Life and Work*," and "*Famous Chemists: The Men and their Work.*" The group of chemists who attended his funeral at Northwood included seven members of the Society, on which occasion, as your representative, I joined with them in paying to him our last respects.

John Webster died suddenly on January 20th, having worked up to the end. He was a contemporary of my own, and I have known him since the days when he was an assistant to Sir Thomas Stevenson. His nature was such that the anxiety and responsibility inseparable from the position of Home Office Analyst must have left their mark upon him. It was fitting that his old colleague, Mr. Hinks, should have represented the Society at his funeral.

While we mourned the loss of our own countrymen, we had an opportunity of expressing a tribute of admiration and respect to the memory of Cannizzaro, whose mortal remains were borne to Palermo; this was on the occasion of the Second National Congress of Pure and Applied Chemistry, in May, when Mr. A. H. Bennett, as the Society's representative, presented an address to our Italian confrères.

Our Journal, THE ANALYST, this year has reached 654 pages—the largest that has ever been. Forty papers were published, 30 notes on analytical and similar subjects, 40 legal notes, 23 reports of public and government analysts at home and abroad, 36 Government reports, 498 abstracts, and 71 reviews.

A list of the papers will be recorded in the Journal in due course.

Of these papers, 15 were concerned with the examination of food and drugs, 2 with organic analysis, 9 with inorganic analysis, and the remainder with apparatus and with subjects of general interest.

During the year, 29 papers were read at meetings of the Society. At a special meeting, held in June, Mr. A. Lucas gave, to a crowded and appreciative audience, an account of his research work among the tombs of Egypt. Egyptologists owe a debt of gratitude to Mr. Lucas for the attention he has given to their problems from a chemical point of view.

The question of the presence of traces of arsenic in foods has been the subject of several interesting communications. In the early part of the year, Dr. H. E. Cox read a paper on the arsenical contamination of apples, and Dr. Shutt—the Dominion Analyst for Canada—followed later with a communication on the presence of arsenic in Canadian apples. Mr. A. Chaston Chapman then added shell fish and crustacea to the foods in which arsenic may be present as a natural constituent, and thus the question arises as to the conditions of combination under which arsenic may cease to be toxic, and, for all we know, become beneficial. Mr. Chapman also showed that shell fish may contain distinct traces of lead and other metallic impurities, and here again the same question as to the condition in which the metal is present requires elucidation. The subject might provide an interesting investigation, but, in the meantime, there is no reason to eschew the delectable shell fish which have nourished our forefathers for ages past. In

this connection mention should be made of a useful method for determining copper in foods devised by Dr. Lampitt and his collaborators.

Among the more noteworthy contributions to inorganic analysis were a paper by Dr. B. S. Evans and Mr. Clarke on the determination of mercury, one on phosphoric acid by Mr. Gunner Jörgensen, and a further contribution by Dr. Schoeller and Mr. Jahn to the analytical chemistry of tantalum, niobium, and their associates.

In December we had the pleasure of hearing an authority on a special branch of science when Dr. Savage gave us a paper on the bacteriological examination of food and water. Papers such as these, by men who are recognised as masters of their subjects, have been welcomed by the Society, judging by the attendances at the meetings, and it is hoped to make arrangements from time to time for other papers on similar lines by well-known authorities.

Last year Mr. Burford suggested that an outline should be published in THE ANALYST of the various standard methods of analysis adopted by different organisations. On consideration, however, it was found impracticable to publish the methods in full, but, as an alternative, a bibliography of them, with references, was decided upon, so that our readers would, at least, know where to turn for such specialised information. The first of these bibliographies—that on the analysis of Leather and Tanning Materials, compiled by Mr. R. F. Innes—was published in the February issue of the journal. The preparation of the bibliographies of other standard methods is in progress.

Articles of scientific interest, having only a very remote analytical interest, frequently appear in scientific and literary journals, and while they are often unsuitable for abstraction, they may contain useful and specialised information. Accordingly, at the suggestion of Mr. Cribb, their titles and a brief synopsis of their contents are now published in THE ANALYST under a special heading, thus providing another new feature.

The publication of the Decennial Index of THE ANALYST for the years 1916–1925 is a fitting memorial to close the fiftieth anniversary of the Society. A comparison of its pages with those of the modest little General Index to the first twenty volumes will show not only the rapid advance made in analytical chemistry during the later period, but also the continual expansion in the interests of the Society.

The Publication Committee paid much attention to the form the entries should take, and decided that for the purposes of the Society the arrangement as now printed had many advantages, although this involved the re-indexing of the ten volumes covered by the Index. This work was done by Miss M. B. Elliott, and the Council were pleased to pass a unanimous vote of thanks to her for the efficient way in which she carried out the task. Every busy man knows how much time is spent in hunting through volume after volume for a particular reference, and in view of the tremendous facilities afforded by an index of this type, it is a matter of regret that not more members of the Society have availed themselves of the special terms offered to them for its purchase. There is, however, still

an opportunity for members to provide themselves with this invaluable book, and I hope that those who do not already possess it will support the Society and its work by ordering it now.

The brief outline I have given regarding THE ANALYST, and its Index, will lead you to seek the guiding hand to which we are indebted for the growing popularity of our Journal, and most of you will have rightly turned your thoughts to the Editor, Mr. C. A. Mitchell. Nothing that I can say can convey to you any idea of the untiring energy which he ever shows in his dual capacity of Editor and Secretary.

I have already referred to Miss M. B. Elliott in connection with the Index, and lest it should be thought that this is her only contribution to the welfare of the Society I must tell you that her interest and activity on our behalf extend far beyond her office of Advertisement Manager of THE ANALYST.

The Analytical Investigation Scheme, which to a large extent fell into abeyance during the War, has fully recovered its vitality, and I am pleased to say that we have six subjects now being investigated, and several under consideration. One communication—that of Mr. Harold Toms, on the crystalline bromides of linseed oil—has already been brought before the Society. This is a particularly interesting piece of work, since it has established the chemical identity of the most insoluble bromide of many drying oils. Pioneer work of this kind is of the greatest value, as it often affords a sound scientific ground upon which analytical tests can afterwards be based.

The financial side of the scheme, however, is not altogether in a satisfactory state, for although the needs are small, the capital fund is not sufficient to provide, out of dividends, such assistance as might, with advantage, be given under the scheme. I, therefore, take this opportunity of inviting subscriptions from those who have prospered by their own analytical investigations. Those not so fortunate can give equally valuable contributions by suggesting subjects for investigation; while those who have the necessary time and skill—and the skill is important—are asked to undertake the investigation of the suggested subjects.

There seems to be an impression among a few that the scheme is in existence solely for the purpose of giving financial aid to investigators; let me correct the idea, for the main object is to provide a mechanism for recording subjects which urgently require investigation, and a means of placing the investigator in touch with those who have knowledge of the special subjects; overlapping of work is thus often prevented and co-operation encouraged. In the great majority of cases, no financial aid is asked or given, but money is nevertheless very necessary in order to purchase expensive chemicals or even apparatus, where such are not accessible to the investigator, and where, in the opinion of the Committee, the case merits such expenditure.

The Standing Committee on the Uniformity of Analytical Methods has now three offspring, in the form of sub-committees dealing with: (1) Essential oils; (2) Milk products, and (3) Dirt in milk.

I should not like the absence of any report to be attributed to a want of activity; quite on the contrary. The Committees have held many meetings and the members thereof have conducted a most prodigious number of analyses with the object of verifying the accuracy of the methods which they propose to adopt. However, before the year is out, it is anticipated that some very useful recommendations will be published, which recommendations will not only be of the greatest help to the analyst but will save manufacturers from many an expensive dispute.

Last year my predecessor referred to the new edition of the *British Pharmacopoeia*, still in course of preparation, and expressed satisfaction that the General Medical Council had invited us to select two representatives of the Society to put forward our views. Since that time our representatives, Mr. A. Chaston Chapman and the Honorary Treasurer, have had an opportunity of attending a conference called by that Council.

It must be realised, however, that there is so much to be done that the publication of the volume at an early date cannot be reasonably expected.

During the past year I have had not only the honour of occupying this chair, but I have been a vice-president of the Institute of Chemistry, and Chairman of the Legal and Parliamentary and the Public Appointments Committees of that Institute. Thus, with a few of my colleagues who are also members of both Councils, I have been present at the discussions on several matters of importance and interest to both bodies. On some of these matters the Institute and the Society have taken independent action, each in accordance with its distinctive position in the sphere of chemical politics—the Institute representing the professional qualifying and registration body of chemists, and the Society acting on behalf of those who are more particularly engaged in analytical and consulting practice. In other matters, where joint action was considered desirable, we have co-operated as in former years. In April last, the Councils of both Societies felt called upon to lodge objections to the registration—under the provisions of Section 20 of the Companies Consolidation Act, 1908—of a company which appeared to be inimical to their interests by constituting a new body with the same or similar objects. The proposed registration does not appear to have been allowed.

Many members will be aware that the Public Appointments Committee of the Institute has collected a great many data regarding the varying conditions of appointment of Public Analysts and other matters concerning the administration of the Sale of Food and Drugs Acts.

The Councils of the two bodies have concurred in the preparation of a series of questions to be submitted for legal opinion before taking action thereon. Public Analysts are particularly concerned with the fact that while the effect of new regulations tends invariably to increase their work and responsibility, little regard—and in most cases no regard—is paid by the local authorities to the amelioration of their terms of appointment. I am hardly in a position to deal with the whole matter at the present stage, but I should like to acknowledge especially the services rendered by Mr. Cribb, Mr. Hawkins, Mr. Hinks and Mr. Richards in collating and reporting upon the returns received in answer to a somewhat

comprehensive questionnaire issued towards the close of 1925. The Society, moreover, concurs with the Institute in the view that the time has come—and indeed is past—when the position of Public Analyst should not be looked upon as coming within the functions of a medical practitioner, unless he has qualifications for the post quite independent of his medical qualifications. While the profession of chemistry is quite as distinct an entity as that of medicine, there is nothing to prevent a man from becoming qualified in both professions, and there are, in fact, a few who successfully combine the two. I go so far as to say that it is the duty of the Ministry of Health to impress upon the local authorities the fact that the training of a medical practitioner does not render him one whit more fit to carry out the analysis of a food, or a water, than does the training of a Public Analyst enable him to prescribe for a sick person.

Fortunately the Medical Officer, as a rule, has an appreciation of the duties of the Public Analyst, and there is now-a-days a general tendency for these two officers to work together in ever increasing harmony, greatly to their mutual advantage and to the public good. There are, however, occasional exceptions. I have in mind a recent surprising case where the function of analyst appears to have been relegated to a medical officer.

Whether this gentleman undertook the task willingly, or not, I do not know, but, as a result, the sanitary inspector refused to give a certificate for the water supply to a new house, basing his refusal on a schedule of unconventional and worthless observations, purporting to be an analysis, wholly inadequate to show whether the water was bad or good. This so-called analysis, which was unsigned, was alleged to have been made by the medical officer, who did the work for nothing, so he at least knew its value. The public analyst subsequently certified the water to be satisfactory, and the attention of the Ministry was directed to the case.

One can only shudder to think of the possibility of a dangerous water supply being passed, as a result of such an examination, and one is led to wonder whether, if the Medical Officer be expected to produce a certificate of analysis, the Public Analyst might not just as well be asked to prescribe for the ailments of the consumers of the water, which is hardly more absurd than what has happened.

The Institute and the Society have also both been consulted in the amendment of legislation as represented in the new Fertilisers and Feeding Stuffs Act, 1926, and I have the honour of representing the Society on the Advisory Committee which has now been constituted in accordance with the recommendations made by the Departmental Committee on which I have already served.

The Minister of Agriculture has shewn a high appreciation of the importance of science in agriculture in appointing on this Advisory Committee of 21 members, 9 scientists, of whom 6 are members of our Society.

In connection with agricultural analyses, the Government Chemist, at the instigation of the Ministry, called a committee, to decide upon a much-needed standard method for the determination of fibre in feeding stuffs, on which Mr. G. Rudd Thompson represented the Society and Mr. Arnaud, the Institute of Chemistry.

The Committee met in April last year and, having surveyed the evidence before them in the light of their own special knowledge, decided upon a process. A uniform sample of feeding stuff was then distributed to the members of the Committee, on which to make a large number of determinations by the agreed process. The results obtained have been treated statistically, and a report will shortly be ready dealing with these results. In addition, the report will contain the results of further experiments, in which the important question of sampling has been explored. This report will increase the debt of gratitude which every analyst owes to the Government Laboratory and to its chief, our Honorary Member, Sir Robert Robertson.

When the Society was founded over 50 years ago, one of the main objects was to promote the efficiency and proper administration of the contemplated laws relating to the repression of adulteration. These laws became the Food and Drugs Act of 1875, the main provisions of which are still in force, though augmented by additional orders and regulations so well known to all of us who are concerned with food and drugs.

This year we enter upon a new era, brought about by the passing of the Preservatives, etc., in Food Regulations, 1925, and I feel that it would be of interest briefly to review the half century recently concluded, by considering the position which existed when the Society was formed as compared with that which exists at the present day with regard to this matter, and the course of event which has led up to it.

We are accustomed to record the 7th August, 1874, as the day when the Society was founded, because that was the day on which 28 Public Analysts attended a meeting at the City Terminus Hotel, Cannon Street, with the object of forming a Society, and, after considerable discussion as to title, they baptised it as the Society of Public Analysts. All analysts *in actual practice* were eligible for election as *members*, and it was provided that those who were *assistants* to such analysts could be enrolled as *associates*.

In passing, therefore, it is of interest to note that in our first Minute Book we find an entry to the effect that a youth, Bernard Dyer by name, was thus admitted as an *associate* member, by reason of his position as assistant to Dr. Augustus Voelcker, and I am sure that the Honorary Secretaries—Heisch and Wigner—who recorded this minute would have felt proud men indeed had they known that they were then entering the name of one to whom the Society would owe more than to any other—a man dear to us all and of whom we are justly proud.

Dr. Dyer has promised to act as historian and is now preparing for us an account of the Society's doings from its inception to the present day, and this is indeed something for us to look forward to.

In those early days the adulteration of food was of a very crude character, and although the methods of chemical analysis then employed were also somewhat primitive, I venture to suggest they were by no means inadequate in exposing fraud; in fact, the analyst was able to diclose the presence of an adulterant much more rapidly than he can to-day. The reason is not far to seek; it lies in the

fact that—with the exception of the addition of water to milk—unscientific adulteration is quite exceptional.

Unfortunately there came a period—now happily passing away—when certain other analytical chemists lent their aid to those who practised the subtle forms of sophistication, thereby engendering a feud between themselves and the public analyst.

This Society can claim a large share in bringing about a better understanding between the official and other analysts, so that in 1907 we find that the preponderance of our members who practised all branches of chemistry was so great as to call for the addition to the original title of the Society of the words: "*and other analytical chemists.*" Those engaged in all branches of analytical, consulting, and technical chemistry who have been banded together in one society have come to a very perfect understanding of each other; they are now accustomed to help each other not only in their scientific difficulties, but have learnt a certain sympathy with one another in all the duties arising out of the profession in which they are engaged.

I feel that I am myself in a unique position in being able to understand the relationship of the Public Analyst to the other analytical chemists, in so far as, not being a public analyst myself, I have for ten years as your Honorary Secretary, fought the battles of the former on many a field, and it has been my privilege also to represent the other and larger side of the Society—the other analytical chemists. Consequently, I can view the scene without undue prejudice in either direction, but with sympathy for both, and what I see is an ever-increasing co-operation between the Public Analyst and the manufacturing chemist. But the position has not yet reached perfection, for it seems to me a pity that a feeling should have grown up in certain quarters that the Public Analyst is the enemy of even the honest manufacturer. This is, to some extent, a result of the extreme position which has occasionally been taken up by individual Public Analysts in opposition to the views of the majority of their colleagues. The position of these extremists reminds one of the pugnacious theologian who asserted, "Orthodoxy is my doxy, and heterodoxy is other persons' doxy." Even now there are several questions upon which conflicting views are held by Public Analysts, and the fact that it is still possible for a tradesman to be prosecuted in one county for the sale of articles which he can sell with impunity—if not with approval—in another county does not tend to increase public respect for either the law or the analytical profession.

I find myself in very great sympathy with the honest manufacturer who is pilloried in the police court in order that some legal point, such as the labelling or description of a perfectly wholesome foodstuff, may be decided, and who is put up as a defendant charged with the crime of adulterating a product honestly sold in good faith.

I should, therefore, like to pay a tribute of admiration and approval to those local authorities who—on the occasions when offences appeared to have been either accidental or unintentional—have seen fit to issue a warning or demand an explanation before deciding to institute proceedings.

We find this line of thought reflected in the new Fertilisers and Feeding Stuffs Act, 1926, in a provision whereby certain deficiencies, which, in the old Act, constituted criminal offences, can now become the subject only of civil claims. Thus a reputable manufacturer is protected from the slur which is inseparable from criminal prosecution.

The application of this principle to the sale of food and drugs is beset with difficulties, but is worthy of consideration.

For years it has been widely urged that there should be a court of reference where disputed points could be agreed under more suitable conditions than before a magistrate, but, until the necessity of this court is admitted by those who have the power to establish it, these unfortunate conflicts of evidence between public analysts seem likely to continue.

In fact, such a court of reference was recommended by the Select Committee on Food Products Adulteration in 1896, and this recommendation was alluded to in the final report of the Royal Commission on Arsenical Poisoning issued in 1903, where a further recommendation was made that a Board of Reference consisting of a scientific body capable of giving advice to the Local Government Board should be elected.

The British people have a world-wide reputation for square dealing. Napoleon Bonaparte called us a nation of shop-keepers; he might have added "and honest ones, too," for those given to fraud are fortunately few.

Moreover, we owe much to the free press of the country, which by its habit of continually deprecating our actions spurs us on to better things. But do not let us be led astray by alarming articles which so magnify the few cases of food adulteration as to lead the public to suppose that we live in the days when Accum wrote the book popularly known as *Death in the Pot*; that was in the year 1820. May I read a few lines from *Blackwood's Edinburgh Magazine* in reviewing Accum's famous book?—"So inextricably are we all immersed in this mighty labyrinth of fraud that even the vendors of poison themselves are forced, by a sort of retributive justice, to swallow it in their turn. Thus the apothecary, who sells the poisonous ingredients to the brewer, chuckles over his roguery and swallows his own drugs in his daily copious exhibitions of brown stout. The brewer, in his turn, is poisoned by the baker, the wine merchant, and the grocer. And, whenever the baker's stomach fails him, he meets his *coup de grâce* in the adulterated drugs of his friend the apothecary, whose health he has been gradually contributing to undermine, by feeding him every morning on chalk and alum, in the shape of hot rolls."

These were the "good old days"; let us see what improvement has been achieved since then:—

No real statistics are available until 1877, when we find as many as 19·2 per cent. of the samples analysed are reported as adulterated. This proportion has gradually fallen, and in 1924 the lowest figure, since the passing of the Food and Drugs Acts, is reached, namely 5·9 per cent. The last report of the Ministry shows a slight increase up to 6·5 per cent., though London and the 40 largest provincial towns yield only 5·5 per cent.

There are now 106 Public Analysts in the country who, last year, examined nearly 120,000 samples, and the officers who took these samples presumably had regard to the likelihood of adulteration in selecting them. Furthermore, it is generally the case that a number of adulterated samples find their source in one particular vendor.

My impression, therefore, is that the food of the country was never in a purer state than it is at the present time, and surely it is a further proof of this that we find quite a large proportion of the prosecutions do not apply to cases of fraudulent adulteration at all, but to such debatable matters as alleged incorrect description or nomenclature.

It is mainly to the efficiency of the Public Analyst that we owe this highly satisfactory condition of our food, and any lapse in his activities or diminution in the number of samples examined would be a retrograde and even dangerous step.

I would urge the manufacturer to avail himself of the services of a chemist, to maintain the purity of his product and to advise him so that he may not unknowingly contravene the law.

In the past, however, there was in the minds of the manufacturers, as well as the authorities, some feeling of uncertainty as to the type of chemical preservative which might be used, and, rightly or wrongly, boric acid was tentatively admitted from sheer absence of information as to whether it was harmful or not. Consequently, the formation of a Committee to enquire into the matter was welcomed by all parties concerned. As a result we have the Preservatives, etc., in Food Regulations, 1925, which have been the subject of much adverse criticism, as may well be expected of any change so drastic as that for which they provide.

For my own part, I am well satisfied that these regulations place both the analyst and the manufacturer in a very much happier position than they were before. Both parties have in these regulations "a book of rules" which prevents an unreasonable analyst from extremes of unreasonableness, and confines each and every manufacturer within limits beyond which his competitors cannot reap an unfair advantage, without risk of punishment.

Such regulations are bound to be full of anomalies and inconsistencies, which however, the Ministry has shewn itself ready to consider and remove where possible. Let us, therefore, help with feasible suggestions for the removal of difficulties, and above all refrain from destructive criticism, for, surely, it is the only wise policy of all, both analysts and manufacturers, to work to the spirit of the regulations where they cannot obey the letter.

An example of what the Ministry is doing to help the analyst is illustrated by the publication on March 1st, 1927, of Report No. 39 by Dr. Monier-Williams, entitled: "The Determination of Benzoic Acid in Foodstuffs" (see p. 153). This report contains a summary and criticism of existing methods and concludes with a detailed description of a new method, which has given good results in the case of certain fruits and vegetables (see p. 229). The method is not put forward as official, but is merely suggested as likely to be of value in the examination of

foodstuffs. This report is to be followed shortly by a further official publication by the same author dealing with sulphur dioxide in foodstuffs.

The increasing interests of the Society have caused me to touch on a wide range of subjects, and I must ask your forgiveness for taking up so much time. There is one subject which I have left to the last because it is the one which has made the greatest impression upon me, and that is the extraordinary good fortune which has given to the Society two such officers as the Honorary Treasurer and the Honorary Secretary; even I, who have known them both for over a quarter-of-a-century, have nothing but praise for them.

When I undertook the responsible position with which you have honoured me, I felt I could count on them for that friendship and support which they have given me in such full measure, and I want to thank them most heartily before you all. I also wish to thank the Council and all the members of the Society who have given me so much kindness and support, and thus made the past thirteen months very pleasant ones for me.

Ladies and gentlemen, I thank you for having listened to me so patiently.

The Immersion Refractometer and Its Value in Milk Analysis.

By G. D. ELSDON, B.Sc., F.I.C. and J. R. STUBBS, M.Sc., F.I.C.

(*Read at the Meeting, February* 2, 1927).

Introduction.—At somewhat frequent intervals during the last few years reference has been made in the Courts, especially in the North of England, to the examination of milk by means of the Immersion Refractometer. Some of the statements seem calculated to convey the impression that the refractometric test is of some outstanding significance, yielding information surpassing in reliability that obtained from the other tests which are usually made in the course of the analysis of milk. Not only this, but in those instances where the solids-not-fat are below the standard, but not to a very great extent, it appears to be claimed that the refraction of the serum of milk will settle without any doubt whether a milk has been watered or is unadulterated but of poor quality. In one case, at least, the printed report of the evidence for the defence in a charge of deficiency of solids-not-fat in milk seemed to bear the construction, without expressly saying so, that the test was of such a nature that it could distinguish extraneous water, although it is true that one of us subsequently obtained a definite admission in published correspondence, that so far as this last point is concerned the test stands on no different grounds from other tests. A similar claim is possibly made by G. A. Ferguson in the minutes of Evidence of the Scottish Inter-Departmental Committee on Milk, 1922, (Question No. 2492).

These claims, explicit or implied, attracted our attention, and we decided to subject the refractometric test to a careful examination with the idea of helping to arrive at its precise value in milk analysis. The practical part of this paper is an account of the work done with this object in view.

Since the Immersion Refractometer was introduced by Zeiss, about 1900, it has been put to somewhat extended use in various directions, although published work on this subject has mostly appeared on the continent of Europe and in America. Very little original work seems to have been published in this country. The delicacy of the indications of this instrument, coupled with its ease of application and its widespread utility for the determination of the strength of solutions, has attracted the attention of numerous workers. Its importance is indicated by the fact that use is made of it by the American Association of Official Agricultural Chemists in an official method for milk analysis (*Methods of Analysis*, 1925, p. 264) and for the determination of the strength of solutions of ethyl alcohol (*Ibid.*, p. 369).

At the present time, though not unmindful of its importance in other directions, we are, for the reasons given above, more particularly interested in the application of the instrument to the examination of milk serum.

The following is a list of the more important papers which have appeared from time to time relating mainly to the application of the Immersion Refractometer to the examination of milk:—

H. Matthes, ANALYST, 1903, **28**, 91.
H. Matthes and F. Muller, ANALYST, 1903, **28**, 241.
A. E. Leach and H. C. Lythgoe, ANALYST, 1905, **30**, 57.
E. Ackermann, ANALYST, 1907, **32**, 117.
C. Mai and S. Rothenfusser, ANALYST, 1908, **33**, 400.
C. Mai and S. Rothenfusser, ANALYST, 1910, **35**, 126.
B. Pfyl and R. Turnau, ANALYST, 1912, **37**, 450.
K. Alpers, ANALYST, 1912, **37**, 317.
G. Shutz and L. Wein, ANALYST, 1913, **38**, 500.
J. McCrae, ANALYST, 1914, **39**, 212.
R. Windisch, ANALYST, 1914, **39**, 256.
E. Ackermann, ANALYST, 1917, **42**, 328.
G. Wilhelm, ANALYST, 1917, **43**, 328.
J. F. Tocher, "*Variations in the Composition of Milk*," 1925.

PART I.

THE REFRACTOMETER.—The instrument used in these tests was the Zeiss Immersion Refractometer. It is probably sufficiently well known to render unnecessary any complete description here, more particularly as full instructions are supplied by the makers. Similar refractometers are made in this country by Messrs. Adam Hilger, by Messrs. Bellingham & Stanley, and by others. The experience of the writers would suggest that a certain amount of caution should be exercised before a final selection is made from the various instruments on the market.

The readings of the Zeiss instrument are on an arbitrary scale (-5 to $+$ 105), and fractions of a division are ascertained by means of a micrometer screw. A

table is supplied for the conversion of the scale reading into refractive index.* The instrument is supported on a stand with the prism at the lower end dipping into a small beaker containing the solution to be tested. The beaker is placed in a water bath maintained at a constant temperature and so arranged that white light is reflected through the bottom of the beaker into the instrument.

THE TEMPERATURE USED.—Wagner in the compilation of his tables used a temperature of 17·5° C., but this is not an easy temperature to maintain, especially in hot weather, neither is it one which has found general acceptance. Several workers have suggested a temperature of 20° C., as being more easily obtainable under all conditions, and this has been adopted by us. It was recommended by the Refractometry Committee of the London Section of the Society of Chemical Industry (*J. Soc. Chem. Ind.*, 1919, **38**, 399T) for general use in non-tropical countries.

The regulation of the temperature of the bath was obtained by means of the arrangement of an adjustable head of water, as suggested by Zeiss. Difficulty in securing a uniform temperature was, however, experienced which was eventually found to be caused by variations in the pressure of the gas supply. It was obviated by using a Gas Regulator supplied by Messrs. Griffins (Catalogue No. X 3605). By this means it was found easily possible to maintain a uniform temperature to within 0·1° C., for as long a period as required.

THE METHOD.—In view of the fact that so little seems to have been published in this country, and also for our own information, we thought that a considerable amount of preliminary work was very desirable and even necessary on the refractometer generally, before passing on to the examination of milk. We have attempted to obtain experimentally some knowledge of the capabilities and limitations of the instrument itself. But since, for our present purpose, it was to be used for milk, we decided that in these preliminary experiments also, milk should be operated on.

The milks used in all these different experiments, both in Parts I and II of the paper, were obtained from widely separated districts in the administrative County of Lancashire. Different sets were used in almost every case for each separate investigation. By this means we hope to have eliminated possible errors due to the abnormalities of any particular milk. In all cases the usual analysis of each milk was also made.

It is impossible to obtain a reading on the refractometer by immersing it directly in milk. It is necessary, therefore, to produce a milk serum. Various suggestions have been made for this purpose, among the more common of which are (1) allowing the milk to become sour and thus produce its own serum, or (2) coagulating with acetic acid, (3) with calcium chloride solution, (4) with carbon tetrachloride and acetic acid, or (5) with copper sulphate solution. For reasons which will appear later, the first is not allowable. Acetic acid does not always

* By changing the prism a wider range can be obtained.

produce a clot from which the serum can be filtered easily, and calcium chloride is troublesome, in that heating is necessary. After some trials the conclusion was reached that the preparation of the serum by the use of copper sulphate solution was, on the whole, the most satisfactory and convenient method. By its use clotting is immediate at ordinary temperatures and the filtration rapid. The copper sulphate method has therefore been adopted. It is also used by the Association of Official Agricultural Chemists, and in the recent work of Tocher. (*Variations in the Composition of Milk*, p. 5.)

PREPARATION OF THE SERUM.—The serum is prepared according to the A.O.A.C. method by adding to a quantity of milk one quarter of its volume of a solution of copper sulphate having a refraction of 36·0 at 20° C. The A.O.A.C. state that such a solution contains 72·5 grms. of crystallised copper sulphate to a litre. We have found that by using *A.R.* copper sulphate, prepared by the British Drug Houses, the correct figure is 71·5 grms. per litre. This amount of *A.R.* copper sulphate is weighed out, dissolved in water, and the volume made up to a litre. This solution should give a refraction of 36·00 and must be adjusted to this, if necessary, by the addition of water or copper sulphate.

For the coagulation, 20 c.c. of milk are mixed with 5 c.c. of copper sulphate solution in a 6 × 1 in. test tube, and the mixture is well shaken and filtered through Munktell's 11 cm. filter paper. When a quantity of about 10 c.c. has collected it is poured into one of the small beakers, which is then placed in the bath. After standing until the serum has attained the temperature of 20° C., the refraction is taken. For a routine method see page 197.

This method answers well so long as the milk is fresh. To measure the milk in the case of sour milks we employed a cylinder cut off sufficiently above the 20 c.c. mark to deliver about 20·5 grms. The question of slight variations in the amount of milk taken is dealt with in Table IV.

THE ACCURACY OF THE READING.—It has been found that the position of the eye makes a slight but noticeable difference in the reading. For this reason it is necessary that the reading should always be taken with the eye in the centre of the eyepiece. In order to show what variations, if any, would be found by different observers operating on the same milk, five operators each prepared six specimens of serum—all from the same milk. The results are given in Table I. The observers did not compare their results until after the whole of the readings had been taken.

TABLE I.

REFRACTIONS BY DIFFERENT OBSERVERS.

Observer.	Scale readings.					
No. 1. ..	37·40	37·40	37·40	37·40	37·40	37·40
2. ..	37·35	37·35	37·35	37·35	37·35	37·40
3. ..	37·25	37·30	37·25	37·30	37·30	37·30
4. ..	37·50	37·55	37·50	37·50	37·50	37·50
5. ..	37·40	37·40	37·40	37·45	37·45	37·45

The sera prepared by one observer were then read by a second one, and the results are given in Table II.

TABLE II.

DIFFERENT OBSERVERS READING THE SAME SERA.

Serum							
Prepared by observer	Read by observer	Scale readings.					
No. 1	No. 5	37·35	37·45	37·45	37·40	37·40	37·45
2	1	37·40	37·45	37·40	37·40	37·40	37·40
3.	2.	37·35	37·35	37·35	37·35	37·35	37·35
4.	3.	37·35	37·35	37·30	37·35	37·35	37·35
5.	4.	37·35	37·35	37·40	37·35	37·40	37·35

There seems to be some tendency, due doubtless to the different methods of setting the instrument, for some observers to read persistently higher than others. The difference is slight but definite. To obviate this, each observer should take the refraction of distilled water at 20° C., which should be 14·5, and make any necessary corrections in all his other readings. With the instrument in proper adjustment consecutive readings by the same person agreeing to within 0·05 of a division can be readily obtained, if reasonable care is exercised.

VOLUME OF LIQUID NECESSARY FOR THE TEST.—A milk serum was prepared and 1 c.c. placed in the beaker. A faint indication on the scale was obtained which was rendered more definite by inserting the prism further, or what amounts to the same thing, by raising the beaker until the edge of the prism rested on the bottom of the beaker. The quantity of serum was increased by 1 c.c. each time and the reading repeated. Two experiments were made and in each case it will be seen that the actual reading was not affected by the quantity of serum taken, but the depth of the part of the field in shadow was deeper as the quantity of serum increased, i.e. the reading was more definite. The results are given in Table III. Ten c.c. is about the most satisfactory quantity to use in the small beakers supplied.

TABLE III.

DIFFERENT VOLUMES OF SERUM.

Series I.		Series II.	
Amount of serum.	Reading.	Amount of serum.	Reading.
c.c.		c.c.	
1	37·5	1	37·5
2	37·5	2	37·5
3	37·5	3	37·5
4	37·5	4	37·5
5	37·5	5	37·5
6	37·5	6	37·5
7	37·5	7	37·5
		8	37·5
		9	37·5
		10	37·5
		11	37·5

THE EFFECT OF CHANGE OF TEMPERATURE ON REFRACTION.—It was thought desirable to investigate the effect of a moderate change of temperature, both above and below 20° C., on the refraction of milk serum. A series was prepared, and the refraction taken at 19°, 20°, and 21° C., and also at 15° C. and 25° C. The copper sulphate reagent solution and distilled water were also tested at the same time. It was ascertained that the effect was to increase the reading for milk serum by an average amount of 0·27 of a division for 1° C. fall, and to diminish it by the same amount for 1° C. rise in temperature. The corresponding variations for the copper sulphate solution and distilled water were found to be 0·25 and 0·20, respectively.

COMPARISON OF REFRACTION IN OPEN AND CLOSED BEAKERS.—For small quantities of liquid there is supplied a closed beaker and auxiliary prism. Two or three drops of serum amply suffice for the test. It is necessary in this case, also, to prepare a serum, for no reading is given if milk is examined directly. In these experiments a serum was prepared and a portion placed in the beaker in the ordinary way, while 2 or 3 drops of the same serum were applied to the auxiliary prism. Six samples compared in this way show that the two methods give identical results.

The disadvantages of using the auxiliary prism for milk serum, as compared with the open beaker, are the necessity of disconnecting the metal beaker and cleaning the two prisms for each observation, and, most serious of all, waiting each time an experiment is made for the combination to attain the temperature of 20° C., which has to be judged without a thermometer being inserted in the serum. Notwithstanding these drawbacks the auxiliary prism could be used if the amount of serum were limited, as the indications in both cases are the same.

PART II.

THE REFRACTOMETER IN THE ANALYSIS OF MILK.

VARIATION IN THE QUANTITY OF MILK TAKEN.—It is easy in preparing the serum of milk to keep the amount of copper sulphate solution constant, but not so easy to keep constant the amount of milk, especially when it is turning or has turned sour. An experiment was therefore made to ascertain what effect on the reading was detectable by a reasonable variation in the quantity of milk taken. Fresh milk was first used for the purpose, 18 c.c., 20 c.c., and 22 c.c. being taken and the serum prepared by adding 5 c.c. copper sulphate solution to each. No difference in reading could be detected with a variation of 2 c.c. above or below 20 c.c. A series of experiments was made in which sour milks were used and rather greater variations in the amounts taken were made, *viz.*, 17, 20 and 23 grms. which were weighed out. The results are given in Table IV.

TABLE IV.

EFFECT OF VARYING THE QUANTITIES OF SOUR MILK ON THE REFRACTION.

Sample.	17 grms.	20 grms.	23 grms.
No. 1	40·00	40·20	40·30
2	39·80	39·90	40·10
3	37·80	37·90	38·10
4	39·20	39·40	39·50
5	39·80	39·90	40·10
6	39·50	39·60	39·70

Here the variation in the amount taken affects the result to a small extent. Experiment has shown that the use of a glass cylinder for measuring sour milk enables a quantity to be taken which is sufficiently near the weight of 20 c.c. of fresh milk to make no appreciable difference in the refraction.

VARIATIONS IN THE QUANTITIES OF BOTH MILK AND COPPER SULPHATE.—It was found convenient in devising a method for routine examination to take 40 c.c. of milk in a 6 × 1 in. test tube marked at 40 c.c., and to add 10 c.c. of copper sulphate solution from an automatic pipette. Experiments were therefore made to ascertain the effect of doubling the quantity both of the milk and of the copper sulphate solution, whilst the relative proportions of each remained constant. It was found that the readings were identical in both cases.

ORDER OF MIXING.—Judging from the instructions given by the A.O.A.C. for preparing milk serum, it might be anticipated that it was necessary to add the liquids in a certain order; otherwise discordant results might follow. We therefore prepared a number of sera (*a*) by adding the copper sulphate solution to the milk, and (*b*) by adding the milk to the copper sulphate. These experiments show that it is immaterial in which order the liquids are added. In all our experiments (except in one half (*b*) of this series) we obtained the serum by adding the copper sulphate solution to the milk.

DO DIFFERENT FILTER-PAPERS AFFECT THE REFRACTION?—It has been suggested that the results obtained might depend, to a certain, extent on the kind of filter paper used to filter off the coagulum. For this reason four quantities of the same milk were taken, the copper sulphate solution added to each and then each portion filtered through a different make of filter paper, *viz.*, Munktell's No. 1 F; Schleicher & Schüll No. 595, Balston's Whatman No. 1 (an ordinary paper), and No. 5 (a very thick paper). The results, given in Table V, show that the use of different filter papers does not affect the refraction.

TABLE V.

THE EFFECT OF THE USE OF DIFFERENT MAKES OF FILTER-PAPER ON THE REFRACTION.

Sample.	Munktell's No. 1 F.	Carl Schleicher & Schüll No. 595.	Balston's Whatman No. 1.	Balston's Whatman No. 5.
No. 1	38·50	38·50	38·50	38·55
2	37·90	37·90	37·90	37·90
3	38·50	38·50	38·50	38·50
4	39·00	39·00	39·00	39·00
5	38·55	38·55	38·55	38·55
6	38·60	38·60	38·60	38·60

THE EXAMINATION OF FRACTIONS OF THE SERUM.—In order to ascertain if at all stages of filtration the serum had a constant refraction, a series of experiments was made and the sera were collected in three approximately equal portions, each portion being examined separately. An independent test for comparison was made in which the whole of the serum was obtained before the refraction was determined. The results are set out in Table VI.

TABLE VI.

REFRACTION OF THE SERUM COLLECTED IN FRACTIONS.

Sample.	Fraction			Whole serum.
	No. 1.	No. 2.	No. 3.	
No. 1	38·30	38·30	38·25	38·30
2	38·30	38·30	38·25	38·30
3	38·40	38·40	38·40	38·40
4	38·10	38·10	38·10	38·10
5	38·35	38·35	38·30	38·35
6	37·65	37·65	37·60	37·65

It will be seen that each part has the same refraction which is identical with that of the whole of the serum when obtained in one portion.

REFRACTION AS A TEST FOR ADDED WATER.—It would naturally be expected that the refraction would be diminished in proportion to the amount of added water in the same manner as the other figures obtained in milk analysis. In the following cases genuine milks were taken, and samples were prepared from them containing varying quantities by weight of added water. The refraction of each was then taken, the figures being given in Table VII. In general, 5 per cent. of added water lowers the refraction by about 1 division.

TABLE VII.

REFRACTION OF MILK CONTAINING ADDED WATER.

Sample.	Added water				
	0.	2·5 per cent.	5 per cent.	7·5 per cent.	10 per cent.
No. 1	38·40	37·85	37·40	36·95	36·40
2	38·45	37·85	37·30	36·80	36·25
3	38·05	37·55	37·00	36·50	36·00
4	37·85	37·35	36·90	36·40	35·90
5	39·00		38·00		37·00

The figures given for the refraction of milks having low solids-not-fat which have been met with in the course of analyses in the ordinary way under the Food and Drugs Acts, and for the corresponding comparison samples are given in Table XXI. In these cases it is seen that the indication of the refraction is in general agreement with that of the solids-not-fat.

REFRACTION AND SOURING.—At the commencement of this work a preliminary experiment was made in which the refraction of the serum of a genuine milk was observed when fresh, and the determination repeated at intervals as the milk went sour and finally putrefied. It was noticed that the refraction rose for the first few days. Again, in the course of the investigation of the milk day by day from a herd of cows for an extended period, it was observed that when the milk was known to be older or on the point of becoming sour, owing to delay in delivery, the refraction was noticeably above the usual range of figures, and only so in those cases.

This appeared to us a point of the utmost importance, and it is one, therefore, to which we have given special attention. On referring to the literature on the subject we find that the fact has been noticed by others. For example, C. Mai and S. Rothenfusser (ANALYST, 1908, **33**, 400) prepared the serum by precipitation with calcium chloride and considered the process only applicable to fresh or slightly sour milks, since it failed to give reliable results when the acidity of the milk was such that 100 c.c. required more than 9·0 c.c. of N/4 alkali for neutralisation. G. Schutz and L. Wein (ANALYST, 1913, **38**, 500) also prepared the serum by the use of calcium chloride and suggested that the increase in the refractometric value due to acidity may be prevented by the addition of a few drops of formaldehyde. E. Ackermann (*Annales de Chimie Analytique* (August 15), 1917, p. 152) speaks of a rise in the refraction which he observed and which he says was attributed by G. Schutz and L. Wein (*Z. Unters. Nahr. Genussm.*, 1913, p. 180), and by Pfyl and Turnau, to the serum and coagulum remaining in contact, thus allowing re-solution of calcium salts. He, himself, thinks it is due to varying amounts of albumin retained in solution.

All these increases, however, refer to the refraction of sera prepared by means other than the use of copper sulphate. In the method adopted by us we add no calcium chloride, and therefore the increase cannot be due to added calcium salts. Again, it cannot be due to coagulum and serum remaining in contact, for in all cases we have filtered within a very short time of precipitation. It appears to us that, while a rise in refraction has been observed, noticeably by Schutz and Wein among others, yet its true significance has been overlooked. We refer to this point again in Table VIII.

Two series of experiments were carried out on the same milks. In one case (Table VIII) the 20 c.c. quantities were all measured out while the milk was fresh and kept in 6 × 1 in. test tubes fitted loosely with paper covers. The milk was therefore not disturbed until the time came for preparation of the serum. This rendered it possible to use a pipette for taking the quantities for examination in

all cases, and the necessity for measuring sour milk did not thus arise. In the other set of experiments (Table IX) portions were taken as required from the bulks (contained in corked bottles), which were thoroughly mixed by shaking before each portion was removed.

TABLE VIII.

REFRACTION OF MILK ON STANDING (SEPARATE PORTIONS).

Sample.		Period in days.							
		0	1	2	3	4	5	8	14
No. 1	Refraction	38·30	38·80	39·90	39·60	39·30	39·00	37·75	36·00
	Acidity ..	1·9	2·8	8·0	9·5	9·8	10·0	9·6	8·3
No. 2	Refraction	38·20	38·40	39·65	39·70	39·60	39·20	38·80	37·85
	Acidity ..	1·9	2·0	7·8	8·2	8·7	9·6	8·0	8·0
No. 3	Refraction	37·55	37·85	38·80	38·80	38·75	38·20	37·20	37·00
	Acidity ..	1·9	2·0	7·0	8·2	8·7	9·6	8·3	7·2
No. 4	Refraction	38·90	39·20	40·45	40·30	40·00	39·50	38·55	37·90
	Acidity ..	1·7	1·8	7·7	8·7	9·2	10·0	10·2	7·9
No. 5	Refraction	37·70	38·00	38·95	39·10	38·95	38·50	37·70	35·60
	Acidity ..	1·6	1·7	6·2	8·4	8·5	9·0	7·9	7·8
No. 6	Refraction	38·40	38·60	39·60	39·30	39·15	38·90	38·00	36·95
	Acidity ..	1·8	1·9	5·6	9·6	9·6	10·7	9·0	8·8

TABLE IX.

REFRACTION OF MILK AFTER STANDING (PORTIONS TAKEN FROM BULK).

Sample.	Period in days.									
	0	1	2	3	4	5	11	18	25	32
No. 1 ..	38·30	38·80	39·80	39·60	39·70	39·60	38·90	38·35	38·00	37·75
2 ..	38·20	38·35	39·40	39·60	39·60	39·50	39·30	39·30	39·20	39·00
3 ..	37·55	37·80	38·70	38·70	38·70	38·60	38·30	37·90	37·30	36·60
4 ..	38·90	39·20	40·35	40·05	40·00	40·00	38·80	37·40	36·60	36·60
5 ..	37·70	38·00	38·90	38·85	38·85	38·85	38·80	38·70	38·20	37·75
6 ..	38·40	38·55	39·50	39·30	39·35	39·35	39·30	38·70	37·80	37·00

Speaking generally, the refraction rises as the acidity increases up to a point. In the case of the milks whose refractions are given in Table VIII, the acidities were also taken by titrating 10 c.c. with N/10 sodium hydroxide solution and phenolphthalein. All the quantities required for acidity were pipetted off at the commencement of the experiment and kept in 6 × 1 in. test tubes loosely covered with paper. The results of these experiments are given in Table VIII above, together with the corresponding refractions.

It will be seen that the refraction reaches its maximum before the acidity does, and that the acidity also falls again after a time. Further, the refraction has risen in every case, the amounts being in Table VIII: 1·6, 1·5, 1·25, 1·55, 1·4, 1·2, and in Table IX: 1·5, 1·4, 1·15, 1·45, 1·2, and 1·1, respectively. In most cases a week or so elapsed before the refraction fell to the initial figure, and in one case (Table IX) it had not so fallen at the conclusion of the experiment.

It appears, therefore, that as a milk turns sour the refraction of the serum rises by as much as 1·5 divisions; even more has been noticed in other experiments. If this is so, it is of profound significance as regards the value of the refractometric test in those cases where a milk is somewhat deficient in solids-not-fat, *e.g.* 8·25 per cent. In a case of this kind the reading of the Immersion Refractometer obtained on the serum has been quoted as the criterion of genuineness; that is, it appears to be claimed that it will definitely prove whether the milk is watered or genuine but of poor quality, as watered milk of this composition should show a slight reduction in the figure for refraction, whilst an unadulterated milk of the same solids-not-fat should show a higher value. As will be seen later, we cannot accept this contention, but in any case the decrease in the refraction of such a milk, if caused by added water, would of course be quite small, and, as souring proceeds even before actual curdling takes place, would soon be masked by the rise accompanying souring. Thus, as will be proved later, as much as 7·5 per cent. of added water might be obscured, and therefore a milk, on standing some time, might pass a standard which it could not do if it were fresh.

This test then becomes of diminished value, if not absolutely worthless, as a means of defence. A milk watered to the extent of 7·5 per cent., by merely standing a few days—exactly how long depends on the temperature, conditions of keeping, etc.—may give a figure identical with that of genuine milk. The fact that in the case of deficiencies in solids-not-fat the refraction is not lowered materially is no proof that a milk is not watered. On the other hand, if the refractive index is low it would support the view that addition of water may have taken place. In short, the test is one from which conclusions may be drawn as to the addition of water, but is of little, or indeed possibly of no value, as a defence against that allegation, unless the milk is quite fresh. As will be shown later, the solids-not-fat, and refraction of a fresh milk appear to vary together.

Rise of Refraction on Souring and Reduction due to Watering.—It is evident from the foregoing that a point must be reached when the rise of refraction on souring and the reduction due to added water must so operate as to produce, on standing, a refraction equal to that which the unwatered milk had when fresh. To determine this point practically, genuine milks were taken, and from them were prepared portions containing the following percentages of added water by weight 2·5, 5, 7·5 and 10. Quantities of these, and also of the

genuine milk, were measured out and the refraction taken daily for 4 or 5 days. The results are given in the following table:—

TABLE X.

REFRACTION AFTER STANDING OF MILK CONTAINING ADDED WATER.

Sample.	Added water per cent.	Days. 0	1	2	3	4	5
No. 1 ..	0	38·40	38·45	38·85	39·60	39·45	—
	2½	37·85	37·90	38·00	38·80	38·90	38·80
	5	37·40	37·40	37·60	38·40	38·35	—
	7½	36·95	36·95	37·05	37·90	37·85	—
	10	36·40	36·40	36·60	37·40	37·50	37·50
No. 2 ..	0	38·45	38·50	38·50	39·80	39·70	—
	2½	37·85	38·00	38·00	39·35	39·30	—
	5	37·30	37·40	37·50	38·60	38·70	38·60
	7½	36·80	36·95	37·00	38·15	38·20	—
	10	36·25	36·40	36·50	37·60	37·70	37·70
No. 3 ..	0	38·05	38·15	38·50	39·70	39·55	—
	2½	37·55	37·65	37·90	38·95	38·95	—
	5	37·00	37·10	37·30	38·60	38·60	—
	7½	36·50	36·65	36·90	38·05	37·85	—
	10	36·00	36·15	36·30	37·25	37·40	37·40
No. 4 ..	0	37·85	37·90	38·10	39·20	39·00	—
	2½	37·35	37·40	37·60	38·80	38·60	—
	5	36·90	36·95	37·10	38·20	38·00	—
	7½	36·40	36·50	36·70	37·60	37·60	—
	10	35·90	36·00	36·20	37·10	37·05	—

From these experiments it is seen that in the case of Sample No. 1, an addition of 5 per cent. of added water is counteracted by standing for 3 days; in that of No. 2, between 5·5 and 7 per cent. of added water is masked in 4 days; in that of No. 3, 7·5 per cent. of added water in 3 days; and in that of No. 4 about 7 per cent.

REFRACTION AND ACIDITY: QUANTITATIVE ASPECT.—When it became apparent that, from whatever cause, there was an increase in the refraction as souring proceeded, an attempt was made to discover whether any quantitative relationship could be discovered, with a view to correcting the refraction for acidity in those cases where decomposition had not gone too far. It cannot be claimed that any very satisfactory relationship has been found as yet. Although the matter is still under investigation, the following results may be of some interest as indicating lines on which work is being carried out.

Although in every case so far investigated increase of acidity is accompanied by an increase in the refraction, this latter increase apparently does not always commence at the same point of acidity, neither is the increase in the one always proportional to the increase in the other. From the observations made up to the present it seems possible that where the decomposition starts at a low temperature the immediate increase in refraction is less for a similar increase in acidity than when the decomposition starts at a higher temperature, thus:—

TABLE XI.

Temp. of Lab.	No. of sample.	Acidity.	Refraction.	Acidity.	Refraction.
About 22°C.	658 S.D.	1·9	38·2	2·0	38·4
	659 S.D.	1·9	37·6	2·0	37·9
	660 S.D.	1·7	38·9	1·8	39·2
	661 S.D.	1·6	37·7	1·7	38·0
	662 S.D.	1·8	38·4	1·9	38·6
About 16°C.	1041 A.D.	1·7	37·5	2·5	37·6
	1044 A.D.	1·7	37·6	2·5	37·7
	1137 W.D.	1·7	38·5	2·0	38·6
	652 Km D	1·6	37·3	1·8	37·4
	655 Km D	1·6	37·9	1·8	37·9

From this table it is seen that when the laboratory temperature was as high as 22° C., an increase in acidity of 0·1 (the acidities are given in c.c. of N/10 sodium hydroxide solution to phenolphthalein for 10 c.c. of milk) may be accompanied by a difference in the refraction of 0·2–0·3 division. When the temperature was about 16° C., an increase in acidity of 0·2 was not attended by any difference in refraction, whilst in two cases where there was a difference of 0·8 in the acidity the increase in the refraction was only 0·1 scale division.

This point, which is being further investigated, obviously makes it difficult to decide at what stage in the acidity the refraction becomes unreliable, but it would appear that where the temperature is kept low this point may be about 2·5, whilst where the milk has been kept at a higher temperature, a noticeable increase has taken place in the refraction before the acidity reaches 2.0.

In order to determine the quantitative connection between rise in refraction and increase of acidity, 44 milks having acidities of 1·7 or less (the acidity of absolutely fresh milk was found to be 1·5–1·6 by the same process) were examined on each of several successive days until the refraction began to fall. In order to find what change in the refraction corresponded to 1·0 of acidity the increase in refraction was divided by the increase in acidity. This assumes, to a certain extent, that the increase in refraction follows the acidity uniformly, but actually this is not so. The greatest increase in refraction appears to take place while the acidity changes from about 3·0 to 5·0, but this depends to some extent on the

temperature of the milk, and therefore probably on the type of fermentation. The following table gives the figures which have been obtained.

TABLE XII.

REFRACTION AND ACIDITY.

Sample No.	(1) Refraction.	(2) Acidity.	(3) Refraction.	(4) Acidity.	(5) Diff. of (1) & (3).	(6) Diff. of (2) & (4).	(5) divided by (6).
647 S.D.	38·7	1·9	40·3	8·3	1·6	6·4	0·23
648 S.D.	38·7	3·3	39·4	8·2	0·7	4·9	0·14
649 S.D.	39·1	1·7	40·3	7·8	1·2	6·1	0·20
650 S.D.	37·7	2·1	38·7	7·2	1·0	5·1	0·20
651 S.D.	37·9	1·8	39·1	6·6	1·2	4·8	0·25
652 S.D.	37·9	1·7	39·2	6·2	1·3	4·5	0·29
653 S.D.	38·4	1·7	39·5	6·7	1·4	5·0	0·28
654 S.D.	37·9	1·6	39·0	6·3	1·1	4·7	0·23
655 S.D.	38·1	2·2	39·2	6·2	1·1	4·0	0·27
656 S.D.	38·7	1·5	40·2	8·0	1·5	6·5	0·23
1086 W.D.	37·8	1·6	38·8	7·8	1·0	6·2	0·16
1093 W.D.	37·8	1·7	39·1	8·2	1·3	6·5	0·20
1098 W.D.	38·5	1·7	39·4	8·2	0·9	6·5	0·14
1102 W.D.	38·0	1·7	38·9	8·2	0·9	6·5	0·14
1104 W.D.	38·2	1·7	39·4	7·4	1·2	5·7	0·21
1126 W.D.	37·1	1·3	39·1	5·4	2·0	4·1	0·49
1034 C.C.F.	38·1	1·6	41·7	7·5	3·6	5·9	0·61
1035 C.C.F.	37·9	1·6	40·7	7·6	2·8	6·0	0·47
347 L. St. A.	37·8	1·6	38·8	5·9	1·0	4·3	0·23
348 L. St. A.	38·0	1·6	38·6	3·0	0·6	1·4	0·43
349 L. St. A.	38·5	1·6	38·8	3·1	0·3	1·5	0·20
1137 W.D.	38·5	1·7	40·2	8·5	1·7	6·8	0·25
315 N.L.D.	37·8	1·7	38·8	6·6	1·0	4·9	0·20
634 S.L.D.	37·8	1·7	39·1	8·1	1·3	6·4	0·20
643 S.L.D.	37·4	1·6	38·8	7·4	1·4	5·8	0·24
946 H.B.D.	38·1	1·7	39·0	8·5	0·9	6·8	0·13
947 H.B.D.	38·3	1·7	39·4	7·0	1·1	5·3	0·21
949 H.B.D.	38·1	1·7	38·8	5·8	0·7	4·1	0·17
956 H.B.D.	38·0	1·7	38·8	6·4	0·8	4·7	0·17
704 R.D.	38·3	1·6	38·8	4·3	0·5	2·7	0·18
705 M.D.	38·4	4·7	39·9	7·8	1·5	6·1	0·25
706 M.D.	38·4	1·7	39·9	7·6	1·5	5·9	0·25
710 M.D.	38·0	1·7	38·9	8·4	0·9	6·7	0·13
723 M.D.	38·0	1·7	38·5	5·6	0·5	3·9	0·13
816 By.D.	37·7	1·7	39·0	10·2	1·3	8·5	0·15
819 By.D.	37·7	1·7	39·0	8·8	1·3	7·1	0·18
753 M.D.	38·5	1·7	39·5	5·5	1·0	3·8	0·26
766 M.D.	37·6	1·7	38·3	4·7	0·7	3·0	0·23
657 S.D.	38·3	1·9	39·9	8·0	1·6	6·1	0·26
658 S.D.	38·2	1·9	39·7	8·0	1·5	6·1	0·25
659 S.D.	37·6	1·9	38·8	7·0	1·2	5·1	0·24
660 S.D.	38·9	1·7	40·5	7·7	1·6	6·0	0·27
661 S.D.	37·7	1·6	39·0	6·2	1·3	4·6	0·28
662 S.D.	38·4	1·8	39·6	5·6	1·2	3·8	0·31

From these results it will be apparent that, on the average, an increase of 1·0 in the acidity accompanies an increase of about 0·2 in the refraction, although this figure is not very constant, whilst in two or three cases very divergent results have been obtained. Until further work has been done the most that can be said is that there may be an increase in refraction where the acidity is something under 2·0, and that, on the average, for acidities between 3·0 and 8·0 the refraction may be corrected roughly for acidity by subtracting 0·2 from the observed reading for each 1·0 of acidity above 2·0. It cannot be claimed that this will always give even approximately correct results, for in three cases the correction was as high as 0·5, in place of the average figure of 0·2.

CAUSE OF THE INCREASE IN REFRACTION.—It is at first sight a little difficult to explain how decomposition of a portion of the soluble solids can increase the refraction of the serum. The most obvious explanation is that the proportion of proteins left in the serum depends upon the acidity of the milk at the time of the preparation of the serum, and that the refraction varies from this cause. To test this, varying quantities of acid and alkali were added to a milk of acidity 1·8 and refraction 38·1, the following results being obtained:—

TABLE XIII.

c.c. N/10 NaOH to 10 c.c. Milk.						c.c. N/10 HCl to 10 c.c. milk.				
3·0	2·3	1·8	1·3	0·8		0·2	0·5	0·8	1·8	2·0
32·2	33·3	34·3	35·2	36·3	38·1	37·8	37·3	37·0	36·4	35·7

It will be seen that, after taking into account the difference due to dilution, the milks to which acid has been added have a somewhat higher refraction than those to which alkali has been added, thus confirming to some extent the suggestion that the acidity at the time of precipitation (and therefore probably the proportion of soluble proteins) has some bearing on the increase in refraction. In order to elucidate this point, if possible, an attempt was made to remove the soluble proteins with phosphotungstic acid. For this purpose a milk (acidity 1·7) was precipitated with copper sulphate as usual, and also with copper sulphate together with 1 c.c. of 9 per cent. phosphotungstic acid solution (such a solution has a refraction of 36·0). The experiment was repeated when the milk was sour; the results are given below:—

TABLE XIV.

Acidity	Refraction.	
	Copper Sulphate.	Copper Sulphate and Phosphotungstic acid.
1·7	37·9	34·7
8·2	39·0	35·6

It will be observed that the rise in the refraction of the copper serum (1·1) is slightly higher than that (0·9) of the copper and phosphotungstic acid serum,

which, so far as it goes, supports the above suggestion, but the amount is so small that it may have little, if any, significance.

There are two points, however, that do not agree. The first is that the refraction of the copper sulphate serum of fresh milk itself increases on standing, and by an amount which is roughly equal to the increase found between the refraction of the fresh milk and that of the same milk prepared after souring and examined immediately. The following table shows this:—

TABLE XV.

INCREASE IN REFRACTION OF COPPER SERUM ON STANDING.

		After period of				
Milk sample.	Immediately.	24 hours.	43 hours.	4 days.	5 days.	11 days.
294 P.D.	38·3	38·4	38·6	38·8	39·0	40·2
295 P.D.	37·3	37·4	37·6	37·8	38·0	38·5
296 P.D.	38·2	38·3	38·4	38·6	38·8	39·4
297 P.D.	39·3	39·4	39·6	39·8	39·9	40·4
298 P.D.	38·7	38·8	39·0	39·3	39·4	40·0
299 P.D.	38·8	38·9	39·1	39·3	39·4	40·1

The sera in these cases were not filtered again after their first preparation. They became slightly cloudy after standing, but this would presumably tend to lower the refraction.

Another point which appears to have a considerable influence on the refraction of the serum is the amount of copper which is retained by the coagulum. Fresh milk gives a coagulum which is quite blue in colour, whilst that from sour milk is much paler. Volumetric determinations show that in the case of a milk of maximum sourness (about 10·0) the serum contains practically the same proportion of copper as that added to the milk, whilst in the case of a fresh milk this is very considerably less and may be only half the amount added. Such a result might be expected, as the bulk of the proteins have already been precipitated in the case of a sour milk by its natural acidity. This would account for the observed increases in refraction, but it is obvious that many factors may be at work, and at the moment no definite conclusions have been reached. The subject is still under investigation.

REFRACTION OF MILKS KEPT FOR FOUR WEEKS.—In the last column of Table IX, figures are given for the refraction of milks kept for 32 days. The samples were contained in corked bottles, shaken each time an observation was made. As the work proceeded a gradually increasing air space was consequently present in the bottle. For this reason two series of experiments were made to simulate as nearly as possible what occurs when a portion of a milk sample, divided in accordance with the Food and Drugs Acts, is forwarded to the Referees at the Government Laboratory.

Samples of milk, after the refraction had been determined, were placed in tightly corked bottles and sealed. They were allowed to stand for 4 weeks at the temperature of the laboratory without being disturbed in any way. One series was done in October when the temperature was moderate, and the other in December when the weather was colder. Two bottles burst in the first series before the expiration of the period. The following table gives the results of the tests.

TABLE XVI.

REFRACTION OF MILKS KEPT UNDISTURBED FOR FOUR WEEKS.

Series 1. Refraction.			Series 2. Refraction.		
Sample No.	On commencement.	After 4 weeks.	Sample No.	On commencement.	After 4 weeks.
1	37·70	39·60	1	37·20	36·40
2	38·30	38·10	2	38·50	39·80
3	37·45	39·20	3	38·10	39·30
4	38·60	38·30	4	39·25	39·00
			5	38·20	39·40
			6	38·00	37·60

The results here are somewhat irregular. In some cases the refraction after 4 weeks has risen, and in others it is about equal to, or less than the original figure. It would appear that such decomposition as affects the refraction takes place at unequal rates in the tightly corked and sealed bottles. It is possible that a decomposition similar to that of milks exposed to air is proceeding, causing first a rise in the refraction and later a fall, but at a retarded rate which in some cases had just reached a point near the "peak" when the 4 weeks had elapsed, whilst in others it had proceeded further. However that may be, it is clear from the above results, that the refraction of milk kept in a confined space does not offer, at the best, any greater promise of determining its genuineness than do the other processes of analysis which have been usually applied in this country.

THE INTER-RELATION OF SOLIDS-NOT-FAT AND REFRACTION.—The refraction of milk serum depends, of course, upon the nature and amount of the solids dissolved therein. The dissolved solids consist, for the most part, of mineral salts, soluble proteins and lactose, the last named being greatly in excess of the others. It would appear likely, therefore, that there is a fairly close relationship between the refraction and the percentage of solids-not-fat, and also, on account of the excess of lactose over the other constituents, between the refraction and the percentage of lactose.

To take the second point first. Not very much work seems to have been done specifically on this point, except that of Ackermann (*loc. cit.*) and of Tocher in his recent monograph. Tocher shows that refraction and amount of lactose are highly correlated, a result which would naturally be expected and which at once throws doubt upon the suggestion that the refraction is a more useful figure than the percentage of solids-not-fat for the detection of watered milks.

The relationship of solids-not-fat to refraction has also apparently escaped serious notice. Tocher (*loc. cit.*) finds that the regression between solids-not-fat and refractive index is linear. We have examined just over a thousand milks obtained under ordinary commercial conditions from all districts in the Administrative County of Lancaster. As has been shown above, the refraction apparently varies to a considerable extent with the acidity, so that the figures have been grouped in Table XVII to XX under their various acidities as well as under the figures for solids-not-fat.

TABLE XVII.

COMPARISON OF REFRACTION AND SOLIDS-NOT-FAT.

Solids-not-fat.	Number of samples.	Range.	Average.
8·4	5	36·6–37·1	36·78
8·5	16	37·0–38·3	37·68
8·6	27	37·1–39·0	37·76
8·7	47	37·2–38·5	37·92
8·8	107	37·2–39·2	38·03
8·9	138	37·0–39·3	38·20
9·0	189	37·6–39·0	38·35
9·1	172	37·3–39·4	38·42
9·2	128	37·3–39·2	38·60
9·3	86	37·6–39·4	38·73
9·4	53	38·2–39·4	38·87
9·5 and over.	29	38·4–40·2	39·17
8·4–9·5	997	36·6–40·2	38·35

The obviously watered samples have been omitted from the above table. In the table following the refractions have been related to the corresponding acidities.

TABLE XVIII.

COMPARISON OF REFRACTION AND ACIDITY.

Acidity.	Number of samples.	Range.	Average.
1·5	17	37·1–38·8	37·89
1·6	24	36·6–38·8	37·83
1·7	69	37·1–39·0	38·00
1·8	159	37·0–39·2	38·25
1·9	186	37·0–39·3	38·32
2·0	197	37·3–39·3	38·39
2·1	137	37·0–39·4	38·51
2·2	96	37·1–40·2	38·67
2·3	36	38·1–39·4	38·74
2·4	28	37·4–39·4	38·71
2·5 and over	48	37·7–39·9	38·66
	997	36·6–40·2	38·35

From Table XVIII it would appear that the refraction of a milk serum is almost as much a function of the acidity of the milk as it is of the solids-not-fat. When working out these averages it was observed, however, that high acidity usually accompanied a high solids-not-fat. In other words, that a milk high in solids-not-fat seemed to sour more rapidly than one with low solids-not-fat. The

percentages of solids-not-fat in milks of each degree of acidity were therefore calculated and are shown in the following table.

TABLE XIX.

ACIDITY AND SOLIDS-NOT-FAT.

Acidity.	Number of samples.	Average solids-not-fat. per cent.
1·7	112	8·91
1·8	160	8·97
1·9	188	9·01
2·0	198	8·99
2·1	140	9·10
2·2	96	9·17
2·3 and 2·4	64	9·19
2·5 to 3·0	34	9·15
Above 3.0	14	9·00

In these circumstances, if acidity and solids-not-fat are in any way related, the regularities shown in Tables XVII and XVIII are due to this extent to the same cause. For this reason solids-not-fat have been correlated with the refraction for each 0·1 increase in the acidity, the results being given in Table XX.

TABLE XX.

VARIATIONS OF REFRACTION WITH SOLIDS-NOT-FAT AND ACIDITY.

Acidity.	Solids-not-fat, per cent.										
	8·5	8·6	8·7	8·8	8·9	9·0	9·1	9·2	9·3	9·4	9·5
1·7	—	37·76	37·75	37·74	38·08	38·26	38·29	38·21	38·55	—	—
1·8	37·57	37·58	37·92	38·03	38·26	38·24	38·44	38·53	38·70	38·83	38·83
1·9	—	37·88	38·14	37·95	38·19	38·39	38·37	38·50	38·56	38·69	—
2·0	—	—	38·14	38·22	38·20	38·35	38·38	38·53	38·61	38·77	39·00
2·1	—	—	38·25	38·13	38·00	38·47	38·39	38·69	38·71	38·81	38·95
2·2	—	—	—	—	38·21	38·50	38·44	38·60	38·62	38·96	39·23
2·3 and 2·4	—	—	—	—	38·46	38·61	38·59	38·79	38·82	38·91	39·13
2·5 to 3·0	—	—	—	—	38·20	—	—	38·88	38·56	—	39·27
Above 3·0	—	—	—	—	—	—	39·08	—	—	—	—

Each of these averages is the result of a much smaller number of samples than those given in Tables XVII and XVIII (averages of less than 4 samples have been excluded); consequently such a definite relationship is not so apparent in Table XX as in Tables XVII and XVIII. It is obvious, however, that the relationship of refraction to solids-not-fat is definite, whilst there is a considerable amount of evidence to support the idea that rise in refraction accompanies an increase in the acidity. For small increases of acidity, however, the refraction appears to follow the solids-not-fat more closely than the acidity.

REFRACTION OF GENUINE MILK SERUM.—The bulk of the published figures for the refraction of milk serum have been obtained by the use of other methods than that (the copper sulphate method) adopted by us. Practically the whole of the published work on the copper sulphate method has been done by the Association of Official Agricultural Chemists in America in collaboration with Leach and other workers, and by Tocher in his recent publication. The A.O.A.C.

considers that a reading below 36 at 20° C. indicates added water; it certainly does, but it would be possible for a considerable proportion of mixed milks to have material additions of water without the refraction of the serum falling below this figure. Tocher's work was all done on the milk of individual cows. He finds an average reading of 38·275 at 20° C. with copper sulphate serum. Only 10 out of his 676* samples gave a reading of less than 36·1, whilst 37 gave readings of less than 36·6. From an examination of Tocher's figures it would appear to be extremely improbable that the milk serum from a herd of cows (say 5 to 10) would give a refraction of less than 36·5 to 37·0. We are of opinion that the figure of 36·0 is very definitely too low, and much lower relatively that the usually adopted 8·5 per cent. figure for solids-not-fat. In the examination of the above-mentioned 1000 samples of the mixed milk of herds of varying sizes taken from all parts of the County of Lancashire the only figures obtained of less than 37·0 are given in the following table (Table XXI).

TABLE XXI.

Number.	Refraction.	Acidity.	Total solids. Per cent.	Solids-not-fat. Per cent.	Fat. Per cent.	Refraction of appeal to cow samples.
303 P.D.	36·7	1·8	11·56	8·26	3·3	—
632 M.D.	35·2	1·5	11·13	7·73	3·4	37·8
633 M.D.	35·1	1·5	10·68	7·58	3·1	37·9
390 S.D.	36·6	1·8	11·45	8·35	3·1	—
646 R.D.	35·2	1·7	11·10	7·90	3·2	37·3
20 D.B.	36·2	1·8	16·60	7·80	8·8	—
678 R.D.	36·6	1·9	11·19	8·39	2·8	37·8
684 R.D.	36·3	2·0	13·05	9·15	3·9	—
686 R.D.	35·5	1·7	11·53	8·03	3·5	37·7
687 R.D.	35·6	1·7	11·64	8·04	3·6	37·7
157 Irlam	35·5	1·8	16·90	7·70	9·2	—
656 Km. D.	36·3	1·7	11·20	8·10	3·2	—
1169 W.D.	36·8	1·8	12·04	8·44	3·6	—
730 S.D.	35·5	1·8	10·84	7·84	3·0	38·1
673 Rs.D.	36·8	1·6	11·08	8·28	2·8	—
683 L.D.	36·6	2·0	15·60	8·20	7·4	—
201 Bootle	34·7	2·2	10·34	7·34	3·0	38·3
202 Bootle	34·9	1·5	10·46	7·66	2·8	38·1
203 Bootle	35·4	1·8	11·06	7·86	3·2	38·2
1028 G.D.	33·6	1·6	10·06	7·06	3·0	38·3
1029 G.D.	35·7	1·8	11·27	7·97	3·3	38·1
598 Bn.D.	36·6	1·7	11·07	7·97	3·1	38·2

It will be seen that, in every case but one, a refraction of less than 37·0 has accompanied a solids-not-fat of less than 8·5 and, further, that in all those cases where appeal to cow samples have been taken much higher figures (always exceeding 37·0) have been obtained. This means that less than one per cent. of 1000 mixed milks taken at random gave a refraction under 37, and of this one per cent. many, if not most, were (on the evidence of the appeal to cow samples), adulterated.

The refraction of genuine fresh mixed milk may be taken as varying between 37·0 and 39·0; few samples will exceed 39·0, whilst few, if any, will fall below 37·0.

* It should not be forgotten that apparently no corrections have been applied to the figures obtained, on account of acidity.

THE RELATIVE VALUE OF THE REFRACTION AND SOLIDS-NOT-FAT IN THE DETERMINATION OF ADDED WATER.—It will be seen from the results given above that the claims made for the refractometric test as a means of detecting added water in milk are greatly exaggerated. It appears to us that, so far as our present results go, the determination of the refraction of the serum offers no advantage over that of the solids-not-fat of the milk; in fact, that it may be even less valuable. In nearly every case the refraction is proportional to the solids-not-fat. In no case have we found a milk having a refraction over 37·1 when the solids-not-fat have been less than 8·5 per cent., such milks having, in general, a lower refraction than 37·0. In only one case, as is shown in Table XIX, has a milk high in solids-not-fat been found to have a low refraction. On the whole, therefore, the use of the Immersion Refractometer, whilst providing a means for the rapid examination of milk and being also useful in comparing poor samples with the corresponding samples from the cows, affords a means of detecting water in an isolated sample of milk, the value of which is no greater than, if as great as, the determination of the solids-not-fat.

THE LANCASHIRE COUNTY COUNCIL LABORATORY,
LIVERPOOL.

DISCUSSION.

THE PRESIDENT said that the investigation had been conducted with such thoroughness and lack of bias that criticism seemed out of the question. The authors spoke of "the defence"—whatever that might be, he (the President) was glad he was not on it. He thought it was very disappointing that so delicate an instrument, capable of giving readings comparable with the fifth place, should prove of so little value. He wondered whether the ratio of albumin to casein was a factor of any importance. He suggested that if the protein were precipitated with phospho-tungstic acid in all cases a new series of figures would be obtained which might throw light on the problem. He noticed that in the last table samples were deficient in solids, but not in fat. He thought that the instrument should serve as a basis for a rapid sorting test and might save determinations of solids-not-fat, provided the results were regarded as indicative and not conclusive.

Mr. A. MORE mentioned that, in a case about six years ago, it was brought to the notice of the magistrates by the Government Laboratory, that the refractometric reading of the copper serum of a milk increased with the development of acidity during storage, that a milk which had developed acidity appeared to be of better quality than the milk in its original condition, and that it was impossible to say whether the reading of the serum in the original sample would have indicated addition of water or not, when judged by the tentative standard of the United States. He, himself, had evidence that the reading in samples increased during storage as much as three degrees, but he had not noticed a subsequent fall. The fall might not occur in hermetically sealed samples. The reading was a measure of the unprecipitated substances in a fresh milk. These substances, however, during keeping, broke down into simpler substances, lactose producing alcohol and lactic acid, and the proteins also undergoing change, and the nature and amount of the substances not precipitated by copper sulphate must of necessity alter. A paper by Mai W. Rothenfusser on the refractometric examination of milk, appeared in the *Zeitschrift der Nahrungs und Genussmittel* (1908, **16**, 7)

a long time ago, in which the rise in the reading was commented upon, and it was stated that the method could not be applied to milks which had reached a certain degree of acidity, and that the rise was due to lactic acid having a greater effect than the lactose from which it was derived. It was doubtful, however, whether this was the correct explanation, and more probably the rise was due to the alteration of the protein into non-precipitable substances, and partly to the greater solubility of the compounds of copper with protein and phosphates in the acid serum. In this consideration it had to be noted that the acidity of a milk which contained buffer constituents was more correctly expressed by the P_H value than by titratable acidity, although the latter was the one generally recorded for milk.

Mr. E. M. HAWKINS asked what the readings were for solutions of lactose, lactic acid and carbonic acid. These, he considered, might give useful information.

Mr. CRANFIELD asked whether the author had determined the refractive index on a series of daily samples from one cow or from a herd. In the case of a herd he understood that it was claimed that the reading was fairly constant, whilst the solids-not-fat percentage might vary considerably from day to day over the same period.

Mr. E. HINKS said that it had been claimed that the refractometric method distinguished between a milk in which the solids-not-fat were low owing to the addition of water, and one in which the deficiency was due to natural causes, *i.e.* an abnormal milk. In his opinion the ratio of lactose to protein to ash was of much greater significance in this connection. He himself, had met the defence referred to, and had been obliged to describe the method as "a rapid way of arriving at an inaccurate conclusion."

Mr. J. W. BLACK said that precipitation was doubtless best effected by copper sulphate, but he wondered if the serum would show the same rise and fall in the absence of residual copper sulphate. The association of residual copper with lactic acid or other decomposition products might lead to combinations subsequently destroyed as the decomposition proceeded, and the effect might be analogous to the effect of molybdenum or uranium salts on the rotation of malic acid.

Mr. ELSDON, replying for the authors, said that the instrument was of value in other directions; it was used in America for the determination of alcohol, and of methyl alcohol in the presence of ethyl alcohol. He had used the heating arrangements suggested by the President. As a basis for a sorting test the instrument was not more rapid than the gravity and Gerber methods, but it was more rapid than the determination of the solids-not-fat by weight; a dozen samples could be examined in an hour. The fall in readings, following a rise, was possibly due to decomposition, evolution of carbon dioxide, and so on. He agreed that titration with $N/10$ alkali was not an accurate measure of acidity in milk, but it gave a comparative figure. The true P_H values would certainly be preferable. With regard to the addition of pure lactic acid, the difficulty lay in the impossibility, hitherto, of obtaining this substances; information of likely sources would be welcomed. They had tested a series of samples from one herd every milking over a period of three months; the refractive index of the fresh milk rose and fell with the content of solids-not-fat. There was evidence to suggest that the test was valueless for distinguishing between watered milk and milk with abnormally low solids. It it were assumed that the lactose varied and the nitrogen remained constant, the refractive index would vary with the lactose and therefore with the solids-not-fat. But according to Tocher's results on individual cows, there was little support for this assumption. The constancy of the nitrogen could not be taken for granted. The refractive index of the serum of sour milk rose when no copper was present.

The Examination of Foodstuffs for Preservatives: A Caution.

By A. CHASTON CHAPMAN, F.R.S., F.I.C.

Since the new Preservatives Regulations came into force at the beginning of this year the search for traces of preservatives in food stuffs, and their determination, if present, has assumed a degree of importance which it has not hitherto possessed. A need has arisen for the close scrutiny of some of the older analytical processes both in respect of their qualitative and quantitative indications, with the consequence that some interesting observations have already been made, and these, with others will, doubtless, in due course be recorded. As an example of this, I may perhaps refer to the following experience.

A sample of caramel was submitted to me with the information that it had been found to contain distinct traces of benzoic acid, and with the request that I would examine it for that substance. I may perhaps add that this sample was submitted to me by the manufacturer, who assured me that the sample could not possibly have come into contact with benzoic acid.

A strong aqueous solution of the sample was thoroughly extracted with pure ether, and the residue left on the evaporation of the ether was subjected in an apparatus of small dimensions to steam distillation. The distillate, having been made alkaline, was evaporated to a small volume, acidified and re-extracted with pure ether. On the evaporation of the ether a slightly yellow, and distinctly crystalline residue, having strong acid properties, was left, and there can be little doubt that this was the substance which had been regarded as benzoic acid. On the addition of light petroleum spirit the greater part of this substance dissolved and crystallised well on cooling, in tufts or rosettes of fine needle-shaped crystals which melted, but not at all sharply, at about 90° C. On recrystallising from light petroleum spirit the melting point was ultimately raised to 122° C., but even after three recrystallisations it was not apparently constant. It will be seen that this is not far from the melting point of benzoic acid. The crystals gave a violet coloration with ferric chloride similar to, but less intense than, that produced by salicylic acid.

On further examination it was found possible to separate this substance into, at least, two constituents, one of which is apparently a phenol giving the violet coloration with ferric chloride, and the other—the larger proportion—an acid which gives neither colour nor precipitate. Bromine reacts readily with this acid, which appears to be unsaturated, but does not form a precipitate, and it is perfectly certain, that whatever else it may be, it is neither benzoic nor salicylic acid. With the quantity of material at my disposal I was not able to prepare more than a few centigrammes of this substance, so that its identification was impossible.

While the above investigation was in progress, a second case of the same kind was reported to me by one of my clients, so that it is clear that from caramel, and probably from certain other carbohydrate materials, an acid (or mixture of acids) is obtained which simulates benzoic acid very closely, and, unless caution is observed, may easily be mistaken for that substance. It is, of course, unnecessary to point out that benzoic acid occurs as a normal constituent of certain fruits, such as the cranberry, bilberry, etc.

FORMALDEHYDE.—At the time of the above enquiry it was observed that caramel distillates gave a reaction which suggested the presence of formaldehyde. This led to a closer investigation, and it was then seen that on steam distillation a distillate was always obtained, giving all the characteristic reactions of formaldehyde and indicating the presence either of that substance, or of some other which resembled it so closely in its analytical characters that it would undoubtedly be mistaken for it. This led to the examination of further samples, and ultimately to the examination of sweets of various kinds.

All the samples examined gave the formaldehyde reaction in greater or less degree, and the same applied to samples of boiled sugar sweets made from pure materials in the laboratory. Thus, it was found that in the case of ordinary sweets numbers up to 20 parts per million of formaldehyde were obtained, whilst in the case of semi-caramelised and caramelised products numbers reaching 50 parts per million were recorded.

For the purpose of the actual determination, Schryver's phenylhydrazine-ferricyanide method was employed, but, as I have stated above, the reaction was obtained not only with that reagent, but with Hehner's and—where it could be applied—the phloroglucinol test. On looking into the literature I found that a good deal of work had already been done on this subject. Thus, Trillat (*Bull. Soc. Chim.*, **1906**, **35**, 681–685) points out that formaldehyde is given off when sugar is heated to 125° C., and that at 150° C. larger proportions still are formed. A. A. Ramsey (*J. and Proc. Roy. Soc. of New South Wales*, **1907**, **41**, 172–175) has obtained similar results, and has stated that traces of formaldehyde are produced even when strong aqueous sugar solutions are heated to 100° C. He also calls attention to the occurrence of formaldehyde in jams. Again, Yoder and Taggart (*Intern. Sugar J.*, **1910**, **12**, 239–245) have confirmed the observation of these workers and have shown that formaldehyde is present in sugar products, even when none has been used in the process of manufacture. They also state that when formaldehyde has been employed in the sugar factory for the clarification of the raw juice, traces may be found in the crystallised sugar, but that the strongest indications are obtained in the molasses.

It is perfectly clear, therefore, that formaldehyde, or some substance resembling it very closely indeed in its analytical properties, is produced on the heating of sugar products, as in the manufacture of sweets, and that formaldehyde reactions, even when pronounced, are not therefore to be taken as indicating the addition of that substance. The importance of this, having regard to the fact that the use of formaldehyde as a preservative is forbidden under the new Regulations,

will be obvious. Recently König (*Chem. Ztg.*, 1926, **50**, 992-994) has shown that the gases evolved when various food stuffs are heated at 100° C. for 2 hours in a stream of nitrogen contain formaldehyde, and states that foods rich in vitamins give very much more formaldehyde than those containing little or none. Other papers dealing directly or incidentally with this subject appear to have been published, but it would serve no useful purpose to refer to these. Incidentally, I may perhaps again direct attention to the natural occurrence of boron in samples of agar-agar and in other edible sea-weeds to which reference has been made in a recent paper by Chapman and Linden (ANALYST, 1926, **51**, 564). Since these materials enter into the composition of certain foodstuffs, caution must be observed in ascribing the presence of traces of boron to the use of boric preservative.

CHEMICAL LABORATORIES,
8, DUKE STREET, E.C.3.

Arsenic in Printing Ink.

BY T. HEDLEY BARRY.

(*Read at the Meeting, February* 2, 1927).

THE question of arsenic in printing ink has become of considerable importance with the rapidly extending use of paper wrappers for food-stuffs and confectionery.

The matter was specifically referred to by G. D. Elsdon (ANALYST, 1924, **49**, 336) who examined 51 wrappers for proprietary breads, and found appreciable quantities of arsenic in nine of them.

As the colours in this case were blue, purple and green, it is highly probable that the arsenic was an essential ingredient of the pigments used, many lakes of these colours being "topped" with arsenic in order to give brilliant shades. As brilliant colours are even more desired in confectionery wrappings, it would appear that arsenical inks are more likely to be used than in the case of bread wrappers.

Various other analysts have reported the presence of arsenic in colours of similar shade. A. F. Shulz (*Chem. Abst.*, 1915, **9**, 2310) examined 311 wall-papers and found arsenic in 80 per cent. of them, whilst C. A. Neufeld, (*Chem. Abst.*, 1913, **17**, 1801) found from 0·1 to 1 per cent. of arsenic in violet, pink and lilac colours.

There does not appear to be any definite ruling or precedent as to the maximum allowable amount of arsenic in confectionery wrappings, and I was therefore led to make some enquiries in dealing with this question from the point of view of the printing-ink maker. Since confectionery is frequently of a moist and sticky nature and comes into the hands of children, it would appear that the limit should be almost as stringent for the wrapper as for the edible contents. At

the present time, the confectioner invariably stipulates for arsenic-free colours to be used by the printer, and he passes the injunction on to the printing-ink maker. This results in a rather unsatisfactory condition, since the essential point is the amount of arsenic on a unit area of wrapper, and this obviously depends mainly upon the amount of printed design per unit area.

Apart from this, there is certainly an equal possibility of arsenic being present in the actual paper, and consequently, from the public point of view, it is useless to concentrate on the printing ink if the paper is neglected. This is by no means a remote possibility, since the printer is in the habit of appealing to the printing-ink maker whenever troubles arise, whilst he is rather inclined to take his paper for granted; for whereas printing ink can be adjusted and modified, paper, when once made, cannot be altered.

Generally speaking, the only standard limit for arsenic known to the printer, is the well-known one for beer of 1 in 700,000 parts, which is, of course, quite impracticable for a mixture of commercial chemical products such as printing ink. Indeed, since many of the B.P. products, *e.g.* zinc oxide, are allowed up to 1 part of arsenic in 100,000, even if inks were made up with pigments of B.P. standard, they would still exceed the limit of 1 in 700,000.

PROPORTION OF WRAPPING TO CONFECTION.—As a first step, the proportion of wrapping to confection was determined in several proprietary sweets. A table of results is appended:—

Confection.	Wrapping.	Weight of wrapping. Per cent.
Chocolate ..	Metal foil	2·5
Milk chocolate	Metal foil	2·5
Toffee ..	Grease proof paper	4·3
"Mixed Fruit"	Grease proof paper, ordinary paper	7·4
Chocolate ..	Metal foil and band of ordinary paper	3·0
Caramels ..	Metal foil, grease paper and band of ordinary paper	5·8

From this it appears that 5 per cent. may be taken as a reasonable proportionate weight of wrapper, since it will be noted that those which approach or exceed this limit possess two or more wrappings.

Taking the ratio of wrapper to confection as 5 per cent. a quarter of a grain of arsenic per pound of wrapping would be equivalent to one part in 560,000 on the confection, or to one part in 28,000 in the wrapper.

In a recent case, (ANALYST, 1926, **51**, 408) 1/64th of a grain of arsenic per pound of apples was held to be a harmless amount, *i.e.*, one part arsenic in 448,000.

AMOUNT OF PRINTING INK UPON PAPER.—An attempt was made to get some idea of the amount of printing ink likely to be found upon paper. This might, of course, vary from almost zero to a maximum. In one case, in which 20 sheets of paper with a fairly heavy design were actually weighed before and after printing, the amount found was about 0·1 grm. per square metre. Some data were also obtained by determining the amount of ink required for a considerable run on a weekly journal; and this gave about 0·3 grm. per square metre,

which of course includes the usual machine-room losses, and is therefore high In one case, that of a paper printed with a solid patch of a heavy green colour, the amount was about one grm. per square metre. It would appear, therefore, that an allowance of 0·5 grm. of ink per square metre would represent the maximum amount likely to be found in the class of work under consideration. It may be taken that the lightest tissue paper used in confectionery wrapping would weigh about 10 lb. per ream: that is, about 2·5 grm. per square metre. Consequently, taking the maximum density of printing ink at 0·5 grm. per square metre, and the limit for arsenic as 1 in 50,000 of printing ink, the proportion of arsenic in the wrapper would be about 1 part in 250,000. Such a wrapper would, of course, represent far less than 5 per cent. of the confection.

OTHER RELEVANT STANDARDS.—In addition to what may be based on these considerations, various standards have been set by users of pigments for paper coating which vary from 5 to 15 parts of arsenic per million on the paste pigment. These would probably be equivalent to 20 to 60 parts per million in printing ink, which, again, is of the same order as that arrived at from the various considerations. I am, however, unaware of the process of reasoning that led to the adoption of these standards. It should also be noted that, whereas the paper coater covers the whole surface, the printer only partly covers it, and this, again, should be allowed for.

The case of clothing is somewhat different, but it may be of interest to note that the limit of 0·01 mgrm. per square decimetre, fixed by some authorities (Massachussetts, *c.f.* Treadwell & Hall, *Analytical Chemistry*, 5th edition, Vol. I, p. 251), if taken as a standard for confectionery wrapping, would allow 1 part of arsenic in 500 of printing ink, on the basis of 0·5 grm. of ink per square metre of wrapper.

SUGGESTED LIMITS.—Taking all these figures into consideration and allowing for the fact that the pigment constitutes only part of the ink, I prefer to divide the pigments for printing ink into three classes:—

Class I.—Those which may be used unconditionally, containing less than 1 part of arsenic in 50,000 of pigment.

Class II.—Those containing between 1 to 20,000 and 1 in 50,000, which may be used in conjunction with those of Class I.

Class III.—Those containing over 1 in 20,000, which are rejected.

As the printing ink contains a considerable proportion of oil medium, the final amount of arsenic in the ink will be less than these limits. A maximum of less than 1 part of arsenic in 50,000 of printing ink is thus ensured.

DISTRIBUTION OF ARSENIC AMONG COLOURS.—With regard to the distribution of arsenic among colours, there appears to be no definite rule, though violets, blues, and some greens are particularly liable to contain large amounts. Pigments of these shades can, however, be obtained which satisfy the limits suggested.

I have generally found that the pigments are distributed indiscriminately between Classes I, II, and III, though where successive batches of one colour

from the same maker are concerned, the variation is seldom sufficient to move it from one class to another.

Thirty-seven colours examined by me were distributed as follows: In the case of Class III the amount of arsenic was still small, generally less than 1 in 10,000.

Class I.		Class II.	
Lead chromes	7	Lead chrome	1
Blue lakes	2	Yellow lakes	2
Green lakes	1	Red lake	1
Red lakes	3	Prussion blue	1
Madder lake	1		
Prussian blue	1	Class III.	
Ultramarine	1	Lead chrome	1
Yellow ochre	1	Lead chrome lakes ..	2
Zinc chrome	1	Yellow lakes	4
Carbon black	1	Blue lakes	2
		Violet lakes	2
		Red lakes	1
		White lead	1

The method of determination was by ignition with lime and magnesia, followed by solution in hydrochloric acid and reduction with sulphurous acid. Arsenic was determined by the Marsh process on an aliquot portion of the filtrate.

It is necessary to test the stain carefully for the presence of antimony when dealing with printing inks and pigments since in addition to the use of tartar emetic as a precipitant for basic dyes, antimony in the form of its oxide is largely used at the present time as a pigment.

Summary.—(1) Arsenic in appreciable quantity may be found in all classes of pigment used in printing ink.

(2) A limit of 1 part of arsenic in 50,000 of printing ink should satisfy the most stringent demands of Public Health authorities.

(3) At the same time, this method of fixing a limit is unsatisfactory, and a definite ruling expressed in terms of weight of arsenic per unit area of wrapper or carton is desirable.

I have to thank Messrs. B. Winstone & Sons, Ltd., for permission to publish these results.

The Laboratory,
1, Gough Square, E.C.4.

Discussion.

The President remarked that at a time when arsenic was very much before the public eye the question of arsenic in such things as wrappers was important. But he did not think it could be dealt with on broad lines: each case had to be considered separately, as it was not so much a question of surface as of solubility.

He mentioned the case of tin paper on chocolates; this invariably contained arsenic, introduced with antimony during the hardening process, but it never got into the chocolate.

Dr. J. A. VOELCKER asked whether the arsenic was in a soluble or an insoluble form. The amount present in the paper was not so important as long as it was not in form capable of being transferred to the food.

Mr. G. D. ELSDON remarked that the wrappings of certain chocolates that had come under his notice contained one third grain of arsenic per square foot. In most cases it was in an insoluble form, though what would happen if the paper were eaten whole he did not know. He had found as much as 10 per cent. in the actual pigment; but there was a growing tendency to ask manufacturers to make their colours more arsenic-free than previously. He thought five parts per million was a stringent enough standard, as the necessity for a strict standard was not so important as in the case of beer, for example.

Mr. J. J. V. BACKES mentioned that he had approached the printers on this subject, and that, although they would not guarantee their inks as "arsenic-free," they were prepared to work to a limit of 1 part of arsenic in 20,000 of ink, or 1/3000 grain per square inch of printed paper. In some instances the question was asked: "What about lead?" Lead might reach a high proportion in some inks, and the printers had suggested 2 per cent. as a reasonable limit, although they were prepared to work to 0·2 per cent. if necessary.

Mr. T. HEDLEY BARRY, replying, said that the question of solubility would hardly arise in the case of printing ink. If present as an ingredient of the pigment, the arsenic would be used as a precipitant for a basic dye and the compound so formed was not likely to be any less toxic than the original arsenious acid.

Apart from the interesting case of tin foil mentioned by the President, the only important case of insoluble arsenic known to him was that of shellac. Arsenic in the form of orpiment was present to the extent of about 3 per cent. in ordinary T.N. shellac. Shellac was used to a considerable extent in tinting metal foils, and probably this also was a case of the insolubility of arsenic rendering it innocuous. When present as an impurity, arsenic probably came either from the chemicals used in the process of manufacture, particularly sulphuric acid, or through contamination with arsenical materials used in the same factory. In these cases the arsenic would almost certainly be present in soluble form.

With regard to the standards suggested it might be pointed out that the limits mentioned by Mr. Backes were not consistent, for 1/3000 grains arsenic per square inch would be equal to about 0·05 grm. per square metre, whereas one part of arsenic in 20,000 of printing ink would be only about 0·0001 grm. per square metre.

He (the author) fully realised that considerably more work was necessary before a final and official limit could be fixed, but the limit suggested appeared to be practical and sufficiently strict to be an adequate safeguard for the public. He fully agreed with Mr. Elsdon that there was a tendency to over-estimate the dangers of arsenical poisoning. Presumably Mr. Elsdon's limit of five parts per million would refer to the wrapper as a whole, and it had been shown that this limit would be more than covered by a limit of one in 50,000 of printing ink.

Unfortunately, public opinion on these matters was spasmodic, with the result that at one time impossible limits were demanded and at others the matter was ignored altogether. It appeared, therefore, all the more important that the question should be discussed and a decision arrived at without the authorities being stampeded by a popular scare.

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.

FORMALDEHYDE IN FISH.

We have read with a great deal of interest a paper by Arnold R. Tankard and D. J. T. Bagnall entitled "The Examination of Fish for Formaldehyde," appearing in the November, 1926, issue of THE ANALYST. The conclusions of these authors seem to be well founded, especially in that it appears that their tests were made directly on extracts of the fish.

We feel, however, that none of the results obtained by us were due to formaldehyde produced by the oxidation of trimethylamine, for the following reasons: (1) The products examined by us contained little or no formaldehyde at the time of canning, this substance developing with age in the canned product, in the absence of oxygen.

(2) Immediately upon opening a can to be tested, the sample was mixed with an approximately equal quantity (c.c. for grm.) of a two per cent. solution of phosphoric acid, and the mixture steam-distilled, the volume of this mixture being kept fairly constant by immersion of the flask containing it in a heated salt bath. Practically all of the tests were made on distillates obtained in this manner.

D. B. DILL and P. B. CLARK.

UNITED STATES DEPT. OF AGRICULTURE,
BUREAU OF CHEMISTRY,
SAN FRANCISCO, CALIF., U.S.A.

DETERMINATION OF CARBON DIOXIDE IN CARBONATES.

The method of Van Slyke as modified by Hepburn (ANALYST, 1926, **51**, 622) has been found very useful in the Central Analytical Laboratory of the British Dyestuffs Corporation, Ltd., for determining the amount of carbonate in dyestuffs.

Owing to the intense colour of the solution, the usual volumetric methods are inapplicable, the methods previously employed being either the gravimetric method with the use of the Schrötter apparatus, or absorbing and weighing the evolved carbon dioxide, or titration by electrometric methods in which the quinhydrone electrode was used.

The former method is tedious and the latter method, whilst rapid and accurate, is not suitable where many analyses are to be carried out by semi-skilled persons.

The method as described by Hepburn has been tried on a number of dyestuffs containing varying amounts of sodium carbonate, and has been found to to give excellent results, agreeing very closely with those given by the above-mentioned methods. Owing to the frothing which usually takes place with such substances on addition of the first few c.c. of acid, it has been found necessary to wet the dry dyestuff with 1 to 2 c.c. of alcohol, which effectually prevents frothing and has no effect on the result. It was found that 6 hours were ample to complete the reaction, but, as a routine method, it is advisable to start the determination late in the evening and complete it the following morning.

T. CALLAN.

Notes from the Reports of Public Analysts.

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

CITY OF BIRMINGHAM.

Report of the City Analyst for the Fourth Quarter, 1926.

During the last quarter of 1926 the total number of samples analysed was 1340, of which 1184 were taken under the Sale of Food and Drugs Acts. Of these 1184 samples, 1123 were bought informally (28 adulterated) and 61 were formal samples (7 adulterated). There were 570 informal milk samples (13 adulterated) and 60 formal samples (7 adulterated).

Boric Acid in Butter and Margarine.—Of the 109 samples of butter examined, 99 were free from boric acid, and the other 10 contained 0·1 or 0·2 per cent. In the third quarter of the year, 90 per cent. of the samples of margarine contained boric acid, but in the fourth quarter the proportion fell to 50 per cent.

Potassium Salts.—The British Pharmacopoeia requires that *acetate of potash* shall contain at least 89·8 per cent. of the pure dry salt ($KC_2H_3O_2$) and not more than 10 per cent. of moisture. Seven of the 10 samples complied with this requirement, containing from 89·8 to 96·1 per cent. of $KC_2H_3O_2$, and from 2·9 to 8·4 per cent. of moisture. One sample was slightly below the limit, the proportions being 85·8 per cent. and 12·4 per cent., respectively. It was passed as "low quality."

Two informal samples contained only 76·5 per cent. and 82·5 per cent. and lost 18·5 per cent. and 16·2 per cent. of moisture, respectively. These samples were in a moist, pasty condition and were obviously unfit for sale.

Nine samples of *citrate of potash* were of good quality, the lowest being 98·9 per cent. in purity. The single samples of *sulphate of potash* and *sulphurated potash* complied with the B.P. requirements, the latter containing 42·6 per cent. of sulphur.

All the above mentioned salts of potash, as well as six samples of *chlorate of potash*, were free from arsenic and contained less than the small amounts of lead allowed by the B.P.

Citrate of Lithia. Four of the five samples were above the B.P. limit of purity and lost from 18·1 to 20·5 per cent. on drying. Another sample had probably been carelessly kept, as it lost 23·6 per cent. of moisture on drying.

Dried Sulphate of Iron.—This drug is required by the B.P. to contain at least 77 per cent. of the anhydrous salt, and 6 samples contained from 77·0 to 84·1 per cent. On drying they lost from 0·6 to 8·4 per cent. of moisture. Five informal samples, which had probably been kept in imperfectly closed bottles, were unsatisfactory. One contained 74·8 and 11·5 per cent. of moisture, C. 3171 had 71·7 per cent. and lost 11·5 per cent of moisture, and C. 3175 had 69·5 per cent. and lost 19·8 per cent. of moisture. In each case, removal of the excess of moisture would have made these samples above the B.P. limit. The vendors of the two latter samples were cautioned.

The B.P. requires that this article shall be "slowly, though *entirely soluble* in water," a requirement which appears too stringent, as none of the samples complied with it.

The amount insoluble varied from 0·3 to 3·3 per cent. The latter had probably been overheated in the preparation. (*Cf.* Abstract 239.)

IRON PILLS.—These pills are required by the last two editions of the B.P. to be made from sulphate of iron and carbonate of soda. The 1914 edition increased the proportion of dried ferrous sulphate by about 11 per cent., and at the same time raised the required amount of ferrous carbonate by a similar proportion, namely, from 20 to 22·5 per cent. Apparently the compilors of the Pharmacopoeia overlooked the fact that with the increase in the *quantity* of dried ferrous sulphate they had allowed a lower *quality* of it to be used. If B.P. materials are used, the pill need not contain more than 20 per cent. of ferrous carbonate instead of the 22·5 per cent. stated.

Twenty-five of the 26 samples contained 0·9 grain of ferrous carbonate, and over, and were passed as genuine, but one informal sample had only 0·6 grain and was probably a four-grain pill sold in mistake for a five grain pill.

Five informal samples from three different vendors had not been prepared, as the B.P. requires, from ferrous sulphate, but from ferrous carbonate, and were, therefore, "carbonate of iron pills" and not "iron pills." The vendors were cautioned and the articles were withdrawn from sale. Iron pills and carbonate of iron pills are two distinct articles, and one should not be substituted for the other.

One of the samples was deficient in carbonate of soda, so that part of the iron was present in the form of sulphate of iron. The B.P. requires that the carbonate of soda shall be slightly in excess. As the pills were coated, experiments were made to ascertain the rate of breaking down, and a great deal of difference was found in the time required to do this.

As a rule, the outer coat, whether pearl, sugar or gelatin, was removed in a short time, but in some cases there was an inner coat which was insoluble in water or warm dilute acid, though it might possibly be broken down during the passage through the human body.

This raised a new problem: Should a pill which was of the correct composition, but slow in breaking down, be described as "not of the nature, substance, and quality of the article demanded by the purchaser?" I felt unable to call such pills adulterated, and reported on them as "genuine, but of inferior solubility." (*Cf.* Abstract, p. 239.)

J. F. LIVERSEEGE.

Legal Note.

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.

SALE OF MIXED GRAIN AS SUSSEX OATS.

ON February 25, a firm of millers was summoned at Stoke-on-Trent for having caused, or permitted, an invoice referring to an article, described as "Sussex oats," sold by the company to a Stoke-on-Trent grocer, to be false in a material particular to the prejudice of the customer, in that the article was not Sussex oats, but a mixture of grounds oats, tapioca meal, ground wheat and barley.

Mr. J. M. Dodds, for the prosecution, pointed out that under sec. 1, sub.-sec. (2) of the Fertilisers and Feeding Stuffs Act, 1893-1906, every person who

sold for the use of cattle or poultry an article artificially prepared must supply with it an invoice stating how the article had been prepared. The official sampler under the Act had taken a sample from an unopened bag of the article, and the bag contained an invoice for "one cwt. Sussex ground oats" which stated: "In conforming to the Fertilisers and Feeding Stuffs Acts (1893–1906) we hereby declare that all feeding stuffs not specially described as pure are prepared from more than one substance or seed. The acceptance of goods under this invoice is to be deemed the acceptance of this condition." Analyses of samples by the Official Analyst and by the Government Laboratories had shown that the article was a mixture and did not consist of Sussex ground oats. The Government certificate showed that the material consisted of 65 per cent. of ground oats, 20 per cent. of tapioca meal, and 15 per cent. of ground wheat and barley.

An official sampler, in cross-examination, said that he quite understood that "Sussex Ground Oats" was a trade name, and that no one who bought the oats expected to get oats ground in Sussex. He would not agree with the use of the term unless the material contained 95 per cent. of ground oats.

Mr. Beresford, for the defence, submitted that the prosecution must prove that the article was prejudicial to the purchaser, and since the invoice clearly stated that unless the goods were stated to be pure they were a mixture, the purchaser could not be said to have been prejudiced.

A director of the firm said that since the firm started in 1845, there had never been any proceedings against them. Sussex oats were not considered to be the best oats, and that would no doubt account for the small percentage of impurity. He had never heard of a standard for Sussex oats.

The stipendiary said that, supposing the invoice was not sufficient for the purposes of the Act, he would be prepared to say that the insufficiency was not due to any desire to impose on the customers of the firm; in order to comply with the Acts, the notice on the invoice should be re-worded. Having regard to all the circumstances, and to make it quite clear that he was casting no reflection on the reputation of the firm, he had decided to dismiss the summons on the payment of costs, amounting to £2 17s.

Parliamentary Note.

THE SALE OF FOOD AND DRUGS BILL, 1927.

THE text of this Bill, which was presented by Mr. A. E. Jacob, M.P. for East Toxteth, Liverpool, on February 16, passed its second reading on February 21, and was passed by the House of Lords on April 6, is as follows:—

The object of this Bill is to give effect to a recommendation of the Departmental Committee on the Use of Preservatives and Colouring Matters in Food.

The Committee recommended that any prohibitions or limitations imposed by the regulations should bind the courts in proceedings taken under the Sale of Food and Drugs Acts.

The text of the Bill "to amend the Sale of Food and Drugs Acts, 1875 to 1907," is as follows:—

Whereas it is expedient to give effect to the recommendations of the Departmental Committee on the use of Preservatives and Colouring Matters in Food: Be it therefore enacted by the King's most Excellent Majesty, by and with the advice and consent of the Lords Spiritual and Temporal, and Commons, in this present Parliament assembled, and by the authority of the same, as follows:—

1. Where any regulations made under the Public Health (Regulations as to Food) Act, 1907, prescribe the composition of any article of food or drink or prohibit or restrict the addition

of any preservative or other ingredient or material to any such article, such regulations shall be deemed for the purposes of the Sale of Food and Drugs Acts, 1875 to 1907, to define the nature, substance, and quality of the article as regards the presence or amount of any ingredient or material specified in the regulations and to determine whether the addition of any such ingredient or material, and, if so, what amount thereof renders the article injurious to health.

2. This Act may be cited as the Sale of Food and Drugs Act, 1927, and shall be construed as one with the Sale of Food and Drugs Acts, 1875 to 1907, and those Acts, and this Act may be cited together as the Sale of Food and Drugs Acts, 1875 to 1927.

3. This Act shall come into operation at the date of the passing of this Act.

Ministry of Health.

REPORT OF THE DEPARTMENTAL COMMITTEE ON THE TREATMENT OF FLOUR WITH CHEMICAL SUBSTANCES.*

ON June 16, 1924, the reference of the Committee appointed to enquire into the use of preservatives and colouring matters in food was extended so as to include the question: "Whether and to what extent the practice of treating flour with chemical substances is objectionable on grounds of health and whether it is desirable in the interests of the public health that the practice should be prohibited or restricted and in the latter case what restrictions should be imposed."

The Committee met on 21 days and heard the evidence of 28 witnesses, including medical men (pathologists, pharmacologists, and medical chemists), agricultural chemists, millers, flour importers and bakers. Certain mills were also visited and the milling and treating processes were seen in operation.

The Report, dated February 14, 1927, first gives an outline of the statutory provisions affecting flour and bread in this country, and of the regulations affecting flour in other countries, and then proceeds to consider the objects for which flour is treated with chemical agents, and the nature of the substances added to flour. The nitrogen peroxide bleaching process, and, to a less degree, the use of phosphates as improvers were described and discussed in Dr. J. M. Hamill's Report in 1911 (Cd. 5613). Since that time the number of improving substances and of processes adopted has been much enlarged.

ACTION OF IMPROVERS AND BLEACHING AGENTS.—These substances are used with the object of adjusting deficiences in the blends of wheat due to natural variations, and to produce in newly milled flour the effects of natural ageing. Various hypotheses to account for the changes in the flour thus effected are described and discussed.

Chlorine is principally used as a bleaching agent on the lower grades of flour, but its use is restricted by the fact that it has no decolorising action on the particles of bran.

Nitrogen trichloride is stated to increase the colloidal properties of the gluten," increase slightly the "Hydration capacity" and to give a livelier fermentation, greater dough stability and greater "oven spring." The addition of ammonium persulphate is said to favour the production of acid, but not gas, by the yeast. Acid calcium phosphate acts as a "strengthener" of the gluten and also acts as a yeast food, whilst benzoyl peroxide is used, for its action as a bleaching agent, in a proprietary preparation which contains 75 per cent. of acid calcium phosphate and 25 per cent. of benzoyl peroxide. Its bleaching action is not immediate, the full degree of whiteness being attained in three or four days.

* H.M. Stationery Office. Pp. 24. 1927. Price 6d. net.

COMMERCIAL CONSIDERATIONS.—The general claims made for the use of improvers and bleaching agents are: (1) That uniform products can be obtained, notwithstanding the variety of wheats used; (2) that millers can use any supplies of sound wheat available, and thus avoid great price fluctuations; and (3) that it enables more English wheat to be used. The Committee do not seriously doubt that the use of improvers has been a valuable aid to home millers in their competition with oversea millers, and that its prohibition might be prejudicial to them. On the other hand, it is possible that, owing to the existence of improvers, millers are less concerned in securing a really well-balanced blend than they were in the days when they had no improvers to fall back upon for correcting the deficiencies in gluten quality.

An argument brought against the use of improvers is that by their means an extra yield of loaves per sack can be obtained, since they enable the flour to absorb more water, and so substitute a certain amount of water for flour in each loaf. On the whole, the figures for water determinations quoted to the Committee do not point consistently one way or the other, and there would appear to be little in this charge.

With regard to bleaching agents, the Committee have little doubt that, by appropriate treatment, flour which would otherwise have to be sold as "household" can be "lifted" into the patent grade. This "lifting" of grades appears to the Committee to be purely a question of commercial propriety concerning which they do not feel called upon to make any comment.

In regard both to improvers and to bleaching agents, the Committee consider that any restriction of the practice of bleaching and improving must apply to imported as well as to home-milled flour.

CONSIDERATIONS OF HEALTH.—Although the Committee's view is that flour should be a product of the milling of wheat without the addition of any foreign substance, they have been confronted by the fact that to bring the great bulk of flour sold for bread-making at the present time within that description would involve interference with practices which have been widely adopted, and which are claimed by traders, with some reasonableness, to be necessary, unless wheat supplies are to be wasted and flour prices inevitably raised. Moreover, in the opinion of the Committee, it seems that, so long as a great demand exists in this country for white bread, some form of bleaching process must be permitted.

Although the majority of the substances used are foreign to flour, it has been urged that acid calcium phosphate, sulphates and persulphates may be regarded as merely contributing to the phosphates and sulphates normally present in the grain; but against this is the fact that the phosphorus in the grain is largely, if not entirely, present in forms of combination other than phosphates in the grain; and the same argument is probably true of sulphur. Nitrogen peroxide, benzoyl peroxide, chlorine and nitrogen trichloride are even more obviously foreign to flour, and there can be no suggestion that their use contributes anything which is normally present in flour.

An obvious method of investigating the presence of harmful compounds or suspected impairment of nutritive properties is by feeding experiments with animals. When such experiments give positive results, they are conclusive, but negative results cannot be regarded as proof that the material tested is certainly innocuous or that its nutritive properties have not been damaged in some subtle but important respect. The results obtained by feeding experiments are merely the summation of a long series of subtle metabolic changes which by themselves elude our present imperfect methods of detection. It is quite possible that the composition of a food material might be so altered in an adverse direction that

although the body would be capable of dealing with it, an extra strain would be imposed upon the tissues and cells of the body which they should not be called upon to bear. The tissues of the body possess marked powers of adaptation to adverse circumstances and exercise their adaptability in dealing with deleterious materials, but it is not desirable to tax this adaptability without necessity.

With a view to ascertaining whether the commercial treatment of flour with chlorine affected the flour so as to produce harmful substances or impair its nutritive value, feeding experiments were carried out on rats with flour treated commercially with chlorine and nitrogen trichloride. Briefly, it may be said that the results obtained were inconclusive. No significant differences were observed between the rats fed on the treated flour and the rats fed as a control on the same flour untreated. A positive result would have sufficed to condemn out of hand the treatment of flour with chlorine. For the reasons already given, however, a negative result in a feeding experiment of this kind cannot be regarded as equally conclusive. Of all the materials used for the treatment of flour, the Committee regard as least objectionable those which produce no obvious change in the composition of the flour, and which in the amounts in which they are used appear to have no deleterious action themselves upon the tissues. Acid phosphates may be taken as an example of such materials.

AN ALTERNATIVE METHOD OF IMPROVING.—Patents have recently been taken out for a process of improving flour by physical means. This process consists essentially in heating the wheat or flour for a definite time, at a definite temperature, with the result that the baking properties of the flour are greatly improved. In the opinion of the Committee the results already obtained are promising, and "if elucidation of the subject should enable the use of chemical improvers to be superseded by physical methods, such an outcome would be very desirable."

CONCLUSIONS AND RECOMMENDATIONS.—"In conclusion, while we consider that a staple and indispensable foodstuff such as flour, the purity and wholesomeness of which are of cardinal importance to the community, should be jealously guarded against unnecessary treatment with foreign substances, we are not prepared, on the present knowledge available, to recommend the complete elimination of the bleaching agents and improvers now in use. Our view is that in the first instance it should suffice to limit the use of these substances to those which appear least open to objection when judged along the lines we have indicated. We think that chlorine, nitrogen trichloride and benzoyl peroxide should not be amongst these.

"It seems to us that much could be done by the trade itself. It has been represented to us that many bakers are opposed to the use of improvers in flour. There are bakers who wish to know the nature of the materials they are using, and who want flour and not a mixture of flour with something else, and insist on being supplied with untreated flour. It is important that this demand should be met, and if it cannot be satisfied by the home millers, it will be met by further recourse to imported flour.

"We have been impressed by the evidence which we have received at a late stage in our enquiry in regard to the possibilities of improving flour by physical rather than chemical methods and by the success which has already attended experiments on these lines on a commercial scale. If improving is necessary, it is in this direction rather than in the use of chemical substances that we should like to see progress made.

"We think that it should be compulsory for the manufacturers of chemical substances for use either as bleaching agents or improvers or both to declare

to their purchasers the nature of the ingredients of which they are composed, and that millers should be required to inform their customers whether their flour has undergone a process of chemical bleaching or improving, or both, and if so, with what substances and in what proportions."

APPENDIX A.

LIST OF WITNESSES EXAMINED.

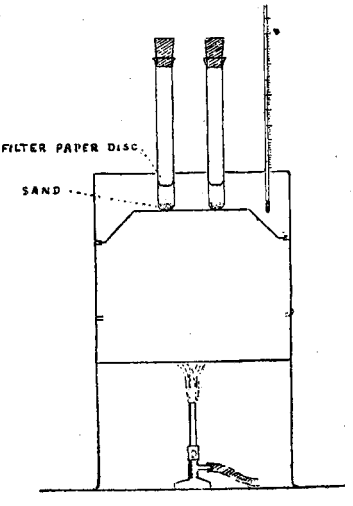
APPENDIX B.

The list of substances recommended by the National Association of British and Irish Millers, Ltd., for use as improvers and bleaching agents, and the limits within which the use of each might, in their opinion, be permitted is as follows:—

Substance.	Amount Per cent.	Equivalent amount per 280 lb. of flour.
Extracts of malt and germ	.. ..	No limit.
Acid phosphate of calcium ..	0·3	= 13½ oz.
Acid phosphate of ammonium ..	0·2	= 9 oz.
Persulphates	0·04	= 1·8 oz.
Chlorine	0·07	= 3 oz.
Bleaching with peroxide of nitrogen: nitrites as sodium nitrite in bleached flour	0·0008 (for Irish Trade)	= 8 parts per million of flour.
,, ,, ,, ..	0·0006 (for English Trade)	= 6 parts per million of flour.

DETERMINATION OF BENZOIC ACID IN FOODSTUFFS.*

The extraction, purification, natural occurrence, and confirmatory tests for benzoic acid are discussed, the most delicate test being that of Mohler (*cf.* ANALYST, 1927, 153). The following method is recommended for the determination of benzoic acid in fruits and vegetables. The sample (30 to 200 grms.) is mixed with water, if necessary, an excess of common salt added, and the mixture acidified with phosphoric acid and distilled in steam for about 1½ hours into a dish containing 10 c.c. of *N* sodium hydroxide solution. The condenser is washed with 25 c.c. of 0·1 *N* sodium hydroxide solution, and the combined washings and distillate (about 500 c.c.) evaporated on the water-bath to about 20 c.c. in 2½ hours. The solution is then oxidised with excess of potassium permanganate solution (6 per cent.) at about 45° C., and the liquid rendered clear and colourless by the addition of sodium sulphite and acidification with sulphuric acid. The solution (about 70 c.c.) is saturated with salt, and extracted in a 100 c.c. cylinder four times with 15 c.c. of a mixture of equal volumes of methylated ether and petroleum spirit (b.pt. 30° to 50° C.). Violent shaking is avoided, and each extract is transferred to a stoppered test-tube by suction through one of two tubes in the stopper. The other tube has the end turned up (as in the Werner-Schmid apparatus for milk analysis), and dips into the non-aqueous layer in the cylinder. The tube is washed with solvent after each



* By Dr. G. W. MONIER-WILLIAMS (*Ministry of Health. Reports on Public Health and Medical Subjects.* No. 39. January 1927. P. 57).

extraction, and this liquid is used subsequently. The test-tube is immersed in water at 30° C., and the solvent removed in a current of dried air in about 1 hour. The benzoic acid is then sublimed by a modification of Polenske's method (ANALYST, **1911**, **36**, 584) in the apparatus shown in the figure. It is mixed with washed and ignited sand (2 grms.) in the lightly stoppered test-tube, the lower 4 cm. of which are firmly fixed in an air-oven, and heated for 1 to 1½ hours at 160° C. The portion of the tube below the disc of filter-paper shown is cut off, and any crystals caught by the paper are returned to the bulk in the top half of the tube. The sublimate is dried in a desiccator, weighed, dissolved out, and the tube reweighed. Errors of the order of 1 to 2 mgrms. were obtained on 20 to 60 mgrms. of added benzoic acid. In the presence of certain substances (*e.g.* cinnamon) benzoic acid may be produced during the oxidation. Saccharin is not appreciably steam-volatile under these conditions. Salicylic and cinnamic acids are determined in the same way, but the oxidation process is omitted, as it destroys the former and converts the latter partly into benzoic acid. The methods of Autenrieth and Beuttel (ANALYST, 1910, **35**, 218) and of De Jong (*ibid.*, 1910, **35**, 129) are recommended for the determination of salicylic and cinnamic acids, respectively, in the presence of benzoic acid or of each other. J. G.

Government of Palestine.

ANNUAL REPORT OF THE DEPARTMENT OF PUBLIC HEALTH FOR THE YEAR 1925.

IN the Chemical Sub-Section of the Report, the Government Analyst, Mr. G. W. Baker, gives an outline of the work done for the different Government departments during the year. The total number of samples examined was 5512, as compared with 4132 in 1924.

Department of Health.

MILK.—Routine examination of milk taken from hospitals and street vendors is carried out in Jerusalem and in nine other municipal areas. Of the 3990 samples examined, 212 were the subject of prosecutions (5·3 per cent., as compared with 7·8 per cent. in 1924).

SEMNI.—In view of the considerable amount of adulteration as indicated in the last report (ANALYST, 1926, **51**, 300), steps were taken to secure a much larger number of samples from the markets and at the ports of entry, and 55 of the 167 samples examined were found to be grossly adulterated. The adulterant is generally added in such large quantities that samples giving analytical values on the border line are of comparatively infrequent occurrence.

Control is complicated by the fact that the adulterated or "mixed" semni is a recognised commodity sold at a much lower price than the genuine semni. It is, however, frequently sold as pure semni at considerable profit. Several successful prosecutions have been instituted in such cases.

ALCOHOL AND ALCOHOLIC LIQUORS.—The denaturing regulations mentioned in the last report (*loc. cit.*) have resulted in a considerable decrease in the amount of alcohol imported into the country, but it is difficult to judge the general effect upon the extensive trade in spurious liquors of local manufacture. It is established that various attempts have been made to produce a potable spirit from the denatured alcohol.

Eight thousand litres of liquor discovered in one distillery contained mineral naphtha and a flavouring essence, the inference being that denatured alcohol had been distilled and then flavoured to imitate cognac. It appears that the local palate will tolerate mineral naphtha (kerosene) in a liquor if it is disguised by the addition of a strong flavouring substance, such as anethol. It has been recommended to the Director of Customs, Excise and Trade that pyridine and wood naphtha should be used as denaturants, and that the resulting methylated spirit be sold under a poison label.

The new licensing conditions, which define the methods and materials to be used in distilleries, are in process of application. It is anticipated that these, in conjunction with the strict Excise control to be instituted in the near future, will greatly facilitate matters.

In the meantime some headway has been made. Essences intended for the manufacture of spurious liquors are now prohibited imports, and imported liquors to be allowed entry must now be adequately labelled with the name of the manufacturer or responsible exporter, the nature of the contents and the country of origin. It is anticipated that this provision will check the growing trade in foreign brands, the quality of which is so indifferent that the manufacturer prefers to remain incognito.

AERATED WATERS AND CORDIALS.—The 334 samples were mainly taken from the local factories and retail establishments by the district inspectors. The chemical examination included tests for lead and other heavy metals and for saccharin. Previous to the introduction of special precautions in the factories excessive quantities of lead, derived from the pipes used, were often found. Now it is rare to find more than one part per million.

DRUGS AND DISINFECTANTS.—The routine examination of the bleaching powder in use for chlorinating water supplies has demonstrated a considerable advantage in favour of the stabilised powder, there being practically no loss of chlorine during storage, whereas the reverse was the case when the unstabilised powder was in use.

Of 19 samples of various makes of acetyl-salicylic acid tablets examined, 18 contained free salicylic acid. Two samples of crystalline acetyl-salicylic acid, out of six examined, contained traces of free salicylic acid when examined by the B.P. test.

Although very large quantities of cocaine, hashish, and opium are confiscated by the Police and Customs Authorities, it is seldom that samples of these are received in the Laboratory for identification. Two samples of suspected cocaine proved to be acetyl-salicylic acid alone in one case, and in the other acetyl-salicylic acid with a very slight trace of cocaine. The paper in which this latter sample was contained was stained in a way which suggested exposure to perspiration from having been carried next to the skin, and it was found that this stained portion was impregnated with cocaine. The inference was that, originally, a mixture of acetyl-salicylic acid and cocaine had been in the packet, but that on becoming damp nearly all the cocaine had dissolved and had been absorbed into the paper.

It is known that cocaine is frequently adulterated with acetyl-salicylic acid, their appearances being similar.

In a mixture supposed to contain 4 minims of hydrocyanic acid dil. in 8 oz. no hydrocyanic acid could be detected. Control tests, however, demonstrated the fact that all the hydrocyanic acid is eliminated from a mixture made up according to the prescription if kept for a week or more in a corked bottle. This led to an investigation of the aeration method as applied to the determination

of very small amounts of hydrocyanic acid and the stability of that acid in medicinal preparations.

A powder examined contained over four times the prescribed quantity of *Fol. digitalis.*

Medico-legal specimens and Criminal Investigations.

COUNTERFEIT COINS.—Over fifty of these have been received. The majority were tendered by passengers on the railway. A close examination of the coin with a lens generally indicates that it has been cast and not stamped, and a low specific gravity generally means a deficiency of silver and excess of copper. Those with a high specific gravity have been found to contain lead. Some coins, undoubtedly counterfeit, are of normal specific gravity and contain the normal percentage of silver, whilst some have been received which contain an excess of silver.

The analyst was called to give evidence in a case where materials suspected of being used in the manufacture of coins had been seized and submitted for chemical examination. The materials included the following: (1) Graphite and clay crucibles; (2) circular iron moulds; (3) ingots of metal, chiefly alloys of silver copper and tin; (4) a plastic substance composed of sand and a heavy mineral oil in which had been incorporated metallic mercury.

One of the pieces of metal found contained gold, and the defence claimed that the accused was not making coins, but had been engaged in the time honoured pursuit of changing base metals into gold.

CRIMINAL CUTTING OF TREES.—This is a favourite form of spite or revenge. In one case 200 olive trees had been cut down, and certain saws in the possession of suspected persons were sent to the laboratory for examination. Deposits of sawdust and dried sap were clinging round the teeth of the saws, and it was found that the tannin in these deposits gave reactions identical with those obtained with olive-wood tannin and differing from reactions given by the tannin from all the eight other species of wood examined. These results, together with those furnished by microscopical examination, were sufficient to establish presumptive evidence that the saws had been last used to cut olive wood.

POISONING.—Out of 13 specimens of viscera, vomit, and excretions in cases of suspected human poisoning, five yielded positive findings. The poisoning agent in two of these was arsenic, 1 mercury, 1 caustic soda, and 1 was zinc sulphate.

Seven specimens of animal viscera were received from Veterinary Officers. In four of these arsenic was detected. The rest gave negative results.

Four specimens of food and drugs suspected of being concerned in cases of poisoning or attempted poisoning have been examined. In all these specimens arsenic was detected.

Special Investigations.

Little time has been found for work other than that connected with the examination of samples received.

FREE SALICYLIC ACID IN ASPIRIN.—An investigation is in hand in connection with excess of free salicylic acid in "aspirin" tablets.

The experiments are being conducted to ascertain whether other ingredients used in the manufacture of aspirin tablets have an influence on the stability of the acetosalicylic acid.

In this connection, note has been taken of the observations of Martindale and Westcott on the hydrolysis of acetosalicylic acid by dilute acid and the sensitiveness of the B.P. test for free salicylic acid.

STABILITY OF HYDROCYANIC ACID.—In connection with an investigation into the stability of hydrocyanic acid in medical preparations an adaptation of the aeration method of determining cyanides was used (ANALYST, 1923, **48**, 566). The investigation is being continued with numerous medicinal preparations in which hydrocyanic acid is an ingredient.

TREATMENT OF TENTAGE.—There has been some further work on the copper soap treatment of tentage against the diamond spot fungus, mention of which was made in the last report.

Certain tents have been examined twelve months after treatment, and it has been reported that there is no new infection on the treated tents, and that the growth of the fungus appears to have been arrested on those tents which were infected before treatment. All Department of Health tentage is now treated annually by this process. Several other departments have made enquiries concerning the treatment.

New Zealand.

AMENDING REGULATIONS UNDER THE SALE OF FOOD AND DRUGS ACT, 1908.

THE amending regulations of 1926 included, *inter alia*, the following:—

LABELLING OF PRESERVATIVES, ETC.—Regulations 10, 11 and 12, of the principal regulations are amended by the addition of clauses specifying the nature of the declaration (and the size of type) on labels to be attached to packages containing preservatives, food colouring matters, and artificial sweetening agents.

MILK BREAD (17) (*a*).—Milk-bread shall be white bread or bread to which milk, skimmed milk, condensed, dried, or separated milk has been added. It shall otherwise conform to the standard of white bread.

IMPORTED EGGS (8).—The regulation shall not apply to the eggs known as "Hahm Dahn" (being salted eggs imported from China coated with a preservative composition mainly of salt and ashes) provided that the importer signs a declaration that such eggs are intended only for sale to and consumption by, Chinese, and provided that the eggs are only dealt with in this way.

MILK AND THE REDUCTASE TEST.—Regulation 42 is amended by revoking paragraph (*b*) thereof, and by the addition of the following two paragraphs:—

(*b*) "In any proceedings for an offence against the said Act, or the principal regulations in relation to milk, the fact that on the application of the reductase test the methylene blue used for the purposes of that test was completely decolorised in less than three hours shall not be sufficient evidence that the milk did not conform to the prescribed standard unless—(i) The reductase test was applied within four hours after the milk was purchased or otherwise procured; and (ii) The milk was continuously kept in an ice-cooled box from the time when it was purchased or otherwise procured as aforesaid until the application of the test.

"*Method of applying Reductase Test.* (*c*) A stock solution is prepared by dissolving one part of powdered methylene blue (not the zinc salt) in 2,000 parts of water. Immediately prior to use one part of this solution is diluted with nine parts of water. One cubic centimetre of the diluted solution is mixed with ten cubic centimetres of the milk in a test tube which is then immersed to at least the level of the contained fluid in water kept between the temperatures of 37° C. and 39° C."

CHEESE PASTE.—10. Clause (5) of Regulation 47 of the principal regulations is amended by adding thereto the following words: "It shall contain not less than seventy-five parts per centum of cheese."

ICING SUGAR is now defined as "sugar to which a farinaceous diluent substance has been added. It shall not contain any other added substance. It shall not contain more than two parts per centum of starch."

JELLY CRYSTALS.—Regulation 64 is amended by revoking clauses (1) and (2) (permitting the use of the term "fruit-jelly crystals" to flavoured confections of gelatin, sugar and citric or tartaric acid), and by the addition to clause (4) of the words: "Expressions or devices which indicate or suggest the presence of fruit or fruit juices shall not appear on any statement or label attached to any packet containing jelly crystals."

NON-FERMENTED BEVERAGES (75), (1), *Pure fruit non-fermented beverages* not otherwise standardised in the regulations shall be composed of the juices, in their natural condition, of sound fruit or fruits, potable water (impregnated or not with carbon dioxide under pressure), with or without the addition of sugar and caramel. They shall not contain any flavouring substance or colouring matter other than those naturally present in the fruits from which they have been prepared, or any added substance other than water, sugar, caramel or permitted preservative (sulphur dioxide not exceeding 1 grain per pint, or salicylic acid not exceeding 0·5 grain per pint).

Flavoured Non-fermented Beverages shall be composed of potable water (impregnated or not with carbon dioxide) and with one or more of the following: The natural or concentrated juices of sound fruit or fruits, vegetable extracts or infusions, citric acid, tartaric acid, harmless colouring matters, sugar, permitted preservative (as above).

Artificial Non-fermented Beverages are composed of potable water (impregnated or not with carbon dioxide) combined with one or more of the following: Harmless flavourings, the natural or concentrated juices of sound fruit or fruits, vegetable extractives or infusions, citric acid, tartaric acid, harmless colouring matters, sugar, permitted preservative (sulphur dioxide, not exceeding 1 grain per pint).

Amending regulations as to the labelling of these beverages are published.

Phosphoric acid or phosphate may be used in flavoured non-fermented beverages and in artificial non-fermented beverages, provided that the word "phosphate" is combined with the name, trade name, or description in lettering of uniform specified size.

Ale and Beer.—Clause 3 of Regulation 81 is amended by the provision that the beer shall not contain more than 50 grains of total chlorides (calculated as sodium chloride) per gallon.

Bibliography: Standard Methods of Analysis.

II. BEER AND BREWING MATERIALS.

OFFICIAL METHOD.

BEER.

ORIGINAL GRAVITY.

Finance Act, 1914. (5 Geo. V. C, 7, S. 10.)
J. Inst. Brew., 1914, **20**, 569–713.
(More concisely presented in *Allen's Organic Analysis*, 1924 edn., Vol. I, 203.

STANDARD METHODS.

BEER.

SULPHUROUS ACID IN.

J. Inst. Brew., 1926, **32**, 170.

MALTS, PALE, BROWN, CRYSTAL AND BLACK.

EXTRACT, MOISTURE, COLOUR, DIASTATIC POWER AND COLD WATER EXTRACT.

J. Inst. Brew., 1922, **28**, 775–786.
Allgem. Zeitsch. Bierbrau. und Malzfab., 1914, **42**, 291.
(Abstract: ANALYST, 1914, **39**, 436.)

CARAMEL.

COLOUR.

J. Inst. Brew., 1922, **28**, 785.

SUCROSE, GLUCOSE, INVERT SUGAR, ETC.

Official Methods of the Association of Official Agricultural Chemists. U.S.A. 1919.

ARSENIC IN BEER, BREWING MATERIALS, COAL, ETC.

Report, Royal Commission on Arsenical Poisoning.
Vol. II, 1903, Appendix 21, 208–215.

T. J. W.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Evaluation of Acid Cream. M. A. Dichno and O. M. Briskin. (*Z. Unters. Lebensm.*, 1926, **52**, 469–475.)—A number of chemical and bacteriological tests have been carried out on samples of cream classified according to quality under four headings. These tests include the number of bacteria, fat content, acidity, specific gravity, water-content, and arbitrary constants obtained from the fermentation and reductase tests. The product of the last two provides a numerical index which varies regularly with the other analytical numbers and

assists in the classification of the samples. The limiting figures for a bad cream are: Specific gravity 1·0135, fat-content 23·46 per cent., acid-content 0·7 per cent. as lactic acid ; a colour is produced in the reductase test in 2 hours, and coagulation in the fermentation test in 2½ hours. The specific gravity was determined on 100 c.c. of the sample mixed with 10 c.c. of ammonia of specific gravity 0·96.

J. G.

Solidification Points of Edible Fats. T. Meyer. (*Z. Unters. Lebensm.*, 1926, **52**, 461–465.)—Time-temperature curves have been plotted, according to the method of Mohr, for the following oils: Palm kernel, coconut, arachis, neutral lard, oleomargarine, butter fat, and hardened whale oil. The solidification points were obtained from the maxima and minima observed on the curves, the curve for each fat being characteristic. The results are correlated with the m.pts. of the high and low m.pt. glycerides known to be present in the fats. A mean deviation of less than ± 0·1° C. is obtained if the experimental conditions are standardised ; *i.e.*, 35 c.c. of the fat at 50° C. contained in a 50 c.c. beaker are placed in a water-bath so that the level of the fat is 2 cm. below that of the water. The temperature of the cooling water (usually 15° C.) influences the nature of the curves, especially in the case of neutral lard. The method will distinguish between margarine and butter, except in the case of a mixture such as oleomargarine (4 parts), palm kernel oil (3 parts) and hardened whale oil (3 parts). J. G.

New Value for the Determination of Butter Fat. F. v. Morgenstern. (*Z. Unters. Lebensm.*, 1926, **52**, 385–388.)—In order to eliminate the influence of the caprylic and other acids, except butyric acid, obtained on saponification of butter fat (*cf.* Kuhlmann and Grossfeld, ANALYST, 1926, **51**, 305), the soap solution may be treated with copper sulphate and filtered. Five grms. of the fat are saponified with 2 c.c. of potassium hydroxide solution (750 grms. KOH per litre) and 10 c.c. of glycerin, and the soap solution cooled and diluted with 100 c.c. of water. The liquid is then cooled to 20° C., well shaken with 10 c.c. of coconut soap solution and 60 c.c. of copper sulphate solution (50 grms. of the crystallised salt per 600 c.c.), and, after 2 to 3 hours, filtered through a large plain filter paper. The filtrate should amount to 100 c.c., and, if necessary, the copper soap must be stirred with a glass rod. The 100 c.c. of filtrate are distilled with 50 c.c. of dilute sulphuric acid (12·5 c.c. of the concentrated acid per litre) and a little pumice in a Reichert-Meissl distilling flask, 110 c.c. of distillate being collected and titrated with 0·1 *N* sodium hydroxide solution. The number of c.c. of the alkali required is the titration value or copper value. Occasionally filtration from the copper soap fails to yield 100 c.c. of filtrate ; in such cases the copper value is proportional to the volume of filtrate titrated.

For copper values 1, 1·1, 1·2 and 1·3, the respective percentages of butter fat in the sample are 0, 0·5, 1·5 and 2. Each further addition of 0·1 to the copper value corresponds with an increase of 1 per cent. of butter fat. Good results are obtained with mixtures of butter fat with coconut butter or rape oil in varying proportions. T. H. P.

Occurrence of Arsenic and Lead in Fruit as a Result of Treatment with Protecting Agents. L. Lendrich and F. Mayer. (*Z. Unters. Lebensm.*, 1926, **52**, 441–457.)—Determinations of the lead and arsenic contents of fifteen types of apples from Canada, the United States and Australia have been made on various portions of the original and washed and dried fruit. The lead was determined colorimetrically by Winkler's method after the destruction of the organic matter and the precipitation of lead sulphate, which was dissolved in ammonium acetate solution. For the arsenic determination the sample was well mixed with magnesia in the proportion 25 : 1, and 10 c.c. of fuming nitric acid added. The whole was dried on the water-bath, heated for 1 hour at 150° C., and the ashing completed over a burner. The residue was dissolved in hot 15 per cent. sulphuric acid. This method gave results with a maximum error of + 4·6 per cent., whereas direct ashing involved a maximum error of − 20 per cent. The arsenic mirrors were dissolved in a 3 c.c. stoppered cylinder filled with 2 c.c. of 0·0005 *N* iodine solution and water. To this solution were added 3 drops of cold saturated solution of sodium carbonate, 3 drops of starch solution, and 2 c.c. of a 0·0005 *N* solution of arsenic trioxide. The solution was then back-titrated with the iodine solution. The titration from a blank experiment must be deducted, and the method (Ramberg's) is capable of determining 0·001 mgrm. of arsenic. In the following table the results refer to 100 grms. of sample.

Type of dried fruit.	Arsenic content (As_2O_3). Mirror.	Titration.	Lead content (Pb).
Pears, unpeeled	0·22 mgrm.	0·34 mgrm.	0·13 mgrm.
,, ,, ...	0·02 ,,	0·029 ,,	0
Apple rings, peeled	0·01 ,,	0·015 ,,	0
,, ,, partly peeled ..	Trace	0	0
,, ,, ,, ,, ..	Trace	0	0

A more complete table shows the results obtained by the two methods for various portions of the apple. In the washed and dried fruit most of the lead and arsenic is derived from the skin, which indicates that it is not easily removed during the handling and treating processes. In most cases lead was absent, and neither lead nor arsenic was found in apples from West Virginia, Canada and West Australia. (*Cf.* ANALYST, 1926, **51**, 291). J. G.

Determination of Benzoic Acid in Foodstuffs. G. W. Monier-Williams. (*Ministry of Health. Reports on Public Health and Medical Subjects.* No. 39. January 1927, p. 57.) See *Ministry of Health Report*, p. 229.

Caffeine Content of Coffee Extracts and their Physiological Action. H. Jesser. (*Z. Unters. Lebensm.*, 1926, **52**, 389–392.)—The view that less caffeine passes into solution when coffee is extracted with sugar solution than when water is used is not substantiated by experimental data. Moreover, the physiological effects of the extracts are the same in the two cases. A cup of coffee holding 150 c.c. and corresponding with 7·5 grms. of coffee, contains about 0·09 grm. of caffeine, and a cup of black tea of similar size, about 0·025 grm. T. H. P.

Determination of Caffeine in Black Tea. W. Stüber. (*Z. Unters. Lebensm.*, 1926, **52**, 393–395.)—Fendler and Stüber's method for the determination of caffeine in coffee (ANALYST, 1916, **41**, 88) may be applied to black tea when modified as follows: From 25 to 50 grms. of the tea are powdered in a porcelain mortar, and 5 grms. of the powder are shaken for 30 minutes in a stoppered glass bottle with 5 grms. of 10 per cent. ammonia solution and 200 grms. of chloroform. The total contents of the bottle are transferred to a large pleated filter, and 150 c.c. of the filtrate are evaporated in a wide-necked flask on a water-bath, the last traces of chloroform being removed by a current of air. The residue is digested on the water-bath, and frequently swirled, with 80 c.c. of hot water, the cooled liquid being treated with 10 c.c. of 1 per cent. potassium permanganate solution and left for 15 minutes. The manganese is separated by addition of 2 c.c. of about 3 per cent. hydrogen peroxide solution containing 1 c.c. of glacial acetic acid per 100 c.c., further additions of 1 c.c. of the solution being made until the red colour of the liquid is destroyed. The flask is placed on a boiling water-bath and the acid peroxide solution added in quantities of 0·5 c.c. until such an addition fails to lighten the colour. The flask is left for 15 minutes altogether on the bath, the liquid being then cooled and filtered through a moist plain filter, about 9 cm. in diameter. Flask and filter are washed with cold water, and the clear filtrate, amounting to about 200 c.c., shaken with 50 c.c., and then with three successive quantities of 25 c.c. of chloroform. The combined chloroform extracts are evaporated in a wide-necked flask, and the residue dried at 100° C. to constant weight, which corresponds with 3·75 grms. of the tea. T. H. P.

Determination of Podophyllin. R. Eder and W. Schneiter. (*Pharm. Acta Helv.*, 1926, **1**, 15–24; *J. Pharm. Chim.*, 1927, **119**, 122.)—Half-a-grm. of finely powdered podophyllin is shaken for 30 minutes with 15 c.c. of chloroform, filtered, and 10 c.c. run into 50 grms. of petroleum spirit. The precipitate is collected on a weighed filter paper of 0·08 m. diameter, the flask and filter washed with 20 c.c. of ether, and the residue dried for 1 hour at 70° and weighed. This weight corresponds to two-thirds of the podophyllin taken and should not be less than 40 per cent. of that weight. D. G. H.

Identification and Determination of Ergot of Rye. A. Tschirch. (*Pharm. Acta Helv.*, 1926, **1**, 89-90; *J. Pharm. Chim.*, 1927, **119**, 122–123.)—The red solution produced by shaking for 2 hours one grm. of the powdered drug with 20 c.c. of ether, 10 drops of ammonia solution, and 20 drops of water, is decanted, the ether evaporated, and the residue taken up with acetic acid and filtered. Sulphuric acid containing ferric chloride is run on to the filtrate, and a blue-violet zone (ergotamine) should form at the point of junction. The acid is colourless or slightly yellow, and the acetic acid layer should show a green fluorescence (ergosterine). If these conditions are fulfilled, 0·02 per cent. of ergotamine is present in the drug and only traces of protein amines, which latter result from defective keeping conditions, such as the presence of moisture.

D. G. H.

Alleged Reaction of Cherry-Laurel Distillate. F. Morvillez and Defossez. (*J. Pharm. Chim.*, 1927, **119**, 97–100.)—Pecker's reagent was not found reliable in its action on natural cherry-laurel distillate in the presence of stannous cyanide, but Meillère's reagent (200 grms. of a 15 per cent. solution of ammonium molybdate, 20 c.c. of 50 per cent. by volume sulphuric acid and 30 c.c. of nitric acid) gave a much more marked coloration. The reducing properties of the distillate are notably augmented by the presence of stannous cyanide, and, whilst the absence of the tin may cause an artificial product to be suspected (since the commercial distillation is carried out in tinned copper vessels), its presence is not a proof of good preparation. D. G. H.

Dried Sulphate of Iron. J. F. Liverseege. (*Pharm. J.*, 1927, **118**, 106.)—Dried ferrous sulphate may absorb water fairly readily, but if kept in properly closed bottles, there should be no great deterioration. The insoluble matter in 12 bought samples varied from 0·3 to 3·3 per cent., and an increase of either temperature (above 150° C.) or time in preparing small samples produced an increase in insoluble matter and decrease in ferrous sulphate, owing to formation of basic and ferric sulphate (*cf.* p. 223). D. G. H.

Iron Pills. J. F. Liverseege. (*Pharm. J.*, 1927, **118**, 106–108.)—As a result of an investigation into the composition of iron pills on the market it is suggested that 5 grain iron pills should contain not less than 1 grain of ferrous carbonate ; that the carbonate present should be chemically equivalent to rather more than the amount of ferrous sulphate ; that talc should not be present in the pill mass ; that variation above and below the mean weight should be within 10 per cent., and that pill coats should be moderate in amount and the pill disintegrate in a reasonable time. Increased accuracy in labels is also suggested. The percentage composition, size and weight in grains of 5 typical samples of Pil. Ferri B.P. are tabulated. The determination of ferrous sulphate by means of 3 *N* phosphoric acid is advocated, with diphenylamine as indicator (*cf.* p. 224). D. G. H.

Biochemical, etc.

Micro-Method for the Determination of the Hausmann Numbers of Proteins. K. V. Thimann. (*Biochem. J.*, 1926, **20**, 1190–1195.)—The analysis for the determination of the Hausmann numbers of the proteins, originally due to Hausmann (*Z. physiol. Chem.*, 1899, **27**, 95) and modified by Osborne and Harris (*J. Amer. Chem. Soc.*, 1903, **25**, 323), has been criticised on several scores. Apart from the early criticisms, which have been answered, there are five main sources of error in the method as usually practised, and these are described. The presence of an uncontrolled amount of salt is shown to be an important factor in the determination of the composition of the precipitate of the basic fraction. A convenient micro-method for the determination of the Hausmann numbers is described which avoids the various errors. It deals with 3 to 6 mgrms. of total nitrogen

in each determination, and hence for the hydrolysis some 15 mgrms. of nitrogen should be used to allow of two determinations, *i.e.*, one-tenth the amount used in the ordinary method. The determination can be done in three days, half the time occupied by the macro-method. Some of the results obtained are given. As a general rule the amide nitrogen is higher, and the diamino-nitrogen lower, than given by the macro-method. The latter result is due to the presence of less salt at precipitation and to more perfect washing on the centrifuge than is possible on a filter. P. H. P.

Comparative Characterisation of Chondrin and Glutin. M. A. Rakusin and K. Braudo. (*Z. Unters. Lebensm.*, 1926, **52**, 397–401.)—Aqueous solutions of chondrin are so opalescent that it is impossible to read through a 15 mm. layer of a 0·2 per cent. solution. The results of adsorption experiments in which the aqueous chondrin solutions were treated with 10 per cent. of their weight of aluminium hydroxide show that a 1·2 per cent. solution undergoes true adsorption, the chondrin molecule being adsorbed as a whole. With the more dilute solutions examined, fission of the chondrin molecule occurs. Thus, with a 0·5 per cent. solution, the filtrate from the adsorbent alumina exhibits the biuret reaction and Molisch's reaction, the biuret complex and the associated carbohydrate constituent being split off. With a still more dilute solution (0·2 per cent.), the adsorption is accompanied by quantitative separation of the chondroitin-sulphuric acid, which forms a faintly acid solution, is precipitable by barium chloride, and has $[\alpha]_D - 46{\cdot}59°$. The percentage composition of gelatin is: Free amino-compounds, 0·50; biuret complex, 49·27; free carbohydrate, 0·96; combined carbohydrate, 23·77; combined chondroitin-sulphuric acid, 25·50 (*cf.* Rakusin, ANALYST, 1923, **48**, 569). T. H. P.

Colorimetric Determination of Silicon in Tissues by Isaac's Method. J. H. Foulger. (*J. Amer. Chem. Soc.*, 1927, **49**, 429–435.)—Experiments have been carried out to test Bertrand's criticisms of Isaac's micro-method for the determination of silicon (*Bull. Soc. Chim. biol.*, 1924, **6**, 157, 656.) The dry tissue (0·5 grm.) is ashed, the oxide converted into the nitrate with nitric acid, and the silica dissolved out in pure sodium hydroxide solution. To the solution, acidified with acetic acid, is added ammonium molybdate solution, and, after 5 minutes on the water-bath, the silicomolybdate is formed, which is reduced by sodium sulphite to a deep blue colour, and determined colorimetrically. The method will detect 0·00115 mgrm. of silicon. In the present paper the method is confirmed and the preparations of silicon free reagents are described. Phosphomolybdates also give the blue colour, but silicomolybdates are reduced by sodium sulphite at a much higher P_H value than is necessary for the reduction of phosphomolybdates. The retarding action of reduction due to phosphates is eliminated for amounts such as are present in the ash of animal tissue by the addition (up to 7 c.c.) of 10 per cent. acetic acid solution before reduction.

J. G.

Determination of Phosphorus in Blood. J. H. Gaddum. (*Biochem. J.*, **1926, 20**, 1204–1207.)—Since the results obtained in the colorimetric determination of phosphorus are known to be affected by a number of factors, more direct methods have been applied to the small quantities of phosphorus present in blood. By the method of Neumann (*Z. physiol. Chem.*, **1903, 37**, 115.) Iversen (*Biochem. Z.*, **1920, 104**, 22.) successfully determined 0·015 mgrm. of phosphorus, with an error of ± 0·003 mgrm. Stewart and Archibald (*Biochem. J.*, 1925, **19**, 484) determined 0·05 mgrm. with an error of ± 0·0015 mgrm. The method has now been simplified by the introduction of various modifications. To determine the "inorganic phosphate" in 0·5 c.c. of blood the precipitate of phosphomolybdate is dissolved in *N*/10 soda and the excess soda is titrated with *N*/10 hydrochloric acid by means of a micrometer syringe described by Trevan (*Biochem. J.*, 1925, **19**, 1111). The precipitate is washed with an ammonium nitrate solution, and the washing is carried out in small centrifuge tubes similar to those described by Trevan and Bainbridge (*Biochem. J.*, 1926, **20**, 423). By this method, which is given in detail, it is possible to determine 0·013 mgrm. of phosphorus with an error of ±0·0004 mgrm. P. H. P.

Effects of Various Agents on Colour Tests for Vitamin *A*. S. G. Willimott, T. Moore and F. Wokes. (*Biochem. J.*, 1926, **20**, 1292–1298.)—Cod-liver oil was exposed to the action of the following vitamin *A*-destroying agents:—Concentrated sulphuric acid, phosphorus pentoxide and ultra-violet light. Various colour tests for vitamin *A* were then applied, and the following results were obtained: The "pyrogallol" test of Fearon (*Biochem. J.*, 1925, **19**, 888; ANALYST, 1926, **51**, 311) is non-specific. This conclusion agrees with the results of Rosenheim and Webster (*Lancet*, 1926, ii, 806; ANALYST, 1927, **52**, 44) who worked on different lines. Concentrated sulphuric acid and phosphorus pentoxide are less sensitive tests for vitamin *A* than arsenic trichloride, as recommended by Rosenheim and Drummond (*Biochem. J.*, 1925, **19**, 753) or antimony trichloride, as suggested by Carr and Price (*Biochem. J.*, 1926, **20**, 497). With the use of the last two tests as criteria of vitamin *A* content, parallel results were obtained when following the course of destruction of the vitamin by ultra-violet light. Antimony trichloride, however, is more suitable for use in quantitative methods, since it gives colours which persist longer. In view of the transient nature of the colours obtained with both these reagents, it is suggested that readings should be taken not more than 30 seconds after mixing. P. H. P.

Vitamins and Other Constituents of Grape-Fruit Rind. S. G. Willimott and F. Workes. (*Biochem. J.*, 1926, **20**, 1299-1305.)—The grape-fruit (*Citrus decumana*) has been little studied either botanically or as regards its vitamin or other constituents; hence this work has been carried out. The results show that grape-fruit rind contains considerable amounts of vitamin *B*, but practically no vitamin *A*. The content of vitamin *C* has not yet been investigated. Maringin, which is the specific glucoside of the grape-fruit, was found

in the rind and in alcoholic extracts of it. The following table gives a summary of data obtained by the authors and other workers on the reaction and content of oxidising enzymes, as compared with the vitamin *A* content in the three *Citrus* fruits: oranges, lemons and grape-fruits.

Vitamins, oxidising enzymes and reactions in ripe Citrus fruits.

		Orange. *Citrus aurantium.*	Lemon. *Citrus medica* var. *Limonum.*	Grape-fruit. *Citrus decumana.*
A.	Rind (flavedo).			
	Vitamin content A	+ +	Slight.	Very slight.
	,, ,, B	+ +	+ +	+ +
	,, ,, C	+	Very slight.	Not yet ascertained.
	P_H	4·4 – 4·8	4·2 – 4·4	4·3 – 4·6
	Oxygenase reaction	–	–	–
	Peroxidase reaction	+	+	+ +
B.	Juice.			
	Vitamin content A	+ +	–	–
	,, ,, B	+	+	+
	,, ,, C	+ + +	+ + +	+ +
	P_H	3·6 – 4·2	2·4 – 2·6	5·1 – 5·2
	Oxygenase reaction	–	–	–
	Peroxidase reaction	±	+	+

P. H. P.

Nature of Fearon's Colour Reaction and its Non-Specificity for Vitamin *A*. O. Rosenheim and T. A. Webster. (*Biochem. J.*, 1926, **20**, 1342–1345.)—Fearon's reaction consists essentially in the production of a stable bright rose colour by the addition of trichloroacetic acid to a mixture of cod-liver oil with a phenol (pyrogallol). The pigment formation is assumed to be due to the condensation of an aldehydic chromogenic constituent of the oil (= vitamin *A*) with the phenol, and is intensified by the addition of an oxidising agent (benzoyl peroxide). Chemical and biological evidence is now presented which shows that Fearon's reaction has no relation to vitamin *A*. The test has been applied to the unsaponifiable fraction of cod-liver oil, known to contain all the growth-promoting material in the oil, with negative results. The fat of a pig's liver gave no colour reaction with Fearon's reagents, whilst its growth-promoting value in the animal test was twice that of a standard cod-liver oil; a sardine oil gave as intense a Fearon's reaction as the standard cod-liver oil, but did not produce growth nor have any effect on xerophthalmia. It is suggested that the chromogen of the reaction is associated with aldehydic oxidation products of unsaturated fatty acids, of the type of clupanodonic acid, $C_{22}H_{34}O_2$. Vegetable oils are negative in Fearon's test. Evidence shows that Fearon's reagents react in many oils with two entirely different chromogens. P. H. P.

Rapid and Reliable Test for Vitamin *D*. H. Jephcott and A. L. Bacharach. (*Biochem. J.*, 1926, **20**, 1351–1355).—Experiments have been carried out which show that albino rats on diets high in calcium and low in phosphorus develop marked faecal alkalinity in 10 to 15 days. This alkalinity is not affected by the administration of certain non-antirachitic substances. The faecal P_H value can be reduced to the acid side of neutrality by means of cod-liver oil, irradiated cholesterol or irradiation. The rise in faecal P_H value can be prevented by irradiation. Graphs show these results. The authors use these facts for the determination of antirachitic activity when a more rapid test than those usually employed is required. Rats are given the basal diet until two consecutive readings of the P_H of the faecal suspension give a mean of 7·3 or more. They are then fed with the substance to be tested, and, after three days, readings are taken until two consecutive readings give a mean value of P_H 6·7 or less. The authors have considered the quantity of antirachitic substance necessary for this purpose to be an antirachitic unit. P. H. P.

Bacteriological.

Studies in Bacterial Nutrition. III. Phosphates and the Growth of Streptococci. H. R. Whitehead. (*Biochem. J.*, 1926, **20**, 1147–1154.) Few chemical data have as yet been recorded on media used for the growing of bacteria. A medium has been examined by separation of the protein substances by the use of ethyl alcohol as a precipitant. A tryptic digest of caseinogen may be separated into three fractions by treatment with ethyl alcohol: (1) that precipitated by 66 per cent. alcohol, (2) that precipitated by 86 per cent. alcohol and (3) residual liquid. In this work the two precipitates were each redissolved in water, adjusted to P_H 7·5 and made equal in volume to the original medium ; the residual liquid was freed from alcohol and made up similarly. It was found that all the inorganic phosphate is present in the fraction precipitated by 66 per cent. alcohol. Streptococci have a lag period of about 24 hours before growth occurs in a medium made from the two fractions containing no inorganic phosphates. Rapid growth occurs if phosphate is added to these fractions. At least three factors appear to be concerned in the growth of streptococci in caseinogen medium ; two are probably protein derivatives and the third is phosphate, preferably in inorganic form. The nature of both anion and kation of added salts is of importance in the growth of streptococci. The darkening and deterioration of caseinogen medium which occur on steaming are probably due to the presence of phosphate. The deterioration is due to a growth factor, present among the simpler protein derivatives, being rendered inert. The change is not an oxidation nor is there a combination of the growth factor with phosphate, but phosphate appears to catalyse the reaction. P. H. P.

Toxicological and Forensic.

Effect of Poisons on the Larvae of Flies. K. Feist. (*Z. Unters. Lebensm.*, 1926, **52**, 466–469.)—In a forensic case it had to be decided whether the stomach

contents of a dog contained strychnine. As the material was fly-blown and living larvae were present, it was argued that this fact was not compatible with the presence of the poison. Toxicological examination, however, proved that strychnine was present, and it was also found that the larvae could live on flesh containing 0·3 per cent. of strychnine nitrate. Experiments with other poisons showed that the larvae were not affected by atropine, colchicine or morphine, but that they were killed by cocaine within 3 hours and by novocaine in nine hours. Methyl alcohol (5 per cent.) had no effect, whereas resorcinol (5 per cent.) killed them within 24 hours. Phenol (5 per cent.) had no action, but cresol killed them within 24 hours. Chrome alum (3 per cent.) had no effect, but potassium dichromate destroyed them within 3 days. Barium chloride (10 per cent.) killed most of them in 24 hours, but tin salt, $Sn(NH_4)_2Cl_4$, (3 per cent.) had no effect.

Agricultural.

Relation of the Magnesium in the Ash and the Lipoid-Protein Ratio to the Quality of Wheats. B. Sullivan and C. Near. (*J. Amer. Chem. Soc.*, 1927, **49**, 467–472.)—The results obtained in the analysis of 20 wheats from various parts of North America are tabulated. Of the mineral constituents, only the magnesium content showed a relation with the strength of the wheat, as determined by its protein content and the quality of its gluten. Stronger wheats contained most magnesium. The lipoid contents varied from 2·53 to 3·84 per cent.; and the ratio of the lipoid to gluten or protein is also a guide to the quality of the wheat. Softer wheats have higher ratios. In the improved method of ash determination described the ground sample is moistened with pure 5 per cent. hydrogen peroxide, dried at a low temperature, and then heated at 610 to 620° C.

J. G.

Composition of Flaked Maize. H. E. Woodman and J. Stewart. (*J. Agric. Sci.*, 1927, **17**, 60).—Samples of several brands of flaked maize have been analysed in order to compare their feeding values. The tabulated results, expressed as percentages, were as follows :—

	A	B	C	D	E	F
Crude protein	10·95	10·99	10·99	11·24	10·93	10·56
True protein	10·18	9·83	10·07	10·96	10·13	10·43
Ether extract	5·12	4·78	4·59	4·67	3·92	4·97
Nitrogen-free extractives	79·80	81·51	81·34	80·89	81·86	81·67
Crude fibre	2·27	1·67	1·69	1·78	2·06	1·59
Ash	1·86	1·05	1·39	1·42	1·23	1·21
Lime (Ca)	0·02	—	—	—	—	Trace.
Phosphates (P_2O_5) ..	0·95	0·45	0·72	0·81	·64	0·59

Sample A was raw maize used in the Cambridge pig-feeding trials; B, C, D, E, and F, well-known brands of flaked maize; C and D are the same, except that C was made from a mixture of 1 part white African maize and 4 parts yellow

Plate maize, whereas D was made wholly from the latter. From the table it appears that flaked maize is a fairly uniform product. Although the percentage of oil has decreased somewhat by the treatment, it is not nearly so low as it was in the flaked maize used for the pig-feeding trials in 1923. The fact that the lime content is so low may be usefully applied in investigations in lime-deficiency in animal metabolism. The moisture-content ranged from 6·61 to 14·43 per cent.; the variation was probably due to different conditions of storage and transport. It is recommended that the drying process should be so arranged that the flakes at the time of weighing into bags should always be of about the same moisture content, *viz.*, 10 to 11 per cent. R. F. I.

Organic Analysis.

Separation of Fatty Acids. A. H. Lewis. (*Biochem. J.*, 1926, **20**, 1356-1363).—Phenylhydrazine reacts with the fatty acids, saturated and unsaturated, and their triglycerides to give the corresponding phenylhydrazides, but, owing to similarity in solubilities, the products cannot be used for a separation of the fatty acids. Hydroxylamine, in the presence of sodium ethylate, gives stable hydroxamic acids with the triglycerides of the fatty acids. The sodium salts of the hydroxamic acids from palmitic and stearic acids are completely insoluble in alcohol under the conditions stated, but, with decrease of molecular weight, the solubility increases, the corresponding compound from lauric acid being appreciably soluble, whilst that from caprylic acid is readily soluble. The sodium salts of the hydroxamic acids from the unsaturated fatty acids and from the hydroxy-fatty acids are completely soluble under the given conditions. Therefore the saturated fatty acids of high molecular weight, certainly from palmitic acid upwards, can be separated from (*a*) the lower acids such as butyric and caprylic, (*b*) the hydroxy-acids, and (*c*) the unsaturated acids, in cases where all these may occur in natural oils in the form of glycerides. Attention must be paid to the concentration of the reagents, a concentration of about 4 to 5 per cent. in the mixed alcohols being the best, and the methyl and ethyl alcohol used must be pure and anhydrous. The separation methods are described. P. H. P.

Fatty Acids of Cod Liver Oil. Y. Toyama. (*Chem. News*, **1927**, **134**, 29.)—A sample of cod liver oil prepared from *Gadus macrocephalus* had the following characteristics: Sp. gr. at 15° C., 0·9261; n_D^{20}, 1·4781; acid value, 0·55; saponification value, 186·1; iodine value (Wijs), 155·9; unsaponifiable matter, 1·28 per cent. *Mixed fatty acids:* Sp. gr. at 30° C., 0·8945; n_D^{30}, 1·4652; saponification value, 194·0, iodine value, 163·2; and ether-insoluble bromides, 41·16 per cent. It is concluded that the fatty acids are composed of saturated acids (10 to 15 per cent.), palmitic preponderating, with myristic and stearic present, and no acids with less than 14 or more than 18 carbon atoms being present. Of the oleic acid series, zoomaric, oleic, an acid $C_{20}H_{38}O_2$ (possibly gadoleic acid of Bull), and cetoleic acid are present. Tetradecenic acid was

not detected. The more unsaturated acids consist chiefly of C_{20} (mostly $C_{20}H_{32}O_2$) and C_{22} (mostly $C_{22}H_{34}O_2$) acids, and possibly C_{18} acids. Acids with the formulae $C_{18}H_{28}O_2$ and $C_{18}H_{30}O_2$ were apparently present. D. G. H.

Determination of the Iodine Value in Aqueous Emulsion. J. Fialkow. (*Z. anal. Chem.*, 1927, **70**, 227–229.)—In the determination of the iodine value of a fat Margosches and Hinner (*Z. angew. Chem.*, 1924, **37**, 202) add water to the mixture of the alcoholic solution of the fat and the iodine in order to further both hydrolysis of the iodine and formation of hypoiodous acid, and also to obtain a large surface of contact between the iodine solution and the fat by distributing the latter throughout the liquid in very fine drops. The author finds that these objects may be attained and the use of alcohol entirely avoided if the fat is emulsified by means of gum arabic. The fat (0·1 to 0·15 grm.) is rubbed in a small porcelain dish with about one-half of its weight of the powdered gum and 1 to 2 drops of water until a uniform emulsion is obtained. This is treated with 5 to 10 c.c. of water, which is added dropwise at first, the mass being constantly stirred during the addition. The turbid emulsion thus obtained is transferred to a flask with a ground stopper, the dish being carefully rinsed. The liquid is mixed with 20 c.c. of 0·2 *N* iodine solution (1 part I ; 1 part KI) and then with sufficient water to make the total volume 200 to 250 c.c., and after 5 minutes the whole is titrated with 0·1 *N* thiosulphate solution. The titre of the iodine solution is determined by a blank experiment similarly carried out. This procedure gives results in agreement with those furnished by Hübl's method, but is not readily applicable to the solid fats, which are difficult to emulsify. T. H. P.

Theory of the Hardening of Oils by Hydrogenation. H. P. Kaufmann and E. Hansen-Schmidt. (*Ber.*, 1927, **60**, 50–57).—A selective hydrogenation, in which the more highly unsaturated glycerides are converted into less unsaturated glycerides, is more suitable for fats intended for food than a complete hardening in which the whole of the unsaturated constituents are hydrogenated. The nature of the changes effected during the hydrogenation of an oil can be studied by means of the thiocyanogen absorption process (ANALYST, 1925, **50**, 577, 634 ; 1926, **51**, 157) used in conjunction with the test for iso-oleic acid, as adapted to hydrogenated oils by Williams and Bolton (ANALYST, 1924, **49**, 460). For example, a sample of arachis oil, with an iodine value of 85·8, and thiocyanogen value of 69·4, was hydrogenated in an autoclave at 200°C., with nickel as catalyst. The samples taken at intervals of 15 minutes gave the following values, the hardening being complete after 135 minutes, when the fat melted at 30·5° C.

Minutes.	Beginning	15	30	45	60	75	90	105	135
Thiocyanogen value	69·3	70·8	70·2	69·1	69·3	72·1	71·0	71·9	72·1
Iodine value ..	85·8	86·1	81·2	79·0	78·5	75·1	74·6	72·0	72·6

It will be seen that the values for the thiocyanogen absorption remained constant (within the limits of experimental error) throughout the hydrogenation. The amounts of saturated glycerides and unsaponifiable matter had also not been

increased during the process, whereas the linolic acid had disappeared, for at the end of the hydrogenation the thiocyanogen and iodine values had become the same. The hardening process therefore had apparently been due to the formation of glycerides of less highly unsaturated fatty acids. A study of the fatty acids separated from the final product by Twitchell's lead salt and alcohol method confirmed this. The hardened fat was calculated to consist of 51 per cent. of glycerides of oleic acid, 32·8 per cent. of glycerides of solid isomers of oleic acid, and 16·5 per cent. of glycerides of saturated fatty acids with unsaponifiable matter. In the hydrogenation of the linolic acid only less unsaturated fatty acids had been formed.

In an analogous experiment with sunflower seed oil (thiocyanogen value, 72·8; iodine value, 117), the composition of a sample taken after 210 minutes, when the thiocyanogen and iodine values had become practically the same, was determined. The liquid fatty acids (separated by the lead salt and alcohol method) consisted solely of oleic acid in an amount practically the same as in the original oil (about 33 per cent.). The solid fatty acids had an iodine value of 49·6 and consisted of 28 per cent. of saturated acids and 34 per cent. of iso-oleic acids, calculated on the total fatty acids. The iso-oleic acids had been derived from the linolic acid, which had also yielded 15 per cent. of saturated acids.

Salts of Linolenic Hexabromide from Lumbang Oil. G. A. Imperial and A. P. West. (*Phil. J. Sc.*, 1926, **31**, 441–449.)—Linolenic acid hexabromide was prepared in quantity from Philippine lumbang oil, which consists almost entirely of the glycerides of linolenic, linolic, and oleic acids. The prepared acid was analysed by determining the bromine content, and salts were prepared from it by first converting the acid into the potassium salts, treating a methyl alcohol solution of the salts with an inorganic salt, such as barium bromide, and purifying the precipitated linolenic hexabromide salt. The barium, zinc and lead salts were thus prepared. The zinc salt gave the best melting point, decomposing sharply at 174° C. Linolenic hexabromide and its salts are not very soluble in ordinary organic solvents; about 10 grms. of the acid dissolves in 200 c.c. of benzyl alcohol at 100° C.; 5 grms. in 500 c.c. of warm isobutyl alcohol, and 21 grms. in 400 c.c. of warm xylene. The salts are still less soluble, but all are soluble (to the extent of 1 to 4 per cent.) in hot benzaldehyde and glacial acetic acid; the barium, zinc and lead salts in hot benzyl alcohol, the zinc salt also in nitrobenzene and pyridine, and the lead salt also in nitrobenzene, whilst the potassium salt is soluble in hot normal propyl alcohol. D. G. H.

Detection of Resins by Brauer's Method. E. Fonrobert and K. Pistor. (*Chem. Zeit.*, 1927, **51**, 139–140.)—Brauer's observations are, in general, confirmed, but the reaction with phosphomolybdic acid is given by a number of substances allied to the resins or used in conjunction with natural resins, and also by phenol and indene (ANALYST, 1926, **51**, 422). The reaction is not a resin reaction and is so faint with abietic acid that it must be attributed to the presence of slight impurity, possibly phytosterol or some other component of the unsaponifiable portion. T. H. P.

Pine-needle Extract. M. Klostermann and H. Quast. (*Z. Unters. Lebensm.*, 1926, **52**, 476–478.)—Commercial pine-needle extracts may be adulterated with sulphite liquors from the manufacture of paper, and tables are given showing the effect of such adulteration on the chemical and physical properties of the oil. The foaming properties of the oil are decreased, and its colour darkened, and the smell of the sulphite is detectable through the true odour of the oil. If the pure oil is ashed a characteristic strongly smelling aromatic decomposition product is obtained which is absent from the adulterated oil. Adulteration raises the sulphate and lime contents of the ash, but lowers the amounts of chlorides, phosphates and oxides of iron, aluminium and magnesium. The excess of total mineral matter over that in the pure oil, is not a measure of the degree of adulteration, which is best gauged from the lime and phosphate content of the ash. A sample of pure oil contained 45·20 per cent. of solids, of which 2·88 per cent. was mineral matter. J. G.

New Reactions of the Mixed Aquo-Ammonocarbonic Acids. L. A. Pinck and J. S. Blair. (*J. Amer. Chem. Soc.*, 1927, **49**, 509–514; *cf. id.*, 1926, **48**, 87.)—Two new mixed aquo-ammonocarbonic acids, unsymmetrical dicarbethoxy-guanidine and carbethoxy-cyanoguanidine, have been prepared by the action of ammonia (in the form of a suspension of ammonium nitrate in absolute alcohol) on dicarbethoxy-cyanamide, and by the action of ethyl chlorocarbonate in an ethereal solution on sodium dicyanodiamide, respectively. These and the reactions of alcohol with guanylurea, biuret and ethyl allophanate illustrate the use of ammonia in the synthesis of one of these compounds from another, and also the reverse type of reaction. The latter is difficult to control in aqueous solution, but in the presence of anhydrous alcohol the hydrolysis is slower, and esters of the more complex acids may be isolated. J. G.

Inorganic Analysis.

Determination of Traces of Nitric Oxide in Air. E. Kohn-Aorest. (*Compt. rend.*, 1927, **184**, 482.)—The air is drawn by suction into an evacuated four-litre flask fitted with a stopper which holds two tubes provided with taps. The flask is filled with the air until atmospheric pressure is almost attained, the pressure before and after filling being determined by means of a pocket manometer. Ten c.cm. of 0·1 *N* sodium hydroxide solution diluted with 50 c.c. of water are now allowed to enter the flask, which is then shaken 5 minutes. Thirty c.c. (one-half) of the solution are then titrated with 0·1 *N* potassium permanganate solution in the presence of 3 c.c. of sulphuric acid diluted to 40 c.c. The permanganate solution should be added drop by drop, the flask being shaken gently after each addition. At the beginning, the decolorisation is almost instantaneous, but towards the end the pink tint persists 1 or 2 minutes; it should persist for more than 3 minutes (1 c.cm. of 0·1 *N* permanganate = 0·0046 grm. of NO_2). The nitrates in the liquid should be determined in order to rule out all cause of error due to ozone or reducing gases, such as sulphur dioxide and hydrogen

sulphide. For this purpose the solution is neutralised with sodium hydroxide and evaporated to dryness, the residue taken up in a small volume of dilute sulphuric acid, and the nitrate determined by Lunge's mercury method. The method provides a ready means of determining the nitric oxide formed in the air of a clinic in which treatment by ultra-violet rays is being carried out. R. F. I.

Colorimetric Determination of Minute Amounts of Compounds of Silicon, of Phosphorus and of Arsenic. W. R. G. Atkins and E. G. Wilson. (*Biochem. J.*, 1926, **20**, 1223–1228.)—In the course of some work upon the minor constituents of sea-water and their utilisation by the phytoplankton it was necessary to use colorimetric methods and to tudy any possible interference of one constituent upon the method adopted for the analysis of another. The reagents used by Diénert and Wandenbulcke (*Compt. Rend. Acad. Sci.*, **1923, 176**, 1478 ; **1924, 178**, 564) for silicate determinations give no colour with moderate quantities of phosphate, arsenate or arsenite. The method of Denigès (*Compt. Rend. Acad. Sci.*, 1920, **171**, 802 ; *Compt. Rend. Soc. Biol.*, 1921, **84**, 875) for phosphate may be used : (*a*) for determination down to 0·0002–0·0004 mgrm. of phosphorus pentoxide in 100 c.c. ; (*b*) for determination of equivalent amounts of arsenate, as arsenic pentoxide, but with arsenite the reagent gives a faint colour only, due to arsenate. The amounts of phosphate and arsenate present in natural waters do not interfere with the method of Diénert and Wandenbulcke for silicate, nor does the silicate interfere with the accuracy of Denigès's method for phosphate. The method of Bell and Doisy (*J. Biol. Chem.*, 1920, **44**, 55) for phosphate is neither so convenient nor so delicate as that of Denigès, though it can also be used for arsenate ; arsenious acid solutions give irregular results. The reaction of Pouget and Chouchak (*Bull. Soc. Chim.*, **1909** (4), **5**, 104 ; 1911 (4), **9**, 649) for phosphates is not given by arsenites, and by arsenates only very faintly in the cold, though on heating, turbidity and a yellow colour develop. With phosphate a strong colour and a marked turbidity appear in the cold and become more intense on heating. The Denigès reaction is not given by glycerophosphate ; triphenyl phosphate does not give it, nor is this readily hydrolysed on boiling the aqueous solution, as is glycerophosphate ; tricresyl phosphate does not give the reaction even after prolonged boiling with water. The Denigès reaction is not given by vanadates, tungstates or phosphotungstates. With the last a purple tint is slowly produced, but this is not a sensitive reaction. P. H. P.

Measurement of the Ionimetric Acidity by the Inversion of Sucrose. Application to Complex Media. V. Vincent. (*Compt. rend.*, 1927, **184**, 338–340.)—The measurement of the ionisation by means of sucrose should be carried out with solutions containing, preferably, 10 per cent. of the sugar and having a titrimetric acidity not exceeding 0·3 grm. of HCl per litre. If the liquid is heated for one hour on a boiling water-bath, a table and curve may be constructed showing for any weight of glucose the weight of acid and the hydrogen ion concentration. The inversion of sucrose by sols does not, however, permit of the measurement of the actual ionisation in complex acid mixtures. T. H. P.

Determination of the Titre of Potassium Permanganate Solution by means of Electrolytic Iron. L. Moser and W. Schöninger. (*Z. anal. Chem.*, 1927, **70**, 235–247.)—Carbon-free electrolytic iron, suitable for determining the titre of permanganate solution, may be obtained by means of an electrolyte containing per 1000 c.c. of water, 100 grms. of $FeCl_2$, $4H_2O$, 180 grms. of sodium chloride, and 5 grms. of boric acid. The anode is of wrought iron (soft iron) wire and is separated by a diaphragm from the platinum or tantalum cathode. The electrolysis is continued for 40 to 50 minutes at 60° C., the voltage being 1·4 to 1·6 and the current density, referred to the cathode, $ND_{\frac{K}{100}}$ 0·5 to 0·7 ampère. From 0·15 to 0·2 grm. of iron is thus obtained. T. H. P.

Volumetric Method for Copper. D. Köszegi. (*Z. anal. Chem.*, 1927, **70**, 297–300.)—The neutral solution, containing less than 0·15 grm. Cu in 50 c.c., is treated with four times as much arsenite solution (made from 4 grms. As_2O_3, 3 grms. K_2CO_3, and 100 c.c. of boiling water) as is required for the reaction $4\,CuO + As_2O_3 = 2\,Cu_2O + As_2O_5$. A 10 per cent. solution of potassium hydroxide is then added, drop by drop, till the pale green precipitate dissolves completely. Separation of cuprous oxide sets in at the ordinary temperature after a few minutes. The solution is boiled and stirred for 5 minutes, the precipitate collected in a Gooch crucible and washed well with warm water. The precipitate is stirred up with a glass rod and dissolved in a warm solution containing 50 grms. of ferric sulphate and 200 grms. of sulphuric acid per litre. The crucible is washed with dilute sulphuric acid and water, and the ferrous iron in the filtrate titrated with 0·1 *N* permanganate ; 1 c.c. = 0·00635 grm. Cu. The results are accurate. W. R. S.

Determination of Bismuth. G. G. Reissaus. (*Z. anal. Chem.*, 1927, **70**, 300–308.)—A rapid volumetric method is based on precipitation of the bismuth as oxychloride, solution of the precipitate in sulphuric acid, precipitation of metallic bismuth by zinc, solution in ferric sulphate, and titration with permanganate. If metallic copper and hydrochloric acid are used instead of zinc and sulphuric acid, reduction proceeds more smoothly and lead is not precipitated. The reduction by copper, carried out under carbon dioxide, provides another volumetric method:—The oxychloride (0·3 to 0·4 grm. Bi) is dissolved in 30 c.c. of hydrochloric acid and 200 of water, and boiled with copper turnings in a current of carbon dioxide. When the reaction is complete, the liquid is filtered through cotton wool into a flask containing carbon dioxide, and the cuprous salt titrated hot with potassium bromate (methyl orange indicator) : $BiCl_3 + 3\,Cu = 3\,CuCl + Bi$. The precipitated bismuth may also be determined gravimetrically, after being dissolved off the copper by shaking with 4 to 5 small portions of hot nitric acid (1 : 2) and rapidly filtered through glass wool. The filtrate is diluted, and precipitated as usual with ammonium phosphate. W. R. S.

Volumetric Determination of Molybdenum. G. Denigès. (*Compt. rend.*, 1927, **184**, 330–331.)—Reduction by means of aluminium brings about

the reaction $MoO_4 \longrightarrow Mo_3O_5$, the resulting product being stable under considerable variation in the conditions. The molybdenum compound, containing 0·005 – 0·040 grm. of the metal in the form of the ion MoO_4, is introduced into a 400–500 c.c. flask, together with 40 c.c. of water, 4 c.c. of sulphuric acid and 0·1 grm. of aluminium leaf. The flask is fitted with a reflux condenser and the liquid is heated rapidly to boiling, which is just maintained for exactly 90 minutes. The aluminium should then be completely dissolved, provided that leaf weighing between 0·25 and 0·3 grm. per 100 sq. cm. was used. The flask is disconnected from the condenser, and the solution *at once* titrated and shaken with 0·1 *N* permanganate. When the liquid clears, titration with permanganate is continued in the usual way. The quantity of molybdenum present is given by 3·6 $(n - c)$ mgrms., where n is the number of c.c. of permanganate used, and c is a correction determined for the particular sample of aluminium by means of a blank experiment.

T. H. P.

Schlagdenhaufen's Reaction for Magnesium. A. Hamy (*Ann. Falsif.*, 1927, **20**, 19–20.)—Schlagdenhaufen's reaction for the detection of magnesium depends on the formation of a red-brown precipitate when a solution of sodium hypoiodite is added to a magnesium solution. If 50 c.c. (or less if necessary) of the sample solution are mixed with 6 to 7 c.c. of *N* iodine solution in 20 per cent. potassium iodide and 5 c.c. of *N* potassium hydroxide solution, and the whole vigorously shaken for 1 minute, the liberated magnesium will have the maximum absorbing power, and the precipitate may be filtered off on a small area and can be decolorised by alcohol; under these conditions as little as 1 mgrm. of magnesium hydroxide in a litre may be detected. Calcium salts do not interfere with the reaction.

D. G. H.

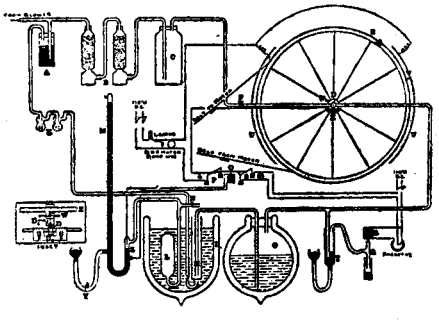
Determination of Perchlorate in Chili Saltpetre by means of Nitron. A. Vürtheim. (*Rec. Trav. Chim. Pays-Bas*, 1927, **46**, 97–101.)—The usual method of determining the perchlorate in Chili saltpetre, based on the decomposition of potassium perchlorate into potassium chloride and oxygen at red heat, is not trustworthy. Nitron (diphenylanile-dihydrotriazole) precipitates both perchlorates and nitrates as sparingly soluble compounds which differ in their crystalline form. The nitrate can be readily reduced by the Devarda method, leaving the perchlorate unchanged. Mixtures of nitrate and perchlorate may be quantitatively separated by reducing the nitrate, precipitating the perchlorate with nitron, and filtering off and weighing the precipitate. Iodates and borates do not interfere with the method.

Fifty grms. of the carefully ground sample of Chili saltpetre are dissolved in 500 c.c. of water, the solution filtered, and 10 c.c. (= 1 grm. of substance) mixed in a 100 c.c. flask with 20 c.c. of water and 2 c.c. of 96 per cent. alcohol. The mixture is kept in cold water and reduced with 2·5 grms. of Devarda alloy and 20 c.c. of 30 per cent. sodium hydroxide solution. This reaction is preferably carried out in two stages, namely, by first adding about 1.25 grms. of Devarda alloy and 10 c.c. of sodium hydroxide solution, and then, after the vigorous evolution

of gas has abated, the remainder of the alloy and of sodium hydroxide solution, the mixture being kept cold all the time. After the whole of the reducing agent has been added and the vigorous evolution of gas has abated, the flask is allowed to stand for one hour, at the end of which the reduction of the nitrate is complete. The mixture is then acidified with about 15 c.c. of strong acetic acid, made up to 100 c.c., shaken (but not too violently, so as to avoid too great admixture with air), and filtered. Twenty-five c.c. (= 0·25 grm. of substance) of the filtrate are then treated in a 100 c.c. beaker with 2 c.c. of 10 per cent. acetic acid and 10 c.c. of a 2 per cent. solution of nitron in dilute acetic acid. After standing for 18 to 24 hours (without shaking) the small, strongly refractive crystals of nitron perchlorate will have separated; these are transferred to a dried and weighed Gooch crucible with the aid of the filtrate, and are then washed with 10 c.c. of cold water which is added slowly and then rapidly drawn through the filter by suction. The crucible is then dried to constant weight at 105° C. (± 1 hour). The number of mgrms. of precipitate × 0·4 × 0·3392 = the percentage of $KClO_4$.

Physical Methods, Apparatus, etc.

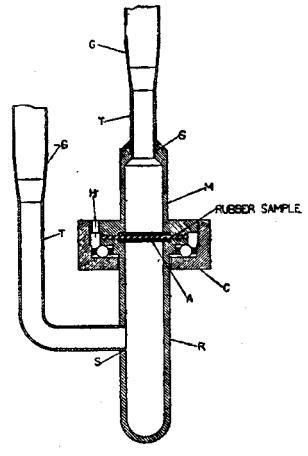
Automatic Low-Temperature Thermostat. O. Maas and W. H. Barnes. (*J. Amer. Chem. Soc.*, 1927, **49**, 360-363.)—The figure shows a thermostat which may be used from room temperature down to that of liquid air, and which will remain constant for any period to within 0·02° C. Air passes through the regulator (A), drying vessels (B), and volume bottle (C) to the tap (D) at the



centre of rotating wheel which, as it rotates, opens and closes (D). Thence it forces liquid air from the 2 litre Dewar flask (G) into the thin copper bulb (H), which serves by conduction to cool the first distillate of petroleum spirit contained in the bath (I). The regulator is a bulb (J), connected by capillary tubing with the closed mercury manometer (M), the level of which just makes contact with a platinum wire at the required temperature. This closes two circuits (at (P) and (S)) by means of the coil (O). The former stops the motor with (D) closed, and the latter allows a current to flow through the solenoid (R), which raises an iron plunger attached to the glass tube (Q), and allows the excess of liquid air evaporating in (G) to escape. The excess of air from (A) is dried in the bulbs

(K), bubbles through the tube (J), and acts as a stirrer. The mercury column (T) enables a column of liquid air always to be kept in the outlet from (G). The contact arrangement at (D) is shown inset. J. G.

Apparatus for Measuring the Diffusion of Gases and Vapours through Membranes. E. E. Schumacher and L. Ferguson. (*J. Amer. Chem. Soc.*, 1927, **49**, 427–428.)—The apparatus consists of two compartments, R and M, held together by means of a screw clamp on ball-bearings (C). These parts are machined from unstainable steel rod and are sealed at (GG) to glass tubes which lead to the evacuating system and diffusion-measuring device. Two rigid porous discs (A) support the diffusion membrane which is cut so as just to overlap the edge of the bearing flange of (R). The space between (C) and the body of (R) is filled with non-hardening putty to retain the mercury, which is poured in at (H), and fills the space around the ball-bearings and membrane. The seal is thenleak-proof. The apparatus has been used to determine the rate of diffusion of distilled water through rubber at a pressure of 0·01 mm., and it may also be used for gases and vapours. J. G.



Reviews.

POTENTIOMETRIC TITRATIONS. By I. M. Kolthoff and N. H. Furman. Pp. 345. New York: John Wiley & Sons, Inc. 1926; London: Chapman and Hall. Price 22s. 6d.

The ordinary methods of volumetric analysis depend on the choice of an indicator which will change colour when the end-point of the reaction between the solutions under examination is reached. Frequently an external indicator has to be used, and hence the results obtained are subject to appreciable errors. It is also well known that the observed end-point of a reaction depends on the indicator used, and an initially coloured solution often presents insuperable difficulties. Further, even with an indicator which is theoretically suitable the personal equation of the analyst must be taken into account. All these difficulties may be overcome by the use of an appropriate potentiometric method for determining the end-point of the reaction; the method is no panacea, however, since it brings a certain number of difficulties in its train, but its chief advantage is that it can often be used in cases where ordinary indicators are inapplicable.

The method of potentiometric titration has justly assumed very great importance within recent years, but until the appearance of the book under review the only account of any length, in the English langugage, of the subject was

that contained in the chapter by Furman in *Taylor's Treatise on Physical Chemistry* (1924, Vol. II, chap. xiii). This author and Kolthoff, the well-known Dutch investigator in the field of electrometric analysis, have now collaborated in the production of a very valuable English text-book on the subject of Potentiometric Titrations.

The book is divided into two parts; Part I, entitled "Fundamental Principles," deals with the theoretical basis of the potential changes which may occur in neutralisation, precipitation and oxidation or reduction reactions, and with the subject of titration errors, whilst Part II—"Practical Applications"—treats of the technique of the electrometric measurements involved in potentiometric titration and describes a large number of applications of this method of analysis. The theoretical portion is very well done, and in fact, somewhat overdone in places; this is a good fault, however, as the book is by no means "heavy" reading. A good selection of numerical examples helps to clothe with flesh the skeletons of mathematical formulae, which might otherwise be fearsome, and adds greatly to the value of the book.

In the second half of the book various types of apparatus, some of them very simple, are described and discussed, and ample references are given for those who desire further information; there is no mention, however, of the electrode apparatus designed by Garner and Waters (*J. Soc. Chem. Ind.*, 1922, **41**, 337). The work is concluded with a very comprehensive summary of a large number of actual analytical determinations covering a wide field. A number of small misprints have been noted, but these are readily detectable; the footnote to page 124 might well be left out, as a similar footnote appears on page 68.

The value of the book is enhanced by an excellent bibliography, in addition to author and subject indexes, and altogether this work forms a valuable contribution to the literature of both analytical and electro-chemistry.

S. GLASSTONE.

INDICATORS. By I. M. KOLTHOFF. English translation of the second German edition, revised and enlarged, by N. H. FURMAN. New York: John Wiley and Sons, Inc. 1926. London: Chapman & Hall, Ltd. Pp. 269. Price 17s. 6d.

The French translation of this work has already been reviewed and praised (ANALYST, 1926, **51**, 218), and the English translation now issued is an advance on the French version. Not only is the general "get-up" of the book very much better, but an omission, which has already been deplored, has now been remedied by the translator's addition of subject and author indexes. Kolthoff has supplied material for further improvements and for a new chapter on amphoteric electrolytes; the bibliographies have also been brought up to date—the end of 1925, apparently. There appears to be no mention, however, of Washburn's early work on the theory of buffer mixtures, although the subject is dealt with very fully.

The translation is, on the whole, very good, although in some cases a little more licence might have been taken and the literary style improved; two peculiarities, among others, noted were "much used" (p. 12) instead of "common," and "table salt" (p. 33) for "common salt." A few misprints were observed, chiefly in the spelling of the titles of German works quoted in the bibliographies; it is interesting to note that the same misprints occur in the French translation, and so may have originated, curiously enough, in the German edition. That is, however, no reason for their perpetuation. Another error which occurs in both English and French translations is the omission of the negative sign in the value of $\frac{1}{2}$ log c on the fifth line of page 113 (English edition). Kolthoff, who is usually so meticulous in his choice of terms, has surely made an error in suggesting "ampholyte" as an alternative to "buffer." It might be well to point out, too, that the translator frequently uses the word "iogen" instead of "ionogen."

Apart from these minor points, the book is excellent and can be heartily recommended to English readers. S. GLASSTONE.

CHEMISTRY OF DYEING. By JOHN KERFOOT WOOD. Pp. iv + 104. 2nd edition. 1926. London: Gurney and Jackson. Price 3s. 6d. net.

The problem of dyeing is one of the most complex in the large field of Dyestuff Chemistry, and Mr. J. K. Wood has been well advised to give a very concentrated survey which enables the reader to form an opinion as to the value of conflicting views. The little book contains practically every possible reference (over 90 items) and it is shown in the different parts of the work how ideas and theories are still shifting according to the momentary standpoint of physics, chemistry and their allied branches—colloid chemistry and physical chemistry. All the many theories concerning the chemical composition and properties of the textile fibres, the properties of dyes and the nature of the dyeing process are discussed. It seems remarkable that the author should not have missed any of the more important theories, if one recalls the fact that his communications cover only 100 pages.

After describing the different theories of dyeing, the author comes to the "General Conclusion" (page 92 *et seq.*) that the electrical theory is the best, but I should like to point out that this view can only be satisfactory to the physical chemist who is satisfied with the statement that electrification is the cause of the absorption of the dyestuff by the fibre. This theory is practically identical with that which Marcel Bader has brought forward in his little book, *Le Problême la Teinture* (1920), and which is to the effect that it is essential that there should be an "Electrophore." It seems evident that the electromagnetic field must play a rôle in some way, but chemists are not satisfied with a word which is meant to include everything and yet does not tell us enough. Probably Bader's work has escaped attention because it is written in French and has never been reviewed in an English journal, so far as I am aware.

Mr. Wood's work must be very welcome to all who are interested in the important problem of dyeing, and that there is still much work to be done is proved

by the excellent experimental publication of K. H. Meyer in the *Naturwissenschaften* (1927, Heft 6, page 129),* where he shows that very many phenomena can be explained by salt formation on the one hand, and by solubility on the other. It is clear that these two processes have some relation to the "electron," but everything is in such a relation, and the question arises: In which way does the electric field make itself manifest? H. E. FIERZ-DAVID.

DYESTUFFS AND COAL-TAR PRODUCTS. By BEACALL, CHALLENGER, MARTIN and SAND. Manuals of Chemical Technology, 4th edition. Pp. xii + 168. Edited by GEOFFREY MARTIN, D.Sc., F.I.C. London: Crosby Lockwood & Son. 1926. Price 16s. net.

This little work is not a textbook in the ordinary sense, but is comparable with the sections of articles in, for instance, the *Encyclopaedia Britannica* or a *German Konversationslexikon*. Therefore, only the most important products are dealt with, and, all things considered, the object has been very well attained. Not only are the intermediates of coal-tar dyes included, but practically all the more important modern dyes (indigo, indanthrene, flavanthrene, etc.) are described and the formulae given. In addition to the dyestuffs, the natural colouring matters, modern inks, saccharin, drugs, and photographic chemicals are dealt with.

It is evident that such a survey can only be of use to a man who wants to obtain information on a substance without going into any details, and traders and engineers will profit much more than a good chemist from such a concentrated extract. There is one thing which ought to be corrected in the next edition, namely the paper. Some of the illustrations have suffered through the softness of the paper, and the "Fourteen-Colour Printing Machine" on page 113 is, in my copy at any rate, only a black spot, which does not convey any idea as to what such a machine can look like. I know, of course, what a Mather and Platt printing machine does look like, but that is not the question. H. E. FIERZ-DAVID.

THE CALENDER EFFECT AND THE SHRINKING EFFECT OF UNVULCANISED RUBBER. By Dr. W. De Visser. Pp. viii + 152. London: Crosby Lockwood & Sons. 1926. Price 15s. net.

When uncured masticated rubber is subjected to calendering under certain conditions, an anisotropic system ensues, which manifests itself (for instance) by the fact that test pieces cut longitudinally to the direction of calendering possess stress/strain characteristics markedly different from those obtained from pieces cut at right angles to the said direction. De Visser has found that there is a further characteristic, namely, stress/strain curves derived from longitudinally cut specimens show no clearly defined vertical direction at any part of the curve, and he proposes to define calender effect on these lines. The author has also observed that calendered sheet from which the "effect" has been removed by warming may still shrink if accorded free movement, and hence differentiates between calender effect proper and "shrinking."

* *Zur Physik und Chemie der Färbevorgänge*, **129**, 134.

The volume under review is a record of the results of an investigation carried out with much skill and ingenuity, and, inasmuch as calender and "grain" problems are of considerable practical importance, Dr. De Visser deserves the thanks not only of his scientific colleagues, but also of those engaged in practical work in the rubber industry.

It may be mentioned that rubber showing the calender effect is doubly refractive, displays dichroism and has a higher specific gravity than isotropic rubber. The various theories as to the cause of calender effect are reviewed and criticised by the author, who comes to the conclusion that it is due to an alteration of internal structure arising from a regular and definite particle orientation.

It is highly probable—in view of modern X-ray work on rubber—that the modification of internal structure takes the form of a partial crystallisation or of a phenomenon on all fours with crystallisation.

The translation of the book leaves something to be desired and suggests that a work on such a highly technical subject should not be translated except by an expert familiar with its scientific and technological aspects.

PHILIP SCHIDROWITZ.

SOIL CONDITION AND PLANT GROWTH. By Sir E. J. RUSSELL, F.R.S. Pp. vi. + 516. London: Longmans, Green & Co. 1927. Price 18s. net.

Originally a comparatively small work on biochemistry, each new edition has increased in size until the present one, the fifth, has extended to rather more than 500 pages. The term "monograph" can be applied to the present volume in its fullest sense, inasmuch as it contains an elaborate account of the scientific investigation of the soil. The author frankly admits that it is not possible for one person to read the mass of papers pertaining to agriculture which now appear in journals and in reports, and he therefore had recourse to the help of a number of colleagues, the outcome being a work of unquestionable merit.

The first chapter is certainly not the least interesting, as it is devoted to a historical review of the science of agriculture, commencing with an allusion to a work of Roman origin and ending with a summary of the modern methods of research as applied at Rothamsted. Attention is drawn to the fact that methods of soil investigation now in vogue, particularly on a field scale, are designed to obviate unwarranted conclusions, for in the past it has frequently been found that the results obtained from a certain line of experiments were good, but deductions were made without the vital influencing factor being considered.

The succeeding chapter deals with the soil conditions which affect plant growth, and at once the reader becomes aware of the difficulty of distinguishing between the factors which in any given field experiment were probably mostly concerned with the result obtained. Of the elements needed to build up plant tissue, carbon is the first to be discussed. It is somewhat remarkable that French investigators are still said to hold the view that plants absorb more than negligible traces of carbon from the soil; however, no proof appears to exist that plant life actually does obtain all its carbon from the atmosphere. The effect of nitrogen,

phosphorus and potassium on the yield of many plants is discussed, and the influence of several other elements is more or less briefly mentioned. A reference follows to the characteristics exhibited by plants suffering from the lack of certain elements. These characteristics, which are often diagnostic, are finally tabulated, and this table should be of service to soil analysts, because when an abnormality of a plant is known, a possible soil deficiency is indicated.

A short article on the effect of radium on plant growth is of interest, because a few fertiliser merchants have advertised particular fertilisers as being specially effective owing to their radio-activity. The investigations of Sutton, and also of Wallis, conducted in pot and field experiments, failed to give an increased yield, though a variety of crops were grown to which radium residues and also minute dressings of radium bromide had been applied.

The composition of the soil occupies an important portion of the book. The varied aspects of this subject are ably dealt with, and as soon as the opening paragraphs on "Soil formation" have been passed, the extremely complicated nature of this branch of study becomes apparent. The physical fractionation of the soil is followed by the properties of these fractions and their influence on the soil. Probably few people could write with the experience of the author on this particular subject. It is now about 20 years since he was first engaged on a soil survey of Kent and of Surrey, and subsequently of Sussex, and his knowledge of the agricultural practices and the results obtained in these counties enables him to draw valuable deductions. The information regarding the influence of soil fractions on the chemical and physical properties of a soil is therefore written largely as the result of actual observation.

The carbon and nitrogen cycles in the soil are represented diagramatically, and for this there is much to be said, because the possible sequence of the formation of various substances found in the soil, as decomposition products, can readily be followed.

As a companion volume deals with the micro-organisms occurring in the soil, this subject has not received any extended attention, but, nevertheless, 54 pages are devoted to this study and the relationship of soil organisms to plant growth.

Present day knowledge is ably summarised in the chapter on "Soil relationship to plant growth" and on "The fertility and exhaustion of soils." The book concludes with an account of the processes used in the chemical and mechanical analysis of soils and a short interpretation of the results so obtained.

The one complaint which might be offered is that a considerable number of results are represented graphically, and the description of the representations is often inadequate, with the result that some time may have to be given if the significance of some of the graphs is to be understood.

From the knowledge of several works by Sir E. J. Russell, it would be anticipated that the book would be carefully written, contain information of recent date, and that results would be ably summarised, and this is certainly the case.

F. W. F. ARNAUD.