APRIL, 1928. Vol. LIII., No. 625.

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 7th, when the President, Mr. E. Richards Bolton, delivered the annual address.

The following were elected as Officers and Council for the year 1928:—

President.—Edward Hinks.

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

Vice-Presidents.—John Evans, Thos. Macara, John White.

Hon. Treasurer.—E. B. Hughes.

Hon. Secretary.—F. W. F. Arnaud.

Members of Council.—A. P. Davson, J. Golding, J. T. Hewitt, E. V. Jones, R. Lessing, Andrew More, W. Partridge, E. K. Rideal, W. H. Roberts, C. A. Seyler.

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

Certificates were read for the first time in favour of:—Frank Rowland Hill, B.Sc., A.I.C., Edward Thomas Illing, B.Sc., F.I.C., Farid Iskander, Harry Bulmer Marston, B.Sc., A.I.C., Reginald James Munro, B.Sc., A.I.C., John Ralph Nicholls, B.Sc., F.I.C., H. Gordon Reeves, D.Sc., Ph.D., F.I.C., George Walsh, B.Sc., A.I.C., Ronald George Warren, B.Sc., William Arthur Waygood, B.Sc., A.R.C.S., A.I.C.

Certificates were read for the second time in favour of:—John Edmund Aps, Edward Eric Billington, M.Sc., Ralph C. Chirnside, Ralph David Owen, A.I.C., A.M.I.Chem.E.

The following were elected Members of the Society:—Andrew R. Buchanan and Arthur Gordon Francis, B.Sc., F.I.C.

The following papers were read:—"Composition of the Fatty Acids present as Glycerides in Elasmobranch Oils," by T. P. Hilditch, D.Sc., F.I.C., and A. Houlbrooke; "Behaviour of Indicators in the Titration of Ammonia, Sodium and Calcium Phosphates, the Methylamines, Pyridine Bases and Boric Acid," by R. T. Thomson, F.I.C.; and "Cacao Tannin and its Determination," by H. R. Jensen, M.Sc., F.I.C.

NORTH OF ENGLAND SECTION.

THE third General Annual Meeting of the North of England Section of the Society of Public Analysts was held in Manchester, on Tuesday, February 28th, 1928.

In the absence of Dr. Dunn, Professor W. H. Roberts took the chair, and about twenty members were present.

The accounts for the year ending December 31st, 1927, were passed, and Messrs. U. Aylmer Coates and William Marshall were unanimously elected Hon. Auditors for the following year. The following officers and committee were elected:—

Chairman.—Dr. J. T. Dunn.

Committee.—W. D. Mackey, J. R. Stubbs, A. R. Tankard, W. H. Roberts, S. E. Melling, J. P. Shenton, R. F. Easton.

Hon. Secretary and Treasurer.—H. T. Lea.

After the election of officers, Mr. John Hanley opened a discussion on the "Rôle of the Chemist in an Improved Milk Case," and an interesting debate followed.

Address of the Retiring President.

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

Delivered at the Annual General Meeting on March 7th, 1928.

LADIES AND GENTLEMEN,

The report I have to make to you this evening, upon the activities of the Society during the past year, is a happy story of unprecedented progress unpunctuated by any very startling event. Were I to turn to the blackboard behind me to illustrate diagrammatically the data which it is customary to give you, the curves I should draw would consist of lines running without exception in a desirable direction. Our little Society, as we have affectionately called it in the past, seems to be reaching the stage when the diminutive adjective ceases to be a correct description. Of course, the psychological moment when a Society ceases to be *small* and becomes *large* is about as clearly defined as when lamb becomes mutton.

We now number 580, as compared with 541 twelve months ago—a very marked increase—and a survey of the names and qualifications of those who have joined us shows at a glance that they are of the type we welcome as likely to uphold the traditions we have so jealously guarded; traditions that have placed our Society in the position of importance it unquestionably occupies.

While only five members have resigned in the past twelve months, 55 new members have been elected, and you will remember that at the February meeting alone we elected 16 new members, a record number, I believe.

Year by year we all grow older, and so many of us each year finish our allotted task upon this earth, some ripe in years, others still in the prime of life.

In the past year we have lost 8 of our members by death; they are:—Arthur William Crossley, John Robinson Leebody, Reginald Charles Pakes, Benjamin Arthur Burrell, Herbert Edward Burgess, Thomas Featherstone Harvey, M. J. Gajjar, Charles James Waterfall.

Some of these have already been the subject of obituary notices in the journal, and I would like to say something about each of them, but perhaps the time is not appropriate. I cannot pass, however, without referring specially to two of the oldest members:—Professor Leebody, who was 87 years of age, and having joined the Society in 1873, was our oldest member; he was not long survived by Mr. Burrell, a member of 47 years' standing. In addition to these members, the Society has sustained a sad loss in the death last month of our old friend, Frederick W. Beck, who acted as our legal adviser. Mr. Beck was a well-known authority upon the Food and Drug Laws, and, I suppose, there is no man living who has appeared more often in legal cases against the public analysts, yet it is significant that no man was on more friendly terms with them. It was characteristic of this man that he never took an unfair advantage; he was never unreasonable, and when one lost a case against him, the first feeling one had was to go and shake hands with him, and say "you have beaten me fairly." His charming personality and sound commonsense won for him the affection and admiration of everyone who had the good fortune to meet him whether as opponent or supporter. One cannot help feeling that he is a man hard to replace, and it was a matter of deep regret to me that I was unable to join Mr. Hinks in representing the Society at the funeral of so old a friend, one for whom I had the very greatest respect.

The North of England Section of the Society can no longer be regarded as a new-born babe, having, on the 28th of last month, held its Third Annual General Meeting. Dr. J. T. Dunn is now Chairman of the Section, and Mr. H. T. Lea its Honorary Secretary and Treasurer.

Our Society has always closely co-operated with the Institute of Chemistry, and we associated ourselves with the Institute recently upon the occasion of the Jubilee Celebrations when I had the honour, as your representative, to present a congratulatory address from the Society.

Our interest in scientific organisations has always extended beyond those in our own land, and upon the occasion of the centenary of the birth of Berthelot our Past-President, Mr. A. Chaston Chapman, representing the Society, presented an address.

The Society has also continued to keep a watchful eye upon all matters directly, or indirectly, affecting the profession, and we have appointed representatives to attend various committees and conferences, the reports on which, appearing in appropriate places, need not be referred to now.

I would, however, like to mention specially a joint meeting with the Institute of Chemistry, held in April to consider the conditions of the appointments of public analysts, as they were affected by recent legislation and regulations. This was followed by a deputation from the two bodies, which was received by Sir H. Kingsley Wood, Parliamentary Secretary of the Ministry of Health. This

deputation afforded an opportunity of airing our views regarding the treatment of public analysts, but resulted as usual in a repetition of the view that the Ministry was totally unable to intervene between the analyst and the local authorities on the question of remuneration. Since then the Public Appointments Committee of the Institute of Chemistry, on which the Society is well represented, has prepared a statement for submission to the Royal Commission on Local Government, and it is probable that this statement may be supported by verbal evidence in the near future.

The main points of our representations are included in the Report of the Council of the Institute of Chemistry, which most of you have recently received.

We have co-operated with the Institute of Chemistry in another matter, namely, the remuneration of Official Agricultural Analysts under the Fertilisers and Feeding Stuffs Act, 1926, and those of you who have official appointments will have received a circular stating the suggested schedule of fees, issued jointly in the names of the Society and the Institute to all local authorities concerned.

I would also like to mention a committee consisting of members of this Society, together with representatives of the British Association of Chemical Manufacturers, which has considered the question of the reasonable limits that should be imposed to restrict metallic contamination in colouring matters. The report of this committee, dealing with the maximum permissible amount of arsenic in colouring matter used in foodstuffs, has been presented to your Council to-day, and will shortly be published; it is worthy of your attention. Further considerations will be given subsequently to other metallic contamination, while another committee has under consideration methods for determining the amount of such contamination.

The Standing Committee on the Uniformity of Analytical Methods, to whose activity I have referred elsewhere, has now published three reports, namely:—

(*a*) The Estimation of Cineole in Essential Oils: (i) Cajuput and Eucalyptus Oils (ANALYST, 1927, **52**, 276),

(*b*) Milk Products, Report No. 1 (ANALYST, 1927, **52**, 402),

(*c*) Physical Constants (ANALYST, 1927, **52**, 530),

to be followed shortly by:—"The Determination of Phenols in Essential Oils," and "Interim Report on the Determination of Alcohols." (See p. 214.)

The investigations are still being actively pursued, and will result in further communications later on.

With regard to the methods put forward for the determination of total solids and fat in condensed milk, it has been suggested that these methods are not the shortest and most convenient that might be devised for the purpose; such a criticism arises from a want of understanding of the main object in view. These methods are put forward as the best within the knowledge of the Committee for the purpose of obtaining results of the highest accuracy, and are consequently intended to be used in cases of dispute; they are obviously too cumbersome for the rapid routine control of the products with which they deal.

The methods for the analysis of condensed milk became necessary owing to the Condensed Milk Regulations, and while the Government Chemist was represented on our Committee, it is a matter of regret that the Department from which these Regulations emanated did not see fit to appoint a representative to serve on the Committee.

Arising out of the First Report of the Milk Products Sub-Committee you will be interested to learn that La Fédération Internationale de Laiterie—which has been in existence about 25 years, is composed of delegates from most of the European countries, and is concerned with international trade in dairy products—is considering, at present, proposals for the unification of standard methods of analysis of condensed milk for international purposes. At the request of the Ministry of Agriculture and Fisheries the Report referred to above was recently submitted, by Mr. A. More, to the Fédération as the contribution from this country to the question of methods of analysis of condensed milk.

In my last address I mentioned with pride that our journal, THE ANALYST, had beaten the record in producing 654 pages. In the past year, however, it has still further increased to 736 pages, and it is all the more satisfactory to know that this increase is mainly due to the publication of papers read before the Society, the total number of papers published being 47, many of which may be regarded as valuable contributions to chemical knowledge.

The journal, the mainspring of the Society, cannot be mentioned without a thought of its editor, C. A. Mitchell, whose energy is, like the pages of the journal, always on the increase, and what he has achieved represents time and thought, notwithstanding that he has, in addition, secretarial duties to perform and, incidentally, his own business to transact. This is a true example of the busy man who always finds time to do something more. With him we must associate Miss M. B. Elliott, who flits round the Society's affairs, giving a capable and helping hand wherever it is needed.

The question of abstracts in the ANALYST has been considered by your Council, who have decided that the requirements of our readers are better served by the preparation of abstracts in sufficient detail to enable new methods to be tried without reference to the original papers. It is for this reason that it becomes impossible for us to pool the abstracts with other scientific journals, who make these abstracts for an entirely different purpose and to serve a different end; and, for the same reason, that no attempt is made to abstract everything published in chemical journals, such papers being adequately dealt with elsewhere. If, therefore, a method is considered by the Editor or the Publication Committee to contain no novel feature, or to be of no utility to the analyst, it is omitted.

We may congratulate ourselves upon the fact that the general standard of the papers brought before the Society is well maintained, and we are glad to think that our journal is clearly recognised by the more intelligent authors as the right medium for the publication of chemical investigations of a purely analytical nature.

Unfortunately there are still some misguided analysts so short-sighted as to imagine that journals having a larger circulation than the ANALYST provide better publicity; but these poor ostriches are really hiding their heads in a sand of chemical chaos not appertaining to analysis, while our own journal provides an oasis of analytical fertility in this desert of heterogeneous chemistry.

The papers read at our meetings cover a very wide scope of analytical interests, and on several occasions in the past we have invited specialists to give an outline of the present position of knowledge on subjects allied to our work. Thus, recently we heard Sir William Willcox, Dr. Roche Lynch, and Dr. Martley give valuable summaries of the present-day methods of serology. On this occasion our members were the guests of Sir Almroth Wright and the Pathological Institute of St. Mary's Hospital, and we showed our appreciation of their hospitality by a crowded attendance.

It has been the general policy, however, to mix the subjects for each meeting so as to provide a potpourri from which every member attending a meeting may find at least one flower to his taste.

To this rule we have made occasional exceptions, as, for example, in the case of the meeting last January, which was devoted entirely to the reports of the Committee of Chemists of the Food Manufacturers' Federation upon the determination of sulphites and benzoates in foodstuffs.

Last year I claimed for our Society a large share in bringing about a better understanding between official and other analysts, and I cannot help once again referring to this subject; for what better illustration of co-operation could be found than that of a group of chemists associated with a trade research association turning its whole attention to the problem of detecting preservatives in food ere the ink is dry upon the Preservatives Regulations. Let me say that no one appreciates this friendly spirit more than the public analysts, to whom this work is offered by the trade to enable them to enforce the regulations.

Standard Methods of Analysis. This brings me to a very important question, namely, the evolution of standard methods of analysis. I know quite well that there are many analysts who take up the attitude that they do not wish to be dictated to upon the conduct of their analytical operations, and who hold the view that when they make an analysis they apply the best method known to them at that time, and reserve the right to alter their method from time to time as their knowledge advances. Hence the man with the longest experience, or the greatest ingenuity, is able to give his clients better service than the man not having these advantages. If, on the other hand, the analyst is bound to operate according to a standard method, his powers are limited in scope, and he is like a skilled bricklayer whose trade union prevents him from laying one more brick than a fellow workman of much more limited capacity. All this is perfectly sound argument, and, up to a point, has my sympathy, but we must face the fact that legal requirements, trade contracts, and various other agreements stipulate that certain products shall be so constituted as

to give certain analytical data under specified conditions, and were each analyst to test the samples by what he considered the most accurate method, the disputes which would arise are not difficult to imagine. It will, therefore, be clear to everybody that conditions have arisen requiring the institution of standard methods for certain purposes, and these standard methods must be provided. The British Engineering Standards Association have published a few standard methods for certain types of chemical analyses; these methods have been well worked out and deserve our appreciative thanks, but it is nothing short of a disgrace to the members of the chemical profession—and to the profession of analysts in particular—that they should have so neglected their duties to the public as to make it necessary for their friends, the engineers, to remedy the effect of their culpable negligence in one or two directions. This state of affairs cannot be allowed to exist any longer; we must set our house in order and tackle this important question in some much more comprehensive and systematic manner than we have done hitherto.

The Standing Committee on the Uniformity of Analytical Methods, to which I have already referred, has now contributed in no small measure to this need in the five reports which I mentioned earlier this evening. What this Committee has done sounds a very small proportion of a very large requirement, but, like the widow's mite, it represents a very great deal from those who gave the work, the time, and the money. Take, for instance, the comparatively short report upon the methods for the analysis of condensed milk; to prepare that report, members of the Committee held 26 meetings and conducted some 4000 analyses, and everyone knows that this work cost money, but many of you will be surprised to hear that one firm alone, whose chemist was a member of the Committee, spent £500 in expenses in connection with carrying out investigations. Other members, in utilising their staffs and organisations, have also spent considerable sums. This being so, it cannot really be said that our Society has not bravely attempted to fulfil the need for standard methods. Obviously this state of affairs cannot go on; neither analysts nor the firms who employ them can labour without some official recognition, and I think it is the duty of our Society to go into the whole question of organisation in detail, and, having prepared a case, to approach the appropriate Government Department for assistance in carrying out this important work.

If the Ministry of Agriculture can produce in a short time the methods of analysis required by the new Fertilisers and Feeding Stuffs Act, surely the Ministry of Health could follow this example and organise some system of standard methods to cope with the regulations they promulgate from time to time; and, moreover, a case might be put before the Department of Scientific and Industrial Research for a grant for this very important purpose.

In the past we analysts have given our time and experience free of charge for public service, but the time has come when those who serve their country should be paid in the same way as lawyers, doctors, soldiers, and others; and it is unreasonable to expect us to look upon scientific interest as the sole reward.

We have good examples to follow in other countries. I might, in particular, refer to the United States of America where there are such organisations as the National Canners Association, which pursues the scientific problems of that particular trade in every necessary direction. There is another American organisation in the form of the Association of Official Agricultural Chemists, whose published methods of official and tentative analysis have to be widely used in this country for want of similar methods of our own. The more one thinks of this subject the more crying does the need appear, and I do hope that it will receive the urgent attention of the active officers of the Society, to whom I leave, as a legacy, my retiring wish that this question of standard methods be actively pursued and placed on a sound business basis.

This will take time to organise, and, in the meantime, we must not neglect our already existing Analytical Investigation Scheme. Last year, I referred to the unsatisfactory state of the financial side of this scheme, and explained that "the capital fund was not sufficient to provide out of dividends such assistance as might with advantage be given under the scheme." I invited subscriptions, and it is a great disappointment to me to find that not one penny has resulted from my appeal. I know that this cannot be from lack of sympathy or interest in the work, but merely because we have all been so busy that it has escaped our attention. I do ask you now to deal with this matter while it is fresh in your minds. Any sum from 2s. 6d. upwards would be of the greatest help, and I would like to suggest to you to give your subscriptions to the new treasurer to-night, as soon as you have elected him. Those who feel their sympathy running to amounts which can only be dealt with by cheque might also put their names, and the amount, on a piece of paper and hand it in to-night, and I can promise you that the treasurer will see those cheques are not forgotten. A sympathetic soul, touched by the want of success of my last appeal, has given 10 guineas to the scheme to-night, just to start the ball rolling—please make it up to 100 guineas.

THE MODERN ANALYTICAL CHEMIST.

During recent years, eminent and learned men have addressed us on the profession of chemistry, the teaching of chemistry, and many branches of science as applied to the common weal; but, when all is said and done, this Society is mainly concerned—though not entirely—with the profession of *analytical chemistry*; consequently, I should like, in conclusion, to say one or two words with regard to that important branch.

Our esteemed Past-President, Mr. Chaston Chapman, in his very able address on "The Teaching of Analytical Chemistry," referred to the establishment of chairs of analytical chemistry by our universities and colleges, and the next year he again made a plea that this should be done. The need still exists. Whatever branch of chemistry we may be concerned with, analytical chemistry is the fundamental key to that and every other branch; even synthesis, which may be held to be the opposite of analysis, cannot be conducted by anyone who is devoid

of analytical skill. Now in these days, when chemists tend to be of the academic type, there is a want of skill in the manipulative side of analytical chemistry. To make my point clear, I can put my finger upon a number of research chemists of a very high order whose analytical work I could only accept with very great reserve. These are the product of the universities who teach chemistry and neglect the art of manipulative chemical analysis, in spite of the fact that the science of analytical chemistry is becoming daily more elaborate and of a higher scientific order.

When this Society started half-a-century ago, the analysis of a product was seldom anything more elaborate than a mere list of the main constituents and their approximate proportions. Nowadays such a schedule is a mere preliminary, and the analyst is expected not only to make observations involving physical measurements but to turn his attention more particularly to the determination of very minute traces of substances, which modern experience has shown to be quite as important to determine as the main components. The more simple of these constituents are perhaps metallic contaminations; whilst the more complicated are of the nature of enzymes, vitamins, and bodies, the structure of which is less understood, but the importance of which is beginning to be fully appreciated.

One further duty will most certainly soon be added to the analyst's lot, namely, the determination of small quantities of lead in the atmosphere, resulting from the use of lead compounds in motor fuel, and this he should proceed to consider at once in order to assist the physiologist who with him will, in accordance with public demand, be asked to conduct more searching experiments to settle definitely the effect on the health of the community of the possible general use of such fuels.

It will be remembered that the sale of petrol containing lead tetraethyl was suspended in the United States of America pending the report of a committee of competent experts, who, having failed to find positive evidence of harmful effects, urged that the investigations should be continued. As this amounted to a verdict of "not proven" it is satisfactory to note that a Government Committee of Investigation is to be appointed in this country to enquire into the matter; but is it not remarkable that while the Government, by appointing such a committee, tacitly admits that ethyl petrol is suspect, and knows, moreover, that such a committee cannot carry out experiments and report for at least a year, it refuses to take any steps in the meantime to regulate the importation or use of the suspected material? Consequently, for a year the public is to be subjected to any dangers that may attend the use of the material, and no form of protection is to be afforded.

Obviously, by the end of the year the public will have made up its own mind, and any report the committee may issue will be without effect, unless it is in the direction of prohibiting use, when it will be too late. The situation has its humorous side, if it were not also dangerous. Analysts must get ready to render assistance promptly when called upon, so that a decision may be reached with the least possible delay, and they will also have to consider not only the determination of

metallic lead, as such, but its condition of combination, for is not lead tetraethyl one of the most powerful poisons known? Let me read an extract from a recent discussion in the House of Lords, as reported in *The Times*:—

> "LORD BUCKMASTER said he did not want an Inter-Departmental Committee, but a Committee composed of eminent scientific men.
>
> THE MARQUESS OF SALISBURY pointed out that the Council of Medical Research consisted of scientific men.
>
> LORD BUCKMASTER.—The Council of Medical Research contains a certain number of doctors; what we want are eminent chemists who know all about this matter."

These thoughts show us that the modern analytical chemist must be a specialist of wide experience having a training of a very high order, and the time will undoubtedly come when not only will chemists require to be registered, but the public will have to be protected more especially from those who assume the title of "analytical chemist" without experience or knowledge to justify them to practice.

In the early days of the Society the profession of analyst was not beset with the difficulties which exist to-day, and we older men can see a very steady change coming over the profession.

In years gone by the medical profession was represented by men very mixed both in skill and culture, and it was not until registration eliminated the quacks that the scientific and responsible nature of the calling drew to that profession the men of culture who are now in the majority. History is repeating itself, and so in our profession, the higher standard of competence is attracting a better type of man, and I am one who regards the time we are now passing through as one of the most critical turning points. Those of my generation, and the generation before me, who have worked to bring about the improvement now clearly in sight, must look to the young men to bring it to fruition, and it is for this reason that I depart from the usual custom in an address such as this to take it upon myself to say a few words upon professional deportment, more particularly addressed to the younger members of the profession—large numbers of whom I am glad to see are joining the Society. These young men will perhaps think me old enough to speak to them on the subject, yet young enough not to be too old-fashioned.

To these younger men I would appeal that they conduct themselves in a manner consistent with a learned profession and not a trade. To them I would say that it is no use crying to the Institute of Chemistry or to this Society to protect them against derogatory treatment meted out to them by Government Departments, by their employers, or by their clients, when the real fault lies in the behaviour of many of the individuals themselves. We hear of many cases where an individual has been subjected to infamous treatment, and when we really go into details we often find that had that individual been a different type of man, no-one would have dared to treat him thus. I do submit that if a man behaves with the dignity consistent with his profession he will command that respect which no organised body can procure for him. We hear complaints of

most humiliating and inadequate terms offered for important public appointments, but we must face the fact that such terms would never be thought of, if it were not that men exist who have so little appreciation of their own value as to accept such appointments.

Remember what Cassius said to Brutus:—

> "Men at some time are masters of their fates;
> The fault, dear Brutus, is not in our stars,
> But in ourselves that we are underlings."

This Society and the Institute of Chemistry lose no opportunities of co-operating in approaching Government Departments and local authorities on matters affecting the status or economics of the profession, and in so doing the officers have the support of the main body of the members. It is, however, heart-breaking to find every now and then that an isolated chemist defeats all the good that could be achieved by these efforts by acting in direct opposition to the considered and agreed policy of his professional brethren. This happens in every profession, but it affects us the more because we are members of a young profession.

This Society cannot teach a public, ignorant of science, that it is short-sighted to employ a man, whatever his qualifications may be, at a rate of remuneration that is inadequate, though the employer really suffers more than the employee.

Let us now consider the type of man who can be really useful to the public as an analytical chemist. Perhaps many of you will not agree with me, but I think a man who sets himself up as an analytical chemist must first and foremost have a sound general education with an aptitude for practical matters; he must also be a psychologist with a sense of proportion in all things. If a man has not these attributes he is wasting his time in studying that branch of chemistry required for the profession of analytical chemist, and I would like to see such men weeded out on these grounds alone before ever they touch a test tube. Given such a man, however, who is trained in the science of his profession, that man is able to approach a problem from a common sense point of view, and to bring all the skill of modern science to the solution of any given problem, and, having solved his problem and accumulated the necessary scientific data, is able to do the hardest of all, namely, set all that science which has helped him aside and put on paper in clear and simple words, comprehensible to his client, the information for which he has been asked—nothing more and nothing less.

Many and many a practical business man is deterred from the employment of an analyst because he has received in the past reports of a high scientific order which he has nevertheless never understood, and which did not contain in available form the information for which he had asked. Of course, I have not lost sight of the fact that in these days when knowledge of science is widespread there are among the public those who can appreciate scientific data. Our analyst, whom I have described as a psychologist, knows when to add such data, but it would be added as an appendix available for those who are competent to understand it.

So complicated indeed are some of the problems set by the more scientific manufacturers that the consulting analytical chemist of modern times—however wide his knowledge may be—cannot hope to be a specialist in each and every branch. Many a young man brings upon himself unnecessary discredit by undertaking work outside his ken, arising from the fear of losing what he can do by refusing what he can't. This is a short-sighted policy harmful to others as well as to himself.

Lastly, I would for a few moments dwell upon an evil of which we are all aware, but of which we never speak. Shall I call it the "curse of the dirty chemist"?

The days have long passed when chemical work had to be secretly conducted in underground cellars with filthy smoking furnaces. A modern laboratory with its equipment of electrical appliances, delicate instruments, and fine chemicals should be as clean as an operating theatre, and, above all, the chemist who occupies it should be, like a surgeon, a presentable person, well-groomed, and cultivating the "chemical equivalent" of a good "bedside manner."

The absence of a generally-recognised title to earmark properly-qualified and educated consulting practitioners permits of the existence of some undesirable camp followers of the profession, and it is a serious menace to the more self-respecting members that they seem to be able to do so little to eject that skeleton from our cupboard as represented by men of mediocre education, ignorant of dignity in chemistry, frequently unable to speak correctly, but oft-times knowledgeable. Such men give the public an entirely wrong impression of the best type of analytical chemist—as represented by the members of this Society.

Fortunately the number of such men diminishes year by year, but they are not yet extinct, and I seriously suggest that we are ourselves to blame for their continued existence, because we associate with them too freely out of respect for their scientific knowledge, and in blindness to their want of culture.

The pursuit of science seldom amasses riches, but even though an analyst be poor he should conduct himself with dignity, for although proud poverty commands respect, poverty and ignorance are not necessarily associated. While dirt and chemistry should now be scheduled as "incompatibles," chemistry and culture should be "inseparables."

Ladies and gentlemen, my time of active service as an officer of the Society draws to a close this evening, and I retire to the less active rôle of a past-president, with the happy memory of 12 years of work which has indeed been a great pleasure. They were years crowded with examples of friendship, support, and co-operation from members of the Society.

As your Honorary Secretary I served under six presidents who set a very high standard, and, consequently, when you did me the honour to elect me to this chair I was filled with fear as to the maintenance of that standard; now that my time has ended I beg to set against my shortcomings the fact that I have given you of my best.

My last duty is to welcome our new President. I am to be followed by one of my oldest friends, a man who can maintain all the best traditions of the Society, a public analyst of the best type, a scientist of no mean repute, and, what to my mind is most important of all, a man of understanding, blessed, in addition, with that knowledge of business so clearly evidenced in the report he has made as Treasurer to-night.

In Edward Hinks you have a worthy President, and in electing him you lose a tried and trusty Treasurer. Fortunately for the Society we have in E. B. Hughes just the stuff from which good Treasurers are made; he will provide an infusion of new blood, and I know that his nomination to the office is nothing short of a piece of shrewd foresight on the part of your Council.

In concluding, words completely fail me to express with any degree of adequacy my grateful thanks to our Treasurer and Secretaries for their co-operation and friendship. They have helped me more than I can say to steer the ship of this Society through the waters of conflicting interests. I, therefore, vacate this chair with the hope that during my time of office I have pleased some of you, for *frustra laborat qui omnibus placere studet.*

Tests for Impurities in Ether.

Part I. Test for Peroxides.

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

(*Read at the Meeting, December* 7, 1927.)

Although known for a considerable time, it is only now becoming generally recognised that one of the most important factors in the production of ether anaesthesia without undesirable after-effects is the purity of the ether used, and, in particular, its freedom from oxidation products such as acetaldehyde and peroxide. For this reason the method of testing ether for these impurities has become a subject of much importance.

Both hydrogen peroxide and an organic peroxide may occur in ether, but in the purified ether used for anaesthesia the latter has been found to predominate. This organic peroxide is considered to be either dihydroxydiethyl peroxide ($CH_3.CHOH.O.O.CHOH.CH_3$)[1] or α-ethoxy-ethyl hydrogen peroxide.[2] It has weak acidic properties, and is formed on exposure of ether to air or oxygen, the reaction being accelerated by light. Clover[2] observed also that the rate of oxidation is increased by the presence of traces of acetaldehyde. Further, he states that, since the peroxide itself decomposes, forming acetaldehyde, the velocity

of oxidation of the ether progressively increases, being at a minimum in the early stages of decomposition; and, consequently, ether which is free from aldehyde and peroxide shows less tendency to oxidise. In the experience of the present authors, samples of purified ether vary considerably in sensitiveness to oxidation; and the authors confirm Clover's views that acetaldehyde promotes the formation of peroxide, but they are not certain that this is the only, or even the chief, factor in the mechanism of oxidation. They have found, moreover, that, in general, specimens of ether which contain peroxide are less stable than those which are free from it.

Numerous tests for peroxide have been proposed during the last fifty years, but (with the exception of the work of Baskerville and Hamer[3] sixteen years ago) direct experimental evidence of the relative value of the various tests is lacking. In the present paper, experiments are recorded with the object of showing by a direct comparison which tests are most satisfactory for general adoption, having regard to the present demand for greater stringency.

Relative Sensitiveness of Tests.—The relative sensitiveness of a number of different tests has been determined, both for hydrogen peroxide and ether peroxide, and in the following table the figures observed for the limits of sensitiveness are given. These figures represent the smallest amount of peroxide required to produce a reaction that is just perceptible. Except where otherwise stated, details of the tests correspond to those given in the original references, but the tests were carried out on 30 c.c. of ether. All determinations were made in duplicate, the mean result being recorded.

Certain of these tests depend upon the reducing action of peroxides, and these also respond to the presence of aldehyde or other reducing substances. For this reason it was necessary to use a solution free from acetaldehyde in determining the sensitiveness to ether peroxide. This solution was prepared in the following way:—

A sample of ether was allowed to stand exposed to light for several weeks. Two hundred c.c. were then evaporated at a temperature not above 30° C. in a current of air, and the residue dissolved in 100 c.c. of pure ether. The peroxide was extracted from this solution by shaking with 20 c.c. of 5 per cent. potassium hydroxide solution, and the solution washed with 50 c.c. of ether and acidified with acetic acid. The acidified solution was extracted with 200 c.c. of pure ether, and the ethereal layer washed with sodium bicarbonate solution and dried with anhydrous sodium sulphate. The strength of this solution was determined by adding 25 c.c. to 25 c.c. of ferrous thiocyanate solution in a flask through which passed a current of carbon dioxide, warming to 30° C., and titrating with standard titanous chloride solution. The ether peroxide solution so obtained was then diluted with a definite amount of pure ether until it gave a just perceptible reaction with the reagent to be tested.

The results show that the organic peroxide and hydrogen peroxide react differently in many cases. The figures obtained are given below in reverse order of sensitiveness:—

	Sensitiveness (calculated as H_2O_2 in parts per million).	
	Ether peroxide.	Hydrogen peroxide.
Uranium nitrate[2]	Over 170	20
Chromic acid[4]	Over 170	10
Cerium nitrate[3]	27	10
Ferric ferricyanide[5]	17	0·1
Ferrous hydroxide[6]	9	—
Ammoniacal silver nitrate (Tollens) ..	9	2
Alkaline permanganate[7]	7	1
Cobalt hydroxide[8]	3·5	5
Vanadic acid[9]	3·5	2
Cobalt hydroxide[10]	1·7	0·4
o-Tolidine-peroxidase	1·4	0·05
Cadmium potassium iodide	1·3	0·2
Titanic acid[11]	0·8	0·5
Cadmium potassium iodide and starch ..	0·6	0·15
Potassium iodide	0·4	1
Potassium iodide and acetic acid ..	0·25	1
Reduced phenolphthalein (Stamm)[12] ..	0·2	0·2
Leuco-methyl green peroxidase	0·06	0·4
Potassium iodide and starch	0·025	0·05
Ferrous thiocyanate	0·017	0·02

Comparison at Different Dilutions.—With a view to employing these reactions in a quantitative way, a more detailed study was made of the three most sensitive—*viz.* the ferrous thiocyanate, leuco-methyl green peroxidase, and potassium iodide and starch tests—comparing them with the potassium iodide test, which is that most generally adopted. The comparison was made by determining the depth of tint produced over the range 0 to 2·5 parts of ether peroxide per million. The tints obtained were evaluated, and the intensity of the different colours compared, with the aid of the Lovibond tintometer. The results are represented in the accompanying graph (Fig. 1).

It will be seen that the curves differ considerably. It is evident that the best quantitative method will be that in which the intensity of colour is most nearly proportional to the amount of peroxide present and that which can be observed over a sufficiently wide range. From this point of view the ferrous thiocyanate test is shown to be the best, the corresponding curve having an approximately uniform slope over a wide range. It should further be noted that the red colour obtained at low concentration of peroxide with this test lends itself better to observation than the yellow colour obtained in the starch-iodide test.

Ferrous thiocyanate.—In the original form of this test, solutions of ferrous ammonium sulphate and ammonium thiocyanate were used. It is not possible, however, to obtain in this way a perfectly colourless solution of the reagent. To render it colourless the solution must be freshly reduced just before use.

Metallic zinc is not suitable for this purpose, as it reduces part of the thiocyanic acid, but a dilute solution of titanous chloride has been found to be a suitable and convenient reducing agent.

The reagent is prepared by boiling 30 c.c. of 10 per cent. sulphuric acid and 100 c.c. of water for a few moments in a flask through which passes a current

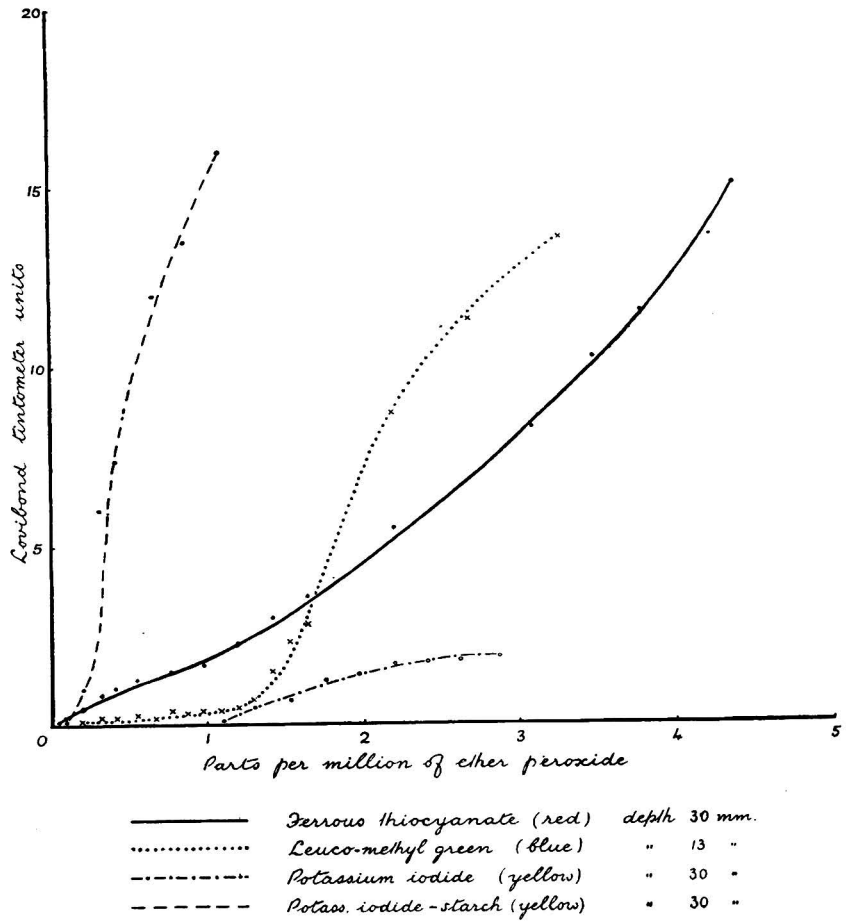

Fig. 1.

of carbon dioxide. Five grms. of pure crystallised ferrous sulphate are added and allowed to dissolve, and, after cooling, 30 c.c. of 10 per cent. potassium thiocyanate solution are added and the solution decolorised by the addition of titanous chloride (about 0·03*N*), drop by drop. Owing to the slow rate of reaction at ordinary temperatures, this reduction is carried out at about 40° C., and care must be taken to avoid the addition of excess of the reducing agent. The reagent

is kept under carbon dioxide and should be decolorised, if necessary, immediately before use.

For the test, small white glass-stoppered bottles of 35 c.c. capacity are used. Thirty c.c. of the ether to be tested are poured into the bottle, followed by sufficient of the reagent to raise the level of the ether to the mid-point of the neck. The stopper is inserted, and the bottle well shaken and allowed to stand in the dark. The colour is observed after a period of five minutes has elapsed.

As an alternative to the above method, one can avoid the use of titanous chloride by preparing the reagent from metallic iron, sulphuric acid, and potassium thiocyanate in an atmosphere of carbon dioxide, no reduction of the solution being then required. The reagent, so prepared, may be kept for several weeks if air is completely excluded, and the method offers the advantage that a stock of reagent may be kept available for immediate use. Further, the risk of error due to the addition of excess of titanous chloride is eliminated.

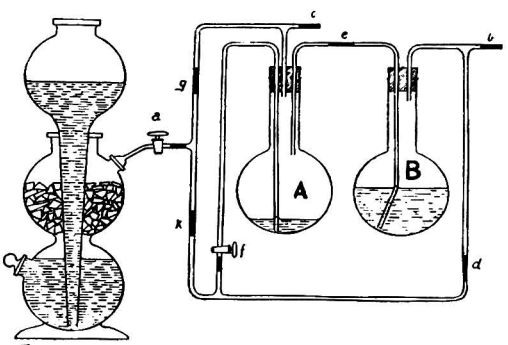

Fig. 2.

The apparatus (Fig. 2) consists of a Kipp's carbon dioxide generator and two round-bottomed 400 c.c. flasks, connected by glass and thick-walled rubber tubing, the latter provided with screw pinchcocks. Below the tap at *f* is a jet through which the reagent is finally run off. In A are placed 60 c.c. of 10 per cent. potassium thiocyanate solution, and in B 100 c.c. of 3·3 per cent. (w/v) sulphuric acid and 2 grms. of bright iron wire. The tap *f* being open, the apparatus is evacuated through *b*, and then filled with carbon dioxide. Dissolved air is removed from the solutions by boiling for two minutes, *e* being previously closed and steam escaping through *c* and *b* respectively, these being closed immediately boiling ceases. B is kept hot until all the iron has dissolved, hydrogen being allowed to flow back into the Kipp's apparatus and released at intervals through *b*. The solution in B, after cooling and allowing the slight deposit to settle, is transferred to A by closing *g* and *f* and opening *e* and *c*. After the two solutions are mixed, the rubber tubing at *f* is screwed down and detached from the tap. On opening the latter the siphon is filled by the gas pressure. The reagent, which must not show any brown colour, is drawn off as required.

A very pure ether will show no colour under this test, and ether employed for anaesthetic purposes should be required to give not more than a very slight colour. This is a commercially attainable standard. It is desirable that a definite limit should be set for the amount of peroxide permissible. We would define that limit by comparing the colour of the ether layer, after standing for five minutes, either with a standard solution of cobalt sulphate or with tintometer glasses. In the former case the tint should be not greater than that of an equal depth of an aqueous solution containing 0·15 grm. of crystalline cobalt sulphate and 2 c.c. of dilute sulphuric acid in 100 c.c., which is equal to a tint of 0·3 units of red on the Lovibond scale in a depth of 30 mm. of liquid. This limit corresponds approximately to 0·15 part of ether peroxide in one million parts of ether.

Potassium iodide and starch.—This test now only finds a place in the Pharmacopoeia of Holland. It was official in the British Pharmacopoeia of 1885, which stated that no blue colour should be produced. Probably the test was abandoned because the expected blue colour was not obtained, for starch and iodine in presence of potassium iodide in a solution saturated with ether give a brown, and not a blue, colour. If this is recognised, the addition of starch to the test is to be recommended, for it increases the sensitiveness of the reaction.

Commercial potassium iodide commonly has a faintly alkaline reaction, but this does not adversely affect the test, as no greater sensitiveness is observed when the solution is acidified with acetic acid. On the other hand, the addition of acid considerably increases the effect of atmospheric oxygen.

Generally speaking, the pharmacopoeias which include the potassium iodide test recommend a period of three hours. Nothing is gained by prolonging the time beyond this limit, as the reaction appears to reach its full extent in less than an hour. It is desirable that this (or any other test for peroxide) should be carried out with the minimum exposure to light.

Leuco-dye tests.—A number of experiments were made in which acidified solutions of various dyes decolorised by treatment with metallic zinc were used. Peroxide restores the colour of the original dye, giving a very sensitive reaction, but, unfortunately, all the dyes which were found suitable were too sensitive to atmospheric oxygen to be of practical use. Although this difficulty can be overcome by protecting the test solution with a layer of petroleum spirit, the care required in the manipulation renders this test unsuitable for general use. The following dyes were tried in this way: methylene green, methylene blue, cresyl blue, Nile blue, thionine blue, fluorescein.

Experiments were next carried out on solutions not sensitive to atmospheric oxidation, but with the use of a peroxidase as catalyst to promote the reaction. As the outcome of work on these lines the following reaction is worthy of mention:—

Leuco-methyl green.—The reagent is made by dissolving 0·02 grm. of methyl green in 3 c.c. of dilute acetic acid and 97 c.c. of water, boiling with 2 grms. of zinc dust till colourless, and filtering. This solution may be kept unchanged for

a week or two. A peroxidase solution is made by shaking 0·05 grm. of dried blood with 50 c.c. of water. (Peroxidases of vegetable origin did not appear to catalyse this reaction). For the test 50 c.c. of the ether are shaken with 1 c.c. of 5 per cent. potassium hydroxide solution, and the aqueous layer separated and made just acid with acetic acid. The reagent (0·5 c.c.) is added, followed by one drop of the peroxidase solution. After 30 minutes a green or blue colour indicates the presence of peroxide. This colour does not fade. The test depends on the acid nature of ether peroxide, which is more completely extracted from ether solution by alkali than is hydrogen peroxide. Since the latter is also considerably less stable in alkaline solution, this test is more sensitive for organic peroxide than for hydrogen peroxide.

The following reactions were also tried in presence of a peroxidase:—Oxidation of benzidine, *o*-tolidine, dianisidine, benzidine disulphonic acid, pyrogallol, metol, *p*-aminophenol, *m*- and *p*-phenylenediamine, guaiacum resin, and mixtures of α-naphthol and *p*-phenylenediamine, aniline and *p*-phenylenediamine, dimethylaniline and *p*-aminophenol, phenol and *p*-aminophenol, and phenol and dimethyl *p*-phenylenediamine. Of these, the only one of any value is given below.

o-Tolidine.—The solution (0·5 c.c.) containing 0·4 grm. of *o*-tolidine in 5 c.c. of dilute acetic acid is mixed with one drop of a peroxidase solution (made by diluting *Succus scoparii*, B.P. with five parts of water), and shaken with 30 c.c. of the ether to be tested. If peroxides are present, there is developed a green or blue colour in the aqueous layer after standing for 20 minutes. The colour disappears after some hours.

Reduced Phenolphthalein (Stamm's reagent).—This is employed as follows:—A solution of 10 grms. of sodium hydroxide in 20 c.c. of water is boiled with 1 grm. of phenolphthalein and 5 grms. of zinc dust till colourless, filtered through asbestos, and made up to 50 c.c. with water. The reagent may be kept and used as required. Four drops of the reagent are added to 3 c.c. of water, followed by 5 drops of a 0·05 per cent. copper sulphate solution, to which has been recently added a little sodium hydroxide. The mixture, which should be colourless, is shaken with 30 c.c. of the ether to be tested, the colour of the aqueous layer being observed after twenty minutes. This test is not affected by atmospheric oxygen.

Tollens' Reagent constitutes an exceedingly delicate test for organic impurities in ether, but is not a distinctive test for peroxide, since it is more sensitive to aldehyde. Only an exceptionally pure ether will give no colour with this reagent. Its use is to be recommended as a general test for indicating the purity of ether, but not as a specific test for peroxide. The figures given in the table (p. 208) show that other tests are much more delicate for the detection of peroxide. Directions for carrying out the test are as follows:—To 5 c.c. of 5·2 per cent. silver nitrate solution are added 1 c.c. of dilute ammonia (10 per cent.) and 2 c.c. of 20 per cent. sodium hydroxide solution. This mixture should be clear, but, if not, one or two drops more of ammonia are added. Five c.c. of the reagent, shaken with 30 c.c. of ether for 2 minutes, should not give more than a pale brown

colour. The reagent should not be kept more than a few hours, as after a short time it gives a deposit which may explode violently without warning.

PHARMACOPOEIAL TESTS.—Since it is to be anticipated that in the next edition of the British Pharmacopoeia the test for ether will be revised, it has been thought useful to include the following table indicating the tests adopted in current pharmacopoeias.:—

Pharmacopoeia.	Edition.	Pot. iodide.	Pot. iodide starch.	Cadmium pot. iodide.	Vanadic acid.	Chromic acid.	Ammoniacal silver nitrate.
British	1885	−	+	−	−	−	−
	1898	−	−	−	−	+	−
	1914	+	−	−	−	−	−
American (10th)	1926	−	−	+	−	−	−
French	1908	+	−	−	−	+	+
German (6th)	1926	+	−	−	+	−	−
Belgian (3rd)	1906	−	−	−	+	−	−
Swedish (10th)	1925	+	−	−	−	−	−
Norwegian (4th)	1913	+	−	−	−	−	−
Italian (4th)	1920	+	−	−	−	−	−
Swiss (4th)	1927	+	−	−	−	+	−
Dutch (5th)	1926	−	+	−	−	−	−
Spanish (7th)	1915	+	−	−	−	−	−
Portuguese	1876	−	−	−	−	−	−
Japanese (4th)	1922	+	−	−	−	−	−
Argentine	1921	+	−	−	−	−	−

\+ Indicates that the test is adopted by the Pharmacopoeia concerned.
− Indicates that the test does not appear.

CHOICE OF TEST.—It is now generally acknowledged that only highly purified ether should be employed for producing anaesthesia; and since, in addition, the experience of the authors confirms the observations of Dunstan and Dymond,[14] of S. van Leeuwen[15] and of Clover, who found that the stability of a specimen of ether is closely related to its initial purity, it is desirable that one of the very delicate tests for peroxide should be adopted officially.

From the results recorded above it will be seen that, judged from this standpoint, all the pharmacopoeial tests for peroxide, with the exception of that of the Dutch Pharmacopoeia, are unsatisfactory. In the authors' opinion, choice lies between the following tests:—potassium iodide and starch, reduced phenolphthalein, leuco-methyl green and peroxidase, and ferrous thiocyanate. If the maximum attainable purity is to be enforced for ether, the most sensitive test must be adopted, *i.e.* the ferrous thiocyanate test. The experience of several years in the routine testing of a very large number of samples by this method shows that the standard suggested, though stringent, is not impracticably high, and that the test is easy to apply and gives uniformly consistent results. It is true that the reagent is fairly readily oxidised by the air, but no appreciable amount of oxidation occurs during the period of the test, and it is easily possible to obtain blank tests with pure ether. The objection that special care and

apparatus are required for the preparation of the reagent cannot be considered a serious one.

SUMMARY.—(1) A comparison of the sensitiveness of tests for peroxide is given.

(2) Two new tests are described.

(3) A new and improved method of preparation of the ferrous thiocyanate reagent is described.

(4) The ferrous thiocyanate test is recommended for official adoption for testing ether, and a colorimetric limit to the amount of peroxide is proposed.

The authors wish to express their thanks to the Directors of The British Drug Houses, Ltd., for permission to publish the above results.

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Determination of Carvone in Dill Oil.

BY J. REILLY, M.A., D.Sc., F.I.C., AND P. J. DRUMM. Ph.D.

(*Read at the Meeting, February* 1, 1928.)

IN an examination of essential oils, carried out in this laboratory, considerable difficulty was experienced in the determination of the carvone content of dill oil. The method of Sadtler (*J. Soc. Chem. Ind.*, 1904, **23**, 303), in which carvone is transformed into a di-sulphonate by means of sodium sulphite, gave higher results than the similar one of Burgess (ANALYST, 1904, **29**, 78), but in the former a satisfactory end point in the control test titration was not obtained. Another method, much used for estimation of this ketone, is that in which the crystalline carvone-phenyl-hydrazone is isolated and weighed after purification. The compound, however, is not stable and decomposes on drying. The neutral sulphite process and the oxime titration method give the most consistent results. After a number of trials, the authors found a new method for the evaluation of carvone in dill oil by means of its semicarbazone.

Two isomeric semicarbazones of carvone have already been described by Rupe and Dorschky (*Ber.*, 1906, **39**, 2112). They do not appear, however, to have employed these compounds in quantitative work. The semicarbazone m.pt. 141–142° C. is obtained on mixing the reagents with external cooling, the semicarbazone m.pt., 163° C. being formed if the temperature is allowed to rise. The pure carvone used in these determinations was obtained through its crystalline compound with hydrogen sulphide. Attempts to prepare it by repeated distillations of commercial carvone gave a ketone admixed with traces of limonene.

METHOD OF DETERMINING PURE CARVONE.—Two grms. of carvone, dissolved in 50 c.c. of alcohol, were added to a solution of 2·25 grms. semicarbazide hydrochloride (Kahlbaum) in 10 c.c. of water. To the cooled mixture was then added 2 grms. of fused sodium acetate dissolved in 12 c.c. of water. The resulting solution was allowed to remain at room temperature for about twenty-four hours, when most of the semicarbazone crystallised out. Water was next added until the total volume of solution was 350 c.c. After standing a short time the precipitated semicarbazone was collected and dried in a vacuum desiccator.

TABLE OF RESULTS.

Carvone taken.	Semicarbazone precipitated.	Semicarbazone in solution.	Total Semicarbazone.	Carvone estimated.
Grms.	Grms.	Solution:	Grms.	Grms.
2	2·54	300 c.c. water, 50 c.c. alcohol, 0·12 grm.	2·66	1·93
5	6·38	750 c.c. water, 125 c.c. alcohol, 0·3 grm.	6·68	4·84

The crude semicarbazone prepared melted at 140°–142° C. The solubility of carvone semicarbazone in a mixture of 300 c.c. of water and 50 c.c. of alcohol was found to be 0·12 grm. In a series of determinations the values all approximated to 97 per cent., and possibly a factor might be used to allow for the deficiency.

DETERMINATION OF CARVONE IN DILL OIL.—The method of procedure was the same as for pure carvone. To a cooled solution of 6 grms. of semicarbazide hydrochloride in 15 c.c. of water was added a solution of 10 grms. of dill oil in 120 c.c. of alcohol and then a solution of 6 grms. of fused sodium acetate in 10 c.c. of hot water. After standing for 24 hours most of the semicarbazone had separated in crystalline form. The solution was diluted to 840 c.c. with water, and after standing for a short time the precipitated carbazone was collected and dried in a vacuum over sulphuric acid. The semicarbazone obtained was pure, a fairly conclusive proof being thus obtained that carvone is the only ketone in dill oil.

A series of determinations of carvone in dill oil gave close results, the extreme figures being given in the following table. No allowance is made for a factor.

Dill Oil taken. Grms.	Semicarbazone. precipitated. Grms.	Semicarbazone in solution.	Total Semicarbazone. Grms.	Percentage of carvone in dill oil.
10	4·94	120 c.c. alcohol, 720 c.c. water, 0·28 grm.	5·22	37·8
15	7·45	0·28 grm.	7·73	37·3

The results obtained by this method (when a factor is employed) are in close agreement with figures from the neutral sulphite method and are approximately 4 per cent. less than those obtained by the oxime titration method.

Carvone semicarbazone is not decomposed on boiling with water for a short time, nor is it volatile with steam. It can be dried at 100° C., without decomposition. It was thought that some semicarbazone might have dissolved in the terpenes remaining in the filtrate after separation of the carvone. On removing the terpenes in a current of steam no appreciable quantity of semicarbazone was precipitated.

Preliminary experiments on the determination of menthone and of the menthone content of peppermint oil have given promising results which it is hoped to communicate in a subsequent paper.

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Some Experiences in the Determination of Very Small Quantities of Iodides.

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(*Read at the Meeting of the Northern Section, February* 11, 1928.)

THE detection and determination of iodide in samples of "Iodised Salt," in which there may be as little as one part in 250,000, presents considerable difficulty, both because of the small absolute quantity present in a sample of any practicable size, and also because of the interference of the relatively enormous quantity of other substances present.

After trying, without success, a number of methods for concentrating the iodide in smaller bulk, such as solution in alcohol, preferential solution in small quantities of water, etc., I found a modification of a method used by Hunter for determining iodine in thyroid glands (*J. Biol. Chem.*, **7**, 336; ANALYST, 1910, **35**, 483) and later by Brubaker and others for the determination of iodides in natural waters (*J. Amer. Chem. Soc.*, **48**, 1502) to work satisfactorily.

The sample, 50 grms., is dissolved in, say, 250 c.c. of water, and a few drops of an ordinary laboratory solution of sodium hypochlorite added. This is followed

by 1 to 5 c.c. of 40 per cent. phosphoric acid. The solution is now boiled till all chlorine has been driven off, for which a few minutes will usually be enough. (With prepared "Table Salts," which may contain carbonates or phosphates, one must see that the solution is acid, or the chlorine will not be completely expelled.) The solution is now cooled, 1 or 2 c.c. of one per cent. potassium iodide added, and the liberated iodine is titrated with thiosulphate solution (about $N/200$); 1 c.c. of $N/211$ solution is equivalent to 0·6 mgrm. of actual iodine, or 0·1 mgrm. of iodine in the sample.

The hypochlorite oxidises all the iodide to iodate, which, in the acid solution, liberates iodine from the added iodide; and the iodine actually titrated is six times the amount originally present, which of course increases the delicacy of the method. It is desirable, if not necessary, to run a blank with all the reagents and a salt free from iodide. Results with solutions artificially made up in the laboratory gave 0·855, 0·900, 0·875 parts instead of 1 part in 250,000, and 0·88 instead of 1 part in 500,000; with a commercial sample 0·875, 0·670, 0·810 parts instead of 1 in 250,000. The discrepancies in these results were, no doubt, due to uneven distribution of the iodide, for the agreement among the results of the laboratory-prepared samples is very close.

Notes.

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

THE STANDARD POLENSKE APPARATUS.

POLENSKE's original paper (*Z. Unters. Nahr. Genussm.*, 1904, **7**, 273, and the abstract made from it (ANALYST, 1904, **29**, 154) give the distance between the adapter of the condenser and the bulb of the still-head as 78 mm., whereas the Official Methods of the A.O.A.C. (2nd ed., p. 292) give 70 mm. Mr. Warburton informs us that the only German authority giving 70 mm. is Benedikt and Ulzer's text-book (1908). Lewkowitsch gives 70 mm., and his block seems to have been derived from Benedikt, and not from *Arb. Kaiserl. Gesundheitsamte*, 1904, 545, which gives the distance as 78 mm. It seems obvious from the reference given by the A.O.A.C. that their block is taken from Lewkowitsch's book and not from either of the German journals.

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DETERMINATION OF SALT IN MARGARINE.

IN the January issue of THE ANALYST (p. 34), Gilmour described an alcohol method for determining salt in margarine. The following method has the advantage of being independent of the water content of the sample:—Weigh 3 grms. of margarine

into a 150 c.c. conical flask and warm till the sample melts. Add 10 c.c. of acetone and a few drops of chromate indicator, and titrate with 0·1 *N* silver nitrate to change of colour. Close the flask and shake well. A few drops more of the nitrate solution will complete the titration.

This method gives the same result as titrating the aqueous extract of the solids-not-fat. Filtering and pipette work are eliminated, as in the method of Flanders (1923) who used chloroform.

D. W. STEUART.

USE OF JANUS GREEN IN THE REDUCTASE TEST FOR MILK.

IN the account of Soep's comparative tests on the substitution of Janus green for methylene blue (ANALYST, 1928, 106) the order of the classification by Christiansen has apparently been reversed. The two scales should be as follows:—

		Barthel and Orla-Jensen's Scale. *Methylene Blue.* Reduction in:	Christiansen's Scale. *Janus Green.* Reduction in: Hours.
Class I.	Good milk. =Organisms, less than 500,000.	5½ hours minimum.	Over 6
Class II.	Indifferent quality. =Organisms, ½ to 4 millions.	2 to 5½ hours.	3—6
Class III.	Bad milk. =Organisms, 4–20 millions.	⅓ hour—2 hours.	1—3
Class IV.	Very bad milk. =Organisms, more than 20 millions, per 1 c.c.	Less than 20 minutes.	0—1

A comparison of the results given by 35 samples of commercial milk with the two dyes in this test has shown that, when worked with due precautions, methylene blue yields similar indications to those obtained with Janus green in respect of bacterial quality. Although the results with methylene blue are stated to be in the direction of leniency, owing to access of oxygen during the test, when the leuco-compound may be oxidised, this oxidation appears to be largely, if not entirely, prevented by the rising of the cream in the tubes, whereby a protective layer is formed. Janus green acts more slowly than methylene blue, and the red colour produced in the first stage of the reduction of this dye is not oxidised by contact with air. It has been noticed that this red colour shows early in the cream when the dye is about to be reduced throughout the milk, and it may show in some instances when the full reaction does not occur within the time limit (6 hours). It is possible that a better differentiation may be obtainable by incubating the tubes at 42° C. instead of 37° C.

It is generally found that when a bacterial count is made on milk showing reduction in either form of the test, the number of organisms present is lower than is indicated on the scales above, although the count is higher than would be associated with bacteriologically satisfactory milk.

A. R. TANKARD.

Reports of the Essential Oil Sub-Committee to the Standing Committee on Uniformity of Analytical Methods.

INTERIM REPORT ON THE DETERMINATION OF ACETYLISABLE CONSTITUENTS IN ESSENTIAL OILS.

As a result of the investigations of the Essential Oil Sub-Committee, evidence has accumulated that even slight variations in the procedure adopted by analysts in carrying out the determination of the acetylisable constituents of essential oils are responsible for serious differences in the results obtained.

The Sub-Committee feel that it is urgently necessary to avoid these differences, and, after extended investigations, the following detailed method of procedure has been agreed upon and is presented with this end in view:—

METHOD OF ACETYLATION.—10 c.c. of the oil, 20 c.c. of acetic anhydride (95 to 100 per cent.), and 2 grms. of freshly fused anhydrous sodium acetate, are mixed in a long necked round-bottomed 200 c.c. Kjeldahl flask; a fragment of broken glass is added, and the contents boiled gently under an air reflux condenser for 2 hours.

The flask should be supported on a sheet of asbestos board, in which has been cut a hole about 1½ inches in diameter, and should be heated by a small naked flame, placed about 1 inch below, and not impinging on the bottom of the flask.

At the expiration of 2 hours, the flame is removed and the flask allowed to cool: 50 c.c. of water are added, and the flask and contents heated on a boiling water bath for 15 minutes, with frequent and thorough shaking. After cooling, the contents of the flask are transferred to a separating funnel and the lower aqueous layer rejected. The acetylated oil is then washed successively with:— (1) 50 c.c. of brine (saturated aqueous solution of sodium chloride); (2) 50 c.c. of brine containing 1 grm. of sodium carbonate in solution; (3) 50 c.c. of brine; (4) 20 c.c. of water. Mixtures 1, 2 and 3 should be shaken vigorously, but the final washing with water must be conducted with gentle shaking only.

If the washing operations have been properly conducted, the aqueous layer from the second washing should be alkaline to phenolphthalein. (Alcoholic phenolphthalein must not be added to the mixture in the separator.)

When the washing is complete, the aqueous layer is removed as completely as possible, and the oil poured out and mixed with about 3 grms. of powdered anhydrous sodium sulphate, stirred for 15 minutes or until one drop of the oil produces no cloudiness when added to 10 drops of carbon disulphide in a dry test tube. The oil is then filtered through a dry filter paper in a covered funnel.

METHOD OF HYDROLYSIS.—About 2 grms. of the dried and filtered oil are accurately weighed into a hard glass flask, 2 c.c. of distilled water added, and the free acidity titrated with $N/10$ *aqueous* KOH, using 1 c.c. of 1 per cent. solution of phenolphthalein in 60 per cent. alcohol as indicator. Forty c.c. of N/2 alcoholic KOH are then added and the mixture boiled under a reflux condenser on a water bath for 1 hour; the flask is then cooled rapidly, 20 c.c. of distilled water added and the excess of alkali titrated with $N/2$ H_2SO_4.

A blank determination of the alcoholic potash must be carried out simultaneously with the hydrolysis of the acetylated oil, and under conditions conforming as nearly as possible with those employed therein.

(*Signed*),

John Allan (Chairman), C. T. Bennett, S. W. Bradley, E. Theodore Brewis, L. E. Campbell, Thos. H. Durrans, T. W. Harrison, Ernest J. Parry, C. Edward Sage, M. S. Salamon, W. H. Simmons.

T. Tusting Cocking (Hon. Secretary).

February, 1928.

THE DETERMINATION OF PHENOLS IN ESSENTIAL OILS.

THE Essential Oil Sub-Committee make the following recommendations with regard to the determination of phenols in essential oils. We are unanimous in our opinion that the most suitable method for general purposes for the determination of phenols in the oils of ajowan, bay, cinnamon leaf, clove, origanum, pimento and thyme, is that of shaking the oil with cold aqueous potassium hydroxide solution and measuring the amount of unabsorbed oil. It has been found that, in order to obtain uniform results, standard conditions must be strictly adhered to.

The determination is carried out in a flask consisting of a bulb of about 150 c.c. capacity with a long neck, of which 10 c.c. is graduated in 1/10ths of a c.c., the length of the graduated portion being not less than 15 cm. Before use, the flasks should be cleansed with strong sulphuric acid and well rinsed out with distilled water.

METHOD OF DETERMINATION.—Eighty c.c. of 5 per cent. aqueous potassium hydroxide solution are placed in the flask, followed by 10 c.c. of the clear oil, and the mixture thoroughly shaken at 5 minute intervals during 30 minutes, at room temperature.

The unabsorbed portion of the oil should then be raised into the neck of the flask by the gradual addition of more of the potassium hydroxide solution, and the separation of the oily layer facilitated by rotating the flask between the hands and gently tapping. After standing for not less than 24 hours, the volume of unabsorbed oil should be read off, taking the bottom line of the meniscus, in each case. The proportion absorbed, multiplied by 10, will give the percentage by volume of the phenolic content of the oil under examination.

Where a small quantity, not exceeding 0·4 c.c., of emulsion is formed between the oily and aqueous liquids, a mean reading of this should be taken. If an emulsion be formed which will not separate, a repeat test should be carried out with the addition of 2 c.c. of xylene* to the test mixture before the initial shaking. This facilitates the separation of the oil; the final reading of unabsorbed oil should be corrected for the added xylene.

The potassium hydroxide solution should be clear and adjusted to contain 5 grams (4·9 to 5·1) potassium hydroxide in 100 c.c.; and whilst the presence of chloride or carbonate does not materially affect the result, yet it is advisable to restrict the amount of these impurities to the proportions which are ordinarily present in good commercial stick caustic potash. It should be free from more than traces of silica and alumina, as these are detrimental impurities, giving rise to separation of flocculent matter.

* Xylene or xylol of commerce with a boiling range of 137° C. to 142° C. is here intended. It should be tested to ensure freedom from impurities soluble in 5 per cent. aqueous potassium hydroxide.

Experiments have been carried out in order to determine:—

(1) The best strength of alkali for absorption.

(2) The more suitable—potassium hydroxide or sodium hydroxide.

(3) Which gives the better results—hot or cold treatment.

For this purpose, mixtures of redistilled eugenol and clove terpenes; thymol and thymene; and carvacrol and paracymene, were employed.

The results obtained indicate that potassium hydroxide is better than sodium hydroxide, that a 5 per cent. solution is the most suitable strength for general purposes, and that, for the above oils, cold treatment is preferable.

Sodium hydroxide does not give good separations.

Small variations in the size of the flasks used made no appreciable difference in the results obtained.

The process has been found to give accurate results with known mixtures of pure phenols and terpenes. With samples of normal oils which were circulated amongst the members of the Sub-Committee, the results of the tests lead us to the opinion that the limits of error are within $\pm$ 1 per cent. This degree of accuracy cannot always be attained with oils that are oxidised, polymerised, or otherwise unusual in character.

BAY OIL.—With this oil a secondary liquid layer frequently occurs at the bottom of the separated non-phenols. This should be included in the unabsorbed portion.

CINNAMON LEAF OIL.—A series of experiments was carried out in order to determine the effect of the presence of aldehydes on the results obtained by absorption with 5 per cent. potassium hydroxide solution. It was found that the addition of as much as 10 per cent. of cinnamic aldehyde to a cinnamon leaf oil did not appreciably affect the determination of phenols by this method.

CLOVE OIL.—In the case of this oil, eugenol and aceto-eugenol are both absorbed.

A number of oils have been tested by all members of the Sub-Committee by the hot method, as well as by the cold method. The results of tests on three typical oils, by the proposed method are given below, and these show a maximum variation of 2 per cent.

Member.	Bay Oil.	Cinnamon Leaf Oil.	Thyme Oil.
(1)	50	78·5	69
(2)	49·5	78·5	67·5
(3)	48	78	68
(4)	49	79	69·5
(5)	49	79	67·5
(6)	49·5	79	68·5
	48·5	—	—
(7)	49	—	—
(8)	50	78·5	68·5
	—	78·7	—
(9)	49·5	77	68
(10)	49	78	69
(11)	50	78	68

(*Signed*)

John Allan (Chairman), C. T. Bennett, S. W. Bradley, E. Theodore Brewis, L. E. Campbell, Thos. H. Durrans, T. W. Harrison, Ernest J. Parry, C. Edward Sage, M. S. Salamon, W. H. Simmons.

T. Tusting Cocking (Hon. Secretary).

February, 1928.

Impurities in Colouring Matters Used in Food.

FIRST REPORT OF THE REPRESENTATIVES APPOINTED BY THE SOCIETY OF PUBLIC ANALYSTS AND BY THE ASSOCIATION OF BRITISH CHEMICAL MANUFACTURERS TO CONSIDER THE IMPURITIES PERMISSIBLE IN COLOURING MATTERS USED IN FOODSTUFFS.

ARSENIC.

THE Sub-Committee appointed to consider this question comprised the following members:—Messrs. E. F. Armstrong, F. Thomas, T. Callan and C. K. Crosland, *representing the Association of British Chemical Manufacturers.* Messrs. E. R. Bolton, L. K. Boseley, H. E. Cox, J. T. Hewitt, E. Hinks, and F. W. F. Arnaud (*Hon. Sec.*), *representing the Society of Public Analysts and other Analytical Chemists.*

The Report was submitted to the Council of the Society on March 7, 1928, when it was decided that it should be published in THE ANALYST.

(1) The appointed representatives have given careful consideration to the question of the maximum permissible amount of arsenic in colouring matters used in foodstuffs.

(2) The Committee would emphasise the fact that colouring matters are added to foodstuffs only in very small proportions and, therefore, it would not appear necessary, from the point of view of public health, that, as regards degree of purity, the colouring matters used in the preparation of articles of food should be regarded as being in the same category as the foodstuffs themselves or as constituents forming a large proportion of foodstuffs.

(3) The limiting proportion of arsenic suggested in 1903 in the Report of the Royal Commission appointed to enquire into arsenical poisoning arising from the consumption of beer and other articles of food or drink was 1/100th grain per pound or 1·4 parts of arsenic per million. This standard of 1·4 parts of arsenic per million places the manufacturers of colouring matters in a more difficult position than the manufacturer of the majority of pure drugs for which an arsenic limit is prescribed in the British Pharmacopoeia, drugs which are frequently taken in relatively large quantities. For instance, the limit of arsenic in purified borax is 5 parts per million, in glycerin 4, in magnesium sulphate 5, in sodium carbonate 2, in potassium nitrate 5; many other pharmaceutical preparations are allowed higher proportions than these, but in only two cases (citric and tartaric acids) is a pure solid substance required to conform to the standard of 1·4 parts per million.

(4) The Committee had evidence that, in order to standardise the tinctorial value and facilitate the use of colours of high tinctorial strength, dilution of the actual colouring matter with certain non-colouring substances was often necessary. Whilst the greater number of commercial colours were not diluted to an extent greater than 1 part of colour to 2 parts of diluent, there were a number of colours used for specialised purposes where the dilution was greater, *e.g.* solutions (alcoholic) of colouring matters. It was considered that in the latter cases the limit of arsenic

should correspond to that generally recognised for foodstuffs themselves; *i.e.* 1·4 parts per million. The Committee appreciated that it would be possible by the mere process of the addition of diluents free from arsenic to bring a colouring matter containing somewhat excessive quantities of arsenic to a low limit, but they were advised that the risk of this at the present time was not serious and was tending to decrease, as users of colouring matters were becoming more insistent on definite statements of actual colour content. An alternative method of dealing with diluted colours would be to calculate the arsenic content on the actual colouring matter present. As, however, the methods available for the determination of arsenic in colouring matters were liable to an error of the order of half a part per million, any such error in the case of a highly diluted colour would be multiplied in accordance with the dilution and would fall entirely on the actual colouring matter. Thus, if a colour were diluted to give a product containing 5 per cent. of colour, an error of half a part per million would calculate back to 10 parts per million on the pure colour.

(5) There is at present no legally enacted limit for the proportion of arsenic permissible in a food colouring matter. The Public Health (Preservatives, etc., in Food) Regulations prescribed no limit, the Minister of Health not having adopted the recommendation of the Departmental Committee or of the Royal Commission mentioned above.

(6) The Committee was informed that a relaxation of the uniform limit of 1·4 parts of arsenic per million for all types of colours would be of material assistance to manufacturers, and the Committee therefore considers that the arsenic content of a pure colour might be of the same order as that laid down in the British Pharmacopoeia for several pure drugs. This would not lead to the occurrence of arsenic in the finished food product in such an increased proportion as would be determinable.

(7) Pending the legal enactment of a limit by such a body as may be empowered to act, the Committee recommend that the following maximum proportion of arsenic (As_2O_3) in food colouring matters should be considered as permissible:—

(*a*) In a pure (straight) colour, 5 parts per million.

(*b*) In colours not diluted to a greater extent than 2 parts of diluent to 1 part of colour, 3 parts per million.

(*c*) In colours diluted to a greater extent than provided in (*b*), 1·4 parts per million.

The percentage of actual colouring matter present in all cases shall be taken as the difference between 100 and the percentage of diluent. The diluents usually used are sodium sulphate, sodium chloride and dextrin, and these can be readily determined with sufficient accuracy for this purpose.

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.

ANNUAL REPORT OF THE CITY ANALYST, 1927.

THE number of samples examined during the year was 5560, of which 4819 were taken under the Food and Drugs Acts. Of these, 152 were adulterated, 12 being by preservatives only.

SULPHUR DIOXIDE IN GROUND GINGER.—Ten of the 16 samples of ground ginger were free from sulphur dioxide. One sample contained a mere trace, and 5 others from 800 to 3000 parts per million. Enquiries from the vendors showed that in four cases the ginger had been in stock from two to four years, but in the fifth case the article had been delivered only a few days, and in this case the wholesale dealer was cautioned.

A letter was sent to the secretary of the local Grocers' Association asking that the attention of the members should be called to this form of adulteration. This suggestion was very cordially received, and the Association took prompt action, and also brought the matter before the Grocers' Federation. The presence of the sulphur dioxide was due to the fact that some varieties of ginger are bleached abroad with this preservative.

When the Regulations were made, the presence of sulphur dioxide in ground ginger was probably overlooked, and no limit was fixed. Unless the Ministry of Health issues a further Regulation permitting the presence of a certain amount of sulphur dioxide, it seems advisable that no action should be taken with regard to this preservative this year, as in many cases the retail sale of ground ginger is very slow, and it will take some time before ginger, treated with sulphur dioxide previous to the issue of the Regulations, has been disposed of. I am informed that the ginger is bleached to supply the public demand for a light-coloured article. Fifteen samples of ginger have been roughly classified according to colour. The two palest samples had not been bleached. Two of the next three contained sulphur dioxide, but it was not present in the next darker class of 5 samples. Three samples containing sulphur dioxide, in one case only a trace, were darker than the previous 10 samples. It is obvious that the bleached samples were not palest in colour, as they occur in classes two and four. With one exception they contained the largest amounts of mineral matter.

FLOUR. SELF-RAISING FLOUR.—Eighteen of the 49 samples of flour and 2 of the 12 samples of self-raising flour contained a persulphate or benzoyl peroxide. Thirty-three per cent. of the flours had been treated, being an increase on the 24 per cent. found in the previous year.

EGG SUBSTITUTE POWDER.—The 9 samples of egg substitute powder examined last year yielded from 2·5 to 9·8 per cent. of carbonic acid gas on addition of water. Two of them, obtained at one shop, were certified as adulterated. These were less than half the strength of any of the others, yielding only 2·5 per cent. and 2·6 per cent. of carbonic acid gas (*cf.* ANALYST, 1927, **52**, 532, 536).

PUDDING SPICE.—This article is rarely analysed, and no previous samples have been submitted. I do not know of any accepted commercial or analytical standard for the spice. The mineral matter in the 14 samples examined varied from 5·2 to 7·7 per cent., and the sandy matter from 0·7 to 2·5 per cent. It is evident that any mixture of suitable spices may be sold under this name, and that sandy grit is an undesirable constituent. Spices of fair quality do not contain much mineral matter or sand. In nutmeg, mace, allspice and cloves, the sandy matter may be about 0·1 per cent.

Two samples contained 2·3 and 2·5 per cent. respectively of sand and were certified as adulterated. They were obtained from two branches of a company's shop, and the vendor was cautioned. He had a warranty from his wholesale dealer, and the latter, in turn, had one from his spice merchants. Either one of the constituents of these pudding spices must have been heavily adulterated with mineral matter, or the spices used must have been of very inferior quality. (See also *Quarterly Reports*, ANALYST, 1927, **52**, 473, 531; 1928, **91**, 152.)

J. F. LIVERSEEGE.

Legal Notes.

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

LINIMENT OF TURPENTINE.

ON January 6, three Islington druggists were summoned at the North London Police Court for having sold liniment of turpentine not of the nature, substance and quality demanded, in that the samples were deficient in rectified oil of turpentine to the extent of 53, 69, and 53 per cent. respectively.

Mr. Robertson, for the Islington Borough Council, said that the Analyst's certificate would show that the articles supplied were not liniment of turpentine according to the British Pharmacopoeia. The sample in one of the cases contained 266 parts of non-volatile matter (by weight), rectified oil of turpentine, 300 fluid parts; distilled water and other volatile matter, up to 1000 fluid parts.

Mr. Glyn-Jones, for the defence, said that the point was whether a member of the public requiring an embrocation wanted the B.P. article. If he received something different, but it was what he wanted, he submitted that there was no offence.

Mr. Robertson contended that there was only one article entitled to be sold as liniment of turpentine, and said that in this case an altogether different article, containing no camphor, was supplied.

Mr. E. Pinchin, F.I.C., the Public Analyst, giving evidence in support of his certificate, said that he had never heard of such a mixture being supplied as liniment of turpentine, commercially or otherwise. In reply to the magistrate (Mr. Basil Watson, K.C.), he said that if a sample were taken under the Food and Drugs Act he thought that the British Pharmacopoeia must be followed.

Mr. C. E. Corfield, F.I.C., said that if asked for liniment of turpentine, the only preparation that he would supply would be that of the B.P. In cross-examination, he agreed that the bulk of the embrocation used by the public was not the B.P. article. Witness was handed a copy of the London Insurance

Pharmacopoeia, which on page 11 gave a formula for white liniment of turpentine which corresponded with the analyst's certificate. He admitted that there would be no material gain to the seller in supplying this article instead of the B.P. preparation, and that the purchaser would suffer no injury, but he had not got what he had asked for.

Dr. Ambrose, giving evidence for the defence, said that if he wished to define the significance of a drug he would turn to the British Pharmaceutical Codex. Personally, he preferred to prescribe the embrocation supplied by the defendant rather than the B.P. article. In his experience, the general public did not use the correct British Pharmacopoeia names. It was quite a common thing for them to ask for liquorice powder when they meant compound liquorice powder. Recognising the difference between white oil and liniment of turpentine, he always specified the one that he required.

The magistrate said that the point was that the purchaser did not get the actual article he required. The evidence for the defence had been absolutely honest, but it had clearly proved the case for the prosecution. As it was a technicality that he had to decide, rather than a case, he thought that it would be sufficient if each defendant paid £5 5s. costs.

Report of the Privy Council Committee for Scientific and Industrial Research

FOR THE YEAR 1926-27.*

THE Report of the Advisory Council deals with matters of policy and of special interest (*a*), and a summary of work undertaken in the various Departments follows (*b*).

(*a*) FUEL RESEARCH.—The Gas Light & Coke Company are erecting a bank of the retorts developed at the Fuel Research Station for producing about 100 tons of smokeless fuel a day. This is a commercial trial for ascertaining facts as to the possibilities of low-temperature carbonisation. As a result of trials with selected British coals, both in Germany and at the Fuel Research Station, it has been shown that a large proportion of the coal substance may be converted into liquid fuels by means of hydrogen under high pressure. Co-operative work with the Tar Distillers' Association has been arranged in connection with the properties of pitch for briquettes, etc.

The investigations of the Air Ministry Advisory Committee on Atmospheric Pollution have been transferred to the Fuel Research Department, and the supervising Committee will be known as the Atmospheric Pollution Research Committee; whilst the former Advisory Committee on Atmospheric Pollution becomes an autonomous Standing Conference for co-ordination between the Departments and Local Authorities.

FOOD INVESTIGATION.—The grant of £35,000 from the Empire Marketing Board to the Low Temperature Research Station at Cambridge makes much needed extension possible. Dr. Kidd left for Australia, at the request of the Australian Government, to investigate special problems of storage and transport of fruit.

* Obtainable at Kingsway House, Kingsway, W.C.2. Price 3s. net.

BUILDING RESEARCH.—The new Building Research Station, occupied for eighteen months, is making headway, and there are definite signs of co-operation from the building industry in problems affecting their products.

FOREST PRODUCTS.—The new Station at Princes Risborough is now occupied.

WATER POLLUTION.—A Water Pollution Board has now been approved, and will have such problems to consider as the establishment of the "activated sludge" process on a real scientific basis; the treatment of the wet solid residue; purification of trade effluents, to avoid the often inevitable destruction of either the industry, or the river, as a source of profitable water.

(*b*) FOOD INVESTIGATION.—*Ham and Bacon.*—Work on the measurement of the heat diffusivity of fat and lean meat has been begun, on the alteration of distribution of water in the tissues by curing, and on the use of frozen pork for canning. A beginning has been made on some of the unsolved problems in bacteriology.

Fish.—Work on the preservation of fish at sea has resulted in the fitting of freezing appliances on some vessels, but large-scale experiments are urgently needed.

Freezing of Tissues.—A Report on the way water is held in tissues is being prepared, and some slow freezing and thawing experiments on quarters of beef completed.

Rabbits.—Discoloration of fat in rabbits was found to be due to an oxidation process, similar chemically to that of linseed oil.

Fruit and Vegetables.—A theory has been formulated relating the good or bad keeping quality of apples to definite features in their chemical constitution; this is based upon accumulated evidence of research work, and suggests that the life expectation of gathered fruit depends upon the amount of living protoplasm contained, and the extent of the sugar reserves accumulated during the growth period. The former determines the intensity of the drain on the latter, death ensuing when the sugar available for the normal process of respiratory combustion in the tissues comes to an end. As a rule, the best keeping apples contain the least protoplasm and the most sugar, and have the lowest respiratory activity.

FOREST PRODUCTS RESEARCH.—*Wood Technology.*—A detailed study of Trinidad timbers is being made; of the structure of ash and its relation to mechanical properties, and of the variation in structure of oak. Specimens sent for identification involve much work.

Mycology.—At the Imperial College, culture work on the identification of wood-destroying fungi has been carried out, and particularly on the fungi attacking aeroplane spruce timber in storage, the latter in conjunction with the Canadian Department of Agriculture.

Chemistry of Wood.—The work at St. Andrew's University has resulted in showing the presence of both ether and ester methoxyl groups in hemicellulose A from beech and oak, so that hemicellulose bears a closer resemblance to the pectic substances and lignin than has been thought. Further, 80 per cent. of hemicellulose A obtained from moist sawdust from beech wood 93 years old was acetylated, but from beech wood 130 years old, and from American white oak heartwood of unknown age, only 20 per cent. was acetylated.

CHEMICAL RESEARCH.—*Low-Temperature Tars.*—Impurities causing darkening of the separated phenols by light have been removed by a method depending on the action of sodium, and the products remain water-tight and sweet after six

months' exposure to light. Some success has attended investigations as to the use of phenols from low temperature tars as "anti-knocks" in petrol for internal-combustion engines, and as germicides and disinfectants. The use of these chlorinated derivatives for protection of fabrics against micro-organisms is to be tested.

Minor Metals.—Cyclo-telluropentane, the parent substance of the cyclo-tellurium compounds, has been prepared, and also co-ordination compounds of gold, silver, copper and other metals with ethylene thiourea. The gold compounds are germicidally as active as sanocrysine and dissolve in water to fairly neutral solutions.

Chemotherapy.—Organisation of research in this branch is pending.

Complex Constituents of Tar.—Two dyestuffs having 2:7-diaminofluorene as intermediate have been submitted to the Dyestuffs Development Committee. The nitration of acenaphthene is being studied, with a view to preparing the three possible mononitro-derivatives and corresponding amines.

Chemistry Co-ordinating Research Board.—Much attention has been given to problems put forward by the Service Departments. The large-scale apparatus for researches on high-pressure gas reactions, with special reference to synthesis of organic compounds, is now in use. The work on the production of higher alcohols from carbon monoxide and hydrogen has now been resumed as the circulatory apparatus is completed.

Corrosion of Metals.—An account of the experimental arrangements has been published (*Proc. Roy. Soc.*, 1927, (A), **116**, 425), and also of the routine preparation of low-conductivity water (*J. Chem. Soc.*, 1927, 2156).

Helium Extraction.—The collection of helium from monazite sand brought over for the manufacture of incandescent gas mantles has been only partially successful, and modifications of the method are being explored.

Synthetic Resins.—Resins obtained by alkaline treatment, utilising a special controlling procedure, have been made up into moulding compositions with wood, etc., and the physical properties of the mouldings examined (*cf.* ANALYST, 1927, **52**, 645).

Production of Cellulose, etc., from Artichokes.—The large scale experiments show that under present conditions, artichokes cannot be economically used for the manufacture of alcohol and good quality cellulose, largely owing to cost of harvesting and transport charges.

ENGINEERING CO-ORDINATING RESEARCH BOARD.—*Light alloys.*—The study of the permanence of dimensions of aluminium alloy objects has reached the report stage, and the first part of the work relates to the "Y" and L.5 alloys and wrought duralumin. In general, the changes due to heat treatment vary considerably with the different specimens. Marked brittleness has often been found in remelted aluminium; simple remelting or bubbling nitrogen through the melt are beneficial as curative processes, and attention is now being directed to hardening elements.

Fatigue of Materials.—In the work on the fatigue failure of single crystals of iron it is noted that, whilst there is no change of density in a specimen composed of a single iron crystal after it has undergone fatigue tests, there is a change after treatment of an aggregate of several crystals, possibly due to effects confined to the neighbourhood of the crystal boundaries.

BRITISH MUSEUM LABORATORY.—A material composed of celluloid and sand, and possessing a certain necessary elasticity, has been successfully used for the

mounting of the large Chinese frescoes now on the staircase in the King Edward the Seventh building. The brittle condition of the "wasted" leather of a roll of Egyptian manuscript dating from 1600 to 1500 B.C. was successfully combated by cautious application of a celluloid preparation to the exposed portion, enabling the roll to be gradually opened out. Objects from the excavations in Ur of the Chaldees, made of gold, silver, lapis lazuli and cornelian, were cleaned and prepared for exhibition. A simple method for roughly estimating the amount of phosphate in English procelain has enabled a distinction to be made between the earliest Bow and Lowestoft products and those from Chelsea.

X-RAY ANALYSIS OF CRYSTALS.—Work on the industrial application of X-ray analysis has been undertaken on magnet steels, particularly on material supplied by the Brown-Firth Research Laboratories; and the scope of investigations is expected to extend to X-ray methods on notch-bar brittleness, clays and refractories.

D. G. H.

"Unsaturated Hydrocarbons" in the Gases from the Carbonisation of Coal.*

THE usual methods of gas analysis provide for the determination of the total percentage only of the unsaturated constituents of gas mixtures produced by coal carbonisation and oil cracking processes, etc., but not for the relative proportions of the individual gases or vapours. In view of the possible commercial utilisation of the "unsaturated hydrocarbons" (those absorbed by bromine or concentrated sulphuric acid, *i.e.* aromatic, olefinic, diolefinic and acetylenic hydrocarbons), a more exact knowledge of the composition of these gas mixtures is desirable. This would also enable more accurate calculations of the calorific values of gas-mixtures to be made.

The methods previously employed are discussed. The separation of unsaturated hydrocarbons by fractional distillation of their bromine compounds is long and tedious, whilst the method based on the fractionation of the liquefied gases is costly and complicated. Dobrjanski's modification of Lebeau's method (*Bull. Soc. Chim.*, 1924, **35**, 489) in which the different gases are absorbed in succession by sulphuric acid of various strengths, would introduce considerable error in the case of coal gas.

The method described, which deals only with gaseous constituents, involves the separation of the unsaturated gases as bromine compounds, and the regeneration of ethylene, propylene and butylene by a zinc-copper couple.

About 10 cb. ft. of gas are scrubbed with 1·5 litres of gas oil (b. pt. 250 to 350° C.) in a vertical tower filled with Lessing rings, and passed into a suitable quantity of bromine (25 to 75 c.c.) covered with a layer (100 c.c.) of a 10 per cent. solution of potassium bromide, contained in a 2-litre Woolf bottle. Two such bottles are provided, and any bromine which escapes is trapped in two spiral condensers cooled by ice and salt. The wash oil from the scrubber is distilled up to its original b. pt., and the small quantities of unsaturated gases removed by the scrubbing are collected in bromine. The bromine compounds are bulked, washed free from bromine with dilute sodium hydroxide solution and weighed, and any

* FUEL RESEARCH. TECHNICAL PAPER No. 17. By A. B. Manning, J. G. King, and F. S. Sinnatt. H.M. Stationery Office. Pp. 19. 1928. Price 6d. net.

bromides recovered from the washings after distillation are included. They are then divided into the three following fractions by distillation under 20 mm. pressure, (*a*) up to 80° C., (*b*) 80–130° C., and (*c*) residue. The presence in (*b*) of bromine derivatives of homologues higher than butylene indicates inefficient scrubbing. Fraction (*a*) comprises 80 to 90 per cent. of the whole, and contains all the propylene and ethylene, and most of the butylene dibromides, whilst (*c*) contains acetylene and butadiene tetrabromides and decomposition products of the distillation.

The olefines are regenerated from 2 grms. of the mixture in a wide (30 c.c.) tube which is previously two-thirds filled with freshly prepared zinc-copper couple just covered with 95 per cent. ethyl alcohol. The tube is fitted with a reflux condenser which leads to the stop-cock of a 300 c.c. gas-measuring tube filled with a saturated solution of calcium chloride, and also with a device to enable it to be filled with water. The reaction is started by gentle heat, and then checked, if necessary, by cooling the tube, the contents of which are finally boiled, and the gases completely driven over into the measuring tube by means of the water supply.

The measuring tube is well shaken to remove any alcohol carried over, and then contains ethylene, propylene, butylene, a trace of hydrogen and about 10 per cent. of air. The volume of gas is measured and the mixture analysed in a Bone and Wheeler apparatus with an additional tube-furnace for combustion over copper oxide (*cf.* J. G. King, *Fuel*, 1922, **1**, 103). The total percentage of olefines is then determined by absorption in fuming sulphuric acid or bromine water. A second portion of gas is burned completely over copper oxide at 700° C. and the carbon dioxide determined, whilst a third is extracted with successive 2 c.c. portions of 87 per cent. sulphuric acid (d. 1·800) for exactly 5 minutes. Since olefines higher than ethylene are absorbed completely in 20 minutes at ordinary temperatures, after which the absorption is due to ethylene alone, the removal of the latter during the first 20 minutes may be determined. The method may therefore be used in the presence of large quantities of ethylene, and is thus superior to the original procedure of Tropsch and Von Philippovich (Brennstoff-Chem., 1923, **4**, 147).

If the acetylene hydrocarbons are determined in the original gas-mixture by any of the usual direct methods (*e.g.* absorption), the butadiene tetrabromide in the residue (*c*) may be determined approximately. Its two forms are produced under the above conditions in equal quantities (m. pts. 118° C. and 36·5° C., b. pts. 265° C. and 180·5 °C., respectively). The former is therefore extracted with hot alcohol, filtered, cooled to 0° C., rapidly re-filtered and weighed. This weight is doubled. It may also be separated as a chlorine compound by the action of chlorine in carbon tetrachloride (Sorokin and Belikov, *Russ. J. Chem. Ind.*, 1925, **1**, 28).

The method gave satisfactory results for mixtures of known composition and for low and high temperature gases from vertical or horizontal retorts. In the last-named case, oil-scrubbing may be omitted, since no appreciable amounts of amylene or its homologues are present. The amounts of gas unaccounted for varied from 0·12 to 0·24 per cent.

The results obtained with low temperature gases (see Sinnatt, King and Linnell, *J. Soc. Chem. Ind.*, 1926, **45**, 385T) are summarised, and show, in general, an increase in quantity of all olefine hydrocarbons, with rise of temperature over the range 400 to 700° C. The retort system, rather than the coal, determines the amounts of hydrocarbons produced. High temperature carbonisation produces most ethylene, but least propylene and butylene, probably as a result of more severe cracking in the retorts.

J. G.

Concentrated Orange Juice.

STATUTORY RULES AND ORDERS, 1927, No. 360.*

MERCHANT SHIPPING.

THE MERCHANT SHIPPING (ANTI-SCORBUTICS) ORDER IN COUNCIL, 1927.

At the Court at Windsor Castle, the 22nd day of April, 1927.

PRESENT:

The King's Most Excellent Majesty in Council.

Whereas by Section 200 and the Fifth Schedule of the Merchant Shipping Act, 1894 (a) power is given to His Majesty by Order in Council to make provision as to the use of anti-scorbutics other than lime or lemon juice of such quality and composed of such materials, and packed and kept in such manner and served out at such times and in such quantities as His Majesty may direct:—

Now, therefore, His Majesty, by virtue of the powers in this behalf by the said Act, or of all other powers enabling Him in that behalf, is pleased, by and with the advice of His Privy Council, to give the following directions, as regards the provision of Concentrated Orange Juice as an anti-scorbutic on board every ship navigating between the United Kingdom and any place out of the same, except in the case of—

(*a*) Ships bound to European ports or ports in the Mediterranean Sea, and—

(*b*) Such ships or classes of ships bound to ports on the eastern coast of America, north of the thirty-fifth degree of north latitude, and to any islands or places in the Atlantic Ocean, north of the same limit as the Board of Trade may exempt:—

1. The concentrated orange juice shall contain not less than 70 per cent. of total soluble solids by weight. It shall be free from signs of alcoholic fermentation and contain no mould growths. It shall be so prepared and stored that there is no material loss of vitamin potency.

2. The concentrated orange juice shall be prepared from the fresh juice of sound oranges free from fermentation and moulds, with the addition of the best cane sugar only, and shall not contain any added colouring matter, chemical preservatives, nor metallic impurities.

The quantity of cane sugar to be added shall not exceed 20 per cent. by weight of the finished product.

The preparation or concentration of the juice shall be carried out *in vacuo*, at a temperature not exceeding 50° centigrade.

3. The concentrated orange juice shall be supplied in glass bottles containing not more than one pint. The bottles shall be filled to the neck, properly closed and secured and shall bear an adhesive label with the date of manufacture and the volume of the contents stated in fluid ounces, and no concentrated orange juice shall be used as an anti-scorbutic for the crew or passengers of any ship after two years from such date of manufacture. The concentrated orange juice both before and after being placed on board the vessel shall be kept in cold storage.

4. Every brand of concentrated orange juice shall be submitted to the Board of Trade for their approval, and no brand of concentrated orange juice shall be deemed fit and proper to be taken on board any ship for the use of the crew or passengers thereon unless it is shown by a certificate under the hand of an Inspector appointed by the Board of Trade to be proper for use on board ship.

5. The concentrated orange juice shall be served out to the crew so soon as they have been at sea for 10 days and, during the remainder of the voyage, during such times as they are in harbour and are there supplied with fresh provisions.

6. The concentrated orange juice shall be served out daily at the rate of one-half fluid ounce each per day to each member of the crew, and shall be mixed with six times its volume of water before being served out. The juice shall not be diluted before the day on which it is to be served and shall be served out in sufficient quantity to each mess or watch at the dinner hour so that it may be obtained by the crew in time to drink during their meal.

7. This Order may be cited as the Merchant Shipping (Anti-Scorbutics) Order in Council, 1927.

(Signed) COLIN SMITH.

(a) 57–8 V. c. 60.

* H.M. Stationery Office. Price 1d. net.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Occurrence of Acetaldehyde in Bartlett Pears and its relation to Pear Scald and Breakdown. C. P. Harley and D. F. Fisher. (*J. Agric. Res.*, 1927, **35**, 983–993.)—Acetaldehyde was found to be a normal metabolic product of pears and was the only aldehyde produced. Bartlett pears were removed from cold storage, some analysed for acetaldehyde at once, others after varying periods, and it was found that pears remaining free from scald at the end of 7 days showed a slight increase in acetaldehyde over those analysed at once, and that the concentration of acetaldehyde increased in proportion to scald severity. The same relation existed with breakdown. No pears containing more than 140 mgrms. of acetaldehyde in 100 grms. of fresh tissues, or 0·014 per cent., were found which did not show either scald, or breakdown, or both. Where scald and breakdown were present, acetaldehyde was found in greater quantities in the scalded skin tissues than in the apparently normal white underlying tissues, but most was present in the broken-down core areas. The disagreeable taste and odour preceding and accompanying scald and breakdown are apparently due to acetaldehyde, and the toxicity of acetaldehyde can be demonstrated by exposing pears to the gas or by injecting weak solutions into the tissues. D. G. H.

Presence of Glycuronic Acid in Wines from Mouldy or Rotten Vintages. D. Chouchak. (*Compt. rend.*, 1928, **186**, 520–522.)—Wines made from sound grapes grown in dry regions contain from 0 to 0·1 grm. of glycuronic acid per litre, whereas those made from mouldy or rotten grapes may contain as much as 1·25 grm. per litre. Corresponding differences are shown by the musts. To extract this acid, the wine is defecated with mercuric sulphate, and the acid then precipitated by means of ammoniacal basic lead acetate solution. The action of hydrogen sulphide on a suspension of the precipitate in water gives a liquid in which the glycuronic acid is easily detectable by its reaction with orcinol, phloroglucinol, naphthoresorcinol, or β-naphthol, or by the characteristic quinine salt. With musts, the lead precipitate must be carefully washed with ammonia water in order to remove the sugars. The acid may be determined approximately by Chiray's or Brule's method. Reduction of Fehling's solution by glycuronic acid falsifies the proportions of reducing sugars found. T. H. P.

Composition of Oil of Oats. K. Amberger and E. W. Hill. (*Z. Unters. Lebensm.*, 1927, **54**, 417–431.)—The literature of the subject is discussed. Oil of oats, which is distributed throughout the whole corn but occurs chiefly in the embryo, was extracted from the ground sample by boiling petroleum spirit, the

solvent removed in a current of carbon dioxide, and the residue re-extracted with freshly distilled acetone. After distillation in carbon dioxide, and re-extraction of the oil with petroleum spirit, filtration, and distillation, the final product was washed with water, and obtained as a yellow oil, with a fresh smell and a slightly irritant taste (yield 2·9 per cent.). Constants :—Sp. gr., 0·9210 ; acid value, 34·55 ; saponification value, 191·4; iodine value (Wijs), 105·3 ; Hehner value 93·9 ; Reichert-Meissl value 0·59, Polenske value 0·32 ; unsaponifiable matter, 1·4 per cent. ; free fatty acids, 17·2 per cent. ; neutral fat, 82·8 per cent. The fatty acids were separated into the solid (10·4 per cent.) and liquid constituents by the methods described elsewhere (Abstract, p. 236). Oxidation of the liquid acid with potassium permanganate yielded di- and tetrahydroxystearic acids from the insoluble and soluble portions, respectively, whilst bromination gave a liquid and solid tetrabromide (m. pt. 114° C.). The solid portion contained palmitic acid, and the percentage composition of the total fatty acids was thus calculated as palmitic acid 10·4, α-linolic acid, 17·2 ; β-linolic acid, 13·9 ; oleic acid, 58·5. This was confirmed from the iodine value and mean molecular weight. Linolenic acid was absent. Ten glycerides are thus possible, and the action of fuming nitric acid on a solution of the oil in acetone at 0° C. for 20 minutes, with fractional crystallisation from acetone of the resulting elaidin, yielded trielaidin and palmito-elaidins. Fractional crystallisation from chloroform, of the hardened oil, produced tristearin and palmito-distearin. Thence the glycerides from the original oil are deduced to be triolein and α-palmito-α-β-diolein (*cf.* ANALYST, 1921, **46**, 238).

J. G.

Fatty Acids of Cohune Nut Fat. T. P. Hilditch and N. L. Vidyarthi. (*J. Soc. Chem. Ind.*, 1928, **47**, 35T.).—About 1300 grms. of the kernels of the cohune palm (*Attalea cohune*, Morris), after being ground and extracted, yielded about 695 grms. of crude fat having the following constants :—Acid value, 0·9 ; saponification value, 251·0 ; iodine value, 9·8 ; unsaponifiables, 0·47 per cent. The mixed fatty acids had a titre of 21·2° C. Two hundred grms. of the fat were refined, and the composition of the fatty acids determined by preparing and examination of the methyl esters, with the following results :—Caproic acid, trace ; caprylic acid, 7·5 ; capric acid, 6·5 ; lauric acid, 46·5 ; myristic acid, 16 ; palmitic acid, 9·5 ; stearic acid, 3 ; oleic acid, 10 ; and linolic acid, 1 per cent. These figures are closely similar to those for the fatty acids of coconut oil and not very different (except in the content of caprylic, capric and oleic acids) from those of the fatty acids of palm-kernel oil.

R. F. I.

Detection of Yeast by the Yeast Gum Reaction in the Presence of the Products of Hydrolysis of Animal Proteins and of Animal Organs. H. Kraut. (*Z. Unters. Lebensm.*, 1927, **54**, 446-449.)—In Micko's yeast-gum reaction (*ibid.*, 1904, **8**, 225) for yeast in meat extracts, the liquid is treated with an excess of ammonia, filtered, and an excess of a mixture of 100 c.c. of a 13 per cent. solution of copper sulphate, 150 c.c. of ammonia, and 300 c.c. of a solution of 14 per cent. sodium hydroxide added to the filtrate. A solution of the resulting

precipitate in dilute hydrochloric acid is then precipitated with 5 times its volume of alcohol, when the yeast gum separates. It is necessary to test this precipitate by reprecipitating it from an acid solution by means of a 3 per cent. solution of sodium hydroxide, when the formation in the filtrate of a precipitate with Fehling's solution denotes the presence of yeast gum, 0·02 per cent. of which is detectable. A case is cited in which the omission of this stage has given misleading results.

J. G.

Determination of Lecithin-Phosphoric Acid in Egg Liqueurs. J. Grossfeld. (*Z. Unters. Lebensm.*, 1927, **54**, 450–462.)—The methods, particularly that of Juckenack (*ibid.*, 1900, **3**, 1 ; 1903, **6**, 827), for the determination of lecithin-phosphoric acid, are discussed and criticised in detail, and an improved procedure suggested for egg liqueurs. The sample (5 grms.) is warmed with phosphorus-free, water-free alcohol till the proteins are precipitated, and then transferred to an extraction thimble with 95 per cent. alcohol. The Besson extraction apparatus (*Chem. Ztg.*, 1915, **39**, 860), in which the thimble is supported in the neck of the flask below the reflux condenser, in the vapour of the boiling solvent, is preferred to that of Soxhlet. After 1 hour, the extract is carefully evaporated in a platinum dish with 1 c.c. of an 8 per cent. solution of alcoholic potassium hydroxide, and 1 c.c. of a 50 per cent. solution of magnesium acetate, acidified with acetic acid, dried for 2 hours at 120° C., and ignited at glowing heat. To the filtered and dilute solution (60 c.c.) of the ash in 5 c.c. of nitric acid (sp. gr. 1·2) are added 20 c.c. of 50 per cent. solution of ammonium nitrate, the liquid boiled, and 5 c.c. of a 10 per cent. solution of ammonium molybdate added. The precipitate is stirred, filtered off when cold in a weighed Gooch crucible, and washed with a solution of 50 grms. of ammonium nitrate and 40 c.c. of 25 per cent. nitric acid per litre. It is carefully heated till it becomes dark blue, cooled and weighed, when the factors 0·0395 and 0·790 give the lecithin-phosphoric acid in mgrms. and as a percentage, respectively. Alternatively, the precipitate may be washed into a flask with a minimum amount of water, boiled for 10 minutes with 50 c.c. of 0·1 *N* sodium hydroxide solution and titrated while hot with 0·1 *N* acid, with phenolphthalein as indicator. The factor 0·254 then gives the result in mgrms. The method is stated to be quick, simple, and to require a minimum amount of sample and of alcohol. J. G.

Biochemical.

Hygienic Evaluation of the Biological Properties of Milk. M. A. Dychno and O. M. Briskin. (*Z. Unters. Lebensm.*, 1927, **54**, 438–446.)—Comparative analyses of ordinary and boiled milk and milk heated below 100° C. (usually between 60° and 70° C. for 30 minutes) show that the heating process improves the biological properties of the milk, but impairs its general quality, probably as a result of physical changes. The sp. gr., solids-not-fat, and acidity are lowered, and the fat and solids raised. The heated milk is little changed, and the effect is greatest for the boiled milk. Rovat and Keller's peroxidase

test, in which 2 c.c. of milk and a drop of 2 per cent. hydrogen peroxide are shaken in the presence of 3 c.c. of a starch and potassium iodide solution, gives a blue, brownish and negative reaction for raw, heated and boiled milks, respectively. Catalase may be due to original excretion from the udder-glands, or to subsequent infection, and the test, which is useful only for sorting purposes and is least marked for the boiled or heated milks, is carried out in a Lobeck catalasometer. This consists of a graduated tube held inside a vertical stoppered cylinder, with the top end open to the air, whilst the other dips into a mixture of 7·5 c.c. of milk and 2·5 c.c. of 1 per cent. hydrogen peroxide. The level of liquid in the inner tube is noted after 2 hours at 37° C. It is shown that, since the reductase test (in which 20 c.c. of milk and 1 c.c. of methylene blue solution are maintained at 38 to 40° C. in a stoppered vessel), depends on the extent to which the milk has been heated and on its acidity (particularly that due to lactic acid), caution must be exercised in the application of the Barthel-Jensen scale (*Milch. Zentr.*, 1912, 14 ; *cf.* ANALYST, 1928, 107), in which the rates of decolorisation are correlated with the number of bacteria per c.c., and the milk thus placed in one of four classes. Fermentation of the milk for 4 hours at 40° C. enables the milk to be classified according to the nature of the curd. The most acid and least fermentable milks decolorise methylene blue most quickly. J. G.

Quantitative Determination of Dihydroxyacetone in Blood and Urine. W. S. McClellan. (*J. Biol. Chem.*, 1928, **76**, 481–486.)—The volumetric method of Campbell (*J. Biol. Chem.*, 1926, **67**, 59) for the determination of dihydroxyacetone in blood has been used and found satisfactory. Known amounts of dihydroxyacetone have been recovered by this method when mixed with whole blood. A method for the quantitative determination of dihydroxyacetone in urine is presented. Dihydroxyacetone is one of a few substances which will reduce acid molybdate solutions. Phosphates show this property; therefore, the phosphates in a specimen of urine are first precipitated and removed according to the method of Miller and Taylor (*J. Biol. Chem.*, 1914, **17**, 531), and then the dihydroxyacetone in the filtrate is determined by the volumetric method of Campbell. Experimental details are given. The accuracy of the method has been tested by the recovery of known amounts of dihydroxyacetone which had been added to the urine. Tables show the results. Of the amount added, 95 per cent. could be recovered. P. H. P.

Determination of Blood Sugar. II. S. R. Benedict. (*J. Biol. Chem.*, 1928, **76**, 457–470.)—The probable factors which contribute toward high figures for glucose in previously described methods for blood sugar determinations are discussed, and a new technique is presented for the determination, which appears to indicate very closely the true glucose content of the blood. The new reagent required is kept in the form of two solutions, A and B, and these are mixed in equal volumes within a day or two of being used. For each c.c. of the mixture one drop of a 4 per cent. solution of sodium bisulphite is added. For solution A 150 grms. of sodium nitrate, 25 grms. of anhydrous sodium carbonate and 10

grms. of alanine are dissolved in about 300 c.c. of warm distilled water ; 4 grms. of crystallised copper sulphate are dissolved separately in about 50 c.c. of water and added to the other solution without shaking, and the whole is cooled and diluted to 500 c.c. Solution B consists of 150 grms. of sodium nitrate and 25 grms. of Rochelle salt dissolved in distilled water and made up to 500 c.c. Phosphomolybdic acid is used as the basis for colour development, since the new copper reagent does not contain an excess of a salt of a weak organic acid. For human bloods the Folin-Wu technique yields figures for the blood sugar which average about 22 mgrms. per 100 c.c. of blood too high. A modified technique for the rapid fermentation of blood by means of yeast is presented, and a new criterion for testing the accuracy of blood sugar methods is suggested. Results of analyses of forty samples of human blood indicate that such blood does not contain appreciable quantities of any fermentable sugar other than glucose. P. H. P.

Vitamin Synthesis in Plants as Affected by Light Source. V. G. Heller. (*J. Biol. Chem.*, 1928, **76**, 499–511.)—This work was undertaken in an attempt to ascertain :—(1) Is vitamin synthesis or activation in plants dependent upon light rays, or is it a function of germination ? (2) If light proves to be the determining factor, are waves of certain lengths responsible for the change ? (3) Are the vitamins which are most susceptible to destruction, likewise most readily activated ? (4) From a commercial standpoint, would it be better to use vegetables grown in the open air or in the greenhouse ? (5) Would it be possible to increase the vitamin content by artificial illumination with some definite light wave ? The results show that the amount of vitamin *A* formed in seedlings seems to depend upon the light rather than upon changes taking place in the process of germination. Some increase is always found in etiolated seedlings. The quantity of vitamin *A* synthesised is dependent upon the intensity of illumination, length of exposure, and the relative amount of shorter wave-lengths, and follows closely the rate of growth of the plant. Vitamin *B* is not increased in the germination and early growth of the seedlings, but forms at a later period of development of the plant. Vitamin *C* is formed more rapidly than either of the other vitamins studied. There is evidence that germination alone, even in the dark, produces a considerable amount of vitamin *C*. It increases in light-grown seedlings to an extent that is greater than can be accounted for on the basis of the increased growth of the seedlings, and its production is accelerated like vitamin *A*, by increased intensities of light. It is shown that plants grown in the open sunlight under intense illumination should be slightly superior sources of vitamin. P. H. P.

Determination of Cholesterol in Small Amounts of Blood. S. M. Ling. (*J. Biol. Chem.*, 1928, **76**, 361–365.)—A modification of the method of Myers and Wardell (*J. Biol. Chem.*, 1918, **36**, 147) for the determination of cholesterol in small amounts of blood has been devised which is simpler and more accurate than the original method, and it has a further advantage in that there is a marked saving

of chloroform and acetic anhydride. The drying oven need not be used. Results show that the Myers and Wardell method gives low figures which are not constant, the difference from the theoretical being as high as 16 mgrms. and as low as 8 mgrms. per 100 c.c., whereas the new method gives the theoretical result when known amounts of cholesterol are determined. A diagram shows the extraction apparatus. It consists of a test-tube (25 × 150 mm.) the lower end of which is drawn out to form a bulb of about 9·5 c.c. capacity, with a 10 c.c. calibration mark on the neck of the bulb. A glass coil condenser, from which a glass cup (20 × 30 mm.) is suspended by fine copper wire, is inserted into the test-tube. Oxalated blood, plasma, or serum, (0·5 c.c.) is pipetted on to two pieces of Whatman fat-free filter paper, the papers are hung on a copper wire over an electric hot plate, and, when dry, folded and dropped into the glass cup which is then hung on the condenser. About 8 c.c. of pure dry chloroform are placed in the large tube, the condenser coil carrying the cup is inserted, and water inlet and outlet are attached. The bulb is then immersed in boiling water and extraction continued for 40 minutes from the time chloroform begins to drip from the condenser. The test-tube is then taken from the water bath and cooled under the tap. The condenser, together with the cup, is removed, and chloroform is added to the 10 c.c. mark. In a similar tube or 10 c.c. volumetric flask are placed 5 c.c. of standard chloroform solution of cholesterol (0·1 mgrm. per c.c.), and chloroform is added to the mark. To each tube are added 2 c.c. of pure acetic anhydride and 0·1 c.c. of concentrated sulphuric acid ; the tubes are corked and the contents mixed by inversion. The tubes are left in a dark place for 15 minutes and then compared in the colorimeter. When the standard solution is set at 20, and R is the reading of the unknown, $\frac{20}{R} \times 0{\cdot}5 \times \frac{100}{0{\cdot}5} = \frac{2000}{R}$ = mgrm. of cholesterol per 100 c.c. of blood, plasma, or serum. In case the unknown solution is diluted with 5 c.c. of chloroform the result is multiplied by 1·5. P. H. P.

Allophanates of Certain Sterols. U. Tange and E. V. McCollum. (*J. Biol. Chem.*, 1928, **76**, 445–456.)—The preparation and properties of the allophanic esters of sterols have been studied in order to discover the possible usefulness of this class of derivatives in the isolation of fat-soluble vitamins. The properties of allophanates of other alcohols have, in general, been favourable for identification ; therefore, should sterols react as do other alcohols with cyanic acid, with the formation of allophanic esters, it would be possible to work with easily formed derivatives which contain nitrogen, and have solubility properties which would be advantageous in the separation and purification of sterols. The allophanic acid esters of cholesterol, sitosterol, isocholesterol, dihydrocholesterol and coprosterol have been produced through the action of cyanic acid upon the sterol dissolved in benzene. The cyanic acid gas was prepared by heating cyanuric acid to dissociation. The property of dissolving in fat solvents is in great measure lost when the sterol is converted into the allophanate. The solubility of these sterols, with the exception of isocholesterol, in most solvents is so low that recrystallisation

can be effected with very little substance remaining in the mother liquor. It appears probable that the allophanic esters of the sterols may prove advantageous in the separation of mixtures of sterols, and perhaps in the isolation of fat-soluble vitamins. The solubilities in a number of ordinary solvents, and the ease of saponification with alcoholic potassium hydroxide and with sodium ethylate, have likewise been studied. P. H. P.

Existence of Two Active Factors in the Vitamin *B* Complex. II. W. D. Salmon, N. B. Guerrant and I. M. Hays. (*J. Biol. Chem.*, 1928, **76**, 487–497.)—Vitamin *B* is now known from recent research to be of a complex nature. There are at least two active substances in the complex ; one is the pellagra-preventing substance which has been termed the P-P factor, and the other the beriberi-preventing substance, or B-P factor. The following three points have been studied during an investigation of the vitamin *B* complex :—(1) the relative adsorption of the B-P and the P-P factors by fullers' earth ; (2) the further purification of the P-P fraction ; (3) the retardation of growth by an insufficiency of the B-P factor. Fullers' earth adsorbed both the B-P factor and the P-P factor of the vitamin *B* complex, but under certain conditions the former factor was more completely adsorbed than the latter. A combination of treatments with small amounts of fullers' earth, of fractionation with alcohol, and of heating, furnished a preparation from an extract of velvet bean leaves, that retained marked properties of preventing pellagra-like symptoms and also of accelerating growth when it was fortified with sufficient B-P factor. The treated preparation alone did not produce any growth and did not prolong the life of rats beyond the average for rats on the basal diet alone. A relation between the amount of B-P factor available and the rate of growth was found to exist. The concentration of the B-P factor in a sample of dried kudzu leaves was too low to permit of a complete utilisation of the P-P factor. A low concentration of either the B-P factor or the P-P factor may limit the rate of growth. It is definitely concluded that any future attempts to determine the vitamin *B* content of a product, which do not consider the complex nature of the vitamin, may be regarded as useless. P. H. P.

Toxicological and Forensic.

Relation of *d*-Gossypol to the Toxicity of Some Cottonseed Products. W. D. Gallup. (*Ind. Eng. Chem.*, 1928, **20**, 59–63.)—The toxicity of cottonseed meal may be due not only to the presence of ether-soluble gossypol as it is found in cotton seeds, but also to the presence of what appears to be a decomposition product formed during the heating of the seeds previous to expressing the oil, and given the name *d*-gossypol. The toxicity of the meal studied was not reduced by removal of the small amount of ether-soluble gossypol which it contained, nor was this form of gossypol present in sufficient amounts to produce toxic symptoms in animals when the extract was given in excessive quantities. When the meal is heated in the presence of moisture, as in autoclaving, it loses its toxic

properties although it may still contain a small amount of *d*-gossypol. Although cotton seeds are extremely toxic, they also may be rendered non-toxic by autoclaving in a wet condition. By heating the seeds for a short time in a dry condition the gossypol becomes partially converted into the insoluble form, and a separation may be made of the two by extraction with ether. The insoluble gossypol so produced is much more toxic than the insoluble form found in cottonseed meal or in seeds which have been subjected to steam heat. The determination of *d*-gossypol by the present chemical methods is not a safe criterion for estimating the toxicity of cottonseed products.

Water Analysis.

A Stable Scale-Standard for Determining Nitrites in Waters. R. Danet. (*J. Pharm. Chim.*, 1928, **120**, 113–114.)—Fuchsine S in feebly acid solution was found the most satisfactory colouring matter for concentrations of sodium nitrite between 0·5 and 2 mgrms. per litre. In 4 similar test tubes are placed 0·3, 0·6, 0·9 and 1·2 c.c. of a 0·1 grm. per litre solution of Fuchsine S, made up to 10 c.c. with water saturated with camphor to which 1 per cent. of acetic acid has been added. The tubes are closed with corks coated with paraffin wax. To make a test, 10 c.c. of the water and 1 c.c. each of the 2 solutions of sulphanilic acid and naphthylamine are mixed, left for 20–30 mins., and compared transversely with the standard tubes, which, under these conditions, correspond to 0·5, 1, 1·5 and 2 mgrms. of sodium nitrite per litre. D. G. H.

Agricultural.

Determination of Nitrogen in Soils in the Presence of Nitrates and Nitrites. C. Olsem. (*Compt. rend. Trav. Lab. Carlsberg*, 1927, **17**, No. 3.)—In a study of the capacity of soils for fixation of free atmospheric nitrogen account must be taken of nitrate nitrogen and nitrite nitrogen, and the usual Kjeldahl method cannot be used. The methods hitherto described give low results, and that of Mitscherlich, though accurate, is too long and difficult for routine work. A method adapted from that of Ulsch (*Z. anal. Chem.*, 1891, **30**, 175) is described. The soil is best sampled in the moist state, and the moisture determined on a 20 grm. portion, dried in a vacuum desiccator for 3 days at 85° C. The moist sample (5 grms.) is gently heated for 45 minutes in a Kjeldahl flask with 10 c.c. of water, 5 grms. of pure *ferrum redactum*, a few drops of octyl alcohol (as an anti-froth), and 30 c.c. of cold dilute sulphuric acid (1 : 2). A mixture of the sulphates of potassium (10 grms.), copper (1 grm.), and mercury (0·75 grm.) is then added with 30 c.c. of concentrated acid, and the usual procedure for the Kjeldahl determination followed. Nitrites are detectable by the action of the Griess reagent on a filtered aqueous extract of the soil, and should first be oxidised to nitrates by the action of 10 c.c. of a 5 per cent. solution of potassium permanganate with 30 c.c. of dilute sulphuric acid. The total nitrogen may then be determined

as described above. Distillation is most efficiently performed in Kjeldahl's original apparatus, and 10 c.c. of a saturated solution of copper sulphate are added as soon as boiling starts (Andersen and Jensen, *Z. anal. Chem.*, 1925, **67**, 427), and washed into the flask to prevent retention of ammonia by the acid solution which remains on the walls. Control analyses of the reagents are essential. Analyses of serum solutions and of humic peat and sandy acid soils, in the presence and absence of nitrates or nitrites, gave satisfactory results. J. G.

Efficacy of Mixtures of Natural and Solubilised Phosphates measured by a Bacteriological Method and the Effect on Higher Plants. G. Truffaut and N. Bezssonoff. (*Compt. rend.*, 1928, **186**, 522–524.)—The assimilability of the phosphates in a medium may be measured by the mass of the nitrogen-fixing bacteria formed in the medium. Determinations of the nitrogen fixed by these bacteria in nutrient media containing natural phosphate (with 34 per cent. of insoluble P_2O_5) alone or mixed in various proportions with neutral phosphate (with 24 per cent. of total P_2O_5, 5 per cent. soluble in water and ammonium citrate solution) show that the best results are obtained when the natural phosphate forms about one-third of the mixture. The results are confirmed by experiments on potatoes grown in poor soil, and show that addition of a small proportion of soluble P_2O_5 facilitates the assimilation of a natural phosphate. A high content in soluble phosphate in a compound fertiliser is thus unnecessary for obtaining maximum crops and, indeed, is sometimes harmful. T. H. P.

Organic Analysis.

Determination of Alcohol in the Presence of Acetone. J. M. Macoun. (*J. Soc. Chem. Ind.*, 1928, **47**, 43T.)—The method is based on the condensation in the presence of potassium hydroxide between benzaldehyde and ketones containing the $\cdot CO \cdot CH_2$ group. About 50 c.c. of the sample are weighed and transferred to a 500 c.c. Kjeldahl flask provided with a reflux condenser. Benzaldehyde is added in the proportion of 3 c.c. for every 1 c.c. of acetone supposed to be present. Solid potassium hydroxide (1 grm. to 10 c.c. of aqueous liquid) is added, and the mixture boiled gently for 30 minutes, when the flask is cooled and 100 c.c. of water are added. The solution is distilled into a separating funnel, in which water, alcohol, and the excess of benzaldehyde are collected till 100 c.c. or more have been distilled. Fifty c.c. of saturated brine are added, with enough solid sodium chloride to saturate the whole, after which the benzaldehyde is removed by shaking with petroleum spirit. The aqueous layer, together with the brine washings of the petroleum spirit layer, is distilled into a tared flask provided with a glass stopper. The distillate (not less than 100 c.c.) is weighed, and its specific gravity determined, from which data the percentage of alcohol is calculated. In those cases where the refractive index of the distillate is required, in order to determine whether methyl-, ethyl-, or iso-propyl alcohol is present, the above procedure is modified in order to remove the last traces of benzaldehyde from the distillate. The use of sodium chloride is omitted, and the petroleum spirit extract is treated with

water only. The aqueous solution and washings are treated with 3 grms. of silver nitrate and enough potassium hydroxide to render the solution alkaline. It is allowed to stand 5 minutes, and the distillation is then proceeded with as before. Tables are given showing the results of the method on mixtures containing known amounts of methyl and ethyl alcohol, respectively. Although they show losses in the alcohol, varying from 0·2 to 2·5 per cent., better results are obtained than by any other known method. R. F. I.

Comparative Investigation of Different Methods for the Separation of Solid and Liquid Fatty Acids. K. Amberger and E. W. Hiil. (*Z. Unters. Lebensm.*, 1927, **54**, 431–434.)—Of methods for the separation of solid and liquid fatty acids, the thallium salt method of Holde (ANALYST, 1924, **49**, 448) and that of Twitchell (*ibid.*, 1921, **46**, 466) were found to give excellent results. Roser's modification of Felser's ether and lead salt method (*Z. Unters. Nahr. Genussm.*, 1919, **38**, 241) was unsatisfactory. The methods were tested by determinations of the iodine values of the acids obtained from mixtures of known compositions and were applied in the case of oil of oats (*cf.* Abstract, p. 228). J. G.

Insect Fats. Fat from Aphidian Parasites of the Terebinth. J. Timon-David (*Compt. rend.*, 1928, **186**, 104–106.)—The plant lice congregate in the large galls of the terebinth and are easily collected. After a preliminary washing with ether they are ground with sand, extracted with ether, and, after distillation of the ether, about 20 per cent. of a pale yellow fat remains, readily forming spherical crystals melting at 35° C. The fat separated had a saponification value of 255 ; iodine value (Wijs), 1·5 ; Reichert-Meissl value, 9·9 ; and Polenske value, 24·7. The insoluble fatty acids had a m.pt. of 38·5° C., neutralisation value 256·9 and mean molecular weight of 218. It was composed of numerous glycerides of the acetic acid series ; soluble volatile acids (butyric group) ; insoluble volatile acids (caprylic group) ; lauric, and higher acids reaching, at least, to palmitic acid. D. G. H.

Yamagobo Oil. M. Ogura. (*J. Soc. Chem. Ind. Japan*, 1928, **31**, 15B.)—The seed of the yamagobo (*Phytolacca acinosa*, Roxb.) contained water, 23·1 ; oil, 10·6; and ash, 1·5 per cent. The extracted oil was of a light orange colour, and had the following characteristics :—Sp. gr., 154° C., 0·9148 ; n_D^{20}, 1·4713; saponification value 186·2 ; iodine value (Wijs), 104·6 ; and unsaponifiable matter, 1·73 per cent. The fatty acids (liquid in summer, partly solid in winter) had a neutralisation value of 198·5 and iodine value of 105·7, and yielded 92·3 per cent. of liquid fatty acids by the lead salt and ether method. On bromination 35·9 per cent. of solid bromide was obtained, soluble in ether, insoluble in petroleum spirit, forming white crystals of m.pt. 113–114° C., and thus corresponding to tetrabromostearic acid. D. G. H.

Inorganic Analysis.

Alkali Earth Metals in Saccharate Solutions and their Use in Alkalinetry. A. C. Shead. (*J. Amer. Chem. Soc.*, 1928, **50**, 415–416.)—As a material for preparing, with a single weighing, a standard alkali solution of definite normality, carbonate-free and ready for use without standardisation by means of an auxiliary substance, the author recommends metallic calcium or barium, which may be converted into oxide or hydroxide and dissolved in 30 per cent. sucrose solution. Calcium which is uniform and of a definite high degree of purity is now obtainable, and the chief impurity is magnesium, which, like its oxide, is almost insoluble in the sucrose solution. The film of tarnish which forms on clean metallic calcium in moist air appears slowly in ordinary air, and is readily removed from the warm metal by sandpaper. Rod, one-eighth of an inch in thickness, would be convenient to use. T. H. P.

Determination of Bismuth as Oxyiodide. R. Strebinger and W. Eins. (*Z. anal. Chem.*, 1927, **72**, 417–429.)—When a weakly acid bismuth solution is treated with potassium iodide, a black precipitate of BiI_3 is formed, soluble in excess of precipitant with yellow colour ($KBiI_4$); dilution determines partial re-precipitation of BiI_3. If the yellow solution is heated, the black precipitate changes into crystalline copper-red BiOI, and the liquid becomes colourless at the point of quantitative precipitation:—$KBiI_4 + H_2O = BiOI + KI + 2HI$. The quantitative precipitation requires the following conditions:—The weakly acid nitrate solution (less than 20 c.c.) is treated with solid potassium iodide until the liquid above the black precipitate shows a weak but distinct yellow colour. The solution is then diluted to 80 or 100 c.c. and heated to the boiling point, when the change to the oxyiodide occurs. Dilution with water and heating are continued until the yellow coloration has almost or completely disappeared. A few drops of methyl orange are added, and the hydrolysis completed by a solution of sodium acetate (25 grms. per litre) added, drop by drop, to neutrality. The precipitate is collected in a porous glass crucible, washed with warm water, dried at 105° to 110° C., and weighed. Bi factor, 0·5939. The results are accurate. If the bismuth solution is too acid, it is best to evaporate the excess of acid previous to precipitation. A separation from lead can be effected by this method; double precipitation is required unless the quantity of lead is very small. W. R. S.

Separation of Arsenic from Antimony. L. W. McCay. (*J. Amer. Chem. Soc.*, 1928, **50**, 368–373.)—From a hydrofluoric acid solution of arsenic and antimonic acids, made slightly but distinctly alkaline with ammonia, silver nitrate in excess precipitates the arsenic completely as silver arsenate, quite free from antimony. The precipitate is washed with water containing 5 grms. of ammonium nitrate and 0·25 grm. of silver nitrate per litre, and then with a little alcohol, the alcoholic washings being rejected, as they contain no antimony. The arsenic is then determined by dissolving the precipitate in nitric acid and determining

the silver present by the Volhard method. The filtrate from the silver arsenate, freed from silver by means of the minimum amount of hydrochloric acid, is evaporated with concentrated sulphuric acid in a quartz dish until it fumes strongly, and then gently boiled for 20 minutes with a piece of sulphur weighing about 2 grms. to reduce the antimony to the trivalent state. The cold liquid is treated with 100 c.c. of dilute hydrochloric acid (20 c.c. of concentrated acid plus 80 c.c. of water), freed from sulphur by filtration, and made up to 200 c.c.; the antimonious acid is determined by titration with either 0·1 *N* potassium permanganate or 0·1 *N* potassium bromate.

For the complete oxidation of antimony to the pentavalent condition, potassium persulphate appears to be the best reagent. Dilute solutions of ammonium fluoride, rendered faintly alkaline, have little action on pyrex glass vessels, which may therefore be used for the separation.

A mixture of arsenic, antimony and tin sulphides is best dissolved in a small platinum or quartz dish in boiling concentrated sulphuric acid containing a piece of sulphur. After dilution of the liquid, addition of hydrofluoric acid, and filtration into a large platinum dish, the arsenic and antimony are reprecipitated as sulphides, which are treated with fuming nitric acid. Most of the acid is expelled by evaporation, and the liquid heated with 2 c.c. of hydrofluoric acid and a little water until clear, diluted to 100 c.c., and treated with potassium persulphate. The separation is then effected as described above. T. H. P.

Detection of Traces of Beryllium and Colorimetric Determination of this Element. I. M. Kolthoff. (*J. Amer. Chem. Soc.*, 1928, **50**, 393–395.)—In faintly alkaline solution a 0·1 per cent. solution of turmeric in alcohol is adsorbed by beryllium hydroxide, with formation of an orange-red colour. To 10 c.c. of a beryllium solution are added 1 drop only of the indicator, 0·5 c.c. of 4 *N* ammonium chloride and 6 to 8 drops of 4 *N* ammonia. A solution containing 0·05 to 0·001 grm. of beryllium per litre gives a flocculent precipitate with a red (orange-red) colour. The sensitiveness may be increased to 0·00005 grm. by using a blank for comparison. The reaction lends itself to the determination of beryllium in concentrations between 1 and 0·05 mgrm. per litre. Potassium, sodium, lithium, calcium, and barium do not interfere with the reaction, but magnesium lowers its sensitiveness somewhat. Aluminium forms a coloured lake, but may be removed sufficiently by preliminary treatment of the slightly acid solution with excess of sodium fluoride, followed, after an hour, by filtration. Ferric salts may be removed similarly or by precipitation at room temperature by excess of sodium hydroxide. In strongly alkaline solutions beryllium does not react with the turmeric, as the beryllium hydroxide redissolves, and under these conditions magnesium gives a colour reaction which is distinct but less sensitive than that with titan yellow. T. H. P.

Detection and Colorimetric Determination of Aluminium. I. M. Kolthof. (*Chem. Weekblad*, 1927, **24**, 447–8.)—A delicate reagent for aluminium is 1, 2, 5, 8, oxyanthraquinone. Ten c.c. of the solution under examination are

brought to a P_H value of about 5·6 and treated with 0·2 to 0·3 c.c. of a 0·1 per cent. solution of the above indicator. On warming the solution to 50° C., or allowing it to stand for half-an-hour, the formation of a violet-coloured lake indicates the presence of aluminium. The test may be made quantitative at concentrations between 0·02 and 0·5 mgm. of aluminium per litre. Iron, copper, tin, antimony and bismuth should be absent. Copper is rendered inert by treatment with thiosulphate ; tin, antimony and bismuth by the presence of Rochelle salt or sodium tartrate. R. F. I.

Titan Yellow as a Reagent for Magnesium in the Micro-Chemistry of Plants. H. Eilers. (*Chem. Weekblad*, 1927, **24**, 448–450.)—The use of titan yellow as a reagent for magnesium (*Chem. Weekblad*, 1927, **24**, 254) in plant microchemistry has been investigated. It has been shown that the reaction is not quite specific for the detection of magnesium in plant tissue, as the dyestuff is also adsorbed by pure cellulose, to give a red colour. For the detection of magnesium in cell-walls it is therefore necessary to make a control test by extracting the tissue with dilute acid and washing it with water to remove magnesium. The section is boiled with a drop of *N* sodium hydroxide solution and an equal part of 0·1 per cent. titan yellow is added. Oils and resins have a disturbing effect and should be extracted before treating with caustic soda.

Pyrophosphate Method for the Determination of Magnesium and Phosphoric Anhydride. A. W. Epperson. (*J. Amer. Chem. Soc.*, 1928, **50**, 321–333.)—The following procedures are recommended as being the most favourable for the determination of : (1) *Magnesium.*—To the neutral or weakly acid solution of magnesium chloride containing not more than 0·1 grm. of MgO, 5 c.c. of concentrated hydrochloric acid and methyl red indicator are added, and, after dilution of the liquid to 150 c.c., 10 c.c. or a five- to ten-fold excess of saturated diammonium hydrogen phosphate solution. The solution is then neutralised by gradual addition of ammonia solution (sp. gr. 0·90), stirred for about five minutes, or until the precipitate is well formed, treated with 5 c.c. of the ammonia solution, and stirred for ten minutes. After being left for at least four hours, preferably overnight, the precipitate is filtered off and washed with water containing from 3 to 5 per cent. of the ammonia solution. The precipitate is dissolved from the filter by warm 1 : 9 hydrochloric acid (sp. gr. 1·02), methyl red and about 1 c.c. of diammonium hydrogen phosphate solution being added, and the precipitation completed, as before, in a volume of 100 to 150 c.c. ; digestion for 4 hours suffices in this case. The precipitate and the wet filter paper, in a platinum crucible, are charred without flaming, then ignited at about 500° C., with the lid open enough for circulation of air until the residue is white, and finally at about 1000° C. to constant weight.

The errors due to a very large excess of precipitant, the presence of potassium chloride, and addition of the precipitant to an ammoniacal solution, are not entirely remedied by reprecipitation ; the first and third of these errors are, however.

avoidable, and that due to excess of potassium chloride is usually removed by a third precipitation. The use of microcosmic salt as precipitant gives high results for a single precipitation, but correct ones when two precipitations are carried out.

(2) *Phosphoric anhydride.*—To the neutral or weakly acid phosphate solution, containing not more than 0·1 grm. of P_2O_5, are added 5 c.c. of concentrated hydrochloric acid and methyl red, the liquid being diluted to 150 c.c. and treated with 10 c.c. or a five- or ten-fold excess of the precipitant, preferably magnesia mixture without ammonia (50 grms. of $MgCl_2$, $6H_2O$, 100 grms. of NH_4Cl, and 5 c.c. of concentrated hydrochloric acid in 1 litre of water) ; the solution, which should then be acid, is neutralised with ammonia solution (sp. gr. 0·90), stirred for about five minutes, or until the precipitate is well formed, treated with 5 c.c. of the ammonia solution, and stirred again for ten minutes. After digestion for at least four hours or, best, overnight, the precipitate is filtered off, washed with water containing from 3 to 5 per cent. of ammonia solution (sp. gr. 0·90), and dissolved from the paper with warm (1 : 9) hydrochloric acid (sp. gr. 1·02). The solution is treated with 1 c.c. of the magnesia mixture in presence of methyl red, and the precipitation completed as before, digestion for 4 hours sufficing. The ignition is effected as described above.

Of the errors not remediable by re-precipitation, those caused by addition of the precipitant to an ammoniacal or a hot solution are avoided by the procedure given, and that due to presence of ammonium sulphate is negligible in all but the most accurate work ; somewhat low and irregular results are, however, obtained in presence of citric acid.

As regards methods suggested for treating the precipitate in order to obtain pure magnesium pyrophosphate : solution in nitric acid, followed by evaporation to dryness and ignition, is without apparent effect, as also is moistening of the ignited pyrophosphate with one or two drops of nitric acid and re-ignition; solution of the ignited precipitate in nitric acid and evaporation of the solution is found impossible. No advantage attends addition of ammonium nitrate to the ammonia solution used for washing the precipitated magnesium ammonium phosphate.

T. H. P.

Separation of Tungsten from Silica and Tin. J. Ciochina. (*Z. anal. Chem.*, 1927, **72**, 429–434.)—Tungstic acid is soluble in hot sodium tungstate solution, yielding metatungstate: $Na_2WO_4 + 3WO_3 = Na_2W_4O_{13}$. The author proposes utilising the reaction to separate the mixed precipitate of silicic and tungstic acid obtained in ore or alloy analysis. The precipitate is washed free from acid with distilled water ; the tube of the funnel is closed, and a hot 20 to 30 per cent. solution of sodium tungstate poured on the filter. The tungstic acid dissolves readily. The filter is thoroughly washed, and the residual silica ignited and weighed. Digestion of silicic acid with a boiling solution of sodium tungstate is inadmissible, as silica dissolves. Metastannic acid, on the other hand, does not react at all with sodium tungstate, and can be separated from tungstic acid by the procedure described.

W. R. S.

Iodimetric Selenium Determination. R. Berg and M. Teitelbaum. (*Chem. Zeit.*, 1928, **52**, 142.)—In the reduction of selenious acid by iodide, the absorption of iodine by the precipitated selenium causes low results, and the coloured precipitate interferes with the recognition of the end-point. The following modification avoids these drawbacks by solution of the selenium and iodine:—The selenite solution is diluted in a glass-stoppered flask to 100 c.c. and acidified with 10 c.c. of 25 per cent. hydrochloric acid. Carbon disulphide (20 c.c.) is added, then 1·5 times the required quantity of 0·2 *N* potassium iodide solution in a thin stream while the flask is given a rotatory motion. After one minute's vigorous shaking, the iodine is measured with thiosulphate, the liquid being well shaken after each decolorisation of the aqueous layer, and finally titrated after addition of starch. The end-point is sharp. Accurate results are given. 1 c.c. 0·1 *N* thiosulphate = 0·00198 grm. Se. W. R. S.

Apparatus.

Pipette for Micro-Analysis. A. T. Shohl. (*J. Amer. Chem. Soc.*, 1928, **50**, 417.)—This consists of a modification of Van Slyke's pipette, with a glass Luer adapter sealed to the bottom end, which is ground and fits snugly into a small gauge (18–23) hypodermic needle, which is cut off horizontally and ground on a stone. Drops of volume about 0·00015 c.c. can be removed from such a pipette and the amount delivered is accurate to within 1 or 2 parts in 10,000. For corrosive liquids a platinum needle may be used. T. H. P.

References to Scientific Articles not Abstracted.

NOTE ON THE DETERIORATION OF BOOK-BINDING LEATHER. By F. P. VEITCH, R. W. FREY and L. R. LEINBACH. *J. Amer. Leather Chem. Assoc.*, 1928, **23**, 9.

Analyses of pieces of leather from old bindings (A.D. 1480—1880) from Windsor Castle—Highest acidity found, 0·53 per cent.—Relatively long life and good condition of binding attributed to absence of appreciable acidity and predominance of a pyrogallol tanning in bindings (other than vellum).

CARBON DIOXIDE THERMO-SALINE SPRINGS. By A. SCHOTT. *Lancet*, 1928, **214**, 546 (March 17).

A study of sub-cutaneous gas tensions by the technique of Campbell, who showed that after the injection of nitrogen or air into sub-cutaneous tissue spaces an equilibrium of tension is established between the injected gas and the tissues.

Errata:—The reference to the atomic weight of titanium on p. 159 should read 47·90, as in the table on p. 160.

P. 187, line 5. *For* "*Liq.* Bismuthi, B.P. j" *read* "*Liq.* Bismuthi, B.P. ℥j."

Reviews.

THEORETICAL AND EXPERIMENTAL PHYSICAL CHEMISTRY. By J. C. CROCKER, D.Sc., and F. MATTHEWS, Ph.D. Pp. vii + 581. London: Churchill. 1927. Price 21s.

Physical Chemistry is rapidly extending its boundaries, and its importance in both analytical and industrial processes is generally becoming recognised. This expansion, however, is not without effect upon elementary text-books, which on revision inevitably become enlarged. The student thus finds it increasingly difficult to isolate and to concentrate on just those parts which are fundamental, and which will enable him to make intelligent reference to the original literature. The book under review has been written, in the words of the authors, "to include, in one volume, what is usually derived by the student from several sources." To the reviewer it appears that the authors have secured a certain measure of success, though to hope to make the student dependent upon one source for his information is not altogether good, so far as training is concerned.

Into a space of 564 pages there has been compressed: change of state, spectrometry (including Tesla luminescence) the correlation of physical properties with chemical constitution, thermodynamics, including the Nernst theorem, the phase rule, electrochemistry, including conductivity in both aqueous and non-aqueous solutions, E.M.F., and electrometric titration, velocity of reaction and photochemistry, and the theory of the atom with a short account of the quantum theory. This is, indeed, an achievement, though the reviewer feels that it has sometimes been done at the expense of (*a*) clearness, (*b*) the omission of some fundamental matter, and (*c*) giving an adequacy of references to the recent monographs and literature on the subject. The book savours very much of "cram books" written expressly for examinations, rather than to assist in providing a thorough chemical training, which should involve a certain amount of reading the subject in the original memoirs. Thus the description of normal electrode potentials is so cursory that it is extremely improbable that any satisfactory idea of what they really are can be obtained; so also is that of the electrodes which are chosen as being of arbitrary zero-potential.

The constitution of the normal hydrogen electrode, given on page 391, namely, Pt_{H2}:N.HCl, is incorrect; so also is the impression, no doubt given, through unnecessary brevity, that a normal electrode potential refers to that of a metal immersed in a normal solution of one of its salts, *e.g.* Zn:N.$ZnCl_2$. It should certainly have been stated that the concentration refers to one gram-mol. of zinc-ions per litre, and that normality in electrometric work refers to gram-mols., and not to gram-equivalents, per litre. It is somewhat disappointing to find that the values of normal electrode potentials should have been taken

from a paper published in 1900, since when many of them have been revised and more satisfactory data obtained, *e.g.* that of silver is given as −0·771, instead of the recent value −0·800.

Although the book contains some account of certain highly specialised branches of physical chemistry, such as Tesla luminescence spectra, the parachor, and the magnetic rotation of polarised light, it seems unfortunate that the treatment of the phase rule should have been curtailed so as to be contained in the "allotted space." Many industrial processes involving crystallisation from aqueous solutions have been improved considerably by means of a knowledge of the phase rule systems involved. Of the nine pages devoted to three component systems, the only examples of ternary systems deal with fusion and partial miscibility of liquids. Modern chemical literature is replete with ternary systems involving crystallisation of double salts, but not a word is devoted to them. Moreover, ternary systems are usually plotted on an equilateral triangular diagram, though sometimes according to either the van't Hoff or Jänecke schemes of rectangular co-ordinates. Although no mention is made of the latter methods of representation, the authors introduce and adopt the recent method of R. Philip (1923), which has, as yet, obtained hardly any application. Some attention should have been paid to the Schreinemaker method of finding the composition of solid phases by means of "tie-lines," on account of their importance to the student in studying the much-neglected subject of inorganic chemistry. It is disappointing that the authors do not recommend either of the standard works on the phase rule, namely those of Clibbens and of Rooseboom.

Some confusion is likely to arise over the disregard, sometimes inconsistent, of the conventions generally adopted in chemistry. The mass law expression giving the value of the equilibrium constant is usually given in the book with the concentrations of products of a reaction in the denominator and not in the numerator, though in the case of thermal and electrolytic dissociation the reverse procedure is adopted. The adoption of the first procedure has involved the change in the signs in the van't Hoff Isotherm, and also when it is applied to oxidation-reduction reactions. The algebraic signs, though in accord with this arrangement, given to electrode potentials, do not accord with the more general practice.

The references to original papers are often to the older literature; for instance, the last reference on the oxygen electrode for titration purposes is to a 1918 paper, though this subject has been more exhaustively investigated by several workers more recently. The diagram of the capilliary electrometer (p. 387) is that of a form which is obsolescent; a better form is now used.

In conclusion, difficulties of the nature indicated above were almost bound to arise in the preparation of such a book, and it must be conceded that the work does provide a valuable and compendious outline of the subject, and one which will be of considerable help to the student in his effort to acquire a wide knowledge, though, in this respect, he will probably find this book not to be altogether sufficient.

HUBERT T. S. BRITTON.

FIRST PRINCIPLES OF CHEMISTRY. By F. W. DOOTSON and A. J. BERRY. Pp. viii + 339 with 43 figures. Cambridge: The University Press. Price 6s.

Since the days of our youth, the general quality of text-books written for the use of the elementary student has steadily improved, and this volume provides evidence that the beginner is well catered for at the present time. The book is designed to cover the syllabus of the School Certificate Examinations of all the English Universities, and will thus provide adequate instruction for the first year of study in many curricula.

Whilst, in general, following on similar lines to those of earlier text-books, several notable improvements have been introduced. Thus the periodic law and the obsolete laws of multiple and reciprocal proportions are omitted, and brief sections on hydrogen ion concentration, colloids and isotopes have been introduced. Further, the study of the metals is greatly facilitated by dealing with similar compounds of the different metals together, thus bringing vividly before the students' notice the relationship between these substances, whilst considerable interest is added to the text by frequent references to industrial and domestic applications of the reactions under consideration. At first sight it appears somewhat unusual to find that paragraphs and sections usually denoted by the first few words being printed in heavier type are without this adornment, but the omission is not detrimental in any way to the use of the book. It is evident that meticulous care has been expended in the production of this volume, for an exhaustive examination has failed to detect any error in the subject-matter, the equations, or the type, or in the titles and page numbers given in the two indexes. Although so essential in a handbook for students, this degree of perfection is rarely attained. The authors have provided a volume eminently adapted for its intended purpose, and it may be confidently recommended to all elementary students and teachers, who will have cause to be grateful to everyone concerned in the production of this adjunct to the work of the lecture room and the laboratory.

T. J. WARD.

THE PHASE RULE AND ITS APPLICATIONS. By ALEXANDER FINDLAY, M.A., D.Sc., F.I.C. 6th edition. Revised and largely re-written. Pp. xv + 326, with 165 figures. London: Longmans, Green & Co. 1927. Price 10s. 6d. net.

For twenty-three years "Findlay" has been the standard English text-book on the Phase Rule. In this sixth edition there is no great change in the actual contents of the book, but the whole has been rearranged on a much more systematic basis, and the titles of chapters and sections altered in accordance with the same logical system. This is a distinct gain. The treatment has also been made generally more quantitative by more frequent application of the Clausius-Clapeyron equation to particular instances.

As regards fresh matter, there is a valuable new chapter (chapter XVI) on "Practical Applications of Equilibrium Diagrams." It may, however, seem a pity that exigencies of space should have made it necessary to cut out the six pages (old chapter XVII) on the iron—carbon monoxide—carbon dioxide system.

References to the literature are very copious. H. R. AMBLER.