

THE ANALYST

The Journal of The Society of Public Analysts and other Analytical Chemists

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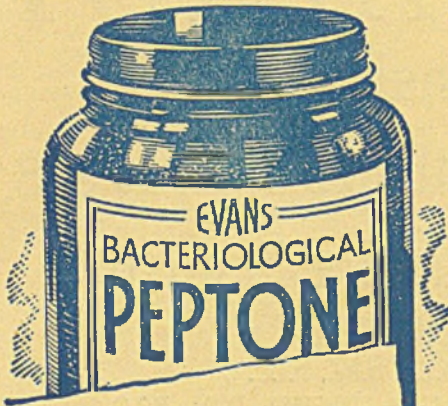
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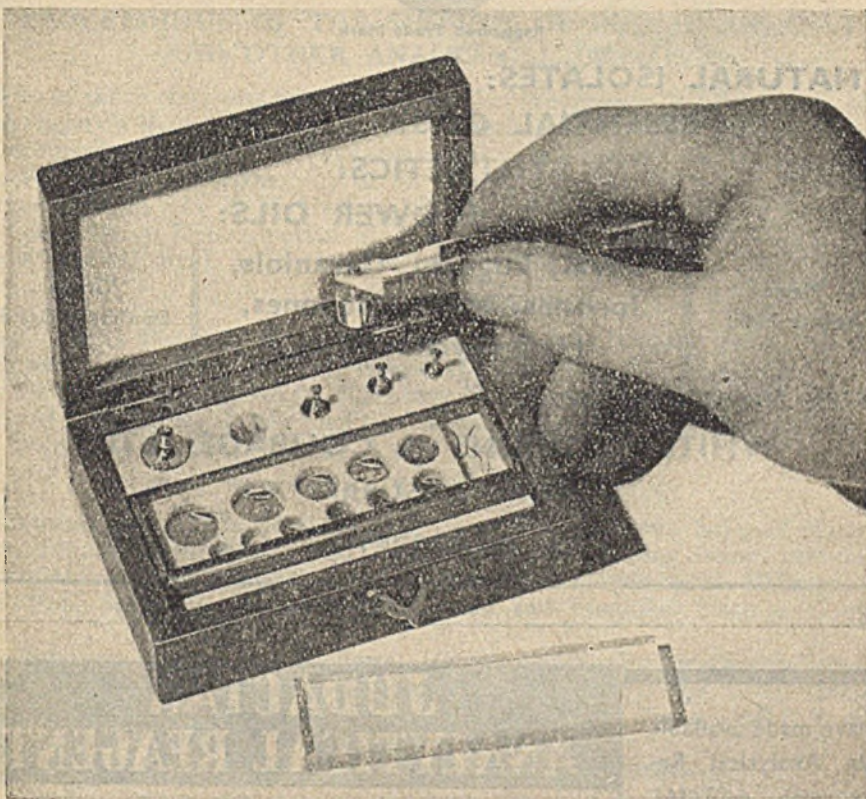
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THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS

A MEETING of the Society was held at 3.30 p.m. on Wednesday, February 2nd, 1944, at the Chemical Society's Rooms, Burlington House, London, W.1. The President, Mr. S. Ernest Melling, occupied the chair. The following papers were presented and discussed: "A Review of Micro-Volumetric Apparatus," by G. H. Wyatt, B.Sc., Ph.D., F.I.C.; "The Analysis of Commercial Lecithin," by H. H. Hutt, A.I.C., and H. Weatherall, F.I.C.; "A Micro-method for the Quantitative Determination of Tannin," by M. Nierenstein, D.Sc., Ph.D.; "An Amperometric Cell," by J. T. Stock, B.Sc., A.I.C., and M. A. Fill.

NEW MEMBERS

James Alfred Beardall, F.I.C., Ph.C.*; Henry John Callow, B.Sc. (Lond.), A.I.C.; Leslie Hilton Easson, B.Sc., Ph.D.*; Gerald Ingram; John Mather Jones, A.I.C.; Herbert Sweeney, A.I.C.*; Gerald Bowling Thackray, B.Sc. (Lond.); Reginald Harold Thompson, B.Sc. (Lond.), A.R.C.S., D.I.C., A.I.C.; William Charles Wake, B.Sc. (Lond.), A.I.C.; Thomas Walter White, B.Sc. (Lond.), A.I.C.

NORTH OF ENGLAND SECTION

THE Nineteenth Annual General Meeting of the Section was held at Manchester on Saturday, January 29th, 1944. The Chairman (Mr. W. Gordon Carey) presided. There were thirty-six members present, including the President (Mr. S. E. Melling).

The Hon. Secretary presented the Report and Financial Statement, which were adopted.

Appointments were made as follows:—*Chairman*, W. Gordon Carey; *Vice-Chairman*, E. Gabriel Jones; *Committee*, A. A. D. Comrie, Dr. W. F. Elvidge, C. H. Manley, H. M. Mason, J. R. Stubbs, R. W. Sutton; *Hon. Auditors*, U. A. Coates and J. R. Walmsley; *Hon. Secretary and Treasurer*, Arnold Lees.

Several subjects of scientific interest were afterwards discussed.

Obituary

JOHN GOLDING

JOHN GOLDING, D.S.O., F.I.C., whose death in his 73rd year occurred suddenly in Reading on September 22nd, 1943, had been a member of the Society since 1902, and had served on the Council in 1908-9 and 1927-8. For the major part of his long and active life he had been engaged in research in agricultural analysis of very varying types, from vitamin assay to cryoscopy, and from soil analysis to determination of milk chlorides. One of his last pieces of published work was on an analytical method of greatest importance to farmers, namely, "a new simplified method for the determination of butter-fat in dairy products." He was never satisfied with any method, however widely used, which he had not himself tested and found sound at every stage, and in fifty years of laboratory work these high standards never flagged.

Golding was born at Plaxtol, near Sevenoaks, of a well-known Kentish family that had owned and farmed land in that neighbourhood for several generations. He had, however, made up his mind to be a chemist, and after leaving Queen Elizabeth's Grammar School, Sevenoaks, he went to London to pursue his professional training. It was perhaps natural that, with his family and background, he should lean towards the agricultural applications of his chosen field, and his first paid post was that of assistant, in the laboratories of the Royal Agricultural Society, to Dr. J. A. Voelcker. He remained there for a relatively

* Through the North of England Section.

short time, being appointed lecturer in agricultural chemistry at University College, Nottingham, at the early age of 23. Here he met Florence Mundella, who was to be his partner through a long and happy life. His next call was to the Midland Agricultural College at Kegworth, where at 29 he became head of the Chemistry Department.

The importance of a sound knowledge of chemistry for every future farmer was being increasingly realised, and Golding's responsibilities steadily grew. Though his teaching duties became heavier, he kept in touch with the advancing fringe of his science by spending his long vacations in different European research laboratories, in Denmark, Sweden and Germany. Some of the contacts he thus made, particularly the Danish ones, were maintained until the outbreak of the present war.

The Development Commissioners and the Ministry (then the Board) of Agriculture established in 1912 the National Institute for Research in Dairying at Reading, and Golding's was the first appointment made to the research staff. The work of the young institute had, however, hardly got under way when the war broke out in 1914. Golding volunteered for military service and went overseas in 1915. He served with distinction in France, was awarded the D.S.O. and also a French decoration, and retired from the Army in 1919 with the rank of Captain. He resumed his work as Head of the small Chemistry Department at the Institute with renewed energy, engaging himself particularly in work on the nutritive value of milk and its vitamin content. In the next 18 years he published many original papers, some in collaboration with workers either at Shinfield or in other laboratories. These publications deal with a wide variety of topics, including the bromocresol purple test for mastitis, fat-soluble and other vitamins in cow's milk, methods of drying whey, utilisation and nutritive value of whey, whey silage, the relative value of raw and heated milk in nutrition, the freezing point of milk, the vitamin requirements of pigs, and the chemical composition and analysis of milk.

Throughout these years Golding's early enthusiasm for the development of accurate analytical methods did not diminish, and he spent much time in working out quicker and more precise methods for the determination, in milk, of those constituents whose values are most needed in practical dairying. In his laboratory was carried out much of the fundamental work on which are founded the B.S.I. standard methods for the Gerber and Babcock tests and for the sampling of dairy products, and specifications for various types of apparatus for use in dairying and the dairy laboratory.

His department steadily grew in size. An analytical section under W. L. Davies (later appointed Director of Dairy Research to the Government of India) and a nutrition section under S. K. Kon (now Head of the Physiology and Biochemistry Department of the Institute) were added. Several men who are now in senior dairy and industrial research posts both at home and abroad spent longer or shorter periods as post-graduate research students either in Golding's own laboratory or in one or other of the two sections of his department, and his influence on the dissemination of accurate analytical methods in the dairy industry was thereby increased.

Still a fit and active man, Golding retired late in 1937, having served one year beyond the normal age of retirement. He had intended to take a long holiday abroad, and then to return to his old home in Kent, where he was fitting up a small laboratory. The first part of this programme was duly carried out, and he spent with Mrs. Golding the greater part of a year touring in South Africa. The second part was interfered with after a few months by the outbreak of war, and his keen sense of duty made him accept the call to return to Reading, this time as Lecturer in Agricultural Chemistry in the University, filling one of the gaps left by the departure of younger men to the Forces. He was still engaged in this work at the time of his death.

John Golding will long be remembered by his many friends and colleagues not only for his work as a dairy chemist but also for his unflinching courtesy and kindness, his cheerfulness in fair or foul weather, his perseverance in face of difficulties and, with Mrs. Golding, for his hospitality. He died as he would have wished, after a full life, but before time had sapped his energy too severely.

H. D. KAY

Nutrition of the Public and Food Legislation

THIS was the general subject of the Joint Meeting of the Society with the Food Group of the Society of Chemical Industry, on December 1st, 1943, at which the following three papers were read and discussed.

Natural Nutrients of Fresh and Manufactured Foods

BY PROFESSOR (NOW SIR) J. C. DRUMMOND, D.Sc., F.I.C.

(*Scientific Adviser to the Ministry of Food*)

As a background for discussion it will be helpful to compare the nutritional position in this country to-day with that of the years before the war. The studies of Lloyd and Sir John Orr revealed that partition of the food supplies on the basis of ability to buy led to disturbing disparities of consumption between the rich and the poor. It was estimated in 1935-6 that at least one-third of the population of Great Britain was unable to afford the quantities of the higher-priced protective foods required for their health, whilst the lower income groups showed more or less serious deficiencies in respect of good quality protein, calcium, iron, and all the vitamins. There was a natural tendency at that time to regard these deficiencies in the light of the increased production and consumption of a number of foods required to bring the intake figures for the lower income groups up to that of those whose consumption of protective foods was satisfactory. So far as I am aware, no one looked into the question of the total food supply of the country considered in terms of nutrients per head of the population, and on the assumption that distribution of the more important foods was controlled on the basis of physiological needs. Here is the pre-war picture as revealed by a nutrient analysis.

TABLE I—NUTRIENTS PER HEAD PER DAY

	Group I. Orr's Survey, 1935 Up to 4/- per head per week on food	Group VI. Orr's Survey, 1935 Over 14/- per head per week on food	Estimated daily requirement	Available in pre-war food supply
Calories	2317	3326	2550	2978
Protein, total, g	63.4	98.3	65	80
Protein, animal, g	22.5	57.8	—	43
Calcium, g	0.37	0.95	0.91	0.67
Iron, mg	8	13.7	11.7	12
Vitamin A, I.U.	774*	2875*	4700	3510
Vitamin B ₁ , mg	0.87	—	1.47	1.1
Vitamin C, mg†	42	116	71	94

* The figures for vitamin A are probably low. They are not strictly comparable with that given in the third column, as different values for individual foodstuffs were used.

† No allowance for cooking losses.

Although these data showed that, given equable distribution, the pre-war food supply would have gone a long way to provide an adequate diet for all, there remained clear-cut deficiencies in respect of calcium and vitamins A, B₁ and C.

Without going into a detailed account of the complex system of food allocations built up by the Ministry of Food on this scientifically sound foundation, I may refer to the efforts that have been made to make good the above-mentioned major deficiencies. Increased consumption of milk solids and addition of calcium carbonate to flour have made the calcium position sounder for all ages and classes. Wheatmeal flour has dispelled all risk of B₁ deficiency. Addition of vitamin A to margarine and encouragement of the eating of carrots and green vegetables have done much to improve the intakes of vitamin A. Finally, to increase ascorbic acid intakes, the consumption of vegetables and, when obtainable, fruit, has been encouraged by intensive propaganda.

Inspection of the figures in Table II clearly shows that the position has been greatly improved, in spite of a reduction of about 50% in the tonnage of imported foodstuffs.

Surely it cannot be contemplated that when the days of relative plenty return we shall ever retrace our steps, having advanced so far along the path that Orr and other

pioneers have opened up. It is a reasonable assumption that this country will continue to have a basic nutritional policy, which will ensure that the poorest family can get the essentials of a good diet. On that assumption it is reasonable to think that the problems of food manufacturers and processors are likely to be fewer and less complicated than they might otherwise have been. Their main objective will be to preserve as much as possible of the original nutritive value of the foods they handle. Many manufacturers have already done a great deal in this direction.

TABLE II—NUTRIENT CONTENT OF FOOD PURCHASED BY THE MIDDLE CLASS AND WORKING CLASS. SAMPLES, COMPARED WITH ESTIMATED REQUIREMENTS

	Amount available		Estimated requirements	
	Middle class	Working class	Middle class	Working class
Calories	2224	2240	2314	2356
Protein, total, g	74.3	73.4	60	62
" animal, g	37.9	33.7	—	—
Fat, g	89.0	86.9	—	—
Calcium, g	0.82 (0.94)	0.71 (0.85)	0.82	0.88
Iron, mg	13.5	13.7	11	11
Vitamin A, I.U.	3650	3140	4420	4360
Vitamin B ₁ , mg	1.54	1.55	1.38	1.41
Vitamin C, mg	132 (40)	128 (38)	66	67

The indiscriminate enrichment of foods with vitamins during the war period has been discouraged, partly to economise supplies of these valuable materials, but more from a desire to guide the public in the direction of selecting good foods and cooking them well. In my opinion, the people of the U.S.A. will one day regret having capitulated almost without a struggle to the "Blitzkrieg" of the commercial interests which in 5 years have pushed the sales of vitamin proprietary preparations up to the fantastic level of about 150 million dollars per annum; probably at least three-quarters of these vast quantities of materials are consumed by well-fed and well-nourished people. I am not decrying vitamins in artificial forms. They have their uses outside the field of medicine. The move by the U.S. authorities to enrich not only wheaten flour but cornflour with thiamin, riboflavin and nicotinic acid may go a long way towards wiping out the black record of deficiency diseases that has for so many years distressed those who had the welfare of the Southern States at heart. Whether this step is the best means of doing so or not is arguable. The rise in the wage levels has already caused a big drop in the incidence of pellagra and other deficiency conditions, and enrichment of foods may well prove to be a very important weapon in our fight against malnutrition in the post-war years. I hope, however, that there may

TABLE III—KNOWLEDGE OF THE VITAMINS

1933			1943			1943		
Vitamin	Chemical nature known	Human needs known	Vitamin	Chemical nature known	Human needs known	Vitamin	Chemical nature known	Human needs known
A	+	—	A	+	+	Pyridoxine	+	?
B ₁	+	—	Aneurine, B ₁	+	+	Pantothenic acid ..	+	?
B ₂ [G]	—	—	Riboflavin, B ₂	+	+	Choline	+	?
PP	—	—	Nicotinic acid	+	+	Inositol	+	?
C	—	—	Ascorbic acid, C	+	+	Biotin (H)	+	?
D	+	—	D ₂ and D ₃	+	+	<i>p</i> -Aminobenzoic acid	+	?
E	—	—	Tocopherol, E	+	+	K	+	?
						Hesperidin, P,	+	?
						Filtrate factor,	?	?
						W	?	?
						Folic acid	+	?

be no recurrence of the ignorance or irresponsibility which led to ill-devised schemes for "improving" manufactured foods by enrichment. We passed through an unhappy period some years ago when the novelty of pure calciferol and its ready availability led to its haphazard addition to every kind of food product. Fuller investigation by manufacturers of the influence of their processes on the nutritive value of their products is needed. Too often there is a tendency to focus attention on a few—the best known nutrients. The field of vitamins in 10 years is strikingly shown in Table III.

The more recently discovered factors in the foregoing table are of great importance to food technologists. To cite only two examples: Since the original studies of Harden and Zilva, so far back as 1918, it has been assumed that the food value of beer could be expressed solely in terms of the calories provided by its carbohydrates and by such of the alcohol as was oxidised in the body. Similarly, because it was found that meat extracts contained no vitamin B—in the days when only one B vitamin was recognised—they have ever since been regarded as possessing little or no nutritive value. But, if one reflects that within the past year barley and malt have been found to contain significant amounts of riboflavin and nicotinic acid, as well as other "B-group" vitamins, and that fresh meat was shown to be an excellent source of nicotinic acid, there was a *prima-facie* case for re-examining these materials in the light of this knowledge.

The results of this re-examination in the laboratories of the Ministry of Food Cereals Research Station (formerly the Laboratories of the British Flour Millers' Research Association) are indeed striking. Dr. Moran has given me unpublished data which his colleagues have recently obtained.

TABLE IV

	Riboflavin mg/100 g	Nicotinic acid mg/100 g
Light ales	0.1	probably good source*
"Old" ales	0.12 to 0.15	"
Malt vinegar	0.04 to 0.08	"
Artificial vinegar	0	"
Meat extracts	probable good source	30 to 90
Yeast extracts	"	60
Cocoa	0.25	—

* Full details are not yet available, but there are indications that beers contain nicotinic acid to the extent of about 1.5 mg per 100 ml.

There is much to be said for continuing to plan our nutrition programme in this country on good natural foods. By this I do not mean foods that have not been processed. Obviously that is neither desirable nor practicable. Food technology clearly has a great future, a future that carries even greater responsibilities for the food manufacturer than in the past. None is greater than that of providing the consumer with products as nearly as possible of the nutritive value of the fresh foods. In all considerations of vitamin losses in food processing, that of ascorbic acid looms large. It may be that loss in treating foods is not preventable. Is appropriate enrichment before treatment the solution? Can we make better use of some of the natural products of unusually high vitamin value?

By way of illustration I suggest that all jams might have a minimum ascorbic acid content, making them equivalent in this respect to "fresh fruit," providing, say, 5 to 10 mg per ounce. Would it be practicable for this purpose to make use of ingredients of relatively very high ascorbic acid content? Here are a few that have come to our attention since the outbreak of the war.

TABLE V

	Ascorbic acid mg/100 g		Ascorbic acid mg/100 g
Rose hips (various species)	500-2500	Winter strawberries	135
Green walnuts	700-3000	Cape gooseberries (dried)	50
Dried guava	500-1000		

I have added dried Cape gooseberries to the list because, although the actual content of ascorbic acid is not high, it is unusual to find a dried fruit of this type with so much ascorbic acid. The main interest in connection with dried Cape gooseberries is their remarkably high carotene content. Samples recently examined by the Ministry of Food contained as much as 6 mg/100 g. This very attractive product has, I think, a future as a "dried fruit." It does not naturally contain much sugar, but this could be made good and one could have, for special purposes, an alternative to raisins and sultanas that would have the nutritional advantages of good A and C content.

But I hope we shall keep a balanced outlook in the future. We do not want vitamins, minerals, etc., added when such additions are inappropriate. We do not wish to have vitamins A and D added to such products as biscuits or custard powders. On the other hand, it is a reasonable aim that foods essentially carbohydrate in character should contain an appropriate quantity of the vitamins related to carbohydrate utilisation, whether provided by the use of natural flours or by enrichment. Food technologists will have a big part to play in achieving a rational nutrition policy in the years to come.

The Rôle of Food Legislation in securing Adequate Nutrition

By H. E. COX, PH.D., D.Sc., F.I.C.

THE Food and Drugs Act is mainly a consolidation, with amendments, of old legislation; most of it was framed nearly 70 years ago when the object was stated to be the prevention of fraud. The food legislation which we now envisage not only aims at the prevention of fraud and gross adulteration, but is intended to preserve and improve the standard of nutrition. It is to prevent or restrict exploitation of the newer knowledge by bogus or exaggerated claims and to stop those insidious, but none the less real dangers which may result from the continued ingestion of impure materials, or excessive or insufficient quantities of important constituents. Most of the regulations made under the Food and Drugs Acts from 1875 onwards have stood well the test of time, but some of them are quite out of accord with modern knowledge. Consider, *e.g.*, the definition of a preservative: "any substance which is capable of inhibiting, retarding or arresting the process of fermentation, acidification, or other decomposition of food . . ." With a few stated exceptions, no such substances may be added to food. A pressing problem to-day is the maintenance of the vitamin content of many foods and the prevention of incipient rancidity in refined fats. We now know that the natural vitamins and pigments in many articles of diet are of much physiological importance and we desire to retain them. Yet to take steps to retain these things may be a criminal offence. We know that some of the natural quinones, anthocyanins, flavones and the like in minute quantities are the best antioxidants; they preserve vitamin content and prevent oxidative rancidity. These innocent substances do the very thing the Regulations say shall not be done—they inhibit, retard or arrest the process of acidification. No doubt we are better without such preservatives as boric, benzoic or salicylic acid, but let us have regulations not in conflict with what is now known to be desirable. Another anachronism is the rule about added colouring matters; six are forbidden; some of these are no longer manufactured as colours and have not been found in food these 40 years. The investigations on which this regulation was based are mostly 50 years old, and some at least were defective owing to the use of impure colours. Since then other colours have been used and are used to-day. A few of these have been investigated physiologically and the verdict on some is adverse, yet the use of these is not prohibited. Surely a permitted list of pure synthetic dyes which have been investigated and proved satisfactory should be devised, and provision should be made for the inclusion of other colours when they are shown to be harmless.

The significance of traces of active compounds is now realised as it was not realised 25 years ago, and experiment on the large scale upon the public should not be allowed. At the present time the law only steps in if it can be proved that the substance is injurious in a particular case; the facts must be argued before a bench of justices, who may be ill equipped for assessing the merits or demerits of a complex chemical compound in the light of controversial evidence. Is it not time that this relic of a bygone age should be deleted? In its place we need a regulation likely to encourage the use of naturally occurring pigments that have anti-oxidant value, and which provides for official approval of substances that have been properly investigated. Anti-oxidants and the like are prime necessities for vitamin-containing foods, such as fats and canned fruits.

What of trace elements? Which are valuable and which are objectionable impurities? How long are we to be dosed with crude phosphates containing x per cent. of fluoride or with jellies containing y parts of zinc and z parts of lead and copper? When difficulties of this kind arise, manufacturers and analysts may reasonably look to the central authority for guidance. It is seldom forthcoming. Forty years ago they did these things better. Arsenic was troublesome and a Royal Commission pronounced upon it after due deliberation. True it is that their finding might now be revised, or at least re-considered, but they did act helpfully at the time.

Such reflections point a moral. Our food legislation needs to be alive and progressive and to be in the hands of a competent authority which takes an active interest in the subject. one that is accessible to the trade and to the food and drugs authority and is in more or less constant session. The chemistry of food and nutrition is active and rapidly developing;

it is encumbered by antiquated regulations. Coke defined law as the perfection of reason; he lived in an era long ago when there was no law relating to food and drugs, and no Schedule of Permitted Preservatives.

REQUIREMENTS OF FUTURE LEGISLATION—Even when obsolete law has been swept away, how is legislation to be improved so as to help in promoting better nutrition? Food is one of the most important factors in our lives. Its care and control ought to be the concern of a separate department and not a minor department of the Ministry of Health. That that Ministry has plenty else to do is evident from its annual reports, of which less than 1% is devoted to food topics, and that 1% is largely a compilation of what others have done—reports of local authorities, of Public Analysts and so forth. Years ago, it is true, we did see a series of very valuable reports on food topics, but even these now rarely appear.

Hence, in my opinion, the first administrative requisite is the post-war establishment of an independent department armed with statutory powers and duties. A first care of a live post-war department dealing with food ought to be the establishment of standards and definitions which secure for the public adequate nutrition. We need calories as well as vitamins. Then it must protect the public not only against fraudulent dilution but also against exaggerated claims. Such standards need to be elastic and progressive, so that meritorious new products are not discouraged and adjustments may be made without undue difficulty. By way of illustration, consider two basic foods, milk and cheese. By virtue of a decision of the courts 28 years ago, any milk drawn from the udder of the cow is genuine, and it is permissible to charge the same price for milk with say 1.5% of fat as for milk of good quality. Children may be under-nourished for the sake of the calf; quality must yield to quantity. It is time that milk should be required to have a certain minimum composition. It is desirable too that some plan should be devised to encourage the production and sale of the higher quality milk. It may be difficult to work this out, but industrial firms have successfully done it. It may be that consequential changes should follow, so that the honest but unfortunate vendor be not stigmatised as an adulterator; but why should the public suffer every time? Cheese normally contains about 30–35% of water; why should the excellent process of pasteurisation be accompanied by addition of gross quantities of water? Of course, the reputable manufacturers do not desire such practices, but the stress of competition tends to an increase in water content. It is time this was regulated. Also, we shall want our beer a little more dehydrated and some more meat in our sausages.

The matters that I have mentioned are quite old-fashioned. The newer problems are due to the recognition of the importance of vitamins and of certain trace elements. Manufacturers must be encouraged to produce foods containing, as far as possible, all their natural vitamins. False or misleading claims must be prohibited, and false advertisements, whether as to nutritive value in general or as to the content of some particular substances. How can it be done equitably? Clearly it is unnecessary for a vendor to declare the content of a naturally occurring food—whether fruit, vegetable, meat or milk; so he must not make claims for them. We know that oranges contain ascorbic acid, but what of jam or lemon squash? If these are prepared from fresh fruit they will contain vitamins; if from old or sulphited pulp they may not, and the manufacturer who so runs his factory as to conserve the vitamins or who has developed processes for so doing is entitled to some advantage over his less efficient rival. A fruit product, for example, should be entitled to be labelled as made from fresh fruit, if so it be; it should then be required to have a stated minimum ascorbic acid content. This would inform the public that it has a dietetic or antiscorbutic value. Alternatively, as Dr. Drummond has mentioned, it could be enacted that all jams should have a minimum ascorbic acid content; this would be attained either by the use of fresh fruit or by addition of a concentrate. This kind of Order could, and indeed should, be applied quite widely. Beer and vinegar should contain riboflavin, nicotinic and pantothenic acids; so there should be a re-definition of vinegar to bring out clearly the merits of the genuine product. Meat extracts should be required to state their content of creatine and of nicotinic and pantothenic acids, if only in their own interest, to show that they have dietetic and nutritive value and are not solely stimulants, as is stated in text-books.

Applying these ideas on a large scale would involve the issue of standards for many foods and needs careful planning to avoid hardship arising from deterioration or sale of stock not so fresh as it should be. But it would stimulate the conservation of the natural virtues of foods and reduce the need for nostrums.

Following on this, no commodity should be permitted to be labelled "rich in vitamins"; the amounts or kinds claimed should be stated. Similarly, mixtures for which special vitamins are claimed should state the quantities of the active constituents on which the claim is based. Such claims are likely to be for vitamins or pro-vitamins; minerals or trace elements are also involved, and these should be present as the result of intelligent planning and not just gross impurities in substances which ought to be pure. So limits should be prescribed for all deleterious elements in the light of experience. It is not sufficient merely that the Minister should have power generally to regulate the composition of any food; he has had such power for many years. The need is to use it. But when one comes to consider how to use it, difficulties arise, partly on account of lack of accurate knowledge. Yet the absence of such detailed knowledge, *e.g.*, of cumulative action, ought not to prevent the promulgation of reasonable standards for impurities. If arsenic or copper is an undesirable substance known to be harmful if in substantial quantity, it can be said that the desirable quantity in any food is none. "None" is an ambiguous word in this connection; some will always be found if the method of analysis is sufficiently sensitive. So the standard should be that proportion which is unavoidable and represents the best obtainable in commerce. If, for example, gelatin manufactured from good materials, under good conditions, can be made so that it contains no more than say 50 or 100 p.p.m. of zinc, such a figure should be chosen as the maximum, and food manufacturers can observe it without undue cost. It ought not to be necessary to prove that 100 parts are injurious to health, whereas 90 parts are not injurious. Such an approach is possible by a committee of chemists representing producers, users and the public and does not pre-suppose any elaborate physiological experiment. Moreover, such standards can be revised from time to time.

Labels and Advertisements—Recent legislation has taken cognisance of labels and advertisements. It is to be anticipated that there will be an increasing number of pre-packed articles for which wide claims will be made. Some difficulty must arise in deciding what generalisations are permissible. Yet claims for specific merits should be supported by a declaration of the constituents on which they are based. The identity and, as far as possible, the quantity of the active substances should be stated in plain terms.

It would seem from the recent issue of the Defence (Sale of Food) Regulations, 1943, that the Ministry of Food contemplates taking action along these lines. But it remains to be seen to what extent Regulations will be operated, for we remember that the Ministry of Health had much the same powers. Evidence that the public conscience is now more fully alive to the need of control of advertisements of this kind and of labelling is afforded by the recently published* decision of the Newspaper Proprietors Association to refuse advertisements, in their papers, weekday and Sunday papers, of medicines and treatments of various diseases. They will also refuse advertisements which are exaggerated and convey false impressions. This is quite apart from the legal prohibitions in relation to venereal and some other diseases. The N.P.A. propose to submit products to medical scrutiny or chemical analysis where necessary.

Consequences of interest and difficulty to the Public Analyst arise from the implementing of such Regulations. Labels will have to state the nature and amount of dietetically important constituents in many foods. It is important that such labels shall state what is actually present when the article is purchased, not merely what was there when it was first produced, although some safeguard as to time may be necessary to prevent injustice to the manufacturer. This involves the implication that the Public Analyst must be ready to check the composition and assay the vitamins, minerals and other factors with reasonable speed and certain accuracy. This may be no small problem, and one in which he will need the help of the central authority in the elaboration of standardised or proved methods. Such are apt to take much time to prepare, and certainly the Society's Analytical Methods Committee will need to be very busy. Vitamin D presents particular difficulties at present.

I think the matter goes further—though some may not agree; in my view the Public Analyst ought to be prepared to advise not only his local authority, which is acting as an inspector or policeman, but also the public and the trader on such matters. He must therefore be prepared to undertake, as far as possible, any such work and provide the answer in a reasonable time and at reasonable cost to any *bona-fide* enquirer. He must be prepared not only to determine constituents, but also to advise or report upon the properties

* *Brit. Med. J.*, 1943, 462.

of foods in general. He must therefore be well informed on recent work both on analytical problems and on the structure and properties of food constituents if he is to retain the place which is his due and not lapse into merely a testing machine. Post-war planning for the Public Analyst needs to be largely bound up with enlarging his knowledge, experience and competence in the science of food and nutrition, and in proving to the outside world his potential usefulness not only as a guardian of a department of the public health service but also to the industry.

Legal Proceedings—In conclusion, I would refer to three small matters relating to legal proceedings and penalties which, in my opinion, are in urgent need of reform. It is clear that offences arising from false labels or advertisements are generally the responsibility of the manufacturer or wholesaler. This is recognised both in the Food and Drugs Act and in the new Regulations, and in both provision is made for proceedings to be taken directly against the manufacturer or in some cases for bringing them in as parties. The fact that the larger firm is before the court and that the offence may, and often does, relate to quite large commercial dealings, make three reforms desirable. First, the penalty needs to be made adequate—the punishment to fit the crime. Under the Food and Drugs Act the penalty for a first offence is only £20, a trivial sum now-a-days in respect of transactions which may involve thousands of packets or pounds and cases which may cost many times that sum to prosecute or defend. Under the new Regulations presumably the penalties are the much higher ones ordained under the Emergency Powers Order, but this is only temporary and will doubtless cease after the war. For a second or subsequent offence the penalty may be £100 with or without imprisonment; the latter is nugatory in respect of a company. The Divisional Court has recently pronounced upon what is a second offence in these matters. It includes any sale of the peccant package after the date of a first conviction. Consider a hypothetical case. An individual producer makes 100,000 packets of an article to which he attaches a label false in some particulars; a packet is sold by retail and the producer is summoned and fined £20; he then tries to recall the merchandise but fails, as fail he always will, to get every packet in. A second case is taken and he is fined £100 and gets three months' imprisonment; when he comes out an enterprising inspector has another case waiting and he gets a further three months. This might continue to the end of his days because, as their Lordships pointed out, the offence charged is the retail sale and it is laid at the manufacturers' door as the person to whose act or default it was originally due. No doubt this is sound law, but it is hardly justice, inasmuch as there was no second conscious or intentional breach of the law. Two amendments seem clearly indicated: a re-definition of a subsequent offence more appropriate to food and drugs cases, and some form of estopper on repetitive prosecutions if it can be shown that the offender has taken all reasonable steps to prevent a recurrence.

The Part to be Played by the Food Supplier in Safeguarding Nutritional Values

. BY E. B. HUGHES, D.Sc., F.I.C.

THE title of this part of the subject of to-day's meeting might seem to indicate that the food supplier is to be required to take a new outlook on his responsibility to the public. The manufacturer of ordinary food products has not in the past considered his products directly in terms of nutrition, but he has considered them and must think of them as requiring to be wholesome products, prepared from wholesome ingredients free from adulteration, from contamination and from preservatives—except such as are allowed by law—and acceptable to the public as food they like and find palatable and satisfying. That, I think, sums up the basis of the business of the manufacturer of ordinary food products. The materials he uses are the food materials generally used in the household, but allowing of more exacting requirement and selection; his cooking or baking processes are mostly only mechanical developments of home-cooking procedure, but allowing of greater precision and uniformity and of scientific control.

What, then, is this further part to be played by the food manufacturer? Obviously it is to ensure that, as far as possible, there shall be no loss or destruction of known natural nutrients in the course of manufacture of food products, and that, where loss does occur, endeavour to reduce it shall be made. These are reasonable requirements but they are

not a new point of view for the food chemist, as the wide range of excellent papers read and discussed before the Food Group in the 12 years of its existence clearly proves; these papers show keen and persistent realisation of duty to the public.

The requirements from those who make specific claims must, of course, be more exacting; whatever claim is made must be capable of verification. None of us would disagree with that. The Food Group, for example, expressed that opinion clearly 10 years ago in the memorandum presented to the Departmental Committee on Food Law.

This further part to be played, then, implies no serious addition to the food chemist's work, but it does imply definite assurance of what the substances (or qualities) are that must be safeguarded and that these can be measured by reasonably practicable scientific means.

Concerning losses of nutritional value in domestic cooking, and also in the preparation of commercial food products, a fair amount of investigational work has already been carried out; information is certainly not complete (one might cite, *e.g.*, the simple case of the cooking of cabbage) and there is undoubtedly a good deal yet to be done in making such investigations more extensive and also more detailed, but we do know at least the broad outlines and the general principles and, as far as can be judged from these, there are no losses that are serious; certainly—in commercial practice—food products are in general at least as nutritive as the home-cooked foods. As further knowledge becomes available—and the consideration nowadays being given to this matter indicates that the importance and urgency are realised—the position should be improved so that the degree of conservation of nutritive values practically attainable should be realised.

To consider in detail what special precautions might need to be taken in the manufacture of food products to conserve nutritional values—which in this discussion mean chiefly the vitamins and minerals—is beyond the scope of this paper. In canning of fruit and vegetables, for example, processes employed, with the exception of blanching, all tend towards minimising loss or destruction of nutritional constituents. This seems to be equally true of meat canning, where the only important losses are probably extracted minerals and vitamins when meat is shrunk by par-cooking before sealing the cans; even riboflavin suffers little loss in the processing. The canning industries are alive to the need for the fullest information, and both in this country and in the U.S.A. special attention is being given to the subject.

In jam practically the whole of the ascorbic acid of the fruit used is conserved, provided that care is taken to avoid contact with certain metals, particularly copper. (Incidentally we can now realise that when copper sulphate was used for the greening of vegetables there must have been complete destruction of the ascorbic acid.) In view of the practice of preserving fruit with sulphur dioxide it is satisfactory to find that the vitamin C of the fruit is well conserved thereby; this beneficial action of sulphur dioxide also occurs in the drying of fruit. Wheat flour is the product that comes to mind as an example of what can be done when the need for safeguarding nutritional values (in this case mostly vitamin B₁) causes a very noticeable change in the accustomed appearance and quality of the material, as in the change from white flour to National 85% extraction flour. The millers have not been content to comply merely with the required extraction, but have made great strides in the production of high extraction flour of surprisingly low crude fibre content and also of improved colour; so much so, indeed, that it seems likely that before very long we shall have white, or almost white, National flour. The baking qualities of this high extraction flour are not equal to those of our pre-war flour but, considering the difficulties and the admixture of a proportion of other cereals, the general standard has been to the credit of the milling industry. In bread-baking there is but little loss of aneurine—either from the flour or the yeast, and no serious loss in toasting; in other bakery products it seems that not more than about 15% of it is lost, provided that the pH does not exceed 7.1 and that the product is not overbaked.

In the reports of the detailed work carried out by the D.S.I.R. on the drying of vegetables and fruits it will be noted that an important feature of the work has been the establishment of conditions for minimum loss of vitamins and extractives—notably, for vegetables—in the method and condition of the scald (serial scald, with sulphur dioxide for some vegetables, and acid pH), and in the packing (generally in inert gas or in compressed block form). In meat drying, care is taken to concentrate and add to the meat before drying the liquor resulting from the pre-cooking of the meat; gas packing or compression into blocks is advisable.

Great improvements have been made during the war in the drying of egg, and the product when fresh is generally excellent and contains its vitamin A unchanged, but dried egg is not imperishable, and in a few months (at best) it undergoes serious loss of culinary qualities and of vitamin A, unless kept in cool or cold store; as for full-cream milk powder, it would seem to be necessary to pack the powder in gas for best results.

The manufacturer of sugar confectionery has no problems of this kind; his products are in the main palate pleasers and calorie suppliers. Milk chocolate, however, occurs to me as a type of product that may require consideration, for, if its contents of milk solids and butter-fat are dietetically significant, then obviously some standard of composition will be advisable—and there are doubtless other instances for like consideration.

From the suppliers of raw or basic foods, particularly some vegetables, no more can be expected than that their materials shall be supplied as fresh as possible and that there should be appreciation of the most suitable means of transport and of storage. Though varietal effects are important, there are so many other considerations—such as season, climate and soil, early or late growth, eating qualities—that unless a variety is exceptionally rich or poor, there will probably be little particularisation for vitamin content.

Cold storage and transport in cold store conditions are not yet applied in this country to anything like the extent that they should be; so far we have hardly gone beyond the stage of cold storing only such products as would otherwise become visibly or palatably bad.

In considering this section of to-day's subject allotted to me I have tried to view it critically—but, as will be realised from what I have said, I have not found a great deal to criticise, certainly no outstanding fault. I feel certain, too, that it need not be feared that the food manufacturer will fail in the duty of giving as much thought and care to the safeguarding of nutritional values as he does to the more obvious qualities. This will ensure one contribution towards the ideal state of a well-nourished nation; another, much less readily realisable, is the education of the public in matters of diet so that, when the time of unrestricted supply and choice comes again, each one of us may be able to eat both wisely and enjoyably.

DISCUSSION

Dr. J. R. NICHOLLS said that it was a difficult discussion to open because of the wide field covered by the three papers, and he would only deal with certain general principles. It had been brought out that food was one of the most important factors in life, and difficulties arose through the great variety offered to, and utilised by, the public. It was generally accepted that a proportion of natural foods in the diet was essential and distributors must bring these to the consumer in the best and most natural condition possible. Manufactured foods were generally less perishable, but they should retain a maximum of their natural value, and exaggerated claims should not be made for them. It was the duty of the scientist to help both distributors and manufacturers with their problems, but it was important that there should be a basic nutritional policy with agreed minimum requirements. Although we now had considerable knowledge of nutrition, it would be agreed that finality had not been reached and caution must be exercised. The scientist must also assist in the education of the public as to what was good for them. It must not be forgotten that taste and flavour were most important items and it was just as unsatisfactory to bring to the public adequate food which was unappetising as to bring attractive food which had lost essential constituents. In war-time, drastic control might be necessary; but in peace-time there should be free choice with proper education of the public in what was best. Legislation required care and should not impose unnecessary hardship on distributor or manufacturer. Three things, therefore, emerged: (1) scientific knowledge must be coordinated, agreed and passed on to the public; (2) manufacturers and distributors must have a free hand for development and a minimum of control; (3) legislation must prevent exploitation of the public and waste of natural nutritional constituents. All three were interdependent and must be considered together.

Mr. H. E. MONK said that if all manufacturers conformed to the creed of Dr. Hughes there would be very little need for food legislation, but there would always be those who did not. The first point that seemed to emerge from all three papers was the need for standards. Who was going to set up these standards? Obviously the Ministry of Food should have all the authority and powers with regard to foodstuffs. How long would the Ministry of Food persist after the war? The most comforting thing that Professor Drummond had said was that we were likely to have a conscious nutritional policy. The speaker hoped that this would be so and that the Ministry of Food would remain to administer it, but after all, rationing—in the widest sense of getting the food into the country and distributing it fairly—was the main job of the Ministry, and rationing was irksome to the man in the street and particularly to his wife.

Any nutritional policy raised large issues, some of them political. For instance, if it were decided that everyone ought to have certain minimal amounts of specified nutrients, how could it be ensured that they got them? By rationing or by subsidies on selected key foodstuffs? Such questions might be outside the scope of the present meeting; nevertheless they raised thorny issues which would have to be solved if a national policy was to succeed.

Another need which had been disclosed was for considerable scientific work to elucidate and to implement any nutritional policy. The closest co-operation between the Public Analyst and the responsible Ministry was called for. Hitherto action by Local Authorities had resembled guerilla raids by local patriots more than coherent military operations. Co-ordination by the higher command had been conspicuously lacking.

Mr. ERIC C. WOOD questioned the suggestion that the publicising, to other than qualified persons, of the vitamin content of foods in quantitative units was *per se* in the general interest. The widely varying intakes of the various articles of diet made it essential to consider the vitamin content per daily intake, not per unit weight, to avoid misleading comparisons; and since the value of the vitamin content could be judged only as a percentage of the normal daily requirement (which the public did not and could not be expected to know), it was in this form that it should be quoted. Even so, a division into qualitative categories, such as "rich," "good," etc., "source of vitamin—" on the basis of the figures as declared to the Ministry would be much better for general use on labels, etc., than the figures themselves; otherwise misleading and even false comparisons between foods of differing vitamin content were unavoidable. A food which provided 10 times as much of a particular vitamin as some other food was not necessarily 10 times more worthy of consumption; but it was almost impossible to prevent any but trained nutritionists from thinking so.

Mrs. S. TRITTON said that the fixing of standards for food, looked at from the manufacturers' point of view, was all to the good. With the diversity of opinion which existed on permissible quantities of impurities in gelatin, for instance, much time, trouble and litigation would be saved if standards were laid down. A doubt existed whether the declaration of vitamin content was entirely desirable. With vitamin C, for instance, it might happen that a product containing at the time of manufacture an adequate proportion of that vitamin might have lost this by the time it reached the consumer or the Public Analyst. This frequently occurred with fruit squashes, and the situation might arise that a manufacturer preparing a cordial from fresh fruit juices and declaring this fact together with the vitamin C content on the label could through legal prosecution lose his good name, whilst an inferior product might escape prosecution through not declaring a fresh fruit juice or vitamin content.

Mr. W. B. ADAM was of opinion that more attention should be given to the improvement of the nutritive value of "natural" foods. The effect of variety, maturity, soil and climate should be studied by controlled experiments and the results be treated statistically. By this means the best conditions might be ascertained for improving nutritive values. Standard analytical methods should be agreed upon and also standard methods of expressing the results.

Dr. J. YUDKIN considered that the direction of food production based on nutritional needs rather than on economic considerations, and also the control of food legislation, could best be ensured by the formation of a Nutrition Council as envisaged at the Hot Springs Conference.

Mr. H. LEE considered that it would take a very long time for the legislative reforms advocated by Dr. Cox to be attained, especially as the Emergency Powers Defence Regulations would not be accepted in peace-time.

Mr. A. L. BACHARACH thought the opportunity was a good one for paying a tribute to the work of Dr. Drummond at the Ministry of Food. He had probably furnished the best example in the lifetime of all those present of the scientifically right man in the right place at the right time doing the right job. He was sure Dr. Drummond did not wish to imply that the consumption of milk, which had been "flattened out" as the result of priority schemes, was satisfactory at its present level of approx. 0.5 pint per day. 1.5 pints per day was the level usually advocated by nutritionists on both sides of the Atlantic. He could not accept Mr. Wood's dictum that disclosures on the label should be withheld on the ground that the public would not understand them. This was dangerously like the attitude of the "superior person," which ultimately led to totalitarianism and all the things we were fighting against. In a democracy there must be adequate machinery for safeguarding the interests of the public, and those in charge of the machinery must have all the information available both to them and to the public that they protected. Simultaneously, a programme of education would make the public increasingly able to understand what it was told about its foods and food-medicinals. The two processes would act and re-act on each other. He fully accepted Dr. Hughes's assurance that the best food manufacturers were prepared to collaborate in any schemes aimed at improving the nation's diet. If manufacturers were unable to secure that particular nutrients would survive in their products during normal "shelf life," then they would have to forego any claims for those nutrients. Finally, he thought that Dr. Hughes had been a little too sweeping in his condemnation of the "enrichment" policy. Every case should be judged on its merits and he, the speaker, had attempted at a joint meeting of the two Societies two years ago (*Chemistry and Industry*, 1942, 61, 57) to lay down the criteria by which any proposed fortification could be judged.

Mr. J. VALENTINE BACKES said that the soundness of the points brought out by the three speakers in their papers was not only irrefutable but expressed an essential national need. Science had accumulated an enormous amount of valuable information, but by reason of its being too scientific and not sufficiently industrial, the community lost the benefit through lack of discreet application. The necessity for ensuring maximum nutrition from food, the necessity for guidance to manufacturers of prepared foods, and the necessity for bringing legislation up-to-date were all recognised, and food chemists regretted the scanty appreciation that Government Departments had given to these matters in the past. The success achieved by war-time control and enforced legislation made the time ripe for scientific principles of feeding to be more universally applied, but great care must be taken that the pendulum did not swing too far the other way.

Dr. DRUMMOND, replying to the discussion, said that he did not deprecate the enrichment of individual foods, but thought that each one must be considered on its own merits. While control might not be maintained in full after the end of the war, he considered that every care must be taken to see that the priority classes were properly fed.

A Review of Micro-volumetric Apparatus

By G. H. WYATT, Ph.D., F.I.C.

(Contribution to the Microchemical Symposium, Sheffield, October 9, 1943)

It is very noticeable that, on the whole, micro-analysts have not paid sufficient attention to micro-volumetric analysis and one finds papers published on apparatus which, with the exception of a few details, has already been described. Micro-analytical methods have a reputation for being more rapid than their macro counterparts, and it is therefore surprising that wider use is not made of micro-titration, since, generally speaking, volumetric methods are quicker than gravimetric. It is to be regretted that most of the apparatus here described is not available commercially in this country. This may account partly for the present restricted application of micro-volumetric methods, and it is suggested that if the position were remedied by the provision of representative instruments, they would find increasing use.

MEASURING FLASKS—The usual macro-pattern (A, Fig. 1) may be purchased in sizes down to 5 ml capacity, but as the size is diminished it becomes increasingly tedious to mix the contents thoroughly; also some of the solution is held by the stopper. Caley¹ therefore suggested the design B, Fig. 1, having an upper chamber with a vol. about 5 times as great as that of the lower, calibrated part. By tipping the flask, the soln. is run into the upper part and is mixed by a swirling movement as it is returned to the lower part. A stopper is required only when volatile solvents are used. This flask suffers from the disadvantage of being top-heavy, a property which is avoided in the flasks C to F, Fig. 1, due to Holmes.² These flasks may be supported in an ordinary test-tube rack; they may be heated on the water-bath and a reflux condenser may be inserted in the top. The last example, F, is made from a centrifuge tube, so that a substance may be pptd., centrifuged, washed, and dissolved to give a soln. of known volume, all without transference being necessary.

PIPETTES—Precision wash-out pipettes (A, Fig. 2) have been described by Pregl and Wostall.³ These have a capacity of 0.1 or 0.15 ml and, after delivery in the usual manner, are rinsed with water or other appropriate solvent. They are accurate to ± 0.001 ml. (cf. Ostwald, Folin type⁶²). A modification (B, Fig. 2) due to Szent-Györgyi⁴ has a side-tube carrying rubber tubing, by the manipulation of which it is easier to adjust the meniscus to the graduation line; the main tube is covered by the finger in the usual manner. A very similar design to that of Pregl and Wostall was used by Linderstrøm-Lang and Holter⁵; it has a jet so fine that the soln. is retained by surface tension until pressure is applied by blowing through rubber tubing attached to the top.

Another micro-pipette, for simple delivery without rinsing, is due to Christensen, Pennington and Dimick⁶ (C, Fig. 2); it is fitted with a rubber teat having a slit at the closed end. The teat is pressed parallel to the slit and the jet is dipped into the solution; releasing the pressure causes the pipette to fill. Delivery is effected by lightly pressing perpendicularly to the slit, so that the contents can drain out. For the filling and emptying of pipettes Schilow⁷ used a simple levelling tube (E, Fig. 2); the pressures involved are as marked, together with the surface tension acting at the jet. Another simple and convenient device for the filling of micro-pipettes was described by Benedetti-Pichler⁸ (D, Fig. 2). A filter-pump is connected as indicated and suction is controlled by placing the finger over the opening at *x*. A more complex apparatus is that of Linderstrøm-Lang and Holter⁹ (F, Fig. 2). The capillary opening at the top of the pipette is adjusted so that the soln. does not escape when both ends are subjected to equal pressures; delivery is effected by application of a pressure equal to about 50 cm of water, the rate being controlled by adjustment of the jet diameter (the jet is bent to touch the side of the titration vessel during delivery). By means of a reservoir above *d* the chamber *e* is filled to a level just above the top of the pipette, taps *a* and *c* being open. Tap *a* is then closed to force the soln. into the pipette, the level of the liquid in which is adjusted by closing tap *c* and opening *a* and *b*. The soln. is delivered by means of the pressure at *d*, taps *a* and *b* being closed. With a 30-cb.mm pipette the error is less than 0.1%.

Wigglesworth¹⁰ has described a micro-pipette (G, Fig. 2) to deliver automatically about 0.3 cb.mm. The capillary tube *a* has the lower end waxed inside and outside and is

cemented in the holder *b*. Once the soln. has passed the waxed portion, capillary forces cause it to rise to the top of the capillary, hence a constant vol. can be obtained without visual adjustments.

The force required to suck up soln. into the pipette and to deliver it may be supplied by the compression of rubber tubing. Standen and Fuller¹¹ attached a capillary pipette of 2 ml capacity, graduated in 0.1 ml, to the lower end of a \sim -shaped tube, the upper end of which carried a rubber tube compressible by a roller. The right-hand part of the \sim -tube contained water, and the soln. to be measured entered the pipette only; thus by means of a set of pipettes with interchangeable ground-glass joints, rinsing of the apparatus was avoided when different solns. were used. Linderström-Lang and Holter⁹ applied a constant pressure *h* (A, Fig. 3) of about 20 cm of water, which was insufficient to expel the contents of the pipette *p* unless the jet was below the surface of the liquid in the titration vessel *v*. To start or stop delivery, *v* was raised or lowered by a rack and pinion movement *r*. The pipette was filled by suction at *s*, the tap *t* being closed. (Capacity of pipette = 6 cb.mm., accurate to less than 0.3%.)

In 1925 Trevan¹² drew attention to the fact that a good vaccine syringe may be used for volume measurement, when it can be set by eye to less than 0.1 mm, corresponding to 0.002 to 0.003 ml, and that there is no parallax or drainage error. By attaching a micrometer screw to drive the piston through 0.5 mm per turn he found it possible to deliver 0.01 ml with an error of less than $\pm 1\%$. Krogh¹³ and his collaborators used a syringe (B, Fig. 3) with a glass plunger ground to fit accurately; no lubricant was necessary. The screw *a*, locked by a grub-screw *b*, serves as a stop so that the syringe may be used for the delivery of volumes from 0.1 to 1.5 ml (accuracy = ± 0.0001 ml). Temperature effects are compensated by the expansion in opposite directions of the steel rods *c* and the glass syringe. Fibre guides *d* prevent rotation of the plunger, which is shown at the bottom of its travel.

Krogh filled his syringe with the soln. to be measured, as also did Kirk.¹⁴ This means that a comparatively large vol. of soln. is required in excess over that actually used. This disadvantage was avoided by Titus and Gray¹⁵ by attaching jets to a 1-ml syringe by interchangeable ground-joints. The pipette was held vertically and 0.2 ml of mercury was drawn into it. This mercury then served as a piston, an air-bubble being left between the mercury and the soln. Similar pipettes have been described by Chambers,¹⁶ Rosebury and Heyningen,¹⁷ Keston, Rittenberg and Schoenheimer,¹⁸ and Benedetti-Pichler¹⁹; in each instance the syringe is filled with water, which is separated from the solution by an air-bubble. Benedetti-Pichler (*loc. cit.*) describes a special apparatus to draw out jets, as shown at C, Fig. 3. These have a diam. of ca. 1μ at the tip; when the jet is held under water and pressure is applied to the piston, it is not possible to blow bubbles of air. The bore of the capillary is measured under the microscope, the magnifying effect of the curved glass being avoided by immersion of the capillary in cedar-wood oil. The vol. of soln. expelled from the pipette may then be calculated from a measurement of the distance travelled by the meniscus. Alternatively, the soln. may be withdrawn from a larger capillary (D, Fig. 3) and magnified $\times 40$ to 60, a graduated eyepiece being used.

Finally, Hadfield²⁰ ingeniously combines the syringe and wash-out types of micro-pipette. The soln. is drawn up or expelled by means of the plunger *a* (E, Fig. 3), which contains water or other suitable solvent for rinsing the pipette. Hadfield describes the method of construction of such a pipette of a given capacity, say, 0.5 ml.

BURETTES—The usual type of burette, having a capacity of 10 ml and graduated in 0.05 ml may be purchased with a Schelbach scale. Such burettes, fitted with a stock bottle and automatic filling device, were used by Pregl²¹ for micro-Kjeldahl determinations (*cf.* Bang⁶³). Numerous papers have been published on more specialised micro-burettes, many of which depend upon principles described above in connection with pipettes. In the first place, however, some attempts to combine the advantages of macro- and micro-burettes in a single instrument may be mentioned. Löber²² described such a burette in 1931 and was soon followed by Dustman²³ and Eissner.²⁴ The instruments being very similar, it is sufficient to illustrate only that of Eissner (A, Fig. 4), in which the macro part consists of a number of bulbs, each of 3 ml capacity, the micro part being a 3-ml burette graduated in 0.01 ml.

Benedetti-Pichler²⁵ in 1928 described a micro-burette (B, Fig. 4) with a long, narrow jet. Control is made more certain by provision of a "capillary brake"—shown enlarged in the figure. This consists of a tube *a* held in a wider tube *b* by a rubber sleeve; *a* is drawn

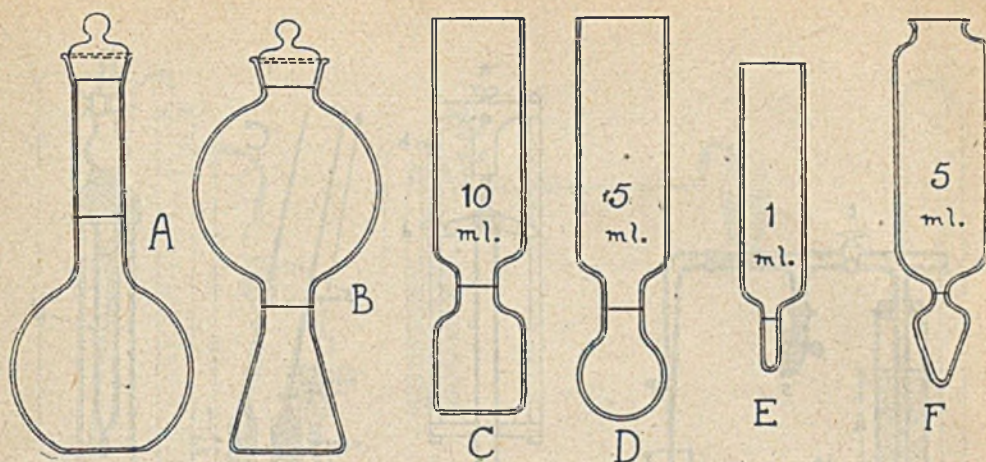


Fig. 1

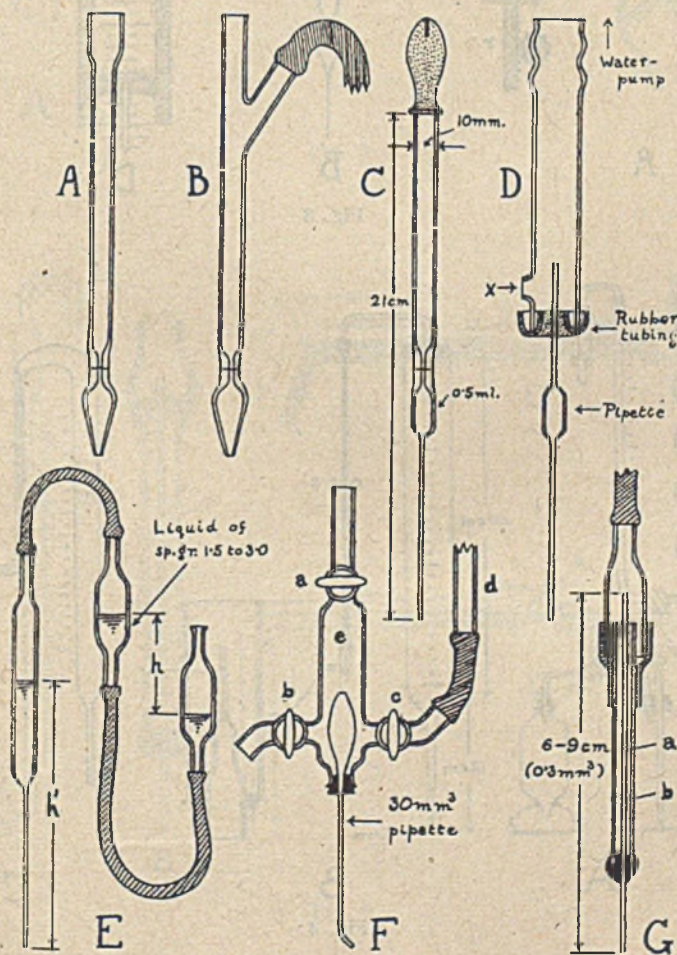


Fig. 2

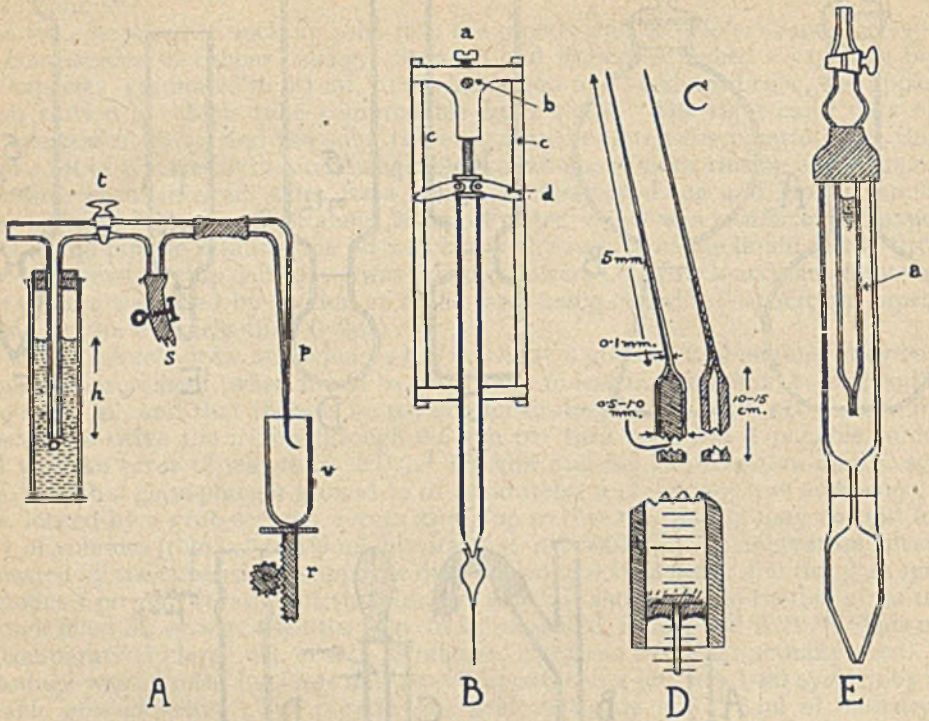


Fig. 3

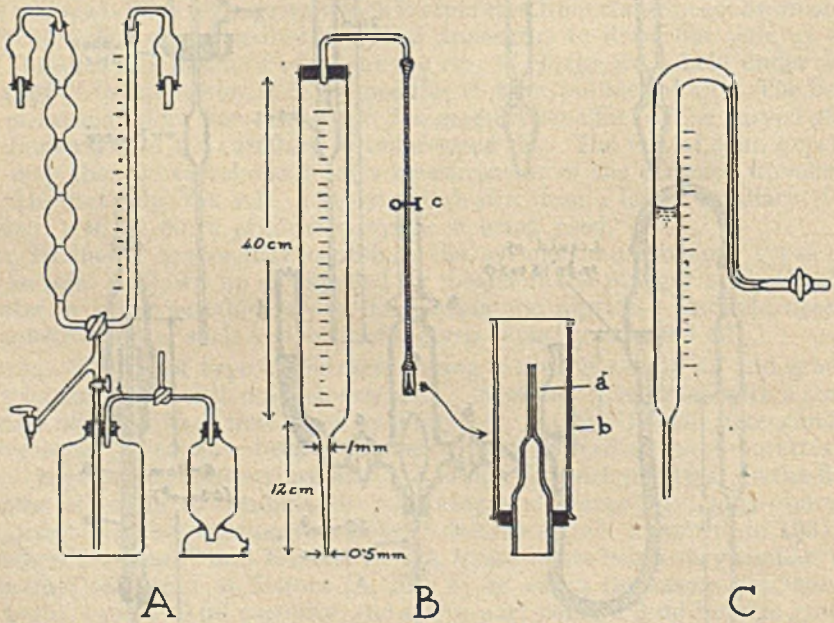


Fig. 4

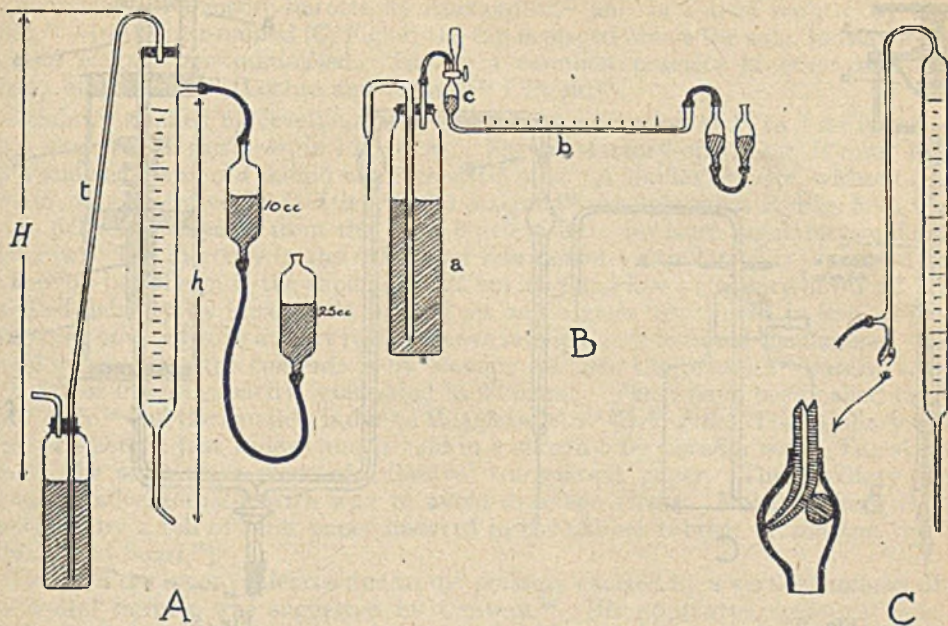


Fig. 5

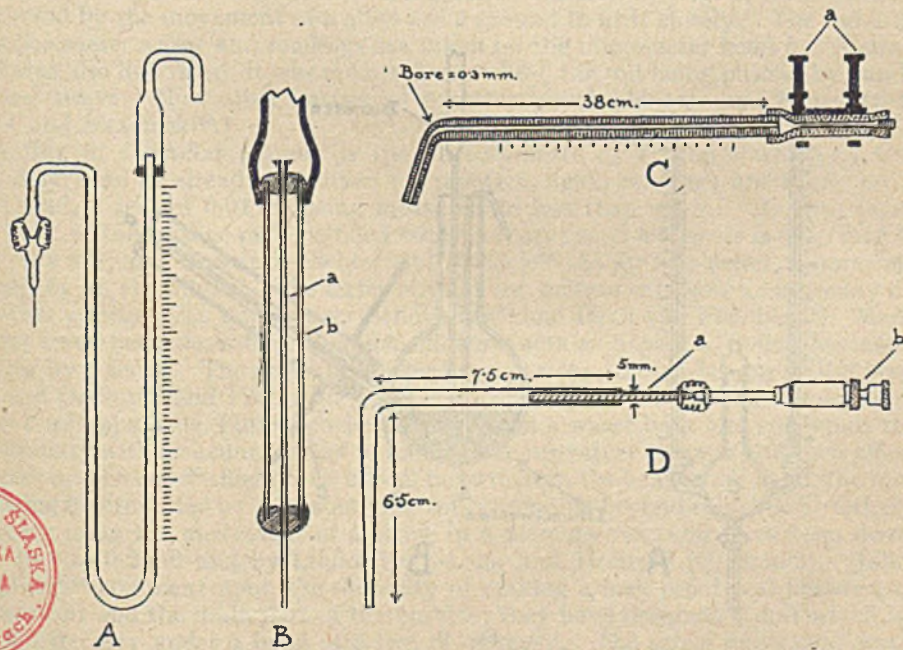


Fig. 6



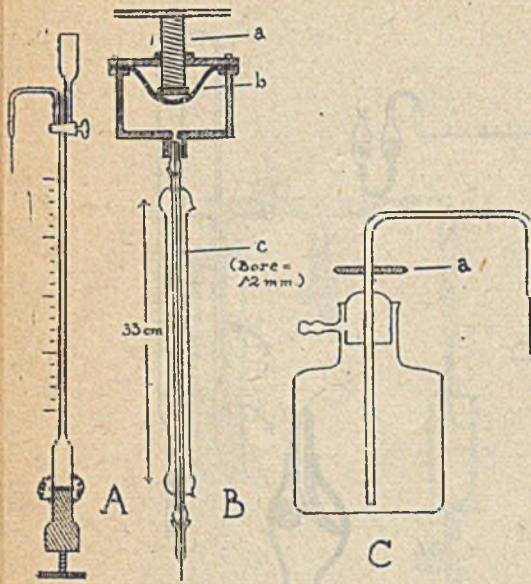


Fig. 7

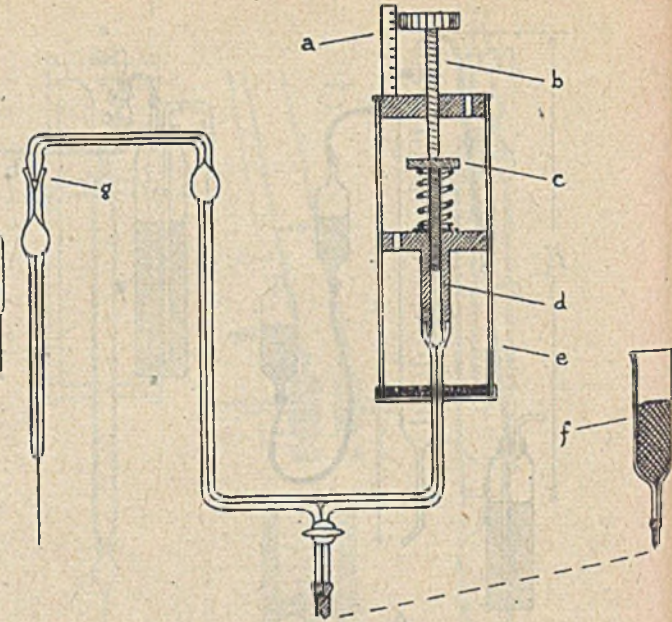


Fig. 8

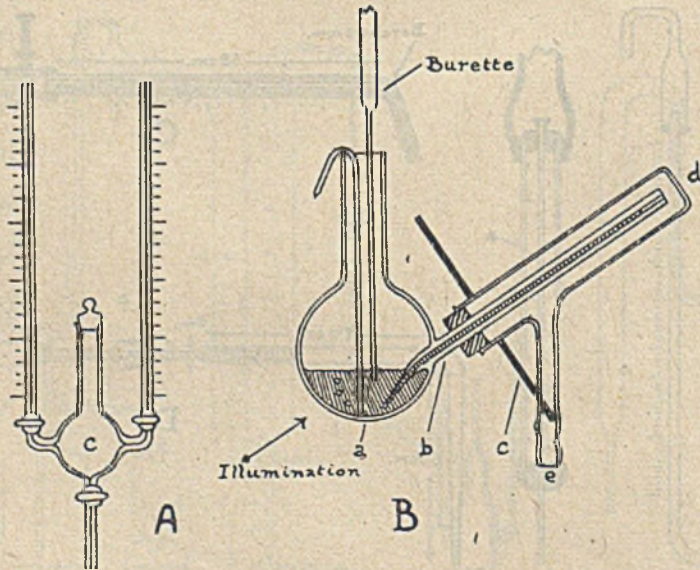


Fig. 9

out to a capillary of diam. *ca.* 1 mm, and this in turn is drawn to a finer capillary (0.01 to 0.03 mm), the length of which is adjusted to give a suitable rate of flow from the burette on opening the pinch-cock *c*. The capillary brake was devised by Mohr²⁶ in 1855 and had been embodied in a macro-burette by Alexandroff²⁷ and in a 1-ml burette by Pauli and Semler.²⁸ In the last-named (C, Fig. 4) the tap is placed above the soln. so that contamination and leakage are minimised. This is a common practice of other micro-burettes differing only in detail (Lochte and Hoover,²⁹ Ormont³⁰).

Schilow³¹ applied his levelling device to the manipulation of 1- to 3-ml burettes fitted with a stock bottle as shown in Fig. 5 (A). For satisfactory operation, *H* must be greater than *h* and the diam. of *l* should not exceed 1.5 mm. A similar burette, without a solution reservoir, was described by Heatley,³² who designed³³ the apparatus B, Fig. 5, in which the soln. is delivered directly from the 2-oz. bottle *a* by a pressure regulator similar to that of Schilow. The mercury in the calibrated thermometer capillary *b* is separated from the reservoir by paraffin in *c*; the capillary is 25 cm long and has a capacity of 0.1 ml. Fuchs³⁴ controlled delivery by means of a hooked jet and a glass bead fused to it (C, Fig. 5), the whole being covered with a short rubber sleeve manipulated between the fingers. A simpler method of expelling the contents is by blowing, as with the Schwarz³⁵ burette (A, Fig. 6) of 0.1, 0.2 or 0.5-ml capacity, graduated in 0.001 ml. These have been called the "wash-bottle" type,³⁶ and the smallest is due to Wigglesworth¹⁹ (B, Fig. 6). The capillary *a* is cut at the point where it just widens and is held in a sheath *b* by paraffin wax. The sheath also contains the scale on a piece of adhesive, transparent paper. The capillary is coated internally and externally with wax to avoid drainage errors. Moisture from the breath is absorbed by a roll of filter paper inserted in the rubber tubing. (Compare the burette of Llacer and Sozzi.³⁶)

To avoid the inconveniences due to the pressure exerted by a vertical column of liquid, a horizontal burette was suggested by Conway.³⁸ His apparatus, however, was rather complex, and a simpler form has been described by Holt and Callow³⁹ (C, Fig. 6), who expel the soln. by compression of a short piece of rubber tubing by means of two screw clips *a*. (This method was also used in a 1-ml vertical model by Ogg, Johns, Hoecker and Hammer⁴⁰). Hadfield²⁰ also devised a horizontal burette, D, Fig. 6, from which the soln. is delivered by the movement of a glass rod *a* ground to fit it closely. The rod is advanced by a micrometer screw and readings are taken on the micrometer head *b*. A simpler 3-ml model was also described; it was mounted vertically, the rod being pushed by hand through a rubber sleeve. (For other horizontal burettes see Hybbinette and Benedetti-Pichler⁴¹; Hawes and Skavinski⁸⁵.)

Acting in a similar manner is the micro-burette of Trevan,¹² which incorporates a medical syringe, as already described for pipettes; again readings are taken on a micrometer head, a vol. of 0.01 ml being measured to less than $\pm 1\%$. As with pipettes, the main body of the syringe may be filled with mercury; such a burette is described by Kirk⁴⁴ (0.1 to 0.2 ml), and Benedetti-Pichler and Rachele¹⁹ use air-free water, separating it from the soln. by an air bubble. The latter authors use minute capillaries, as already described. (For other syringe-type burettes see Scholandar⁴⁶ and Dean and Fletcher.⁶⁷) Many micro-burettes have been described in which mercury acts as a piston, being displaced from a reservoir by a screw. The best known, perhaps, are the similar designs of Rehberg,⁴² Widmark and Ørskov,⁴⁵ and Pincussen⁴³; the last (capacity 0.1 ml) is shown at A, Fig. 7. In a similar 3-ml apparatus, Links⁴⁴ encloses the soln. in a water-bath and surrounds the graduated capillary with a vacuum jacket to minimise temperature changes and their effects, while Widmark and Ørskov⁴⁵ shield their 0.2-ml burette from the heat of the hand, the micrometer head being manipulated by a "distant control" extending beyond this shield. Other burettes depending upon the movement of a screw in a mercury reservoir have been described by Kirk⁴⁴ (0.1 to 0.2 ml) and by Linderstrøm-Lang and Holter^{5,9} (60 cb.mm). Johnson and Shrewsbury⁴⁶ comment upon the difficulty of making a leak-proof seal between the screw and reservoir and the main part of the burette; they have designed a unit which is claimed to be satisfactory under a head of 5 feet of mercury. The screw movement was used by Krogh⁴⁷ for the control of fluids in gas analysis; the idea of a screw-controlled syringe appears to have been due to Lister in bacterial research and to Kelvin for the constant pressure gas thermometer (see Trevan¹²).

Attention is drawn to the burette of Gorbach⁴⁸ (see B, Fig. 7), which has the advantages of screw control without the difficulties involved in the presence of a second liquid,

such as mercury or water. It is essentially a greatly refined form of Schilow's "pneumatic control."⁴⁹ The screw *a* presses against a rubber diaphragm *b* which controls the movement of the soln. in the graduated tube *c* (enclosed in a vacuum jacket). By using interchangeable ground joints, one screw mechanism is made to control a number of burettes. For my own work I am using a micro-burette based on the pipette of Rosebury and Heyningen.¹⁷ A spring-loaded piston *c* (Fig. 8) moves in the tube *d* and is controlled by the micrometer screw *b*, readings being taken on the micrometer head *a*. The reservoir of mercury *f* is for ease in filling, and jets may be attached by interchangeable joints at *g*; hence the instrument is readily cleaned and only one calibration is necessary for all jets. Although a total vol. of ca. 0.75 ml can be delivered, smaller vols. may be measured to ca. 0.0001 ml; leakage is prevented by filling the casing *e* with mercury.

Finally, a number of micro-analysts prefer the so-called gravimetric or "weight" burettes which are weighed before and after the titration to obtain the titre. It will be appreciated that for such an instrument a normal soln. will contain equiv. of active agent dissolved in 1 kg of soln., not in 1 litre. The neatest vessel is that of Mika⁵⁰ (C, Fig. 7), which holds 30 ml, *i.e.*, sufficient for about 6 titrations. Delivery from the jet is obtained if the ground joint is held down by the glass lugs *a* during the blowing through rubber tubing attached to the side-arm. (Compare Schmitt,⁵¹ Geilmann and Höltje,⁵² and Ormont³⁰.) Szebelledy and Clauder⁵³ use a syringe burette as a weight burette.

It should be noted that the drops falling from a burette jet have a diam. greater than that of the jet if the soln. "wets" the material of which it is made. To avoid this uncertainty of drop-size, some workers^{24,25,29,30} grease the tip, whilst others^{21,30,38,52} remove fractions of a drop on a fine rod, with which the soln. is stirred. It is customary in more modern instruments to use a fine capillary jet which is kept below the surface of the liquid being titrated—a practice which seems to have been first applied by Linderstrøm-Lang and Holter.^{5,9} In some circumstances^{9,14,20,32,48,55} it is then necessary to be able to raise and lower the titration vessel with respect to the burette. (The idea of having a detachable jet for ease of cleaning originated with Rehberg.⁴²)

TITRATION VESSELS—The titration vessels recommended include small test-tubes,⁴² weighing bottles,^{7,30} and microscope slides.^{10,14,19} Pilch,⁵⁴ in an early micro-burette (A, Fig. 9), included a titration chamber *c* in the design, and Benedetti-Pichler and Siggia⁵⁵ describe a special vessel (B, Fig. 9) which is very useful for the accurate observation of the end-point. It consists of a small bulb *a* with a sealed-in capillary *b*, which is viewed endwise from *d*, so that a good depth of colour is seen. This viewpoint is shaded from direct light by the screen *c*. When the colour change is observed, the soln. in the capillary, which has not been titrated, is forced into the bulb by blowing *via e*, and the end-point is approached again cautiously; the process may be repeated. Photoelectric observation of the end-point is discussed from theoretical and practical aspects by Müller,⁵⁶ who gives references to earlier literature. The method appears to be sound, but somewhat tedious for routine operations. (Other aids are suggested by Alber and Harand.⁵⁷) Potentiometric micro-titration has been carried out successfully by Schwarz,⁵⁸ and Cunningham, Kirk and Brooks⁵⁹ (see also ^{14,29} and a review by Furman⁶⁰).

Stirring of the solution is nearly always effected by means of a stream of air or inert gas bubbles from a capillary (*cf.* B, Fig. 9)—a method apparently due to Pollak.⁶¹ Wigglesworth¹⁰ stirs a drop on a microscope slide by causing it to rotate rapidly in a tangential jet of air. Electromagnetic stirring devices have also been advocated.^{9,14}

For the methods of calibration of the various burettes and pipettes and the errors inherent in their use, reference may be made to the literature, which also contains numerous papers on individual micro-volumetric determinations.

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Notes

THE DETERMINATION OF SULPHUR DIOXIDE IN GELATIN

THE published methods stipulate soaking the gelatin until swollen in water containing acid and then removing the sulphur dioxide from the boiling liquid in a current of carbon dioxide, oxidising it in the distillate, and determining it either volumetrically (iodimetric or acidimetric) or gravimetrically (BaSO_4). The soaking of the gelatin and the slow distillation add considerably to the time needed for the determination. In industrial laboratories where many samples may have to be tested in a day there is need for a simple, rapid and accurate method. The following method has been found to meet these requirements. It depends for its success on (1) rapid heating of the liquid to b.p. by a current of steam; (2) rapid expulsion of air from the flask by the steam; (3) the fact that solution of the gelatin is delayed until the liquid is at, or nearly at, b.p., when the air will have been swept from the flask by the steam. Thus the sulphur dioxide is held by the gelatin until the latter begins to dissolve at the boiling-point.

METHOD—Reagents—Sulphuric acid: 50 ml of H_2SO_4 (sp. gr. 1.84) diluted to 250 ml with water; hydrogen peroxide: 10 vol. strength; sodium hydroxide, $N/20$; indicator: 1 g of methyl orange and 1.4 g of Xylene Cyanol FF. dissolved in 500 ml of 50% alcohol.

Apparatus—A special flask (500 ml), round-bottomed, with long neck bent at a right angle and hole in bulb, with fittings (see Fig. 1); double surface condenser (10 in.); a 10-ml burette reading to 0.05 ml; a conical flask (250 ml) as receiver.

Procedure—Place 75 ml of water in the flask without wetting the neck, add 20 g of the gelatin (broken sheet or powder) followed by 25 ml of the dil. sulphuric acid, and swirl. Fit the flask to the condenser, put 20 ml of hydrogen peroxide (neutralised, if necessary, to the indicator) in the receiver, and adjust the receiver so that the end of the adapter is covered by the liquid. Immediately pass a rapid current of steam into the flask and collect 100 ml of distillate within 10 min., assisting the distillation with heating the flask with a small flame. Titrate the distillate with $N/20$ sodium hydroxide, using the indicator recommended, to a neutral-grey end-point (1 ml \equiv 0.0016 g of SO_2 or 80 p.p.m.)

NOTES—(1) Sulphuric acid is not carried over by entrainment with the steam.

(2) Addition of sulphuric acid in excess of the quantity stated gives slightly high results, possibly owing to slight charring of the organic matter, with consequent reduction of the H_2SO_4 to SO_2 .

(3) The use of phosphoric acid in place of sulphuric acid yields low results.

(4) Hydrochloric acid is unsuitable, as it is carried over either volumetrically or gravimetrically; if volumetrically, the receiver must be stoppered and fitted with a trap containing glass-wool moistened with potassium iodide soln.

(5) The end-point can be judged with an accuracy of at least 0.1 ml. This corresponds with 8 p.p.m. of sulphur dioxide.

(6) Results agree well with those obtained by the gravimetric method in which hydrochloric acid and carbon dioxide are used.

(7) The method is satisfactory for gelatin in sheet or powder form.

(8) It is important to bend the end of the steam delivery tube so that the flow of steam into the liquid is directed obliquely against the walls of the flask. This gives a rotatory motion to the liquid, reduces frothing and prevents splashing.

The special " SO_2 flask" may be obtained from Messrs. Stanley Belcher & Mason, Ltd.

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A. C. FRANCIS
A. J. PILGRIM

OXIDATION OF SULPHUR COMPOUNDS WITH IODINE IN ALKALINE SOLUTION

ALTHOUGH the oxidising power of hypiodites has long been known, their use for the volumetric determination of sulphur compounds does not seem to have been recently investigated. Methods requiring back-titration have been described for sugars (Hinton and Macara, *ANALYST*, 1924, 49, 2), formaldehyde (Romijn, *Abst. ANALYST*, 1897, 22, 221) and thiourea (Cuthill and Atkins, *J. Soc. Chem. Ind.*, 1937, 56, 5r), but inorganic sulphides are usually determined with iodine in acid solution, when $S'' \equiv 2I$. The advantages of using iodine in alkaline solution are that $S'' \equiv 8I$, that the titration of alkali soluble sulphides is possible, and that the reagent, iodine in potassium iodide, is stable compared with hypochlorites and hypobromites.

ARSENIC—Oxidation of As_2S_3 to arsenate and sulphate occurs in strongly alkaline soln. (above about 4 N), so that $\text{As} = 14I$. Methods involving back-titration necessitate acidification of the reaction mixture and the heat developed tends to cause loss of iodine. This is accentuated by the effervescence upon subsequent addition of sodium bicarbonate, whilst sodium acetate gives variable results. After a large number of oxidation-reduction indicators and dyestuffs of various types had been unsuccessfully tested, I found that a 0.02% soln. of alizarin or quinalizarin in dilute sodium hydroxide soln. could be used as an internal indicator permitting direct titration. As an alkaline quinalizarin soln. is soon decolorised in air, alizarin is to be preferred.

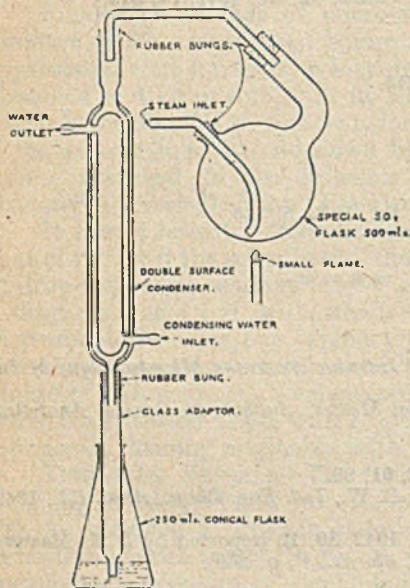


Fig. 1

Method—Ppt. As_2S_3 from acid arsenite soln. by brief passage of hydrogen sulphide. Add paper pulp to coagulate any colloidal sulphide, filter through a Gooch crucible with paper-pulp pad, and wash well with dil. hydrochloric acid. Blow the pad into a beaker, add 10 ml of 10% sodium hydroxide soln., swirl to break up the pulp and add 10 ml of 50% w/v sodium hydroxide soln., shaking thoroughly. (The pulp swells and appears to dissolve and, if shaking is not thorough, As_2S_3 may be occluded within a mass of swollen alkali cellulose.) Add 1 ml of indicator and titrate with iodine soln. (*N/10*, i.e., 12.7 g of I and 20 g of KI in 1 litre) until the soln. is pale yellow. It is advisable to check the end-point by adding a further ml of indicator; not more than 2 drops of iodine should be required for decolorisation.

Antimony may be similarly determined. 1 ml *N/10* iodine \equiv 0.000535 g As or 0.000870 g Sb.

	Arsenic	Antimony
Taken ..	6.64, 3.50, 1.66 mg	17.40, 8.70 mg
Found ..	6.64, 3.50, 1.66 \pm 0.05 mg	17.40, 8.70 \pm 0.08 mg

MOLYBDENUM—The sulphide MoS_3 was pptd. from alkaline molybdate by passing hydrogen sulphide, acidifying, and heating. As the ppt. was not readily sol. in dil. sodium hydroxide soln., asbestos was substituted for paper pulp and 50% sodium hydroxide soln. was used to effect solution. In other respects the method is the same as for As (1 ml of *N/10* iodine \equiv 0.000400 g of Mo).

ALKALI SULPHIDES AND THIOSULPHATES are oxidised to sulphates.

Method—To 20 ml of soln. add 10 ml of 50% w/v sodium hydroxide soln., and 1 ml of indicator. Titrate with *N/10* iodine until pale yellow. A turbid solution indicates incomplete oxidation and insufficient alkali.

1 ml of *N/10* iodine \equiv 0.00140 g of $S_2O_3^{2-}$ or 0.000400 g of S^{2-} .

As S^{2-} and $S_2O_3^{2-}$ require 2 and 1 atoms of iodine respectively in acid soln., and 8 each in alkaline soln., the method may be of use in the analysis of mixtures of sulphur acids.

UREA, FORMALDEHYDE, THIOUREA—The reaction was too slow, even with hypobromite, to permit direct titration of the first two, but the use of an internal indicator was possible with thiourea. A strongly alkaline soln. gave low results, and even at the alkalinity recommended by Cuthill and Atkins the direct titration method tended to give rather low results. Theoretically, 1 mol. of thiourea \equiv 8 atoms of iodine.

USE OF ALIZARIN WITH OTHER HYPOHALITES—Titrations with hypochlorites and hypobromites were not very satisfactory. It was, however, observed that, although hypochlorites alone had no action on the indicator, decolorisation was immediate in presence of an iodide.

HARRIS TECHNICAL COLLEGE
CORPORATION STREET, PRESTON

H. F. FROST
November 24, 1943

A MICRO METHOD FOR THE ESTIMATION OF TANNIN

(Read at the Meeting on February 2, 1944)

IN a recent communication a new qualitative test for tannin was described in which filter-paper soaked in gelatin was used.¹ This qualitative test gave good results, and has now been developed into a quantitative method in connection with a biological study in progress. By this new method it is possible to estimate tannin in *single* leaves, such as young rose-leaves. The results so far obtained are interesting, as they show the effect of light and temperature on tannin-metabolism; they support, in the main, the admirable study of Kraus,² which, however, suffered from the disadvantage that large quantities of leaves were required for each analysis. The botanical data are to be published elsewhere.

METHOD—Soak strips of thin white cardboard (5 cm \times 2 cm) prepared from postcards, for several hours in a 1% soln. of white gelatin, dry and soak again for 24 hr. in solns. containing from 0.25 to 0.1 mg of Mitchell's glucose-free gallotannin.³ Dry these strips and immerse them for 24 hr. in a 1% soln. of anhydrous ferric ammonium oxalate. When they are perfectly dry, mount these coloured strips on a glass plate, cover them with a second glass plate and seal the plates together. They serve as analytical standards for matching and should be kept in the dark when not in use. A set of 12 such strips with a wide range of tannin content was used in my expts.

For the analysis, dry the individual leaves at 100° C., powder and extract them for 1 hr. in a Soxhlet extractor with a mixture of chloroform and carbon tetrachloride (1:1) (to extract chlorophyll, waxes, etc.). Evaporate adhering solvents, and extract the leaf-powder with boiling water for 25 to 30 min. in a Soxhlet apparatus. Immerse strips of cardboard (prepared and treated with gelatin, as described above) for 24 hr. in the cold soln., dry and stain for 24 hr. with a 1% soln. of anhydrous ferric ammonium oxalate (oxalate is used to avoid the introduction of sulphuric acid). Dry the strips and match them against the standards.

In expts. with aba-turug bark, algarobilla, babool bark, fir bark, willow bark, mangrove bark, oak bark, pistacia, sumach, teripods, valonia, tea, jak-bark, *Aleurites Fordii* bark, Paraguay tea (*Ilex paraguensis*, Don.) a guava-bark and barbatimao bark the results agreed fairly well with the data recorded by Harvey⁴ and by Greenway,⁵ which were obtained by the official hide-powder method.

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THE UNIVERSITY
BRISTOL

M. NIERENSTEIN
January, 1944

DISCUSSION

Dr. C. A. MITCHELL, who, in the absence of the author, gave an outline of the method, said that the new test, which combined the fixation of tannin by gelatin and its colorimetric estimation after fixation, promised to be very useful in biochemical work. Its weak point was that the standards for colorimetric comparison had been based on a natural tannin which was not generally available. To overcome this drawback, he suggested that any tannin selected as a standard should itself be standardised on the lines described in the following note.

STANDARDISATION OF THE TANNIN USED FOR COMPARISON IN NIERENSTEIN'S MICRO METHOD OF ESTIMATING TANNIN

THE natural tannin chosen by Nierenstein as a standard is the remarkable specimen (derived from Chinese galls) that I discovered some 20 years ago and described to the Society.¹ When dried at 100° C. its composition is: water, 1.2; gallic acid, 10.5; gallotannin, 88.3%. When hydrolysed on the water-bath for 54 hours with 5% sulphuric acid it yielded less than 0.5% of glucose and when treated for 8 days with *Penicillium* sp. the hydrolysate had an optical rotation of only $\alpha_D = +0.2^\circ$. In comparative tests with this tannin in the air-dry condition by the hide-powder method and by Chapman's cinchonine pptn. method Hooper² found 77.8% of tannin by the former and 76.8% by the latter method. Basing the calculation on the proportion of pyrogallic groups I found that its composition agreed most closely with the "long-chain" formula of Nierenstein's polydigalloyl-leuco-digallic anhydride,³ omitting the glucose. Schiff's formula,⁴ $C_{14}H_{10}O_2 \cdot 2H_2O$, adopted by Chapman⁵ for calculating the composition of his cinchonine ppts., is obviously not correct, for no natural tannin has been found with a molec. weight of less than 600. Again, although Fischer's synthetic tannin, pentadigalloyl glucose,⁶ has many of the characteristics of natural tannins, it differs from them in some of its chemical reactions. It is evident that natural tannins are not uniform products, and all the later evidence tends to support the view that commercial "pure" tannins are usually mixtures of different glycosides, but mainly di-digalloyl glycoside, with a digallic anhydride of the type described by Nierenstein.

The only component in the constitution of natural tannins that can be looked upon as fundamental is the pyrogallic grouping, and it should be possible to standardise tannins on that basis. Since Nierenstein has made my exceptional glucose-free tannin the standard for his new test, the simplest plan would be to standardise other tannins against it. This could be done by estimating the total pyrogallic groups in terms of gallic acid, determining and deducting the pyrogallic groups due to the gallic acid, and multiplying the difference by the empirical factor 2.2 (*cf.* Mitchell, *loc. cit.*).

Another method of making the Nierenstein standards generally available would be to use Lovibond glasses corresponding with the respective colours in the scale. The standards sent to me by Dr. Nierenstein range from 1 mg to 0.25 mg of glucose-free tannin. The colours of these standards correspond with the following Lovibond units determined by means of Osborn's comparison microscope.

	1 mg	0.75 mg	0.5 mg	0.25 mg
Red.. ..	2.0	1.5	1.1	0.1
Blue.. ..	1.0	0.8	0.5	—
Yellow ..	2.5	1.8	1.4	0.6

The reagent (ferric ammonium oxalate) gives no colour with 1 mg of gallic acid or pyrogallol.

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THE CLOSE, WEEDON
AYLESBURY, BUCKS.

C. AINSWORTH MITCHELL
February, 1944

EYE INFLAMMATION AS THE ONLY SYMPTOM OF INCIPIENT HYDROGEN SULPHIDE POISONING

I WAS recently asked to investigate an outbreak of eye trouble in a tannery. The only symptom appeared to be a painful soreness of the eyes with severe photophobia and tears which "burned the cheeks." There was no history of any respiratory or other trouble, and at the Eye Hospital where the men were treated it was suggested that their eyes had been splashed with acid. Their occupation, however, precluded this, and I therefore enquired whether there had been exposure to formalin, sulphur dioxide or other obvious eye irritants, but there had been none.

Reference to Legge's "*Industrial Maladies*" suggested that hydrogen sulphide might be the cause. (Sulphides were being used in an adjoining building.) As the D.S.I.R. booklet with colour chart for estimating hydrogen sulphide was out of print, a pump was improvised to give qualitative tests. This was found to be unnecessary; a piece of lead acetate paper held stationary in the hand where the men were working darkened as it was watched (without any aspiration) at a rate reminding one of the darkening of exposed printing-out paper in bright sunlight. It seems probable that the men's freedom from more serious symptoms was due to the building in which they worked being partly open to the air. Steps were at once taken to prevent the permeation of the hydrogen sulphide, whereupon the trouble disappeared and has not recurred.

This note is submitted because eye trouble, such as that described, may be a timely warning of possible serious or fatal poisoning, and it does not seem to be sufficiently recognised as an early symptom of hydrogen sulphide poisoning.

THE LABORATORY
4, QUEEN SQUARE, BRISTOL

H. S. HOWES
December, 1943

Ministry of Food

STATUTORY RULES AND ORDERS*

1944—No. 1. **The Flour Order, 1944**, dated January 1, 1944. Price 6d.

This Order is, in the main, a consolidating Order, replacing the Flour Order, 1943 (S.R. & O., No. 11) and three amending Orders (S.R. & O., 1943, Nos. 625, 797 and 1275). *The Order contains minor alterations relating to transport and prices.*

— No. 69. **The Saccharin (Control and Maximum Prices) Order, 1944**. Dated January 20 1944. Price 2d.

This Order consolidates the Saccharin (Control and Maximum Prices) Order, 1942 (S.R. & O., 1942, No. 2455, as amended by 1943, No. 669), and introduces some amendments.

The 4 principal alterations are: (1) *Tolerances for the saccharin and dulcin contents of sweetening tablets are introduced. The saccharin content must be not less than 0.081 grain and not more than 0.099 grain and the dulcin content not less than 0.054 grain and not more than 0.066 grain.* (2) *The regulation prohibiting the retail purchase or sale of saccharin soln., saccharin powder and soluble saccharin does not apply to authorised sellers of poisons.* (3) *Retailers of saccharin tablets or sweetening tablets must keep detailed records of every purchase of such tablets. Retention of an invoice will meet the requirement. The record must be kept for a year from the date of the transaction.* (4) *The prohibition on the manufacture or preparation by way of trade of any product containing saccharin tablets or sweetening tablets does not apply to a caterer using the tablets in the preparation of food or drink served by him in the course of his catering business.*

The British Pharmacopoeia, 1932: Alterations and Amendments

We have been requested to publish the following amendments to the Schedule.

ointments—(see Sixth Addendum to the British Pharmacopoeia, 1932, page iv, Notice Concerning Ointments). The period during which ointments prepared according to the original formulae of the British Pharmacopoeia, 1932, or Addenda, may be dispensed or supplied is extended to include the six months ending August 1st, 1944.

EXTRACTUM BELLADONNAE FOLII LIQUIDUM—Liquid Extract of Belladonna Leaf contains 0.75% w/v of the alkaloids of Belladonna Leaf, calculated as hyoscyamine (limits, 0.70 to 0.80).

Belladonna Leaf, in moderately coarse powder, 1000 g; Alcohol (95%) and distilled water, of each a sufficient quantity.

Exhaust the Belladonna Leaf by percolation with a mixture of 5 vols. of alcohol (95%) and 1 vol. of distilled water, reserving the first 200 ml. Remove the alcohol from the remainder of the percolate, at a temperature not exceeding 60° C., add the reserved liquid, remove the alcohol from the mixture at a temp. not exceeding 60°, and adjust the vol. of the residual liquid to 1250 ml either by concentration at a temp. not exceeding 60° or by addition of distilled water. Set aside at a temp. of about 2° for two days; filter through a small filter and thoroughly wash the residue and filter with distilled water. Evaporate the filtrate and washings to 300 ml at a temp. not exceeding 60°, and add 100 ml of alcohol (95%). Determine the proportion of alkaloids in the liquid, thus obtained by the assay described below. To the remainder of the liquid add sufficient of a mixture of 14 vol. of distilled water and 5 vol. of alcohol (95%) to produce a Liquid Extract of Belladonna Leaf of the required strength. Set aside for not less than 12 hr.; filter, if necessary.

Assay—Carry out the assay described under "Extractum Belladonnae Liquidum."

Doses—(Metric) 0.015 to 0.06; (Imperial) $\frac{1}{4}$ to 1 minim.

EXTRACTUM BELLADONNAE LIQUIDUM (Liquid Extract of Belladonna)—When Liquid Extract of Belladonna is prescribed, or demanded, Liquid Extract of Belladonna Leaf may be dispensed, or supplied.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS

Food and Drugs

Ash Determinations in Foods with an Alkaline Balance: Reactions of Alkali Carbonates with Calcium Phosphates. H. J. Wichmann (*J. Assoc. Off. Agr. Chem.*, 1943, 26, 522-559)—The reactions of monocalcium phosphate, dicalcium phosphate and pptd. basic calcium phosphate with sodium and potassium carbonates, respectively, were investigated. Monocalcium phosphate (0.2 g) was dissolved in water in a platinum dish, sodium carbonate solns. of known content were added, and the mixture was evaporated on the steam-bath. The residue was heated for 15-min. intervals at increasing temp., usually from 200° C. to 800° C., in a controlled muffle furnace. The residue was treated with 50 ml of water, and the mixture was heated to b.p. for 5 min. during which the insol.

residue was crushed and disintegrated by means of a rubber-tipped glass rod. The mixture was further heated in a 100-ml flask for 10 min., after which it was cooled, diluted to 100 ml and filtered. Water-sol. phosphate was determined in the filtrate by the volumetric molybdate method. The insol. residue was washed free from sol. matter, ignited with the filter-paper, first at 550° C. and finally for 10 min. at 800° C., and weighed. The residue was dissolved in dil. hydrochloric acid, and the soln. was made up to 100 ml. Calcium oxide, sodium oxide and phosphorus pentoxide were determined in aliquot portions of this soln. The method was applied to mixtures of the other two phosphates of calcium with sodium carbonate, and, with some slight modifications, the series of expts. was repeated with potassium carbonate in place of sodium carbonate. The investigation showed that alkali

* Obtainable from H.M. Stationery Office. Italics signify changed wording.

carbonates can react with calcium phosphates, in both the wet and the dry states, to form different compounds the composition of which depends principally on the carbonate/phosphate ratio and the temp. of ashing. The wt. of residue or total ash varies similarly to some degree, but the composition of the ash, and particularly of the insol. ash, is more definitely influenced. Soluble phosphates are produced in amounts varying with the temp. and type of reaction. The insol. ash always contains sodium if sodium carbonate is one of the reactants, but may or may not contain potassium in analogous potassium carbonate reactions. If the carbonate/phosphate ratio is low, the insol. ash consists of insol. double calcium-alkali phosphates, double phosphates containing carbon dioxide (rhenanites) or mixtures of these, and these carbonated double phosphates do not lose their carbon dioxide appreciably at any temp. up to 800° C. Increase of the carbonate/phosphate ratio and increase of temp. tend progressively to force the reaction towards the hydroxyapatite equilibrium with decrease in water-sol. alkalis. In presence of sodium carbonate the hydroxyapatites contain sodium, but the corresponding potassium compounds are less stable. Insol. carbonated hydroxyapatites tend to lose carbon dioxide between 500° and 800° C. When sodium or potassium enters the hydroxyapatite molecule, free calcium carbonate appears in the insol. ash, and is decomposed at 600°-700° C. Excess of alkali carbonate and high temp. (800° C.) decompose hydroxyapatites and release water-sol. phosphate. A similar study of the magnesium phosphates is in progress. A. O. J.

Detection of Olive Oil in Edible Oil Mixtures.

J. Fitelson (*J. Assoc. Off. Agr. Chem.*, 1943, 26, 499-506)—Squalene is concentrated in a fraction obtained by selective adsorption treatment of the unsap. matter and is determined by halogen absorption. The adsorption column is prepared in the following manner—Place a small wad of cotton wool in the constricted end of a glass tube 0.8 cm inside diam. and 30 cm long. Add chromatographic adsorption alumina (80-200 mesh) in ca. 10 portions to form a column ca. 10 cm high. Compress each portion lightly with a flattened glass rod, applying gentle suction, place a small wad of cotton wool on top of the column and wash with light petroleum. Keep the top of the column covered with light petroleum until ready for use. *Method*—Saponify ca. 5 g of the oil with 3 ml of conc. potassium hydroxide soln. (60 g in 40 ml of water) and 20 ml of 95% alcohol under reflux for 30 min. Extract the unsap. matter by the usual procedure, using light petroleum (b.p. 63°-70° C.) as solvent. In the final evaporation remove the last traces of solvent by warming in a current of carbon dioxide. Dissolve the unsap. matter in 5 ml of light petroleum and transfer the soln. to the adsorption column, using light petroleum to rinse in the last portions. Collect the filtrate in a glass-stoppered iodine absorption flask, applying gentle suction to regulate the emergence of the filtrate to ca. 1 ml per min. Continue to add solvent and always keep the top of the column covered until 50 ml of filtrate have been collected. Evaporate the filtrate, removing the last traces of solvent in an atm. of CO₂. Dissolve the residue in 5 ml of chloroform, add enough pyridine sulphate and bromine reagent (*infra*) to provide at least a 50% excess and leave the mixture in the dark for 5 min. Add 5 ml of 10% potassium iodide soln. and 40 ml of water.

Mix thoroughly, rinse iodine from the stopper and titrate with 0.05 N sodium thiosulphate, using starch indicator near the end-point. Correct the titration by means of a blank determination on the pyridine sulphate and bromine reagent. Each ml of 0.05 N sodium thiosulphate = 1.71 mg of squalene. To prepare the pyridine sulphate and bromine reagent, dissolve 8 g of bromine in 20 ml of glacial acetic acid and prepare another soln. by gradually adding 5.45 ml of conc. sulphuric acid to a cooled mixture of 20 ml of glacial acetic acid and 8.15 ml of pyridine. Mix the two solns. and dilute to 1 litre with glacial acetic acid. The squalene content of 44 samples of olive oil from 10 countries ranged from 136 to 708 mg per 100 g with an average of 330. Ordinary edible oils gave the following average figures (mg per 100 g)—cottonseed, 8; peanut, 28; corn, 28; soya bean, 11; sunflower, 13; tea seed, 12; sesame, 3; rape, 28. The higher squalene content of olive oil serves to distinguish it from other common edible oils, but the wide range prevents the use of these data as a diagnostic criterion for the purity of olive oil. In conjunction with the usual oil analysis the method should be of value in confirming the presence of small amounts of olive oil in many types of oil mixtures. (*Cf.* next abstract.) A. O. J.

Occurrence of Squalene in Natural Fats.

J. Fitelson (*J. Assoc. Off. Agr. Chem.*, 1943, 26, 506-511)—In the investigation of methods of determining squalene in olive oil (*cf.* preceding abstract) it was found that other vegetable oils contain small amounts of unsaturated matter resembling squalene. The presence of squalene was confirmed in these oils by the formation of characteristic squalene hexahydrochloride crystals from a fraction of the unsap. matter. Since squalene has been reported to be limited to relatively few oils, mainly those of certain marine animals, the unexpected presence of this compound in some vegetable oils led to the examination of a variety of natural fats. The fat (100 g) was saponified for 2 hr., as described in the preceding abstract, the vol. of reagents used being proportionally increased both in the saponification and in the subsequent extraction. Selective adsorption was carried out through an alumina column 35 cm high and 75 ml of filtrate were collected. After removal of solvent the procedure was as follows—Dissolve the residue in anhydrous ethyl ether and treat the soln. with dry hydrochloric acid gas for 1 hr. in a centrifuge tube immersed in ice. Remove the tube from the ice and maintain the stream of gas until the ether has evaporated. Add 1 ml of light petroleum (b.p., 35°-60° C.), heat to boiling, cool and place the tube in a refrigerator. Very small amounts of squalene hexahydrochloride may require 24-48 hr. for pptn. Centrifuge the ppt., decant the solvent and wash the residue with light petroleum. Dissolve the residue in a small vol. of hot acetone, cool, and place in a refrigerator until crystals appear (1-2 days for small amounts). Recrystallise, if necessary, and examine the crystals at ×100 for typical diamond or hexagonal plates of squalene hexahydrochloride. Squalene was found in the following fats in addition to those mentioned in the preceding abstract, the figures being the mg of squalene per 100 g by the method of the preceding abstract. Mustard, 7; rice bran, 332; grapesced, 7; almond, 21; coconut, 2; linseed, 4; butter, 7; cod-liver, 31; seal, 35; chicken, 4; lard, 3; beef, 10. Squalene was not found in cocoa butter. A. O. J.

Removal of Mustard Gas from Fats. H. C. Lockwood (*Chem. and Ind.*, Feb. 5, 1944, 50-51)—The mustard gas is decomposed by steam distillation, and the decomposition products are removed by washing with hot water. Melt 100 g of the fat contaminated with, e.g., 50 mg of mustard gas (ca. 1 lb. per ton), add 50 ml of warm water and steam distil for 30 min. Separate the lower aqueous layer, wash the fat with 2 lots of hot water, and separate. Combine the washings, titrate the thiodiglycol and compare the result with the amount of thiodiglycol in the distillate. Test the treated fat by the extraction and titrimetric method previously described (*ANALYST*, 1941, 66, 480). The following results give an indication of the amount of mustard gas volatilised before decomposition.

Material	Mustard gas p.p.m.	Thiodiglycol, %		Residual mustard gas
		in washings	in distillate	
Cocoa butter	539	89	11	nil
Biscuit fat	441	95	5	"
"	493	91	9	"
Butter	591	97	3	"
Margarine	418	94	6	"
Lard	620	95	5	"

These results show that ca. 10% of the mustard gas is volatilised, and an efficient condenser must therefore be used, especially when the method is applied on a large scale. Any volatilised mustard gas is hydrolysed to thiodiglycol in the condensate. Blank tests on the treated and untreated fat were seldom identical, but the differences were significant only with cocoa butter and biscuit fat, the pronounced yellow colour of which masks the end-point in the titration (*loc. cit.*). Cocoa butter has a somewhat rancid taste after the treatment and would require deodorising. The decontaminated fats are suitable for human consumption.

Component Fatty Acids of *Vateria Indica* Fat [Malabar Tallow]. C. Venkatarao and M. Narasingarao. (*J. Indian Chem. Soc.*, 1943, 20, 239-242; 298-300)—Seeds from *Vateria indica*, an evergreen tree found in Mysore, South Canara, Malabar and Travancore, yield on boiling with water about 20% of "Malabar tallow," a greenish-white edible fat which was formerly exported from Bangalore for use as chocolate fat. It is also suitable for yarn sizing and compares well with high-class hydrogenated fats and animal tallows. A sample contained 92.6% of insol. mixed fatty acids (60.7% solid, 39.3% liquid). They consisted of:—myristic, 0.7; palmitic, 13; stearic, 43.1; oleic, 42.5; arachidic, 0.4; linolic, 0.1; lower acids, 0.2%. The fat had: m.p., 36-37°C.; sp.gr. at 40.3°/33.5°C., 0.8989; n_D^{40} , 1.4578; sap. val., 191.1; iodine val. (Wijs), 36.72; unsap. matter, 0.588%; Hehner val., 92.6; acid val., 2.88. Iso-oleic acid, mentioned but not

confirmed by Puntambekar and Krishna (*J. Indian Chem. Soc.*, 1933, 10, 205) was not found. From the last fraction of the solid methyl esters of the fatty acids pure arachidic acid was isolated and identified, but the lignoceric acid mentioned by Puntambekar and Krishna (*loc. cit.*) was not detected. Linolic and myristic acids were probably not detected by Jones, Hilditch and Salotore (*J. Soc. Chem. Ind.*, 1931, 4681) owing to the small amount of fat at their disposal. The unsap. matter is mainly sitosterol with a trace of a new sterol the bromo-acetate of which melts at 120-122°C. to a dark-coloured liquid. Steam-distillation of the fat yields 0.05% of a terpenoid oil with iodine val. 148.

The glyceride structure of the fat, determined by the usual methods, was calculated to be tristearin, 2.5; oleostearin, 44.9; oleopalmitostearin, 16.6; oleodipalmitin, 7.1; dioleostearin, 15.8; dioleopalmitin, 13.0; triolein, 0.2%. This fat is one of the solid seed fats which follow even distribution of fatty acids having an association ratio on the total fatty acids of 1.3 to 1.6. Its glyceride structure shows other similarities with that of Borneotallow (*cf.* Hilditch and Priestman, *J. Soc. Chem. Ind.*, 1930, 49, 1977). E. B. D.

Determination of the Alpha Resin Content of Hops. F. Rabak (*J. Assoc. Off. Agr. Chem.*, 1943, 26, 481-485)—The alpha resin, the most important brewing resin in hops, is usually determined gravimetrically by pptn. of its lead salt from a methyl alcohol soln. of the soft resins by means of a 1% (by vol.) soln. of lead acetate in abs. methyl alcohol. The procedures recommended (*J.A.O.A.C.*, 1942, 25, 292) are: (a) preliminary titration, by means of a spot test of the amount of lead acetate soln. required for complete pptn. and (b) direct addition of 7 ml of the lead acetate soln. to ppt. the salt. Ford and Tait (*J. Inst. Brew.*, 1932, 38, 351) showed that the lead ppt. is somewhat soluble in excess of the lead acetate soln.; on the other hand, insufficient reagent means incomplete pptn. In applying the spot test to determine the right amount to be added great care is necessary not to transfer traces of the pptd. lead salt, together with the drop of supernatant liquid to the filter-paper, and thus give a false indication of an excess of the lead acetate soln. To obtain information as to the rate of pptn. and the solubility of the alpha lead salt in excess of the reagent, a series of tests was made on methyl alcohol solns. (representing 1 g of hops) of the soft resins from 6 different samples of hops, containing widely different proportions of both total soft and alpha resins, by using varying quantities of lead acetate solns. for the pptn. It was found that the reductions in the result due to the solubility of the alpha salt in 1 ml excess of the reagent were relatively small, ranging from 0.19 to 0.34%, but with each ml of further excess the loss was greater. The results are given in the following table.

Sample	Total soft resins %	With spot test		Without spot test										Reduction % caused by excess of Pb A soln.			
		Alpha resins, %	Lead acetate soln. ml											1 ml	2 ml	3 ml	4 ml
				3 ml	4 ml	5 ml	6 ml	7 ml	8 ml	9 ml	10 ml						
1	22.28	8.14	8.25	—	5.19	5.36	6.79	7.60	8.17	7.97	7.73	0.20	0.44	—	—		
2	18.14	6.50	7.25	—	4.61	5.50	6.51	6.62	6.33	6.03	5.71	0.29	0.59	0.91	—		
3	17.24	5.35	6.75	—	4.18	4.90	5.27	5.40	5.19	4.80	4.35	0.21	0.60	1.05	—		
4	15.60	4.78	6.50	—	4.26	4.57	4.95	4.61	4.26	3.96	3.68	0.34	0.69	0.99	1.27		
5	14.37	2.79	3.75	2.68	2.80	2.61	2.21	1.92	1.66	—	—	0.19	0.59	0.88	1.14		
6	13.18	1.74	3.50	1.71	1.80	1.51	1.05	0.89	0.69	—	—	0.29	0.75	0.91	1.11		

The results show that from 4 to 8 ml of lead acetate soln. was required for complete pptn. according to the alpha resin content of the particular sample. Of the 6 samples, Nos. 2, 3 and 4 may be regarded as hops of average composition (15.60 to 18.14% of soft resins); with these the use of 7 ml of lead acetate soln. gave results agreeing closely with those obtained by the spot test method (deviations +1.8, +0.9 and -3.5%* from the actual content). Sample 1, with exceptionally high soft resin content (22.28%), with a deficiency of lead acetate soln. differs by -6.6%*, whereas Nos. 5 and 6, which were unusually low in soft resins (14.37 and 13.18%), show much greater deviations (-31 and -48.4%)* when 7 ml of lead acetate soln. (an excess of 3 ml) were used for the pptn. The data in the above table enable an estimate to be made from the total soft resin content as to the correct quantity of lead acetate soln. to be used for the rapid determination of the alpha resin content by direct addition of the reagent.

Biochemical

Report of 1942/3 Methods of Analysis Sub-Committee on Riboflavin Assay. J. S. Andrews (*Cereal Chem.*, 1943, 20, 613-625; *J. Inst. Brew.*, 1944, 50, 51)—The results of 18 collaborators assaying 4 samples of cereal products—wheat flour, patent flour, patent flour enriched with 2.64 μ g per g, and bread made with the enriched flour—are tabulated and discussed. The methods used for comparison included a specified fluorimetric procedure, other fluorimetric methods, and modifications of Snell and Strong's biological method (*Ind. Eng. Chem., Anal. Ed.*, 1939, 11, 346). When the specified method was used notes were taken of the effects of acid extraction and sample size, and several of the results confirmed the author's observation that the use of small samples and of extraction with acid in place of water gave higher values. On the average, the microbiological results tended to be higher and more erratic than the fluorimetric results, but both methods are capable of giving satisfactory recoveries of riboflavin, though the cause of the discrepancies with the microbiological method in some laboratories is not yet clear. Several of the laboratories obtained nearly identical results with the wheat and patent flours, but widely differing results with the fortified flour and bread. Notwithstanding considerable variations in the results, the author considers that present procedures should not be condemned until it has been proved that the prescribed method has been followed exactly and has failed to give good recoveries.

Further Studies of Factors Influencing the A.O.A.C. Chick Method of Vitamin D Assay. I. Motzok and D. C. Hill (*J. Assoc. Off. Agr. Chem.*, 1943, 26, 516-521)—The effect of minor variations in procedure in the A.O.A.C. method for vitamin D assay by determination of the ash content of bones ("*Methods of Analysis*," 1940, 371) was studied. Storage of bones with freezing was compared with storage in ethanol, 69 pairs of right and left tibiae being used. The left tibiae were immediately freed from adhering tissue, extracted and ashed according to the recommended procedure. The corresponding right tibiae were stored unprepared at -12° C. After 12 days 36 bones were allowed to thaw, cleaned, extracted

and ashed, and after 54 days the remaining 33 bones were treated similarly. For the second method the left tibiae of 111 pairs were cleaned and stored in 95% ethanol for 24 hr. and were then extracted and ashed. The corresponding right tibiae were divided into 3 groups and stored for 24 hr., 1 week and 2 weeks respectively, the 1st group serving as a check expt. Freezing caused a significantly lower ash content, the difference being attributed to the less complete extraction of lipid material from bones that had been frozen. Prolonging storage to 54 days had little additional effect on the amount of ash. Preservation in ethanol up to 2 weeks had no effect on the % ash, and, when bones cannot be extracted immediately after dissection, storage in alcohol is preferable to freezing. The suggestion in the official method that crushing may facilitate lipid extraction was examined. With 157 pairs, the right tibiae were crushed (*i.e.*, broken in half transversely and the halves split longitudinally, each crushed bone being wrapped separately in filter-paper) before extraction, whilst the left tibiae were extracted whole. Extraction consisted in boiling the bones under reflux for 20 hr. in 95% ethanol followed by extraction with ether in a Soxhlet extractor for 20 hr. Crushing had no significant effect on the amount of lipid matter extracted either at low or high levels of calcification. To remove adhering tissue, the official method suggests immersion of the bones in boiling water for not more than 2 min. The right tibiae of 205 pairs, after dissection and removal of most of the tissue, were divided into 3 groups and immersed in boiling water for 1, 2 and 4 min. respectively, and the remainder of the tissue was removed. The corresponding left tibiae were completely freed from tissue without boiling-water treatment. Treatment with boiling water for 2 and 4 min. had a significant effect on the % ash, but treatment for 1 min. had no appreciable effect. It seems advisable to restrict boiling time to 1 min. or, if a long time is used, to control the time of boiling so that all bones receive identical treatment. Finally, the ashing time of 1 hr. at 850° C., as recommended in the official process, appeared adequate even for charges of 111 bones and 248 bones in large crucibles containing 15-20 bones each in a muffle of internal dimensions 7½ × 5½ × 14 in. After incineration at 850° C. for 1 hr. the ash was weighed and re-heated for 4 successive 30-min. periods at 850° C. The loss in wt. of the ash never exceeded 1%.

A. O. J.

B-Vitamins in Honey. G. Kitzes, H. A. Schuette and C. A. Elvehjem (*J. Nutrition*, 1943, 26, 241-250; *B.C.A.*, 1944, AIII, 45)—Samples of honey showed considerable variations in their vitamin B contents. The following values were recorded: pantothenic acid 0.55, riboflavin 0.26, nicotinic acid 1.1, thiamin 0.044, pyridoxine 0.10, biotin 0.00066, and folic acid 0.03 μ g per g. On ageing, the amount of pantothenic acid decreases. Pollen and royal jelly contained respectively: pantothenic acid, 27.0, 320.0; riboflavin, 16.7, 28.0; nicotinic acid, 100, 111; thiamin, 6, 18; pyridoxine, 9, 10.2; biotin, 0.25, 4.1; folic acid, 0, 0.5 μ per 100 g. The high proportions of biotin and pantothenic acid in royal jelly may be connected with the metabolism of the young bee.

Microscopical Properties of some of the Crystalline Water-soluble Vitamins. G. L. Keenan (*J. Assoc. Off. Agr. Chem.*, 1943, 26,

* % calcd. on the amount of α resin determined.

514-516)—The following optical data and micro-chemical tests are of value for the rapid identification of some water-sol. vitamins. The optical constants, including the refractive indices, were determined by the immersion method, which is often applicable to tablet material as well as to the pure substances. An intermediate refractive index (n_i) is found with nicotinic acid and nicotinamide. To prepare the gold bromide reagent, add 1.5 ml of 50% hydrobromic acid (or sat. sodium bromide soln.) to 1 g of gold chloride and dilute the soln. to 20 ml with hydrochloric acid. The test with silver nitrate is made by adding a small fragment of silver nitrate to an aqueous soln. of the vitamin on a microscope slide. The refractive indices of riboflavin and thiamin hydrochloride are difficult to determine by the immersion method.

tetrathionate. Chalk is of doubtful value as a buffer, as it inhibits the growth of *B. typhosum*, but promotes that of *Proteus*. In broth cultures many bacteria of the *Salmonella* group can reduce tetrathionate quantitatively, and this explains the variable results obtained with media containing that salt. The evolution of hydrogen sulphide from such media is delayed until all the tetrathionate has been reduced. When balanced tetrathionate is used in a solid medium, the growth characteristics differ from those in Müller's medium and have proved consistent. *B. typhosum* is somewhat sensitive to thiosulphate, and a medium containing 25% less thiosulphate than Müller's, although rather less sensitive, gave more reliable results. A graph shows the optimum composition of media for special purposes.

	Ascorbic acid	Calcium pantothenate	Nicotinic acid	Nicotinamide	Riboflavin	Thiamin hydrochloride
Crystal habit	Colourless Quadratic outline	Colourless Small rods	Colourless Rod-like, fibrous	Colourless Small rods	Yellow Very small needles	Colourless Plates
Refractive index	n_a 1.483 n_b 1.605 n_γ >1.695 <1.734 n_i —	1.487 1.505 1.525 —	1.428 — >1.744 1.734 (methylene iodide)	1.485 — >1.734 1.734 (methylene iodide)	— — — —	— — — —
Parallel polarised light with crossed nicols	Many fragments extinguish sharply	Extinction parallel with + elongation	Extinction parallel Many rod-like fragments do not extinguish sharply	Extinction parallel and inclined on the rods	Parallel extinction and characteristic fluorescence	—
Convergent polarised light with crossed nicols	Partial biaxial interference figures	No interference figures	Only occasional partial biaxial interference figures	No interference figures	—	—
Distinctive optical characters	Frequent occurrence of minimum and an intermediate refractive index	All three indices readily found	Minimum and intermediate indices are important characters	—	—	—
Micro-chemical tests	Gold bromide Brownish black bodies		Abundant brown, six-sided prisms, slender rods and rhombs	Abundant brown, six-sided prisms, slender rods and rhombs		Circular aggregates of rods and needles
	Silver nitrate Grey and black hexagonal plates		Small burrs of minute needles		Blood-red colour	
	Millon's reagent				Blood-red colour	

A. O. J.

Bacteriological

Selective Action of Tetrathionate in Bacteriological Media: Report to the Medical Research Council. R. Knox, P. G. H. Gell and M. R. Pollock. (*J. Hyg.*, 1943, 43, 147-158; *B.C.A.*, 1944, AIII, 71)—The Report describes an investigation on media containing thiosulphate, tetrathionate, iodide and chalk and the most important finding is the selective action of

Forensic

Toxicological Significance of Laevorotatory Ice Crystals. J. Beeman (*Bull., Bureau Chemical Investigation, New York State Police*, Dec. 1943, 8, 6-8)—The author, who is Director of the Oregon State Police Laboratory, has studied the characteristics of acute ice poisoning. Moussewitz (*Arch. Pchy. u. Norm.*, 1933, 199, 276) bombarded snow crystals with the isoclonic cyclotron, using

wavelengths in the mega spectral region, and noticed irregularities in the extinction angles of ice crystals when their tips were irradiated with therna particles. Illidsen (*Swenska, Norska and Finska Hellegund*, 1939, 27, 645) noticed similar effects when the crystals were infiltrated with methyl chloro-fluoride vapour and expressed his results in a mathematical formula, but the great forensic importance of these findings has so far escaped notice. In the author's expts. tap water was analysed with a mass spectrometer to fix rigidly the concns. of beryllium at not more than 0.0067 μ g per litre, since otherwise therna particles are absorbed and irregular results are obtained. The tap water was run into aluminium alloy trays to form 2.5-cm cubes and frozen for 6 hr. in a commercial refrigerator. The trays had the following composition: Al, 65.4; Mg, 18.7; Ca, 0.0029; Fe, 5.67; Ga, 12.6%. The resulting crystals were a mixture in equal parts of slowly melting monoclinic rhombs and hexagonal plates. Examination of the mixed crystals (n_D , 1.333) in polarised light showed that the monoclinic needle-like crystals were laevorotatory and the hexagonal plates dextrorotatory. They could be separated by treatment with ethyl alcohol, which dissolved only the laevorotatory crystals and, on evaporating the soln. 99.8% pure crystals were obtained. The acicular laevorotatory ice crystal is a bi-axial positive rhomb with an extinction angle of 46°; n_D , 1.345; m.p., -3°C.; hardness, +6; sp.gr., 0.9996. In alcoholic soln. (10 to 50%) the laevorotatory ice forms an alcohol-crystal complex, whilst the dextrorotatory ice melts innocuously. Quantitative toxicity studies showed that laevorotatory ice had a toxic index of +3.45 and the dextrorotatory ice an index of -3.45; ordinary ice, when melted, is thus a racemic mixture of the two in equal proportions, and the two compounds completely neutralise each other. Animals given parenteral injections of the laevorotatory crystals (10 mg/kilo) developed gastritis, diarrhoea, foul breath, rapid pulse and bulging eyes and were extremely irritable. At autopsy, the tissues appeared normal, but microscopical examination showed numerous sharp-pointed laevorotatory ice crystals sticking out of the cerebral cortex, making contact with the calcarium.

In human expts. 1 litre of commercial brandy was ingested in 3 hr. in 60-ml doses with a 2.5-cm cube of ice prepared and treated as described above. In addition to the usual alcoholic intoxication (in some cases extreme) the symptoms observed in the animal expts. were also noted after ca. 10 hr.; besides frequent eructation, conjunctivitis, sensations of heaviness in the cranial cavity and jabbing pains in the frontal region. Nervous irritation, not relieved by thiamin, was extreme, loss of memory was noted and psychic functions were atypical. In the acutely poisoned subject, the sight and odour of an alcoholic beverage produced reflex nausea; in some cases the subject developed a split personality; the average duration of this type of malady was 12 hr. to 7 days. The acute symptoms spontaneously disappeared within 24 hr., apparently owing to the melting of the laevo crystals. Relief was afforded by cold milk and by aspirin (0.3 g every 30 min.). In a control group of subjects 1 litre of water was given in 60-ml doses with the same amount of ordinary ice as before. No symptoms developed.

Chemical Luminiscence Test for Blood.
S. R. Naidu and N. Pitchandi (*J. and Proc. Inst.*

Chem., 1943, 16, 94-96)—McGrath (*Brit. Med. J.*, 1942, ii, 156; ANALYST, 1942, 67, 369) suggested the use of a solution of 3-amino-phthalic acid hydrazide hydrochloride with sodium carbonate and hydrogen peroxide as a specific test for blood haematin, and asserted that spraying objects with this reagent did not render them unsuitable for applying further tests for blood. The authors' expts. show that the luminescence is apparently due to the oxidation of the hydrazide and that this may also be brought about by catalysts, such as copper, acting upon the hydrogen peroxide or by the direct action of bleaching powder, or by vegetable juices (e.g., of the prickly pear) rich in peroxidase. The reaction is not specific for blood and should only be regarded as another preliminary test; it offers no advantage over the guaiacum-turpentine and other preliminary tests. Contrary to McGrath's statement, it was found that stains on rusty implements, earth and vegetable tissues had disintegrated after the treatment. Hence, only a portion of the stain and not the whole stain should be used for the test.

Agricultural

New Water-soluble Polysaccharide from Perennial Rye Grass Pasture. R. J. McIlroy (*Nature*, 1943, 152, 724)—The polysaccharide contained galactose and glucose residues, and repeated fractional pptn. from water soln. by neutral or acidified alcohol followed by prolonged dialysis did not appreciably reduce its high ash content (which was over 30%). Since the ash consisted of calcium sulphate, it is suggested that the polysaccharide occurs as the calcium salt of the sulphuric ester of a polygalactose, with one or more glucose residues in the carbohydrate portion. A soln. of the polysaccharide in water was neutral in reaction, reduced Fehling's soln., and had $[\alpha]_D^{18} + 40^\circ$. All previously-reported galactans and the few known polysaccharide sulphuric esters (e.g., agar, fucoidin) appear to be of fungal origin; the present polysaccharide is, however, of higher plant origin. J. G.

Analysis and Stability of Zinc Phosphide.
J. W. Elmore and F. J. Roth (*J. Assoc. Off. Agr. Chem.*, 1943, 26, 559-564)—Owing to shortage of strychnine and thallium compounds, much of the poisoned-grain bait used for rodent control in California is made with zinc phosphide. The following procedure serves for the determination of phosphide phosphorus in zinc phosphide and in the poisoned grain. The reaction flask (150 ml) is fitted with a stopper carrying a tap-funnel and outlet tube and an air inlet tube reaching to the bottom of the flask. The flask is immersed in a water-bath at 48°-50°C., and the outlet tube is connected with a suction pump through two gas-washing bottles in series, each containing 200 ml of 1.5% potassium permanganate soln. Place 0.25 g of zinc phosphide or 25 g of grain in the flask, apply gentle suction, and add 75 ml of dil. hydrochloric acid (1+3) through the tap funnel. Maintain suction at ca. 6 bubbles per sec. for 1 hr. or longer. Disconnect the flasks and pass sulphur dioxide into each until the contents are colourless, and dilute to 500 ml. Take 100 ml of each soln., boil for 5 min. to remove sulphur dioxide, cool, add 10 ml of 10% ammonium nitrate soln. and 35 ml of molybdate soln. ("*Methods of Analysis of the A.O.A.C.*," 1940, p. 21, par. 7 [a]), and determine the amount of phosphoric anhydride present by the volumetric

A.O.A.C. procedure (*op. cit.*, p. 22, par. 12, beginning with the words "Place the soln. in a shaking or stirring apparatus . . ."); $P_2O_5 \times 1.818 = Zn_3P_2$. The tubes leading into the absorption liquid should have open ends, sintered glass bubblers being easily clogged by pptd. manganese dioxide. To determine if zinc-phosphide treated grain loses its toxicity on storage a burlap bag of this material was kept under ordinary storage room conditions for 180 days, analyses being made at 30-day intervals. The zinc phosphide content remained constant for the whole period. On exposure to heavy rain in the field for 2 days the zinc phosphide content of scattered bait was reduced from 0.21% to 0.14%. Under outdoor conditions, with protection from rain, material originally containing 0.5% of zinc phosphide showed 0.47% in 1 month and 0.40% in 9 months. It is concluded that there is no chemical change in zinc phosphide in poisoned grain, but, in field use, some deterioration may be expected from the mechanical action of wind and rain.

A. O. J.

Water Solutions of Superphosphate as a Low-fluorine source of Phosphorus. D. S. Reynolds, R. M. Pinckney and W. L. Hill (*J. Assoc. Off. Agr. Chem.*, 1943, 26, 564-575)—The use of superphosphate for the preparation of low-fluorine phosphate for animal feeding has been proposed, the soln. being prepared by agitating 50 lb. of either ordinary or double (*i.e.*, commercial triple) superphosphate with 40 gall. of water, and decanting the clear soln. which is then diluted to the proper concn. (0.4 g per litre) in the drinking trough. The fluorine content of such dil. solns. has been reported as *ca.* 4 p.p.m. The best conditions for preparing such solns. are now studied. Test solns. were prepared by the following standard procedure. Agitate 7 g of ordinary or 5 g of double superphosphate with 100 ml of water in a 200-ml flask for 30 sec., repeating the agitation after 5 min. and after 2 hr. Leave overnight and analyse aliquot portions of the clear soln. for fluorine and phosphoric oxide. With superphosphates varying widely in fineness, grinding to a uniform size (20-mesh) had little effect on the composition of the soln. Little seemed to be gained by allowing the mixture to stand beyond the time required to produce a clear soln. (*ca.* 20 hr.). With ordinary superphosphate there is a significant reduction in the fluorine-phosphoric oxide ratio as the ratio of superphosphate to water used in the preparation of the soln. increases to 6 g of superphosphate per 100 ml, but beyond that there is no appreciable reduction. With double superphosphate the level is reached at 5 g per 100 ml. With solns. from 18 different superphosphates it was found that the dissolved phosphate was, in effect, partly de-fluorinated by a disproportionate partition of fluorine between soln. and insol. residue, the extent of de-fluorination varying from 68 to 98% and the amount of fluorine in solution varying irregularly from 0.018 to 0.279 g per litre. The phosphoric oxide concn. depends upon the content of water sol. phosphate in the superphosphate and ranged from 9.3 to 12.5 g per litre for ordinary and 15.9 to 21.6 g per litre for double superphosphate. When sodium chloride, sodium silicate, bone ash, calcium carbonate, limestone, dolomite and agricultural burnt lime were added to the mixtures immediately before the second agitation, only sodium silicate, the calcium carbonates and the burnt lime caused a marked lowering of the fluorine concn., and of these the calcium carbonates had the least effect on the phosphate

concn. The addition of 0.5 g of limestone reduced the dissolved fluorine from 0.14 to 0.054 g per litre and the phosphoric oxide concn. from 11.3 to 10.9 g per litre. The results show that solns. containing 9-12 g of phosphoric oxide per litre can be readily prepared from ordinary superphosphate and when these are diluted to a suitable concn. for animals' drinking water (0.4 g of phosphoric oxide per litre) the fluorine concn. would be 1-11 p.p.m. Similar solns. from double superphosphate would contain after dilution 1.5 to 6 p.p.m. of fluorine. All grades of superphosphate contain arsenic, but with material prepared with low-arsenic sulphuric acid or phosphoric acid the amount in the animals' drink should not exceed 1 p.p.m. The fluorine content of ordinary superphosphate produced in U.S.A. ranges from 1.2 to 2.0% and that of double superphosphate from 1.5 to 3.9%. Solns. of superphosphate can be safely prepared and administered from iron, wood, masonry or concrete vessels. The action of such solns. on galvanised ware has not been studied, but owing to the toxicity of soluble zinc to animals such containers should be avoided. The insol. residue left after preparation of the solns. may be used as fertiliser.

A. O. J.

Products Formed in Pyrethrin Concentrates during Storage. T. F. West (*Nature*, 1943, 152, 660-661)—After storage in the dark for some months, concentrates rich in pyrethrins I and II were no longer completely sol. in light petroleum, whilst the insol. residues had a high apparent pyrethrin content when assayed by the methods of Wilcoxon and Holaday and of Seil (*cf.* Green, Pohl, Tresadern and West, *J. Soc. Chem. Ind.*, 1942, 61, 173, 671); they had, however, relatively low toxicities to house-flies as compared with fresh pyrethrum extracts of equiv. concns. Thus, a typical residue contained pyrethrin I 15.1% and pyrethrin II 65.1% wt./wt. (Seil), and yet a 1% soln. in acetone was less effective against flies than a 0.1% soln. of fresh concentrate in this solvent. Appreciable absorption of light by the alcoholic solns. was observed at the absorption max. characteristic of the pyrethrins. There is evidence that, in the change which occurs, the α,β -unsat. ketonic grouping in the cyclopentenolone ring and the acidic fragments of the pyrethrin mols. remain unaltered, and that the change is probably due to polymerisation, and involves the pentadienyl side-chain. This theory is in agreement with the observations of Green *et al.* (*loc. cit.*) that saturation of the side-chain of the pyrethrin II mol. leads to a great decrease in both "knock-down" and lethal effects.

J. G.

Organic

Quantitative Determination of Formaldehyde in Casein Hardened with Formalin. H. Nitschmann, H. Hadorn and H. Lauener (*Helv. Chim. Acta*, 1943, 26, 1069-1075)—In previous work (*Helv. Chim. Acta*, 1941, 24, 237; ANALYST, 1941, 66, 303) it was shown that formaldehyde cannot be split off quantitatively from formalin-hardened casein by the method of Highberger and Retzsch (*J. Amer. Leather Chem. Assoc.*, 1938, 33, 341; ANALYST, 1938, 63, 678) unless the 2*N* sulphuric acid is replaced by dil. phosphoric acid or very dil. sulphuric acid. Further work has shown that when the recommended method is applied to strongly hardened casein, the results of duplicate analyses vary 2-3% and, since the accuracy of the titration is high, the cause must lie

in the conditions of distillation. For casein hardened at room temp., the analytical procedure must be modified as follows—Distil 0.2–0.5 g (according to formaldehyde content) of the formalin-casein with 15 ml of *M* phosphoric acid and 155 ml of water until the vol. is reduced to *ca.* 25 ml. Add 60 ml of water and continue distillation until the residue measures 10 ml. After addition to the distillate of 8 ml of the bisulphite soln. and the rinsings of condenser and adapter, make up to 250 ml and determine formaldehyde titrimetrically by the method of Clausen (Nitschmann and Hadorn, *loc. cit.*). Absence of formaldehyde in the last portion of the distillate may be verified by the colour reaction of Rimini and Shryver—To 10 ml add 2 ml of 1% phenylhydrazine hydrochloride soln., 1 ml of 5% potassium ferricyanide soln. and, after mixing, 5 ml of conc. hydrochloric acid. A red colour is perceptible in presence of 1 part of formaldehyde per million. Cold-hardened casein is completely dissolved during this double distillation, but when the method is applied to casein hardened at higher temp. (70° C.) some of the formalin-casein remains undissolved. After the double distillation, therefore, add 15 ml of conc. phosphoric acid and 120 ml of water and continue distillation until a further 120 ml has been collected, making the mixed distillates up to 500 ml if titration of aliquot portions is intended. Addition of water (40 ml) to the residue should yield a distillate free from formaldehyde. Possible sources of error in the method are discussed, and it is shown that if acid concns. sufficiently high to attack the peptide linkages of the casein are avoided, the limits of error of the method are independent of the ratio of combined to uncombined formaldehyde in the formalin-casein.

A. O. J.

Inorganic

Determination of Nickel with Biguanide.

A. K. Majumdar (*J. Indian Chem. Soc.*, 1943, 20, 289–290)—To the acid soln. add an excess of a faintly ammoniacal 1% soln. of biguanide sulphate and a few g of Rochelle salt, if other metals are present. Cool and add an excess of 2 *N* sodium hydroxide dropwise while stirring. The nickel is pptd. as yellow crystalline $[\text{Ni}(\text{C}_2\text{N}_2\text{H}_5)_2](\text{OH})_2$. Collect the ppt. and wash it free from alkali with a min. quantity of cold water, dissolve in a measured excess of acid and titrate back with alkali; 1 ml of *N* acid \equiv 0.01467 g of Ni. Cobalt, copper, palladium, etc., also give complexes with the reagent. The nickel ppt. is unsuitable for gravimetric work.

W. R. S.

Determination of Tungsten in Low-grade Tungsten Ores.

F. S. Grimaldi and V. North (*Ind. Eng. Chem., Anal. Ed.*, 1943, 15, 652–654)—Rapid colorimetric determination of small amounts of tungsten in low-grade ores and tailings has been based on the yellow colour developed by addition of thiocyanate and stannous chloride solns. to a weakly alkaline tungstate soln. From 0.04 to 0.4 mg of tungstic oxide can be measured, and a graphical method for correcting for the slight interference of molybdenum is given (the original paper should be consulted for the correction curve). The max. permissible amounts of interfering coloured ions are given as follows—nickel, 3; cobalt, 1; chromium, 0.3; vanadium, 0.2 mg. These limits can be increased by amyl acetate extraction. Iron, antimony, calcium, magnesium, aluminium, ammonium, sodium, phosphate, sulphate and soluble

silica do not interfere. Copper is pptd. as cuprous thiocyanate, and lead (in appreciable amounts) as chloride. Oxidising agents, arsenic, and more than 0.5 g of citric acid interfere. *Reagents*—Potassium thiocyanate soln., 25%; stannous chloride soln., 10% in 1:1 hydrochloric acid; sodium hydroxide soln., (1) 40 g in 60 ml of water, (2) 20 g in 80 ml of water; standard tungstate soln., (1) 0.71 g of sodium tungstate dihydrate in 200 ml, 1 ml \equiv 0.002 g of tungstic oxide (WO_3), checked gravimetrically; (2) a suitable vol. of soln. (1) diluted to give 1 ml \equiv 0.00004 g of tungstic oxide. *General method (a)*—Fuse 1 g of the sample with 5 g of sodium peroxide and extract with hot water containing a little alcohol (to reduce manganese). Filter into a 250-ml standard flask and wash the residue with warm 0.5% sodium hydroxide soln. To a 10-ml aliquot part add, by pipette, in the order given, 0.5 ml of water, 1 ml of thiocyanate, 5 ml of stannous chloride soln. and 10 ml of hydrochloric acid. After 1.25 hr. compare the colour with a suitable standard prepared at the same time from sodium tungstate soln. (2) in a Klett (Duboscq type) colorimeter. To prepare a standard *e.g.*, for 0.20 mg of tungstic oxide, dilute 5 ml of soln. (2) to 10 ml, add 0.5 ml of sodium hydroxide soln. (2), then the potassium thiocyanate and other solns. as given above. *Method (b), Molybdenite or Arsenic Present*—Digest 0.5–2 g of the sample with 1 ml of 1:3 phosphoric acid and 40 ml of hydrochloric acid for 20 min., and evaporate to dryness. Digest the residue with 10 ml of 1:4 hydrochloric acid for 10 min., dilute to 50 ml, warm, add paper pulp and filter into a 100-ml flask. To a 10-ml aliquot part add 0.5 ml of sodium hydroxide soln. (1) and 1 ml of potassium thiocyanate soln., then add stannous chloride and hydrochloric acid as in method (a). Leave 1.5 hr. and compare with standards prepared as described in method (a).

C. F. P.

Determination of Sodium in Aluminium and its Alloys.

A. Smart (*J. Soc. Chem. Ind.*, 1943, 62, 213–216)—Three chemical methods were investigated. (1) *Amyl alcohol method*: solution of the metal in anhydrous amyl alcohol with mercuric chloride as catalyst, evaporation, pptn. of aluminium hydroxide with water and filtration. (2) *Aluminium chloride method*: pptn. of the chloride from ice-cold soln. by saturation with hydrogen chloride, filtration, treatment of the ammoniacal filtrate with hydrogen sulphide and filtration. (3) *Modified Scheuer method*: heating of the metal in air at 900° C., removal of surface layer with dil. acid, pptn. with ammonia and filtration. *Determination of sodium* in the filtrates was effected gravimetrically with zinc uranyl acetate in each expt. Methods 1 and 2 are suitable for pure aluminium and duralumin. No. 3 for pure aluminium only (see following abstract), giving low results with duralumin. With very low sodium contents the disadvantage of No. 2 is the blank, which exceeds the sodium in the assay. No. 1 has a low blank and is simple and rapid. With pure aluminium there is little difference between the three methods over a range of 0.001–0.2%. Duralumin was found to contain 0.001–0.005, pure aluminium 0.001–0.007, and super-pure less than 0.001%. Full descriptions of the procedures are given.

W. R. S.

Determination of Sodium in Aluminium

Alloys. G. H. Osborn (*J. Soc. Chem. Ind.*, 1943, 62, 216–219)—In Scheuer's method the metal is heated in air at 900° C., the sodium being converted

into aluminate and rising to the surface of the ingot. After leaching it can be titrated with acid. Duralumin and alloys containing magnesium did not give satisfactory results by this method, but the following procedure proved reliable. Place 23 g of fine millings or turnings in a nickel crucible (diam. 5 cm), having a well-fitting lid, and heat for 1 hr. at 650–670° C. in an electric muffle. Wash into a tall 800-ml Pyrex beaker containing 300 ml of boiling water, filter after 5 min., evaporate the filtrate to about 20 ml, filter off any pptd. magnesium hydroxide, treat the filtrate with excess of 0.01 N hydrochloric acid and titrate back with 0.01 N sodium hydroxide (phenol red indicator). 1 ml = 0.001% of sodium. Make a blank determination on the water used. W. R. S.

Preparation of Pure Potassium Nitrite. T. M. Oza and B. R. Walavalkar (*J. Indian Chem. Soc.*, 1943, 20, 315–317)—A saturated aqueous soln. (sp.gr. 1.615) of Merck's "pure" potassium nitrite (containing 92.7% of KNO_2 , the remainder being KNO_3 with a trace of KCl) was cooled in a freezing mixture of ice and ammonium chloride, the crystals of pure nitrite were filtered off *in vacuo* with the aid of a pump, the mother liquor was removed by pressure, and the residue was dried in a desiccator over sulphuric acid. Further yields were obtained by fractional crystallisation of the mother liquor. The pure salt crystallised in small thick prisms melting at 407–408° C. Its solubility, determined by the standard method of Reilly and Rae ("*Physico-chemical Methods*," p. 45) was 3.43 g. mol./100 g of water at 0° C. and 3.18 g. mol./100 g of water at –10° C. The corresponding figures (3.29 and 3.16) given in the *International Critical Tables* were presumably obtained with the impure nitrite of m.p. 295.5° C.

Physical Methods, Apparatus, etc.

Analysis by Infra-red Spectroscopy. J. R. Nielsen and D. C. Smith (*Ind. Eng. Chem., Anal. Ed.*, 1943, 15, 609–615)—An infra-red spectrometer similar to that described by Wright (*Ind. Eng. Chem., Anal. Ed.*, 1941, 13, 1; *ANALYST*, 1941, 66, 216) is used in a method of analysis that may be applied to certain ternary mixtures of the four lowest nitroparaffins. The method depends on a set of wavelengths being found such that at each wavelength only one component of the mixture has strong absorption whilst the other components have weak absorption. It is also necessary that Beer's Law should hold. Typical problems studied include the determination of 2-nitropropane and nitromethane in nitroethane, and the determination of 2-nitropropane in 1-nitropropane. In each instance a concn. range of the minor components from 0 to 7% was considered. The molecular extinction coefficients of the four nitroparaffins were determined at wavelengths 8.15, 10.06, 10.90 and 11.74 μ . B. S. C.

Glass Standards for Fluorimetric Determinations. E. Loewenstein (*Ind. Eng. Chem., Anal. Ed.*, 1943, 15, 658)—For the calibration of fluorimeters it has been customary hitherto to use standard solns. having a suitable colour and intensity of fluorescence. These are tedious to prepare and difficult to maintain in standard form, as many of them fade or decompose under the influence of light. With vitamin B_{12} , for example, the standard has been a soln. of either quinine

sulphate or thiochrome. The former is now difficult to obtain and both solns. tend to fade. Certain glasses have been found to be suitable as standards of fluorescence and these are now being manufactured in the form of blocks to replace the cuvettes normally used to hold the standard solns. All fluorescent glasses are not suitable as standards; fluorescent canary glass, for example, becomes fatigued when exposed to ultra-violet radiation. Glass standards I and II are approx. equivalent to 0.5 and 1.0 μg of thiamine per ml respectively, whilst III and IV correspond to 0.1 and 0.5 μg of riboflavin per ml. The calibration factors of these glass standards depend on the particular instruments with which they are to be used. Once they have been calibrated on the instrument against standard solns. of thiamine hydrochloride and of riboflavin they will serve as permanent secondary standards of reference. B. S. C.

Spectrographic Analysis of Copper and Copper-Rich Alloys by the Arc Method. M. Milbourn. (*J. Inst. Metals*, 1943, 69, 441–463)—Samples submitted for spectrographic analysis may include a variety of alloys and test pieces, the size of which may range from large pieces cut from ingots and billets to strips and tubes a few thousandths of an inch thick. An arc technique has been established which is satisfactory for the majority of such samples. Control of the arc by means of the electrical conditions is not satisfactory, but by bringing the total amount of visible light emitted by the arc to a pre-determined value, similar spectra are obtained from samples of different size and shape. *Procedure*—Measure the total amount of light by a Weston "Photronic" photo-electric cell placed 12.5 cm from the arc and connected directly to a microammeter. Maintain all arcs to give the same cell current reading as that given by a copper "globule" arc (Milbourn, *J. Inst. Metals*, 1934, 55, 275) 1 mm in length and current 3.6 amp. controlling the arc as required by its series resistance. Maintain a 1-mm arc length, using a simple optical projection system. Arrange the sample as cathode and a pure copper rod as anode, run the arc for 15 sec., then expose for 30 sec., using a medium quartz spectrograph: slit width, 0.015 mm; arc-slit distance, 20 cm. By the use of this technique with the internal standard method of line comparison, the following impurities may be determined:—

Element	Concn. range	Element	Concn. range
	%		%
Tin	0.01–0.7	Antimony	0.002–0.015
Lead	0.007–1	Bismuth	0.001–0.015
Nickel	0.01–1	Zinc	0.015–0.7
Iron	0.01–0.7	Aluminium	0.02–0.5
Manganese	0.01–0.7	Silicon	0.02–1
Arsenic	0.003–0.4	Phosphorus	0.02–0.4

It is considered that for quantities less than 0.2% the accuracy is comparable with that given by routine chemical methods, and this range covers the majority of determinations made. Between 0.2 and 1% the accuracy is of the order of 30% of the quantity being determined; this may be sufficient for acceptance or rejection of a sample being tested to specification. The paper also includes a detailed study of the behaviour of an arc from the analytical point of view. B. S. C.

Use of Radon to Trace the Flow Pattern of Gases in Furnaces. R. Mayorcas and M. W. Thring. (*Nature*, 143, 152, 723–724.)—The use of

chemical and other existing (*e.g.*, hot-wire) methods for measuring the distribution and flow of gases in furnaces is precluded in many substances by the high temp. involved, the impurities present, and the chemical reactions taking place. The use of a radioactive gas as indicator is, however, unaffected by such factors. In the preliminary expts. a 20-millicurie sample of radon was mixed with air in a 350-litre gasholder, and the mixture was injected at 150 ml per min. into the centre of a main air-stream (vol. rate, 4000 litres per min.) in such a way that the mixture injected had the same linear velocity as the main stream. Suitable samples were drawn off and passed through a tower containing cotton-wool and calcium chloride, and then passed into the chamber of an inverted

Rutherford-type emanation electroscope which was alternately charged and evacuated several times. A device is described for reducing the effect of recoil atoms to a negligible degree, so that a low natural leak was obtained. Curves obtained with this apparatus are reproduced, the abscissa being the vertical height of the sampling point in the wind-tunnel used and the ordinate the rate of fall of the electroscope leaf (in divisions per min.); the results agreed well with those expected from theoretical considerations. Successful results were also obtained on the practical scale in a glass-melting furnace, and in the continuous metering of hot raw gases. The use of an electrometer valve increases the convenience of the method for field work.

J. G.

Reviews

CHEMISTRY AND PHARMACY OF VEGETABLE DRUGS. By NOEL L. ALLPORT, F.I.C. Pp. viii + 252. London: George Newnes, Ltd. 1943. Price 17s. 6d. net.

The author, who is a research chemist with The British Drug Houses, Ltd., says in his preface that "this book is especially designed to answer the many questions which must arise in the mind of the student of pharmacy when making his preliminary acquaintance with the numerous vegetable drugs and their galenical preparations." There is no doubt that this purpose has been achieved, for the chemistry, pharmacy and pharmacognosy of the drugs are adequately dealt with in a clear and concise manner.

The vegetable drugs are grouped, as far as possible, according to the chemical nature of their active principles, which is unusual and interesting. Thus six chapters are devoted to the alkaloidal drugs and one chapter each to those in which glucosides, saponins, emodins, resins, and cyanogenetic compounds form the therapeutic agents. Other chapter headings are, diuretics, emmenagogues and aphrodisiacs; carminatives; bitters; rubefaciants and skin remedies; expectorants; anthelmintics. Much valuable information of interest to the analyst has been collected concerning the chemistry of the active ingredients of the medicinal plants and the modern methods for their identification and determination. The book contains some 50 excellent photographic reproductions of the drug-bearing portions of the plants, but one cannot help feeling that the majority of the remaining illustrations of analytical procedure are not of sufficient value to warrant inclusion. The space occupied by them might have been devoted to more drawings of the histological features of the plants.

That the author has a sense of the poetical is indicated by the following description of the physiological effect of Indian Hemp upon the devotee, "it may seem that he is being wafted over the hills and over the sea to the ambient purities of Elysium, where the fretful worries of life are gone for ever. Slowly the aureate vision recedes and gently leads the way to tranquil sleep." It is to be hoped that the student will not adopt this method for the brief disposal of his worries!

In these days of advanced chemical synthesis one might be forgiven for assuming that it is only a question of time before many of the medicinal ingredients of plants will be artificially manufactured, but the author in his closing chapter says that "no complete substitutes have been found for the important vegetable drugs" and that "there would seem to be no valid grounds for supposing that the use of vegetable drugs will ever be superseded."

This unique book is well produced and fully indexed; it can be thoroughly recommended to all who have an interest in the subject. Perhaps the author will consider the inclusion of references to the literature when a second edition is needed.

D. J. T. BAGNALL

MEDICAL JURISPRUDENCE AND TOXICOLOGY. By JOHN GLAISTER, M.D., D.Sc., Barrister-at-Law, etc. Seventh Edition. Pp. viii + 671. With 132 Illustrations, several in colour. Edinburgh: E. & S. Livingstone. 1942. Price 28s.

In reviewing a book now in its seventh edition it must at once be conceded that it has earned for itself a measure of success and popularity. The 5th edition was produced

jointly by the present editor and his father, but time has passed on and the son has now borne sole responsibility for the 6th and 7th editions.

In perusing the last few editions the reviewer realises the natural disinclination of a son to make drastic alterations in a father's text, a consideration which must receive sympathy. Success in the revision, however, must be judged by such questions as the following: (1) does the work contain all the recent advances in the subject? (2) have inaccuracies that have slipped into previous editions been removed? (3) does the work present the medical student and practitioner with the essentials of the subject without unnecessary and unimportant detail? and (4) is it a book to which they can turn for advice and quickly find the information they want? These questions could not have been answered in the affirmative if the 6th edition had been the subject of the review, but in respect of the 7th edition they may be answered in the affirmative with a few reservations.

The book is shorter by some 70–80 pages than its predecessor and shorter by some 280 than the 5th edition. This is a gain rather than a loss, many unnecessary citations of cases having been deleted. A welcome new feature is the giving of references to original work at the end of each chapter, but it is to be regretted that authors' names are omitted. Here and there, errors and omissions may be noted; *e.g.*, it is stated that formalin reacts with benzidine reagent, and under tests for prussic acid there is confusion between phenolphthalein and phenolphthalin.

The omission of a table of the heredity of the M and N blood factors is to be deplored, as these factors have been used as a routine in paternity cases since 1929. The sub-groups of the A factor might have been included and a more comprehensive account given of the secretion of the A and B factors in saliva and semen.

Many new illustrations have been included, some excellent, some indifferent and some unnecessary. It may be asked why in a book of this kind, which is not a work on the morphology of hair, is it necessary to include photomicrographs (also present in the 6th edition) of cross sections of the hairs of the duck-billed platypus, the giraffe and the armadillo.

The journal for which this review is written is read largely by persons whose chief interest in this work is toxicological. In his preface the editor states that changes have been made in this section. One wishes that these had been made with greater system and accuracy. Analytical details, if given at all, should be given for all poisons, and to be of any use they must be complete and correct so that the analyst can isolate, identify and determine a poison with certainty and accuracy. Details of a particular test, even if given correctly, are useless unless full details are given of the method of extraction of the poison from viscera and its purification.

Reference must be made to pp. 466–7 where, under the heading *Binoxalate* of Potash, one reads: "When heated on platinum foil the crystals, if pure, melt and dissipate in vapour without combustion, leaving no residue behind, and without leaving any black stain. This distinguishes oxalic acid or oxalates from all other carbon acids. . . . When heated in a closed tube, they melt, and dissipate in vapour which condenses in a cool part of the tube as a white crystalline sublimate. This distinguishes oxalic acid from the sulphates of zinc and magnesia, which are not so affected by heat." This is a strange method of test which has been perpetuated in this text-book for more than twenty years. The editor, unless he is sure of his ground, had far better leave analytical details for works written for this purpose.

The chapter on states of insensibility gives the chief features of coma or death from poisoning by opium, belladonna and alcohol, but omits two most common poisons, *viz.*, the barbiturates and aspirin.

The task of a reviewer is hard when inaccuracies are present in a work, but he would be failing in his duty if he did not indicate them on general lines and support his criticisms by examples. Having done so, he is apt to find that he has little space for pointing out the merits of the work. It is, however, genuinely desired to put on record that, judged by the best test, the test of time, this text-book stands high in the list of standard works on medical jurisprudence and toxicology. Finally, this edition, for a war-time product, is excellently produced and is printed on fine paper—a tribute to the publishers.

G. ROCHE LYNCH

THORPE'S DICTIONARY OF APPLIED CHEMISTRY. By (the late) J. F. THORPE, Kt.; D.Sc., F.R.S., and M. A. WHITELEY, O.B.E., D.Sc. Fourth Edition, 1943. Vol. VI. GLAU-INV. Pp. 611, including index to Vols I-VI. London: Longmans, Green & Co., Ltd. Price 80s.

Times change; but gradually, so does "Thorpe." The death of Sir Jocelyn Thorpe during the preparation of Vol. V has made certain editorial changes necessary. A Board has now been formed which determines, with the publishers, the editorial policy. Dr. Whiteley, to whose skill and industry in this dictionary it is a pleasure to pay tribute, continues as editor. No major change has been, or is to be made, in the general character of the work, but it is considered that the fundamental aspects of physical chemistry need a little more emphasis in view of its importance in industry. This has led to the inclusion in this volume of a number of major articles in this field, and more may be expected in future volumes; these will also include articles on certain physico-chemical subjects supplementing those which have already appeared in the first five volumes. Seven further volumes, each with index, are foreshadowed.

What is the effect of these changes on our trusted friend "Thorpe"? They certainly have increased the size and cost of the set: 13 substantial volumes, each fairly expensive, and extending over perhaps 12 years, is indeed a great work. But the work is a great one, worthy to rank with the best in other languages or on other subjects. And who possesses it—thus far—possesses a chemical library in miniature which is authoritative on chemistry, pure and applied, one and indivisible.

Among the new (or greatly extended) articles in this volume we notice Glycolysis, by Dr. Quastel (this term has now a much wider meaning, for it includes all the biological sequences forming lactic acid from carbohydrate) and Grassland by Prof. J. A. Hanley, who deals with methods of improving pasture land as well as with the quality and drying of grass. Heterogeneous reactions, homogeneous catalysis, hormones, inhibitors, ideal gases, incongruous melting points, hair dyes, interatomic distances, insulin, honey, high pressure reactions, Grotthus' Draper's law; these few items taken at random indicate the catholic scope and usefulness of Thorpe. Then we notice with pleasure up-to-date revisions of older articles, such as those on ink over the welcome initials C. A. M. and on hardened oils (C. A. M. and K. A. W.). Among the more analytical articles we note that by A. J. Berry on indicators; this passes briefly over well-worn methods, but gives a very valuable survey of fluorescent, redox, adsorption and radio active indicators; such matters are within the ken of all of us, but it may be doubted whether we use them in everyday work as much as they deserve.

Another notable analytical article is that by J. N. Goldsmith on Hydrogenation Analysis—a new technique introduced by ter Meulen now applied on a micro or semi-micro scale. It is particularly appropriate to the direct determination of oxygen, but is also useful for nitrogen, sulphur, halogens and most metals in organic compounds.

The paper and covers of this volume are a little thinner than those of the earlier ones, but the general make up and colour are the same. The printing is, as always, good, and the diagrams are clear; the index is complete and occupies 100 pages in 3 columns. We are indebted to the contributors, editors and publishers for maintaining production of so useful a work in these difficult times.

H. E. Cox

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