DECEMBER, 1933 Vol. LVIII., No. 693

THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS

An Ordinary Meeting of the Society was held at the Chemical Society's Rooms, Burlington House, on Wednesday, November 1st, the President, Mr. F. W. F. Arnaud, in the chair.

Certificates were read in favour of:—Willard E. Baier, B.S. (California), Adam Dunsmore, A.I.C., Donald Clarence Garratt, B.Sc., Ph.D., F.I.C., Colston James Regan, B.Sc. (Lond.), F.I.C., Robert Henry Slater, D.Sc., Ph.D. (Edin.), F.R.S.E., A.I.C.

The following were elected Members of the Society:—Harold Grange Coles, B.Sc., M.A., A.I.C., Alfred Francis Colson, B.Sc., A.I.C., Claude Alexander Dunbar-Mitchell, M.A., A.I.C., Alfred Dudley Husband, F.I.C., George Vaughton James, B.Sc., A.I.C., John Brown McKean, F.I.C., John George Peirce, B.Sc., John Rae, V. Venkatachalam, M.A., Allan Miles Ward, D.Sc., Ph.D., A.I.C.

The evening was devoted to a discussion on "The Chemical (as distinct from Physiological) Tests for Vitamins."

Mr. A. L. Bacharach opened the discussion with a paper on "The Present Position of the Chemical Evaluation of Vitamins and its Importance to Analysts."

Dr. Leslie Harris discussed the methods of applying certain tests for vitamins *A* and *C*. Professor Drummond described his experience with the antimony trichloride test, and Dr. R. J. McWalter demonstrated the use of a simplified ultra-violet spectroscope (Hilger) in the estimation of vitamin *A*.

Mr. Norman Evers pleaded for uniformity in the methods of expressing results.

Mr. J. K. Crews and Mr. S. J. Cox read a paper on "The Relationship between the Carr–Price value and the $328m\mu$ Absorption Coefficient of Preparations containing Vitamin *A*."

Mr. A. H. Bennett contributed a note on the "Titration of Vitamin *C* in Citrus Juices."

Other speakers included Dr. K. H. Coward, Dr. F. H. Carr, Mr. E. Hinks, and Mr. F. K. Donovan.

Obituary

EDWARD THEODORE BREWIS

By the death, on October 12th, 1933, of Edward Theodore Brewis, at the age of 72, the Society has lost a much-respected member. Brewis, according to the Society's list, was not a member of very long standing, having been elected in 1915. It is, indeed, difficult to believe that he was, comparatively speaking, so junior a member, but, though he joined the Society rather late in life, he made up for this by unflagging interest in its affairs to the end of his term. He was present, as one would have predicted he would be, at the opening meeting of this winter's session, on October 4th, only eight days before his death. He leaves a widow and a son.

Brewis was born in York, and at a very early age went to Dublin; those who have heard him speak will remember him as of Dublin rather than as of York. He was "intended" for the medical profession, but became a chemist. Whether that original intention was his or another's the writer does not know, but traces of that intention can be seen in his life's work in drugs, in pharmaceutical matters and in essential oils. After holding positions in England, with Messrs. Southall Bros. & Barclay at Birmingham, and again in Dublin, he settled down in the early nineties as chemist to Messrs. Stafford Allen & Sons, working at the farm at Ampthill and in their laboratories in London. This firm he faithfully served for a period of very nearly forty years. He joined the Society of Chemical Industry in its first year, and was elected a Fellow of the Institute of Chemistry in 1888. His published work includes papers with J. C. Umney, on cubeb and lavender oils, and for a number of years he served on the British Pharmaceutical Codex Committee.

For the two years, 1924–25, Brewis served on the Council of the Society. He was chosen by the Council as one of the original members of the Standing Committee on Uniformity of Analytical Methods, and was nominated by the London Chamber of Commerce to the Society's Essential Oils Sub-Committee convened under the Uniformity scheme. To the work of these Committees Brewis gave much time and thought: he contributed a full share to the ten reports of the Essential Oils Sub-Committee so far published, and to the extensive experimental work which they involved.

Brewis was ever ready to put at the disposal of others the benefit of his special experience, whether at the formal meetings of Council or Committee, or during discussion at the monthly open meetings, or in response to personal enquiry. His colleagues and friends will long remember him, and on behalf of them all this appreciation of him is written.

E. Hinks

JOHN DAVID ROBERTS

It is with deep regret that we record the death, on September 25th, of J. D. Roberts, at the early age of 47. Roberts was a man of genial disposition, and one who was always ready to give his time to the service of his profession, and the Society has lost a valued member in his passing.

He matriculated from his school at Llanelly, and was a student of chemistry at the University College of Wales, Aberystwyth, from 1904 to 1908. In 1908 he came to London as assistant to Messrs. D. T. Williams & Co. In the following year he was appointed assistant to the Public Analyst for the Metropolitan Borough of Stepney, and from that time onwards he remained faithful to the chemistry of foods and drugs. Successively he held appointments at the Government Laboratory, the Great Western and Metropolitan Dairies, Ltd., and finally with United Dairies, Ltd. Roberts made his mark in the field of dairy chemistry and bacteriology, and only in 1932 did he resign his appointment with United Dairies to start in private practice as a consultant. In this venture he was not given the opportunity of travelling very far, for, shortly after securing a laboratory and starting work he had a severe illness and could never recover true health.

He was elected a member of the Society in 1927, and was nominated by the Council as its representative on the Empire Marketing Board's Dairy Research Committee dealing with analytical apparatus. He was also appointed as a member of the Milk Products Sub-Committee on Uniformity of Analytical Methods. To the work of these Committees he was able to bring valuable aid, arising from his wide experience in dairy matters. It is a source of grief to his colleagues in this work that they must continue without his help, and without that pleasant personal association with him that they so much appreciated and had expected to continue to enjoy.

C. A. Adams

The Occurrence and Origin of Lead in Canned Sardines

By L. H. LAMPITT, D.Sc., F.I.C., and H. S. ROOKE, M.Sc., F.I.C.

(Read at the Meeting, October 4, 1933)

In common with any goods where contact, however slight, with solder may have occurred, canned sardines have for years past been examined in our laboratories for the presence of metallic impurities, including lead. Generally speaking, canned sardines will be found to contain traces of tin of the order of 0·1 or 0·2 grain per lb., and, obviously of more serious import, traces of lead. On referring to our records, for example, we find that certain consignments of sardines offered to us in 1928 contained as much as 70 parts of lead per million. It is the general belief of the provision trade that sardines, on account of the oil which surrounds them, are not subject to metallic contamination from their containers, but, notwithstanding this belief, our original conclusion was that the lead we found was derived from the solder exposed in the can, particularly so, as some of the cans examined by us were very crudely soldered—even showing lumps of exposed solder in the tin.

As a result, we paid particular attention to the nature of the soldering, and rejected any consignments when samples showed evidence of careless work.

Some figures may here be of interest:

Lead Parts per million	Number of samples examined
0 to 5	220 (37·0 per cent.)
6 ,, 10	194 (32·6 ,, ,,)
11 ,, 20	112 (18·8 ,, ,,)
21 ,, 30	27 (4·5 ,, ,,)
31 ,, 40	25 (4·2 ,, ,,)
41 ,, 50	7
51 ,, 60	3
61 ,, 70	3
71 ,, 80	3
81 ,, 90	1
150	1
	596

These figures show the common occurrence of lead in canned sardines and the serious amounts frequently present.

A further fact which suggested that the lead found in sardines was due to the exposed solder was that French-packed sardines generally had considerably lower content of lead, as would be expected, since, in France, the solder used in food containers is required to contain not more than 2 per cent. of lead; solder in Portuguese sardine cans has been found to contain lead in large proportions, even up to 80 per cent.

That the solder was responsible for the contamination seemed a fair assumption, but, nevertheless, there are obviously three possible sources of the lead:—(*a*) natural to the fish; (*b*) introduced by the method of preparation; (*c*) introduced by the method of packing. These three possibilities were investigated.

(*a*) LEAD CONTENT OF THE FISH.—Samples of raw sardines were sent to us in lead-free glass bottles. On analysis, the fish were found to contain no lead.

(*c*) INTRODUCTION BY THE METHOD OF PACKING.—Sardines were supplied packed direct in cans having "sanitary" seals both at the top and bottom, the only solder being a little on the outside of the joint at the side of the tin. These sardines were found to contain 10 to 15 parts of lead per million, although the cans were internally free from exposed solder.

Further samples were then sent to us at our request, packed direct into pressed tins with sanitary sealed top. The tinplate was free from lead, as was the rubber gasket, and no solder was present anywhere. The sardines in four cans contained, respectively, 8, 15, 15, 20 parts of lead per million.

It was thus made clear that solder exposed internally in the can, though it might be contributory, and objectionable on this account, was not the fundamental cause of the presence of lead in the sardines.

We had observed, too, that there was no regular relationship between the amount of exposed solder in the cans and the amount of lead found in the sardines. A sample of sardines with no exposed solder in the can had a lead-content of

40 parts per million, a second sample with a little exposed solder gave a figure of 64 parts per million, whereas a third sample with much exposed solder along the seams of the can had only 10 parts of lead per million. Other samples of sardines, with varying amounts of solder exposed in the cans, had lead-contents of 16 to 52 parts per million. In other words, the presence of much exposed solder did not always accompany high lead-content in the fish, and *vice-versa.*

Furthermore, samples of sardines, in cans having exposed solder, which we had kept for 12 months, did not show any material increase in the lead-content when examined after that period. Also, some tins of French sardines, though containing much exposed solder—even in the form of lumps—after 3 years of storage contained only 2 parts of lead per million.

These facts forced us to the conclusion that the contamination occurred at some stage in the process of preparing the sardines before actually packing in the cans.

(*b*) INTRODUCTION BY THE METHOD OF PREPARATION.—With the object of tracing this contamination to its source, one of us (L. H. L.) investigated at the packing houses the whole process, which may be briefly described as follows:

The fish are shovelled from boats into baskets, together with salt, and taken to the packing house, where they are placed in wooden troughs and covered with more salt. The head and viscera are then removed, and the eviscerated fish are pickled in a salt solution. Two different methods of cooking are employed, according to the French or Portuguese method. It is to be understood that these terms are applied to the "processes," and that the French method may be, and is, used in Portuguese factories.

(i) *French Method.*—In four factories we have visited the fish are placed, tail upwards, on the grills, which are essentially wire struts arranged in a series at an angle of about 60°. The fish on the grills are then dried either in the sun or in an air tunnel, and the cooking is carried out by frying for about three minutes in oil heated by means of a steam-coil. The cooked fish are drained, placed in tins and covered with oil, after which the tins are closed and processed in boiling water for 2½ hours.

(ii) *Portuguese Method.*—Three factories were visited. The fish on the grills are heated in a steam-chest at about 110° C. for a length of time depending on the size of the fish. After cooling, the process of filling the cans is the same as in the French method, processing being carried out in autoclaves, at about 108° C. for 1 hour on two successive days. The Portuguese method gives a lighter coloured product.

As, prior to canning, the fish come into contact with metal only on the grills, the composition of these was studied. They are said to be tinned, but the metal coating used in certain factories visited was generally found to contain a large amount of lead, and was in reality ordinary soft solder. This is evidently the source of the lead contamination, and proof was afforded by the following experiment:

Sardines were cooked on ordinary so-called tinned grills, and a further lot on grills newly coated with block tin; the two samples were then packed in identical sanitary cans. On analysis, it was found that the sardines cooked on the old grills contained 44 and 57 parts of lead per million, but the fish from the grills newly tinned with block tin gave results of 7 and 10 parts per million. Further samples, prepared under supervision, using pure tin on the grills, thus ensuring that under factory conditions the possibility of contamination was reduced to a minimum, contained only two parts of lead per million. It was furthermore found that old grills which had been re-tinned with pure tin are only satisfactory until the new metal surface wears away, and that then lead from the previous treatments is liable to contaminate the fish.

These results definitely prove that the presence of lead in the metal used for tinning the grills causes serious contamination of the sardines during the cooking process. The liability to contamination, if impure tin is used, is much greater in the method where steam is used than in the frying method, and this is borne out by the higher lead-content of sardines prepared according to the Portuguese method.

METHODS OF DETERMINATION OF LEAD

PREPARATION OF THE SAMPLE.—The sardines, without the free oil* in the can, are thoroughly minced.

1. *Chemical Method.*—Five grms. of the minced sample are warmed in a Pyrex Kjeldahl flask with 20 c.c. of concentrated nitric acid until the initial violent reaction has ceased; 10 c.c. of concentrated sulphuric acid are added, and the mixture is heated, with frequent additions of nitric acid, until the liquid is straw-coloured. Four grms. of potassium sulphate are added, and the heating is continued until the solution is colourless (at least 4 hours). A control test on the reagents is carried out simultaneously. The clear liquid is washed out of the flask, and the acid is neutralised with ammonia (2:1), any excess of the latter being boiled off.

After this, the procedure is essentially that described in Scott's *Standard Methods of Chemical Analysis* (4th Ed., p. 284).

2. *Photospectrometric Method.*—Ten grms. of the material are destroyed with sulphuric acid and nitric acid, as in the chemical method. The mixture is then twice boiled down with water until white fumes appear, to remove traces of nitric acid, and the heating continued until crystallisation commences.

The method of Francis, Harvey and Buchan (ANALYST, 1929, **54**, 725) is then followed to the stage where the dry sulphates of lead and copper are obtained, after which the procedure is as follows:

To these are added 10 c.c. of water, and then 0·880 ammonia solution, drop by drop, until the solution is blue. Fifteen c.c. of water containing 1 c.c. of concentrated nitric acid are added, and the solution is electrolysed in a U-tube at 70 to 80° C., using 1·5 to 2 volts and a current density of 0·3 to 0·4 amps. per 100 sq. cm.

The cathode is a platinum wire, and the anode is platinum foil which is arranged so that lead peroxide is deposited on only one side of the foil. The

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* The amount of oil in the can varies with the packing, size of fish, etc. In the example given in this paper the oil comprised about 13 to 20 per cent. of the total contents of the can (13-oz. size). The amount of lead in the oil we find to be negligible. For example, samples of sardines were examined after keeping for 4 years; the oil then contained 2 parts of lead per million, whereas the sardines contained 45 parts per million.

electrolysis is continued for one hour and the anode then well washed with water. The anode is sparked, using a platinum wire for the other electrode. The spectrum so obtained is photographed, and, on the same plate, spectra obtained from various amounts of standard lead solution treated in the same way are also photographed. Comparison of the intensity of the lead lines of wave-length 405·8 and 368·4mμ indicates the amount of lead present.

More recently the method has been rendered more sensitive by evaporating the precipitated sulphides (lead and copper) with sulphuric acid in a platinum trough, the residue being sparked directly from this without electrolysis, using a wedge-shaped piece of platinum for the other electrode.

The following are some results obtained by the two methods.

		Chemical method Parts per million	Photo-spectrometric method Parts per million
Sardines	Sample I ..	33	30
,,	,, II ..	45	40
Uncooked sardines	,, III ..	0	0
Sardines	,, IV ..	3	–
Sample IV, plus lead salt added equivalent to 30 parts per million		35	–
Sample V, plus lead salt added equivalent to 25 parts per million. Lead found (added) ..		26	25
Sample VI, plus lead salt added equivalent to 30 parts per million. Lead found (added) ..		34	30

We wish to express our thanks for permission to publish this paper to Messrs. J. Lyons & Co., Ltd., in whose Laboratories the experimental work was carried out.

DISCUSSION

The PRESIDENT congratulated the authors on solving the problem of the occasional occurrence of lead in sardines. Their work suggested the possibility that the presence of lead in other foodstuffs might also be traced to the utensils in which they were prepared, and not to the material in which they were ultimately packed. He had always assumed, for instance, that when traces of lead occurred on the outside of cheese it was due to impurity in the foil wrapping, but now it seemed possible that the lead might originate in the utensils with which the milk, curd or cheese came into contact.

Mr. E. HINKS said that this paper showed the benefit that arose from a firm and its chemists working on a matter such as this. The metallic contamination had been tracked to its source in a way that was not generally open to public authorities.

Mr. E. LUSH asked why the solder was so very much less effective than the lead on the grill.

Dr. H. E. COX asked if Dr. Lampitt could say about how many sardines he thought people ate. It was quite right to decide on certain figures as a proper maximum, but if it were to be considered necessary to have a legal standard, the question of the injury to health arose. If a sardine weighed, say, 25 grms., how many sardines per week, had a man to consume to take, say, the maximum of 2 mgrms. per day, which had been suggested by Sir G. Newman as the safe limit?

Mr. A. L. BACHARACH asked whether, in those instances where a definite, if small, increase in lead-content on storage had been established, they had been able to detect any correlation between the amount of the increase and the acidity of the oil, which appeared in most instances to protect the sardines from the solder.

Mr. SAGE enquired whether the brine on the fish might not be responsible for the presence of some of the lead found in the sardines, for not only might that help to form the oxychloride on the metal hooks, but it might conceivably pick up lead from enamelled vessels in which the fish were handled, if the glaze was not free from that element.

Dr. LAMPITT, replying to the query why the solder on the grill contaminated the fish, said that, as pointed out, the Portuguese method was one whereby the biggest contamination took place, and this was probably due, so far as they had found out, to condensation of the steam on to the grills and subsequent contamination of the fish. It was quite likely that, as had been suggested, the salt present in the fish contributed to the solution of lead, particularly where the sardines were cooked by steaming.

They were not in a position to say that the exposed solder was of no influence. It might be a contributory factor, although they had had no evidence of this. In the sardines which had been kept with exposed solder the increase in lead was practically negligible, and they had therefore assumed that the exposed solder was of no very material importance.

The Chemical Examination of Furs in Relation to Dermatitis

BY H. E. COX, M.SC., PH.D., F.I.C.

PART II

RESULTS OF TESTS ON FURS ALLEGED TO HAVE CAUSED DERMATITIS

(*Read at the Meeting, October* 4, 1933)

SOME time ago I described methods of analysis which had been found useful in the testing of furs suspected of causing dermatitis (ANALYST, 1929, **54**, 694). The present paper sets out the result of a rather extended experience of the examination of furs, and of experiments which were designed to throw some light on the causes or possible causes underlying the disease. Since 1929 much has been published, both on the chemical and medical aspects; perhaps the more useful contributions have been those of Forster and Soyka (*J. Soc. Dyers and Colourists*, **1931**, **47**, **99**); Percival (*Lancet*, 1931, **417**, and *Brit. J. Derm.*, **1933**, **49**); Mayer and Sulzberger (*Arch. für Derm.*, 1931, **163**, 245); Mayer (*ibid.*, 223), and Ingram (*Brit. J. Derm.*, 1932).

All those who have experience of the investigation of cases of alleged fur dermatitis will agree that it is often very difficult to ascertain the true cause of the skin trouble suffered by the injured party. There is often doubt whether the irritation was really caused by the fur, and, if so, whether it was caused by a dyestuff, and, if so, by what dye? Further, was it due to imperfections in the materials or processes, or to the idiosyncrasy of the individual?

Controversy ranges round the question of why certain dyed furs cause dermatitis; some medical authorities merely say it is due to idiosyncrasy, others, that the reaction is allergic. The essence of an allergic reaction seems to be the production in the living cell of specific antibodies by the stimulus of foreign substances; according to Bloch the contact between the invading substance and its specific cellular antibody results in an inflammatory reaction. So far as I am aware, no real evidence has ever been adduced of the formation of such substances in relation to dyes or intermediates.

It is generally agreed that natural undyed furs do not cause dermatitis, assuming, of course, that they do not contain other irritant chemicals from dressing or bleaching operations. Percival expresses the opinion quite clearly that the irritant properties are not due to mechanical action. If this be so, it would appear from Table I that the clinical diagnosis is not always quite justified; the dermatitis may be certain enough, but the prefix "fur" may not be accurate, and I venture to think it cannot, in general, be so without a chemical examination of the suspected fur, in order to establish: (i) whether the fur was in fact dyed; (ii) if so, with what type of dye; and (iii) whether there is any unoxidised intermediate present? The reason for this is shown in Table I, which gives the results of my examination of 216 furs, all alleged to be dyed furs which had caused fur dermatitis. The results do not show what proportion of free base, if any, was present, but merely that the fur had or had not been dyed with a particular substance:

TABLE I

	No.	Percentage
Para-phenylenediamine	98	45·4
Meta-phenylenediamine	27	12·5
Textile dyes	3	1·4
Vegetable colours	20	9·3
Lead salts	3	1·4
Pyrogallol	3	1·4
Para-amino-phenol	4	1·8
Para-amino-dimethylaniline ..	9	4·1
Unknown amino compounds ..	12	5·6
Natural undyed furs	37	17·1
	216	100·0

It will be noted that the phenylenediamines head the list. The textile dyes are applicable only to certain types of dressing, and are not often met with in dermatitis cases. Of the vegetable colours, I have included only those which were free from topping with phenylenediamine or other compounds; such topping is fairly common in logwood dyeing, and is, in my opinion, the cause of the dermatitis which has sometimes been attributed to logwood. There are 12 cases in which amines were indicated, but I could not be sure of their identity.* I particularly wish to call attention to the fact that there were 37 cases of natural undyed furs, in which could be found no trace of irritant material. Is it the fact that some of these furs would cause irritation in the absence of dye, or is one to suppose that the disease was caused by some totally different agent?

I have sometimes heard it suggested that the outbreak may be due to some obscure reaction from contact with the skin of a particular animal—usually the rabbit—towards which the patient is sensitive. With this possibility in mind, it is interesting to observe the types of skin involved in these 216 furs examined. Table II shows the kinds of fur involved and their relative frequency:

TABLE II

	Dyed	Undyed	Total
Rabbit	89	4	93
Hare	5	1	6
Fox	2	2	4
Lamb	28	2	30
Opossum	11	3	14
Moleskin	7	3	10
Musquash	1	7	8
Seal	5	0	5
Skunk	4	5	9
Cat	5	0	5
Squirrel	4	6	10
Marmot	5	1	6
Pony	0	1	1
Monkey	0	1	1
Goat	2	0	2
Mink	0	1	1
Sable	1	0	1
Unknown	10	0	10
	189	37	216

Although the humble rabbit heads the list, there is no reason to think that it is specifically irritant; it is the cheapest and most frequently dyed fur, and seldom appears in its natural undyed state. Lamb wool comes next in order of frequency. It is difficult, indeed, to think of anything specific in wool which could cause dermatitis, and 28 out of 30 of the furs in these cases were dyed. Lamb dyed with phenylenediamine mixtures, appearing as nutria, is very popular, and lamb blackened with logwood and suitably treated passes as astrakhan.

Opossum is very often dyed to represent skunk, which probably accounts for its frequent appearance. Musquash is not often dyed, and seal is sometimes the subject of a special process involving textile colours. Specimens of lynx and susliki have been included as cat and marmot respectively, as they belong to those families, and there are 10 of the identity of which I was not certain. It is of interest to notice that pony and goat fur, which are rather harsh to the skin, do not often appear. There seems no reason to think that the particular type of fur has anything to do with dermatitis if it is not dyed.

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* The probable reason for this failure to establish the identity of the amino compounds will appear in Part IV.

In view of the preponderance of the *p*-phenylenediamine cases, it seems desirable to study this substance in more detail. Out of about 100 cases examined, the amounts of *p*-phenylenediamine found in an unoxidised state varied from 0·15 per cent. by weight of the hair down to the merest trace. If it be taken that a black-dyed hair contains about 5 per cent. of oxidised *p*-phenylenediamine, it will be seen that this represents a residue of about 3 per cent. imperfectly oxidised and not washed out. In some cases the amount of free and incompletely oxidised base is so small as to raise doubts whether it could possibly cause dermatitis.

The next point is as to the mode of action of *p*-phenylenediamine. Is it a definite chemical or pharmacological action, or must one invoke the mysterious word allergy? Percival, in 1931, definitely expressed the opinion that it is an allergic action. I notice that in 1933 he remarks that this theory "presupposes the existence of cell-fixed anti-bodies . . . but as there is no direct evidence of the participation of anti-bodies in the eczema reaction the factor of individual susceptibility or idiosyncrasy so constantly associated with it, does not permit of the theory being adopted for all cases."

Ingram (*loc. cit.*) adheres to the theory of allergy. He shows that on application of *p*-phenylenediamine as a patch test about 4 per cent. of normal individuals react after a latent period varying up to 24 days. Babalien and Reitlinger oppose the theory of allergy, and regard the effect as a purely local eczema. One may be permitted to ask, if a reaction to a low concentration is allergic, is a reaction to a high concentration also allergic, or is it pharmacological? It has been observed by Bloch and Steiner Wourlisch (*Arch. für Derm.*, 1926, **152**, 283) that when the concentration is increased non-sensitive individuals will react to certain potential irritants; even with primula extracts, to which most people are insensitive. 100 per cent. of positive reactions were obtained with a sufficiently strong extract applied to 12 persons.

It seems to me that we ought not to have recourse to the word allergy until all other possibilities have been considered.

It is generally agreed that *p*-phenylenediamine is a strong poison. The experimental basis for this need not be recited in detail, for there is much evidence in the literature. It is a blood poison producing oedema, salivation, urticaria, eczema and various other unpleasant effects. Sartory and Rousseau (*Bull. Sci. Pharmacol.*, 1912, **19**, 338, 520) show that, upon intraperitoneal injection into rabbits, these various ill-effects and death result, whereas the oxidation product, Bandrowski's base, is harmless when so injected. Among many papers dealing with the physiological effects may be mentioned those of Erdmann and Vahlen (*J. Soc. Chem. Ind.*, 1906, 68), Erdmann (*Z. angew. Chem.*, 1905, 1377), Soltmann (*J. Pharm. Exp. Therap.*, 1917, **9**, 391), and O. S. Gibbs (*ibid.*, 1922, **20**, 221). Opinions differ as to whether the diamine itself or an intermediate oxidation product is the true irritant.

I propose to discuss in detail the identity and properties of the oxidation products of *p*-phenylenediamine in another paper, but it is convenient, before passing on to the reactions of *p*-phenylenediamine with the blood, to refer to the detection and determination of traces of the free unoxidised base in furs.

The detailed constitution of the final oxidation product of *p*-phenylenediamine on fur is not yet fully known, but it will be shown in Part IV of this series that, under ordinary conditions, there is always included some Bandrowski base on the surface of the fibre. It will be found that, on treating furs dyed with diamines with mineral acid, some of this base is stripped off and a brown solution is obtained which gives all the reactions of the Bandrowski base and some of those of the original phenylenediamine. Further, the colour may be reduced either to a leuco compound, or even to the diamine itself, by strong reducing agents—a procedure which is often very useful in the identification of the amine. Such decomposition does not occur with water at blood-heat, or appreciably with faintly acidified solutions. At 37° C. in water the brown base is hydrolysed only to the extent of 0·2 per cent. in 24 hours; with 0·1 per cent. acetic acid and normal saline, which forms a convenient artificial sweat mixture, having p_{H} approximately 3, the hydrolysis at blood-heat is about 2 per cent. Sheepskin dyed with *p*-phenylenediamine, and washed free from the base was found to yield 0·2 per cent. of apparent *p*-phenylenediamine after 2 hours on the water-bath with *N*/10 hydrochloric acid.

In order to study the degree of decomposition of Bandrowski's base it is necessary to have a means of differentiating it from *p*-phenylenediamine and determining the latter in the presence of the former. Forster and Soyka describe the indamine and certain other reactions of Bandrowski's base in a manner which would imply that one cannot differentiate the two. I find this is not the case if all traces of *p*-phenylenediamine are first removed.

The base was carefully prepared by the method given by Erdmann (*Ber.*, 1904, **37**, 2908), and was repeatedly washed with dry ether to remove traces of unoxidised diamine; it had m.pt. 242° C. The base so prepared was crystalline and soluble in cold water to the extent of 0·13 grm. per litre. The pure base gives neither the indamine nor the indophenol reaction, and does not form a dichlor-di-imine with hypochlorite. The reactions of a saturated aqueous solution, as compared with a corresponding strength of *p*-phenylenediamine, are tabulated below:

Reagents added to 10 c.c.	Bandrowski's base	*p*-Phenylene-diamine hydrochloride
1. Indamine [3 drops of aniline chloride (1 per cent.) and 3 drops ferric chloride]	No colour	Intense green-blue
2. Indophenol (3 drops of 1 per cent. phenol, 3 drops sodium hypochlorite)	No colour	Intense violet
3. Hydrochloric acid and sodium nitrite	Slight brown with acid, then bleaches	Yellow brown
4. Sodium-R salt added to 3	Very slight orange	Strong red
5. One drop of *N*/1 hydrochloric acid and 1 c.c. of alcoholic solution of *p*-dimethylaminobenzaldehyde	Dirty brown	Deep red
6. Ferric chloride and hydrogen sulphide solution	No change	Lauth's violet

Bandrowski's base is decomposed when boiled with hydrochloric acid, and can be reduced by stannous chloride or zinc dust.

p-Phenylenediamine, in appreciable quantities, can be titrated with sodium hypochlorite solution in the manner described by Callan and Henderson (*J. Soc. Chem. Ind.*, 1919, **38**, 408T); the limit of sensitiveness is about 1 c.c. of 0·01 *N*, which is equivalent to 0·18 mgrm. of *p*-phenylenediamine. In smaller quantities the indamine reaction is most convenient, though it is not specific for this diamine. It may be applied quantitatively by direct comparison or, with a little less accuracy, by the use of the tintometer, if due attention be given to the p_{H} value of the solution. A solution containing 2 parts per 100,000 gives the following colours when 3 drops of a 1 per cent. solution of aniline hydrochloride and 3 drops of 1 per cent. ferric chloride solution are added to 10 c.c., and the colour is observed in a 1-cm. glass cell in Lovibond's tintometer:

p_{H}	.. ..	1·2	2·0	3·0	4·0	5·0	6·0	7·0
Colour	Blue ..	2·2	5·5	8·5	10·0	10·0	9	–
	Yellow	1	2	3	3	3	3	–

The maximum colour is seen at approximately p_{H} 4·5; it is developed within a minute, and lasts several minutes before changing. Dichromate is not suitable as the oxidising agent, as it gives too green a colour. Working at p_{H} 4·5, the approximate concentrations of *p*-phenylenediamine are determinable colorimetrically by reference to a table:

p-Phenylenediamine, parts per 100,000		1	2	3	4	5	6	7	8	9	10
Colour	Blue	6	10	11	12	13	14	15	16	17	18
	Yellow	2	3	3	3	3	3	4	5	6	8

One cannot expect a high degree of accuracy in such a colorimetric process, as there is some room for variation in judgment of the colours. At concentrations a little above 1 in 10,000 blue flecks of the indamine appear, and colorimetry becomes impracticable. Using this colorimetric process it was found that Bandrowski's base, when properly prepared and purified from traces of free *p*-phenylenediamine, gave no indamine reaction when extracted with cold water; when incubated with natural human sweat at blood-heat for 48 hours it showed only a trace of indamine, indicating a hydrolysis of less than 0·1 per cent. The concentration used for this test was 5 mgrms. of base with 0·5 c.c. of sweat.

The evidence available seems to show that Bandrowski's base is harmless, and that it is not decomposed on the fibre by sweat, or by moisture, such as may result from exposure of the dyed fur to rain. This leads to the corollary that if a fur be once properly washed, so that all free *p*-phenylenediamine is removed, none will subsequently be formed by either of these agencies, and, in the absence of any other toxic intermediate products, there can be no question of dermatitis being caused as a result of anything in the fur.

PART III

THE ACTION OF *p*-PHENYLENEDIAMINE ON THE SKIN

The question of what physiological reactions may arise if free *p*-phenylenediamine is present in a fur is worthy of the closest study because, as has already been shown in Part II, it is by far the most frequent cause of fur dermatitis; and, secondly, it may be regarded as the typical member of a group of potentially irritant substances used as fur dyes. The mode of action of *p*-phenylenediamine, if it can be traced, is likely to provide the key to the mode of action of the other members of the group, including the other substituted diamines and aminophenols.

Quastel (*Biochem. J.*, 1931, **25**, 629, 899) has shown that certain dyestuffs are toxic to bacteria, and that certain enzyme actions are inhibited at very low concentrations, of the order of 1 in 10^5 or 10^6; it seems reasonable to suppose that such an action must depend upon the absorption of the substance into the cells. Thus there seem to be three aspects to consider in connection with *p*-phenylenediamine:

> (i) The absorption or penetration of the diamine; (ii) the reactions of *p*-phenylenediamine with the blood or serum; (iii) the presence in the blood or serum of abnormal substances or normal constituents under different conditions.

(i) THE PERMEABILITY OF THE SKIN.—If the finger be dipped in a 1 per cent. solution of *p*-phenylenediamine for a few minutes, a dark brown stain is produced which cannot be completely washed off, so that evidently the surface layer or epidermis is affected. Dead skin, likewise, is permeable; experiments with a 1 per cent. solution showed that it diffused out through a sheep's bladder, the skin of which was 0·13 mm. thick, in less than a quarter of an hour. The coloured oxidation-products likewise passed through quite rapidly, and the skin was dyed black.

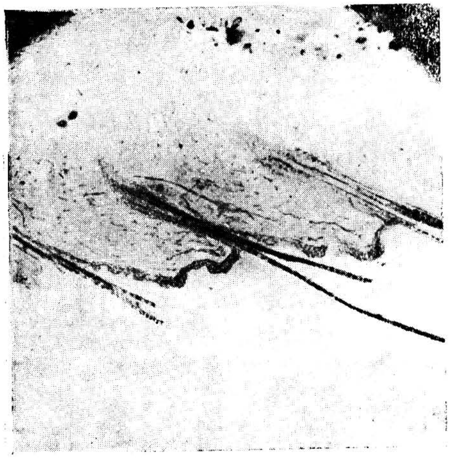


Fig. 1

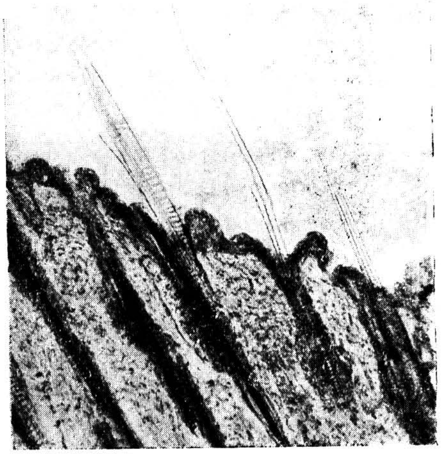


Fig. 2

This does not appear to be the case with living tissue. The same 1 per cent. solution was applied to two white mice; the skin of one was washed with ether, to remove any fat, and then painted with the diamine, whilst the other was painted without any de-fatting. The first mouse licked itself vigorously, and, in consequence, died in a few hours; the second did not lick, and suffered no inconvenience. It was killed after 24 hours and the skins of both animals were examined. The epidermis was stained a brownish-black, but tests on the inner surface with aniline and ferric chloride showed no trace of *p*-phenylenediamine in either case. Transverse sections were cut, using the freezing method to avoid any extraction by a solvent; these were mounted, and I endeavoured to develop any diamine absorbed as an indamine, which shows as a blue-green colour. Fig. 1 shows the result of such treatment; it is unstained with any dye, and it is clearly seen that only the outer horny layer is coloured at all. There is no evidence of any penetration, even with the de-fatted skin of the white mouse which died. Fig. 2 shows another section of the same skin stained with haematoxylin, with the object of observing any change in the tissue or cell structure. It will be seen that there is no abnormal appearance, a fact borne out by study under a higher magnification.

These observations are in agreement with those recorded by Mayer (*Arch. für Derm.*, 1931, **163**, 223), who experimented on a number of guinea-pigs, applying a 10 per cent. emulsion of *p*-phenylenediamine in vaseline to the shaved skin. With non-sensitised animals there was no absorption of colour except at the surface in the horny layer, whereas in sensitised animals there was evident infiltration, with destruction of epidermal cells, though Mayer does not appear to have actually identified the amine or imine in the tissues.

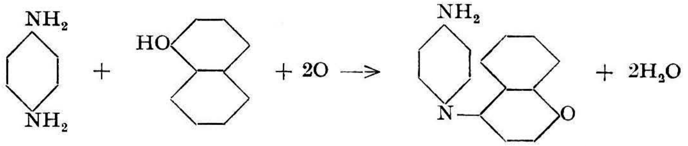
I have also experimented on dead skin, since it is well known that dead cells are more easily stained than live ones. A mouse was killed (by coal gas), and a piece of its skin removed 24 hours later; this was tied over the end of a smooth glass tube, and a few drops of the 1 per cent. solution of *p*-phenylenediamine were added to the outer surface. After 24 hours a distinct indamine could be developed on the inner surface, and examination of a section showed that the *p*-phenylenediamine had penetrated. This confirms the view that, whilst living skin does not absorb the diamine, dead tissue offers no particular resistance to diffusion.

Experiments on the human subject are, no doubt, desirable to enable definite conclusions to be drawn, but the evidence available seems to show that *p*-phenylenediamine does not normally pass into the cells of the living skin; when it does do so, further reactions ensue which may or may not produce dermatitis.

(ii) REACTIONS WITH THE BLOOD OR SERUM.—*p*-Phenylenediamine is so reactive a compound that it is hardly likely that its introduction into the blood or cells could fail to cause at least a local disturbance. It is not necessary to suppose a general absorption into the blood stream, such as is followed by a general toxaemia, *e.g.* as caused by burns. Its addition to blood causes de-oxygenation, and spectroscopic examination showed the reduction of oxyhaemoglobin to methaemoglobin. Szent-Györgyi (*Biochem. Zeitsch.*, 1925, **157**, 79) shows that the purple oxidation compound of *p*-phenylenediamine is reduced to a colourless compound by reducing systems in the tissues, and that this change can be brought about by living muscle tissue and lactic acid. The first oxidation product, quinone-di-imine, is reduced back to the amine by substances in cellular tissue. Keilin (*Proc. Roy. Soc.*, 1929, [B], **104**, 206) shows, amongst other things, that this amine greatly increases the oxygen-uptake of heart muscle.

There can be little doubt that anything which interferes with oxidation-reduction processes or enzyme action in the living cells is deleterious or irritant. Harrison (*Biochem. J.*, 1929, **23**, 982) shows that *p*-phenylenediamine is oxidised to quinone-di-imine by hydrogen peroxide formed in the living cell, and may be reduced again therein by reducing systems in the cell. The poisonous properties of quinone-di-imine are known to be intense; its production may be recognised by the formation of the characteristic coloured compound.

The indophenol reaction has been much used for the study of enzyme oxidation-reduction processes; indophenol is produced from the diamine and phenolic compound only in the presence of a suitable oxygen carrier, in accordance with the equation:



The reaction can be brought about by weak hypochlorite, but not by hydrogen peroxide, except in the presence of an oxidase.

I have made experiments with human blood, blood serum and albumen, and the results show that *p*-phenylenediamine or quinone-di-imine reacts with blood serum in a manner similar to its reaction with albumen, and that the presence of the red corpuscles supplies the oxygen necessary for other reactions, such as the production of indamine and indophenols; the reactions are not instantaneous, but take place in an hour or so at 37° C. Blood serum, to which a 1 per cent. solution of *p*-phenylenediamine is added, develops a deep red colour, then the serum sets to a jelly, and later a slight precipitate forms. A similar action takes place with white of egg. When blood serum is mixed with saturated aqueous solution of α-naphthol and 1 per cent. *p*-phenylenediamine is added, there is no formation of indophenol, but, on adding hydrogen peroxide, indophenol results, showing that the serum provides the oxidase. If red blood be used instead of serum, indophenol results, without the addition of peroxide, showing that the haemoglobin supplies the necessary oxygen, which, with the oxidase from the serum, brings about the reaction. Control tests with normal saline showed that the precipitation which ultimately follows is not due to haemolysis.

Quinone-di-imine coagulates albumen or blood serum, as does *p*-phenylenediamine, but there is a further interesting point, that a dark red jelly is produced which does not proceed to give Bandrowski's base, whereas quinone-di-imine added to water quickly produces this base. Some of these reactions are tabulated below:

Temperature 37° C.; 1 per cent. solution of *p*-phenylenediamine added to:

1. Blood Serum

(*a*) Normal saline	No change, slight brown colour
(*b*) Blood serum	Deep red colour. Jelly forms and slight precipitate
(*c*) Blood serum + α-naphthol	Slight colour slowly develops
(*d*) Serum + α-naphthol + H_2O_2	Strong indophenol reaction
(*e*) Serum + H_2O_2	Reddish-black colour, then ppt.; serum jellified

2. Human Blood

(*a*) Blood	Haemoglobin reduced to methaemoglobin; sets to a jelly and darkens in colour
(*b*) Blood + α-naphthol	Slight indophenol reaction
(*c*) Blood + α-naphthol + H_2O_2	Strong indophenol reaction; dark brown or black ppt.
(*d*) Blood + tyrosine	Intense violet indophenol

3. White of Egg

(*a*) Albumen	Red colour; jelly forms.
(*b*) Albumen + tyrosine	Slight violet colour
(*c*) Albumen + hydrogen peroxide	Dark brown-black jelly
(*d*) Albumen + α-naphthol	Slight colour only

4. Quinone-di-imine added to:

(*a*) Water	Brown solution gradually forming black ppt.
(*b*) Blood serum	Dark reddish colour
(*c*) Blood	Coagulates, and darkens in colour
(*d*) White of egg	Dark red jelly forms

The main point arising from these experiments seems to be that the diamine reacts with the proteins of albumen or serum to form a jelly and an oxidation product; cell oxidases are present and, if active oxygen be supplied, either by red corpuscles or by peroxide, indamines or indophenols are produced when the necessary amine or phenol is also present.

In view of the de-oxygenation of the cells and the coagulation of proteins which evidently follows the introduction of *p*-phenylenediamine into the tissue, it seems remarkable that, if there is penetration, an irritation is not the rule rather than the exception.

(iii) ABNORMAL CONSTITUENTS OF THE SERUM.—The last point to which I wish to draw attention is the possibility of variation in the constituents of the blood or human serum arising from individual health, diet and unknown causes. The wide range of reactivity of *p*-phenylenediamine and its imines suggests how varied are the possibilities. If dermatitis were a necessary consequence of the passage of the irritant base through the skin and its action on normal blood or cellular tissue, it would be possible easily to demonstrate it by inducing the disease by injection or applying the amine or imine to the broken skin. I have tried this and found that with an individual who did not react to a 1 per cent. solution applied to the unbroken skin there was no typical dermatitis produced after scarification. The 1 per cent. solution was applied for 2 hours; it caused some irritation and a sore place, but the characteristic pustules were not formed. Percival likewise has recorded (*loc. cit.*) that application of his Ursol* by scratch did not generally produce dermatitis—a fact which he cites in support of the theory of allergy.

It seems possible, even likely, that diet affects the substances present in the serum, and so has important bearing on the development of dermatitis or its absence. The experiments of Mayer and Sulzberger (*Arch. für Derm.*, 1931, **163,** 245) are most striking in this connection. They worked on about 300 guinea-pigs, using organic arsenicals, as well as *p*-phenylenediamine in rather high concentration, and found that by changing over from summer fodder (with an alkaline ash) to dry winter feed the percentage reaction changed from 12 per cent. to 100 per cent. That this was not due to vitamin-shortage was proved by supplying many of the animals with a vitamin concentrate containing *A*, *B*, *C*, and *D*.

Just to explore one of the many possibilities, I have tried the reactions of *p*-phenylenediamine and quinone-di-imine on tyrosine. This substance, as is well known, is a widely distributed hydrolytic product of proteins, which is not normal in blood, but occurs in some pathological states; it is found in muscle whenever there is any decomposition or putrefaction. It seems possible, therefore, that it might arise when such a deoxidising agent as *p*-phenylenediamine is introduced. It is known that tyrosine decomposes in the body into phenol and cresol.

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* The Ursol A which Percival used was probably acetyl-*p*-phenylenediamine.

The colour reactions of *l*-tyrosine with *p*-phenylenediamine were found to be as follows when applied to a saturated aqueous solution (*i.e.* about 1 in 2000):

1.	Tyrosine+*p*-phenylenediamine	Slight brown only
2.	Do. do. +ferric chloride	Strong green indamine quickly changing to brown precipitate
3.	Do. do. +dichromate	Green changing to safranin red
4.	Do. do. +sodium hypochlorite	Intense indophenol violet
5.	Do. do. +blood serum	Slight violet colour
6.	Do. do. +blood serum+peroxide	Intense indophenol violet quickly formed

It will be seen that the reactions observed with blood or with serum and *p*-phenylenediamine are similar in type to those given when tyrosine is added, though they take place more slowly. It is not suggested that this proves anything, but it does illustrate the possibilities arising in differing individuals or resulting from different diets.

In conclusion, I think it may be said that the available experimental evidence points to free *p*-phenylenediamine as the potential irritant which may act through its early oxidation products and condensations. Normally these substances do not pass into the cells or living tissues of the skin, but merely stain the horny layer. Dermatitis seems to arise only when the irritant passes into the epidermis and reacts with the serum as a result of special conditions. These conditions include the abnormal permeability of the outer layer and an abnormal chemical or physical constitution of the serum. The coagulation and oxidation effects would preclude its passage into the blood-stream or action at places remote from the site of penetration.

SUMMARY.—Details are given of the kind of fur and mode of dyeing of 216 furs suspected of having caused dermatitis; of these 37 were undyed, and it is thought that they could not have caused the disease. Chemical tests for the differentiation of *p*-phenylenediamine from Bandrowski's base are given, and a colorimetric method for determining small quantities of the diamine. Experiments are described showing that *p*-phenylenediamine penetrates the dead skin readily, but does not pass through or into living skin under normal conditions. Some of the reactions of *p*-phenylenediamine with blood and serum are described, and it is shown that it coagulates the proteins and becomes oxidised at their expense; the oxidases present in blood suffice to bring about certain reactions, and, if haemoglobin is present, these reactions proceed further. Tyrosine with albumen produces colour reactions similar to those observed with blood. It is suggested that irritation is determined by the abnormal penetration of the diamine through the skin, followed by its local reaction with certain constituents of the blood or serum yet unknown.

11 BILLITER SQUARE
LONDON, E.C.3

The Determination of Methyl and Ethyl Alcohols in Mixtures containing Acetone and its Homologues

BY RONALD W. HOFF, A.I.C., AND JOHN M. MACOUN

THE determination of methyl and ethyl alcohols in the presence of acetone and its homologues is frequently important for fiscal purposes. None of the methods hitherto proposed has been reliable. The precipitation of acetone by the use of mercuric sulphate fails in the presence of more than 2 per cent. of ethyl alcohol. The conversion of alcohols into alkyl iodides gives results consistent among themselves only because the errors nearly balance one another. The method proposed by one of the present authors (*J. Soc. Chem. Ind.*, 1928, **47**, 43T) shows errors, where methyl alcohol is in question, so serious as to make it inapplicable in an important class of cases. Obviously any method based on refractive index and density is inapplicable where three constituents (methyl alcohol, ethyl alcohol and water) are present in addition to the acetone.

The reaction between acetone and an excess of formaldehyde in the presence of alkali has for some time been known to result in the formation of non-volatile products, and we have now found that it proceeds quantitatively to the elimination of acetone. On these facts it has been possible to base the method we now propose.

The following is a description of its application to a liquid free from volatile oils or any excessive proportion of acetone.

The sample, which must be of such volume that it does not contain more than 20 ml. of alcohol, may best be measured into a 500-ml. Kjeldahl flask, and diluted with water to a volume of approximately 100 ml. For each 1 ml. of acetone supposed to be present, 1·5 grm. of paraformaldehyde should be added, and followed by 50 ml. of a saturated solution of common salt and 20 ml. of *N* sodium hydroxide solution.

The flask is fitted to a vertical Liebig condenser not less than 60 cm. in length (or to some other pattern of condenser of equivalent efficiency), and the mixture is brought, slowly, just to the boiling point, by which time the reaction will be complete. An electric hot plate is the most convenient source of heat.

To the just-boiling mixture, excess of Fehling's solution (usually 50 to 60 ml. suffice) is added through the top of the condenser, after which the flask is cooled to room temperature. The supernatant liquid must remain distinctly blue.

The condenser is rinsed down with a little water, the flask removed, and an open glass tube, about 15 cm. long, slid down into it to facilitate gentle boiling during the ensuing distillation. The flask is arranged for distillation through a foam-trap and a vertical condenser fitted with a straight adapter. The contents of the distillation flask are brought to the boiling point with a small flame, which, when the initial slight frothing has subsided, may be increased. The distillate is collected in a 100-ml. graduated flask. The specific gravity of the distillate is then determined at the standard temperature for such purpose.

Paraformaldehyde of the usual medicinal grade contains small amounts of nitrogenous bases which are carried over into the distillate. Unless their absence is assured, the reagent must be purified before use by washing, first with dilute hydrochloric acid, then with water till the removal of acid is complete, and finally by air-drying.

It was found that the addition of salt to the alcoholic solution before the alkali caused the resin to form in smaller particles, and hence minimised the danger of occlusion of alcohol.

Where the acetone-content is high, or where volatile oils are present, the alcoholic liquid should be treated with petroleum spirit in the manner described by Thorpe and Holmes (*J. Chem. Soc.*, **1903**, **83**, 314). This is notably the case where the material under investigation is wood spirit.

In examination of this latter, the analytical result is the percentage of "free" and "combined" alcohol together, since the Thorpe–Holmes extraction does not remove any appreciable proportion of the methyl esters always present. This is shown by the fact that the same result is given whether this extraction is made on the sample at the outset of the analysis or is omitted till after the treatment with alkaline solutions and distillation therefrom.

TABLE I

ETHYL ALCOHOL

Original		Distillate	
Sp.gr.	Alcohol Per cent. by vol.	Sp.gr.	Alcohol Per cent. by vol.
0·9721	23·8	0·9723	23·6
0·9720	23·9	0·9723	23·6
0·9720	23·9	0·9721	23·8
0·9843	12·0	0·9845	11·8
0·9843	12·0	0·9845	11·8
0·9843	12·0	0·9844	11·9
0·9916	5·9	0·9917	5·9
0·9916	5·9	0·9917	5·9
0·9916	5·9	0·9917	5·9

METHYL ALCOHOL

Original		Distillate	
Sp.gr.	Alcohol Per cent. by vol.	Sp.gr.	Alcohol Per cent. by vol.
0·9683	24·8	0·9690	24·3
0·9683	24·8	0·9690	24·3
0·9681	25·0	0·9686	24·6
0·9834	12·5	0·9838	12·2
0·9833	12·6	0·9833	12·6
0·9833	12·6	0·9836	12·4
0·9879	9·0	0·9881	8·8
0·9879	9·0	0·9881	8·8
0·9915	6·2	0·9915	6·2

The foregoing figures show the degree of accuracy that may be expected from fairly simple alcoholic liquids when all manipulative precautions are observed. In each case a known volume of alcohol was taken and acetone was added to it, the resulting mixture being submitted to the treatment already described. The amount of acetone employed varied in each determination. The specific gravity of the alcohol used was determined for each group of samples. After the final distillation, the distillates were free from acetone as indicated by Legal's nitroprusside test, and showed only a trace of formaldehyde by Schiff's test. The very delicate and more specific Schryver test for formaldehyde also showed the presence of a mere trace.

The errors are evidently increased as the alcoholic strength increases. Everything points to this being due to loss of alcohol-vapour during digestion.

Readings by the immersion refractometer were obtained on the original solution and on the distillate in most of the above cases, and the alcohol indicated thereby agreed satisfactorily with that obtained from the specific gravity.

TABLE II

MIXTURE OF ETHYL AND METHYL ALCOHOLS (approximately 25 per cent. total alcohols)

Original solution				Distillate		
Sp.gr.	Refractometer reading	Methyl : ethyl alcohols, as indicated by Sp.gr. and Ref. read.		Sp.gr.	Refractometer reading	Methyl : ethyl alcohols, as indicated by Sp.gr. and Ref. read.
0·9719	49·5	11·8 : 88·2	(i)	0·9722	49·0	11·6 : 88·4
			(ii)	0·9723	49·0	11·6 : 88·4

The calculations involved are based on the tables of Specific Gravities and Refractometer Readings, published in *Alcohol*, by Charles Simmonds (Macmillan & Co., Ltd., London, p. 285).

Experiments were made to determine whether, under the conditions of the above method, the formaldehyde underwent any conversion into methyl alcohol (and formate) according to the Cannizzaro reaction. About 150 ml. of water, containing 3 ml. of acetone, were digested with 4 grms. of paraformaldehyde and 20 ml. of *N* sodium hydroxide solution, the method above given being followed to its conclusion. The specific gravity and refractometer reading of the final distillate were determined, resulting as follows:

Sp.gr. of distillate	Refractometer reading of distillate	Refractometer reading of water
0·9998	15·5	15·4

The distillate was free from acetone, and contained a trace from formaldehyde. These figures show that no methyl alcohol formation takes place under the conditions given. This is confirmed by a comparison of the refractometer readings of ethyl alcohol solutions, before and after treatment to remove acetone. The following are examples:

Ref. reading of original before addition of acetone	Ref. reading of distillate after removal of acetone
52·3	52·3
32·3	32·4
32·3	32·3

All the above specific gravities and refractometer readings were determined at 60° F.

A typical application of the foregoing method is the determination of alcohol in wood spirit.

In this instance, 20 ml. of sample were run from a burette into 50 ml. of petroleum spirit. The Thorpe–Holmes method of extraction was applied to the mixture. To the resulting 17 ml. of alcoholic saline solution, 2·5 grms. of paraformaldehyde and 20 ml. of *N* sodium hydroxide solution were added (an iodimetric determination had shown about 10 grms. of acetone to 100 ml. of the original spirit, but this proportion is considerably reduced by the extraction). The determination was carried through in the manner already described. The final distillate (100 ml.) had a sp.gr. of 0·9794 at 60° F. corresponding with 78·8 per cent. by volume of methyl alcohol in the sample.

We wish to acknowledge our indebtedness to Dr. Alfred Tingle, Chief of the Customs–Excise Laboratory, at whose suggestion this work was undertaken, for advice and encouragement during its progress. He wishes us to state that, subject to any criticism which may follow publication, this method will be adopted as a standard procedure in this Laboratory.

Customs-Excise Laboratory
Department of National Revenue
Ottawa, Canada

A Volumetric Method for the Determination of Barium and of Sulphates

By J. C. GIBLIN, B.Sc., A.I.C.

The use of rhodizonic acid, in the form of its sodium salt, for the detection of barium and strontium, was suggested by Feigl (*Mikrochem.*, 1924, **2**, 188). This reagent, when spotted on filter-paper, gives a reddish-brown colour with barium or strontium in neutral solution. On acidification, the barium spot becomes bright red or pink, whereas the strontium colour is destroyed.

I have endeavoured to apply these reactions to the volumetric determination of sulphates and of barium in acid solutions. Preliminary work showed that the reagent did not work well when used as an internal indicator, or even when drops were placed in depressions on a white tile. Finally, the following method was found to be suitable:—Drops of the indicator were spotted on filter-paper, thereby forming a yellow stain. (*a*) Standard sulphuric acid was placed in a titration flask, barium chloride solution was added from a burette, and the contents of the flask were well mixed. The precipitate was allowed to settle, and drops of the supernatant liquid were removed and spotted on to the filter-paper. The appearance of a pink or red colour indicated the completion of the reaction. (*b*) Barium chloride solution was placed in the titration flask, and sulphuric acid was added from the burette until drops taken from the supernatant liquid ceased to give a red colour on the filter-paper.

In a preliminary experiment to test the possibilities of the method, separate portions of a solution of 12·20 grms. of barium chloride crystals in 1 litre were given to three different observers. The following results were obtained:

Observer A.—The barium solution (25 c.c.) required:—(i) 27·1 c.c.; (ii) 27·1 c.c., of sulphuric acid (1·097 $N/10$).

Observer B.—The 25 c.c. required:—(i) 27·0 c.c.; (ii) 27·0 c.c., of sulphuric acid. (Mean of A and B = 27·05 c.c.)

Observer C placed sulphuric acid in the flask.—The 25 c.c. of sulphuric acid (1·097 $N/10$) required:—(i) 22·5 c.c.; (ii) 22·6 c.c., of barium chloride solution. (Mean = 22·55 c.c.)

According to these results A and B found the solution to contain 6·945 grms. of barium per litre, and C found 6·777 grms. (Mean = 6·88 grms.) Weighed quantity 6·85 grms.

An arbitrary solution of barium chloride was then made. To it was added an excess of hot solution of pure sodium carbonate (A.R.). The precipitated barium carbonate was thoroughly washed with water and with ether, and was then dried in a steam-oven, and, finally, in a desiccator. A standard solution of barium was prepared by dissolving a weighed quantity of the carbonate in dilute hydrochloric acid and diluting with water.

Titrations made by two observers, working independently, gave the following results:

Observer A.—(*a*) Sulphuric acid (10 c.c. of 1·097 $N/10$) required:—(i) 10·28 c.c.; (ii) 10·32 c.c., of barium chloride solution. (Mean = 10·30 c.c.)

(*b*) Barium chloride solution (10 c.c.) required:—(i) 9·95 c.c.; (ii) 9·80 c.c.; (iii) 9·90 c.c.; (iv) 10·05 c.c., of sulphuric acid (1·097 $N/10$). (Mean = 9·92 c.c.)

Observer B.—(*a*) Sulphuric acid (10 c.c. of 1·097 $N/10$) required:—(i) 10·45 c.c.; (ii) 10·40 c.c.; (iii) 10·45 c.c., of barium chloride solution. (Mean = 10·43 c.c.)

(*b*) Barium chloride solution (10 c.c.) required:—(i) 9·90 c.c.; (ii) 9·85 c.c., of sulphuric acid (1·097 $N/10$). (Mean = 9·87 c.c.)

From these results the amount of barium per litre of solution was calculated: *A*.—(*a*) 7·30; (*b*) 7·45; aver. 7·375 grms. of barium per litre. *B*.—(*a*) 7·20; (*b*) 7·42; aver. 7·31 grms. of barium per litre. Theory, 7·336 grms. of barium per litre.

CONCLUSION.—It appears that the use of rhodizonic acid in the form of its sodium salt, under the conditions described above, is suitable for the rapid determination of barium salts and of sulphates, even in acid solutions. The method is not less accurate than other volumetric methods employing external indicators. By reversing the process of titration and taking the mean of the two results we get a final result of reasonable accuracy. The solutions employed were all of less than 1 per cent. strength. Stronger solutions would probably give a clearer end-point.

My thanks are due to those pupils who assisted me in this work, and especially to T. G. Boaz, Exhibitioner of Trinity Hall, Cambridge.

PERRINS LABORATORY
ROYAL GRAMMAR SCHOOL
WORCESTER

Notes

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

PEARL BARLEY INFECTED WITH SMUT

DURING the last three years I have examined a considerable number of samples of pearl barley, including some imported from abroad, and I noticed that a few of the latter contained dark-coloured grains suggesting that the barley from which they had been produced had been affected by smut. With one exception the proportion of these fungus-affected grains did not appear sufficiently serious for me to condemn the barley. In one lot, however, I observed a fairly large number of suspicious grains, and I therefore determined to make a fuller examination of the consignment, which consisted of 25 1-cwt. bags. I had 10 of these examined at the factory where they had been delivered, and had the dark-coloured grains picked out. Most of them were affected with the barley smut, and some were also affected with ergot. I certified that, in my opinion, the pearl barley had been prepared from damaged barley, which had been affected by smut and ergot. My opinion was disputed by the exporters, and they requested that a sample of the pearl barley should be sent to them; this was done. In their reply they stated that their analyst could find no evidence of smut or ergot, and requested me to have a sample sent to an independent continental analyst. I sent a portion to him, together with some of the pickings which I had obtained from the bulk, but nothing further was heard, and the consignment was shipped back.

I took a portion of the pickings to Professor McLean Thompson, of Liverpool, and he confirmed my finding, and stated that the ergot was not the usual one found in rye, but was one that affected barley, and that the smut was *Ustilago hordei*.

I did not regard the amount present in the barley, when received, to be serious, but in my certificate I expressed the view that the use of damaged grain, even though it undergoes decortication for the preparation of pearl barley, is reprehensible, especially as this is an article used for preparing a diet for invalids.

2 BLACKBURNE PLACE
LIVERPOOL

P. H. JONES

THE FORMALIN TITRATION OF PROTEINS

IN the October ANALYST (p. 605) Mr. J. C. Harral discusses the formol titration of re-constituted milk and the ratio between the total nitrogen and the formol titration.

In work of this kind it would appear to be necessary to distinguish between "drum" powders and "spray" powders. Krause spray-dried milk powder is dried at a very low temperature. On the Continent Krause milk (re-constituted) has given much trouble to analysts (*cf.* Tillmanns and Strohecker, *Z. Nahr. Genussm.*, 1924, **47**, 377).

In 1917–18, I determined the "ratio" values for a number of commercial protein substances, with the results given below. Richmond (*Dairy Chemistry*) states that casein and albumin do not give the same aldehyde figure. My "ratio" values, calculated for milk of ordinary composition, happen to correspond with a "ratio" of 23—something between that of fresh milk (18·7) and Mr. Harral's figures for re-constituted milks (about 27).

The "ratio" values are very useful as an indication of the buffer action of bacteriological peptones. As these values decrease, the buffer action (for the growth of lactic bacteria) increases. It was possible, by the action of pepsin on casein (or albumin), to make a peptone with a better buffer action than that of Witte's peptone; the "pepsin" itself was a good substitute for peptone in this respect, and capable of some improvement by auto-digestion.

	Nitrogen Per Cent.	Formol titration $N/10$ alkali per 2 grms. c.c.	Ratio
Gelatin	14·35	5·1	40
Casein	13·38	8·7	22
Albumin	11·98	5·2	33
Peptone, W.	14·34	12·9	16
Peptone, A. & H. ..	12·66	10·5	17
Pepsin	13·16	17·5	11
Pancreatin	11·14	34·0	5

D. W. STEUART

STANLEY AVENUE
THORPE ROAD, NORWICH

ELON AND SODIUM SULPHITE AS REDUCING AGENTS IN THE COLORIMETRIC DETERMINATION OF PHOSPHORUS

IN the original method of Bell and Doisy (*J. Biol. Chem.*, 1920, **44**, 55; ANALYST, 1921, **46**, 13) and that of Briggs (*J. Biol. Chem.*, 1924, **59**, 255), later modified by Green (*J. Agric. Sci.*, 1928, **18**, 373), sodium sulphite and hydroquinone are used as reducing agents in the colorimetric determination of the inorganic phosphorus of blood. Van der Lingen (*17th Rept. Director of Vet. Services, S. Africa*, 1931, p. 475) showed that this method could advantageously be adopted for the colorimetric determination of the phosphorus-content of plant ashes when a suitable aliquot portion of the nitric acid extract, as used for the determination of the chlorine-content, is taken.

The extract is prepared by treating 10 grms. of plant material with an amount of absolute alcohol sufficient to wet it thoroughly. The alcohol is then evaporated on a steam-bath, and the plant material is treated with approximately 10 c.c. of 10 per cent. calcium acetate solution. After being dried on a steam-bath the contents of the silica dish are ashed at a temperature just below glowing heat in an electric muffle. The material is then extracted with 10 c.c. of 50 per cent. nitric acid, the extract is filtered into a 100-c.c. graduated flask, the residue washed four times with hot distilled water, and the filtrate and washings are cooled and made up to the mark.

It has been found that if a 3·1275 per cent. solution of elon (*p*-methylaminophenol sulphate) is substituted for the 1 per cent. solution of quinol, the effect of the nitric acid is much less pronounced. Since in the original article (*loc. cit.*) the temperature at which the colour was developed was not stated, it was deemed advisable to repeat this experiment. In both cases the temperature of the solution was 26·5° C. A comparative series of determinations with quinol sulphite and elon sulphite were carried out, and the results recorded below were obtained. The time of reduction in the first case was 30 minutes, and in the second 20 minutes. The tubes each contained 10 c.c. of phosphorus standard (0·2 mgrm. of P_2O_5), 1·5 c.c. of Reagent A (sulphuric acid and ammonium molybdate reagent), 1·5 c.c. of either 1 per cent. quinol or 3·1275 per cent. elon solution and 1·5 c.c. of 20 per cent. sodium sulphite (crystals) solution, whilst the final volume was made up to 16·5 c.c. by adding the required amounts of nitric acid and distilled water. A 1 per cent. solution of quinol and a 3·1275 per cent. solution of elon contain the same amount of reducing hydrogen.

QUINOL SULPHITE METHOD

Tube No.	Nitric acid 50% (approx.) c.c.	Average colorimetric reading mm.	Error Per Cent.
Standard	0	30·0	0·0
1	0·25	31·2	4·00
2	0·50	32·5	8·33
3	0·75	33·9	13·00
4	1·00	35·2	17·33
5	1·50	40·4	34·66
6	1·75	57·0	90·00
7	2·00	Tinted	—

ELON SULPHITE METHOD

Tube No.	Nitric acid 50% (approx.) c.c.	Average colorimetric reading m.m.	Error Per Cent.
Standard	0·0	30·0	0·0
1	0·25	31·1	3·66
2	0·50	31·5	5·00
3	0·75	32·0	6·66
4	1·00	32·6	8·66
5	1·50	34·8	16·00
6	1·75	35·8	19·33
7	2·00	39·0	30·00

It should also be stated that the colours produced when elon is used are much clearer than when quinol is used. The amount of trichloroacetic acid normally present in blood filtrates introduces no error in the determination. For general purposes I would recommend the use of a 3 per cent. solution of elon and a colour development of twenty minutes.

G. VAN DER LINGEN

FUEL RESEARCH INSTITUTE
PRETORIA, S. AFRICA

Official Appointments

THE Ministry of Health has approved the following appointments:

RHYS PENDRILL CHARLES as Public Analyst for the County Borough of Bournemouth, in place of R. A. Cripps (retired) (October 19th, 1933).

FREDERICK WILLIAM EDWARDS as Public Analyst for the Metropolitan Borough of Hammersmith, in addition to P. A. Ellis Richards (October 27th, 1933).

Ministry of Health

ANNUAL REPORT OF THE CHIEF MEDICAL OFFICER FOR THE YEAR 1932*

THE Report of the Chief Medical Officer of the Ministry of Health (Sir George Newman) on the state of the public health in England is divided into twelve chapters, which deal with the various branches of the public health services, including general epidemiology, the insurance medical service, work of the Ministry's pathological laboratory, etc. Chapter VII, on The Relation of Food to Health and Disease, has several sections on subjects of special interest to Public Analysts and food chemists.

A NEW PROBLEM IN FOOD ADULTERATION.—It is shown in a diagram that a remarkable improvement has taken place during the last fifty years, the adulteration rate having gradually fallen from 20 per cent. to about 5 per cent. There are, however, a number of practices relating to the scientific treatment of food which have presented a new problem in supervision and in the administration of the Acts controlling the adulteration of food.

Among instances in which a commercial process may possibly involve a definite risk to health or debasement of nutritive value, the following are cited: (*a*) Fumigation of food with toxic gases to destroy insect pests; (*b*) the presence of metals derived from material or containers used in manufacture or storage; (*c*) the increasing use of poisonous insecticides and fungicides in agriculture; (*d*) the treatment of flour with bleaching and "improving" agents; (*e*) "fortification" of foods by artificially added vitamins; (*f*) uncontrolled and unregulated exposure of food to ultra-violet rays; (*g*) the introduction of new synthetic colours and flavours.

SYNTHETIC DIACETYL IN BUTTER.—The best dairy butter may contain about 0·0005 per cent. of diacetyl, to which its aroma is due. An aqueous solution of synthetic diacetyl is used to impart aroma to butter deficient in this respect. Since diacetyl has a definite oxidising action on fats, it is conceivable that the accelerated oxidation brought about by it may have some destructive effect upon the vitamin *A* in butter.

COLOUR IN FOODS.—The bleaching of flour may involve the partial destruction of the lipochrome in flour. Carotene has been shown to be a valuable foodstuff, and it is probable that the lipochromes of butter and egg-yolk have similar properties.

Logically, the labelling system adopted for margarine should be extended to custard powders coloured golden yellow. The public does not understand the subtle trade distinction between "egg powder" and "powdered egg."

ADDITION OF VITAMINS TO FOODS.—Foods are often advertised as of *specially high vitamin-content*, but frequently these claims have little foundation in fact. On the other hand, manufacturers are empirically adding vitamins to various foods, and such indiscriminate dosing of foods may lead to wide-reaching disturbances in the balance of nutrition.

Bread containing irradiated ergosterol (vitamin *D*) is already on the market, and recently an important firm of bakers contemplated adding sufficient vitamin *D* to their bread to make it equivalent in this respect to butter. It is desirable that the administration of vitamin *D* should be under control, and, if such additions as irradiated ergosterol to bread become at all general, it might be desirable for

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* Obtainable from H.M. Stationery Office, Adastral House, Kingsway, London, W.C.2. Pp. 265. 1933. Price 4s. net.

local authorities to consider the application of the Bread Acts or the Food and Drugs (Adulteration) Act for the protection of the public.

LEAD POISONING FROM CIDER.—A further outbreak of lead poisoning from drinking cider was reported from Devonshire (*Lancet*, October 1, 1932, p. 717). As in former cases, the outbreak was traced to the use of lead pipes for conveying cider from barrels to the engine at the bar. The pipes were stated to be tin-lined, but were probably tin-washed, the film of tin being insufficient to protect the pipe from the action of the acid in the cider.

SAMPLING OF FOOD.—It is suggested that samples submitted to Public Analysts are not always taken to the best advantage, since it is sometimes not realised that a number of samples may really represent the same article supplied by a single manufacturer to several retailers in the same district.

The total number of samples analysed by Public Analysts in 1932 was 137,981, and of these 7019 (5·1 per cent.) were reported as adulterated or not up to standard. Of the 72,840 samples of milk examined by Public Analysts, 5307 (7·3 per cent.) were reported as adulterated or not up to standard. No statistics are available of samples tested informally by officers of Local Authorities.

MILK AND CONTAGIOUS ABORTION.—Few dairymen are aware of the danger to man of milk infected with *Br. abortus*, although a large proportion of the milk supplies in this country contain this organism. Preventive inoculation of the cattle does not protect the customer from infective milk; blood agglutination tests and segregation of positive cases form a much more satisfactory method.

UNDULANT FEVER.—Between 1929 and 1932 there were 115 well-authenticated cases reported, but it is certain that many cases are not diagnosed, and that many diagnosed cases are not reported. Undulant fever is notifiable in most States in U.S.A., with the result that the cases reported have increased from 1 in 1922 to 1545 in 1931. Information is available in 112 of the reported English cases, and this shows that the disease occurs in all parts of the country.

PASTEURISATION OF MILK.—Quite recently "in-bottle" pasteurisation has been adopted on a commercial scale. The necessity for efficient pasteurisation is emphasised by the fact that a case was investigated in which tubercle bacilli were found in "pasteurised milk," owing to the heated milk having been passed over a cooler which had previously been used for cooling ordinary raw milk.

DRIED FISH INFESTED WITH LARVAE OF THE LEATHER BEETLE.—Dried fish are sometimes infested with the larvae of *Dermestes vulpinus*, which are covered with hairs liable to irritate human skin. Fumigation of the fish might destroy the larvae, but, as it would leave the irritating hairs, would not render the infested fish suitable for food.

USE OF ANTI-FLY PREPARATIONS FOR BACON.—The use of an extract of pyrethrum has been suggested for counteracting the attacks of the bacon fly, but, in view of its high physiological activity, pyrethrum extract should not be used in connection with any food material.

SALMONELLA INFECTION OF EGGS.—Fifty-five reports of food poisoning were received by the Ministry, and in 36 of these there was circumstantial evidence incriminating the food. Most of the outbreaks in 1932 were of the usual type—the consumption of food infected with salmonella. The "toxin" type of outbreak associated with the consumption of canned food was rare, as contrasted with its frequency in earlier years. In 24 cases living "salmonellas" were isolated from the suspected food, and in no less than 8 of these cases the infection was traced to eggs. In each outbreak, cultures of *B. aertrycke* were obtained from one or more of the sufferers, and in 3 outbreaks, each involving one person (one fatal), the same species of bacteria was also isolated from one or more eggs, obtained from the same flock of ducks as the incriminated egg.

In six of the eight outbreaks (*cf.* Scott, *J. Path. Bact.*, 1932, **35**, 655) the eggs suspected or proved to contain *B. aertrycke* were from ducks; in one, the kind of egg was not ascertained; and, in the last case, a hen's egg of Polish origin had been used in a milk pudding. Research by Dalling in the Wellcome Physiological Research Laboratories has shown that ducks in apparent health can lay eggs containing large numbers of the Gaertner or aertrycke bacilli.

CONTAMINATION OF FOOD WITH "VIRUS" PREPARATIONS.—In November, 1931, there were 38 cases of food poisoning, with one death, which were traced to the use of a "virus" preparation for destroying rodents in a bakehouse at Wigan. Several similar outbreaks are recorded in German literature, as, for example, the four cases ascribed by Boecker and Kauffman (*Deutsch. Med. Woch.*, 1930, p. 1339) to infection with "ratin."

Report on the Burma Food and Drugs Acts, 1928

WITH RECOMMENDATIONS FOR FOOD STANDARDS

THE Public Analyst to the Government of Burma (Mr. E. H. Bunce, F.I.C.) has supervised continuous research and experimental work since April, 1930, to enable him to recommend food standards for official adoption under the Burma Food and Drugs Act, 1928.

This Act was based on the British Food and Drugs Act, 1875, and various amendments in the procedure are suggested, including one now incorporated in the British Food and Drugs (Adulteration) Act, 1928, to the effect that in any prosecution it shall be no defence to allege that the purchaser, having bought only for analysis, is not prejudiced.

SAMPLING.—The usual formal practice of dividing a sample into three parts is recommended. Also, when a food or drug is contained in a sealed receptacle, a sample, complete within its sealed receptacle, may be submitted to the Public Analyst.

NECESSITY FOR FOOD STANDARDS.—Adulteration of food in Burma is widespread and general. The milks of the cow and buffalo are mixed indiscriminately, with or without the addition of water, and buffalo's milk is frequently watered to imitate cows' milk. Ghee is widely adulterated; coffee in the loose condition is almost invariably mixed with ground roasted gram (*Cicer arietinum* L., the chick pea) or with chicory, or both; the more expensive edible oils are rarely pure, and the cheaper oils are often adulterated with mineral oil. Crude methods of adulteration have been replaced by carefully controlled sophistication requiring the use of all available scientific methods for its detection.

FOOD STANDARDS RECOMMENDED.—It was soon discovered, in the course of the investigation, that the standards adopted in India and elsewhere were not necessarily applicable in Burma, and the work carried out has resulted in the recommendations of a series of standards to be added as an Appendix to Section 8 of the Burma Food and Drugs Act, 1928. These proposed standards include the following:

Milk.—As a result of systematic study over a long period (*cf.* ANALYST, 1932, **57**, 449) the following standards are recommended: For cows' milk: 3·0 per cent. of fat and 8·5 per cent. of solids-not-fat. For buffalo's milk: 5·0 per cent. of fat and 9·0 per cent. of solids-not-fat.

The recommendations as to procedure include one to the effect that an addition of "formalin" (40 per cent. formaldehyde solution) may be made to a sample of milk, when purchased for analysis, in the proportion of one drop to 2 ozs. of milk, and that each sample thus preserved in a condition convenient for analysis should bear a label stating that it has been artificially preserved.

Condensed Milk.—Standards similar to those contained in the British Public Health (Condensed Milk) Regulations, 1923, are recommended for Burma, with the omission of unsweetened condensed skimmed milk. A declaration of the volume of standard milk, to which the contents of the tin are equivalent, is also necessary. For this purpose, milk is defined as milk containing not less than 12·4 per cent. of milk solids, including not less than 3·6 per cent. of fat.

The revised system of labelling added in 1927 to the British Regulations is also recommended for adoption.

Dried Milk and Infant Foods.—It is recommended that only full-cream dried milk should be allowed to be imported into Burma. Three-quarter cream, half-cream, and quarter-cream dried milks should be prohibited, on the ground that confusion as to their meaning would be created in the public mind, and skimmed dried milk on the ground that it is of poor value as a food-stuff.

As in the case of the British Public Health (Dried Milk) Regulations, 1923, a minimum of 26 per cent. of milk-fat for dried milk (full cream) is desirable, but it is suggested that the British limitations should be omitted, and that the Burma regulations should apply to dried milk to which no other substance has been added, and to the dried milk contained in any preparation intended for consumption by infants.

With regard to the standard of 26 per cent. of milk-fat, it has been stated that the poor keeping properties of a full-cream dried milk in hot climates present an almost insuperable difficulty to the merchant. This assertion is hardly reconcilable with the fact that there is a successful dried milk powder containing 29 per cent. of milk-fat sold in Rangoon.

It is further recommended that the receptacle containing dried milk intended for human consumption (other than dried milk to which sugar or some other substance has been added) should bear a label, in specified form, declaring that it is dried full-cream milk, and stating the number of pints of milk to which the contents of the tin are equivalent. The word "sweetened," "modified," or "compounded" (as applicable) is to be added to the heading in the case of dried milk to which sugar or some other substance has been added. In all cases where a dried milk or dried milk preparation is intended for infants, it is recommended that the percentage composition should be clearly stated on the container.

GHEE AND BUTTER STANDARDS.—Ghee must be prepared exclusively from the pure clarified fat of the cow or buffalo, or both. If the butyro-refractometer reading is less than 40 or more than 43 at 40° C., or the Reichert–Meissl value is less than 24, it is to be presumed, until the contrary is proved, for the purposes of the Act, that the ghee is not genuine, by reason of the addition thereto of extraneous fat or oil.

"Butter" must be prepared exclusively from the milk or cream of the cow or buffalo, or both, with or without the addition of salt or of innocuous colouring matter. If the fat does not conform to the standards prescribed for ghee, it is to be presumed, until the contrary is proved, that the butter contain extraneous fat or oil. The presence of more than 16 per cent. of water in butter should also raise a presumption that the butter is not genuine, until the contrary is proved. No preservative, other than common salt, must be added to butter.

Edible Oils.—The fixing of physical and chemical standards for individual oils is not recommended, but it is suggested that each oil should be clearly defined. The addition of "white oil" to edible oils, generally, should be prohibited.

Tea.—The adoption of standards similar to those in other provinces of India is not recommended, since values consistent with the standards could be obtained with adulterated samples. The presence of partly extracted tea or of foreign matter should be prohibited.

Coffee is defined as the seed of *C. arabica* or *C. liberica*; it must not contain any foreign substance.

Coffee and Chicory.—It is recommended that the mixture should contain not less than 50 per cent. of coffee, and should be free from foreign substances. Such mixtures are allowed to be sold only in the roasted and ground condition.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS

Food and Drugs Analysis

Polarimetric Method for Determination of the Saccharogenic Power of Flour. H. C. Gore. (*J. Assoc. Off. Agr. Chem.*, 1933, **16**, 403–414.)—Rumsey's method for determining the saccharogenic power of flour (ANALYST, 1923, **48**, 218) is subject to errors from two sources: the cuprous oxide formed is somewhat impure, and, what is more serious, the reducing power of the filtrate from the flour suspension gradually increases, owing to hydrolysis of sucrose, raffinose, or other fructoside, by the sulphuric acid added to stop the diastatic action.

It is suggested that a polarimetric method may prove satisfactory, and the following tentative procedure is proposed: 25 grms. of the flour are mixed with 241·5 c.c. of Sörensen's phosphate mixture of p_H 5·288 (53·116 grms. of KH_2PO_4 and 1·78 grm. of Na_2HPO_4, $12H_2O$ in 6 litres) at 37° C., and incubated for 1 hour at 37° C., with mixing every 15 minutes. The mixture is then cooled rapidly to about 20° C. in a bath of ice-water; 7·5 c.c. of sodium tungstate solution (15 grms. of Na_2WO_4, $2H_2O$ per 100 c.c.) are then mixed in, followed by 1 c.c. of sulphuric acid, added with continuous stirring. The suspension is filtered at once and the filtrate is polarised, preferably not more than 30 minutes after the addition of the sulphuric acid. T. H. P.

Acetylmethylcarbinol and Diacetyl in Wood Vinegar. J. Pritzker. (*Chem.-Ztg.*, 1933, **57**, 793–794.)—These products, which occur in relatively large amounts in wood vinegars, may be determined as follows: *Diacetyl.*—Ten c.c. of the liquid are neutralised, diluted to 50 c.c. and transferred, together with a little pumice, to a 300-c.c. Jena glass flask. The flask is then fitted with the distillation arrangement used by Polenske in the determination of Reichert–Meissl and Polenske values. The receiver (a 25 to 30-c.c. cylinder with ground-glass stopper) contains (per 0·1 grm. of diacetyl) 2 c.c. of 20 per cent. hydroxylamine hydrochloride solution, 3 c.c. of 20 per cent. sodium acetate solution, and 1 c.c. of 1 per cent. nickel chloride ($NiCl_2$) solution. The lower end of the condenser tube is brought as near as possible to this liquid, and about 20 c.c. are carefully distilled over. The receiver is stoppered, kept for an hour in a water-bath at 80° C., and allowed to cool. The precipitate is collected in an Allihn tube, washed with water, dried in a water-oven, and weighed: weight × 0·5962 = diacetyl.

Acetylmethylcarbinol.—Ten c.c. of the vinegar are neutralised and introduced into the distilling flask, with 50 c.c. of 30 per cent. pure ferric chloride solution and pumice. The procedure described above is then followed. If no pre-formed diacetyl is present, multiplication of the weight of the precipitate by 0·610 gives the acetylmethylcarbinol. Allowance must be made for any diacetyl present by subtracting the corresponding weight of nickel dimethylglyoxime from the weight of the precipitate.

Five samples of crude wood vinegar contained, respectively, 12, 4·45, —, 0·5, and 4·39 grms. of acetylmethylcarbinol, and 0·3, 0·3, 0·1, —, and 1·92 grm. of diacetyl per litre. T. H. P.

Goats' Milk and the Composition of Goats' Blood. F. E. Nottbaum and K. Philippi. (*Z. Unters. Lebensm.*, 1933, **66**, 289–301.)—Complete analyses have been made of two series of goats' milk: (1) 24 samples of milks shown at an agricultural exhibition in the spring of 1929, and (2) 15 samples collected, mostly in the winter, in the neighbourhood of Hamburg. With most of the constituents no marked divergence from the proportions present in cows' milk is observable. The chlorine-contents of the goats' milk are, however, about double that of cows' milk; for series (1), from 0·1065 to 0·1846 (mean 0·1337) and for series (2), from 0·1207 to 0·181 (mean 0·1522) grms. of chlorine per 100 c.c. of the milk were found. The high chlorine-content is not necessarily accompanied by a correspondingly high sodium-content, the amounts of chlorine found being considerably greater than those required to combine with the total sodium present.

In view of the close relationship existing between milk and blood, the ash of goats' blood and its serum has been examined. The composition of the ash of the serum does not differ greatly from that of the ash of cows' or sheeps' blood serum, but the goat serum has an appreciably higher potassium-content than the serums of other ruminants, and in this respect lies between those of the sheep and pig. During the past fifteen years various cases of infantile anaemia have been traced to the use of goats' milk for feeding. It is suggested that this anaemia may be caused by the high chlorine-content of the milk, since goats' milk containing 0·180 grm. of chlorine per 100 c.c. would supply about six times as much chlorides as an equal quantity of human milk. T. H. P.

Butters from the Milk of Sheep and Goats. T.-G. Stathopoulo. (*J. Pharm. Chim.*, 1933, **125**, 287–289.)—The analytical values of 7 authentic samples of sheeps' butter, and 8 of goats' butter, collected in the country districts of Greece, were determined. All the butters examined were found to be very rich in vitamin *A*. The maximum, minimum and average values obtained for the fats of (*a*) sheep's and (*b*) goats' butter were: Sp.gr. at 100° C., (*a*) 0·8669, 0·8656, 0·8666; (*b*) 0·8676, 0·8653, 0·8662; butyro-refractometer reading at 40° C., (*a*) 42·3, 40·9, 41·63; (*b*) 41·4, 40·6, 41·0; saponification value, (*a*) 228·0, 223·8, 226·4; (*b*) 230·9, 228·1, 229·5; iodine value, (*a*) 31·1, 28·7, 30·02, (*b*) 29·5, 24·5, 29·7 [*sic*]. Reichert–Meissl value, (*a*) 23·37, 22·81, 23·83 [*sic*], (*b*) 24·71, 21·70, 23·42; Polenske value, (*a*) 2·12, 1·51, 1·83, (*b*) 4·04, 1·92, 3·08. D. G. H.

Composition of Cocoa Butter. E. Lewkowitsch. (*J. Soc. Chem. Ind.*, 1933, **52**, 236–238T.)—Owing to an error in calculation, the constitution of cocoa butter, as deduced by Amberger and Bauch (ANALYST, 1925, **50**, 77), differs from that published by other workers (ANALYST, 1929, **54**, 243). The author has re-calculated Amberger and Bauch's results, and the amended figures are in better agreement. The fatty acids would be present as palmitic acid, 23·4, stearic acid, 35·7, and oleic acid, 40·9 per cent. The glycerides are present in the following proportions:—Tristearin, trace; β-palmitodistearin, trace; palmito-stearo-olein, 55; oleodistearin, 25; palmito-diolein, 20 per cent. W. P. S.

Adulteration of Cocoa Butter. Determination of the Azelaic Acid Value.* G. Schuster. (*Compt. rend.*, 1933, **197**, 760–762.)—The average azelaic acid value for cocoa butter is 98·7, and that for shea nut butter 131·1, whilst the average value for cocoa butter mixed with 25 per cent. of shea nut butter was 106·6, and with 50 per cent., 114·9. The proportion of shea nut butter in cocoa butter can, therefore, be estimated in a simple mixture, whilst in more complex mixtures the value will aid in the detection of adulteration. The value is determined by the following method, modified since it was first put forward (*Thèse Diplôme Super. Pharm.*, Paris, 1932, Pichon):—Forty grms. of the cocoa butter, dissolved in 400 c.c. of acetone, are oxidised with 160 grms. of potassium permanganate (*cf.* ANALYST, 1932, **57**, 191). The acid mixture is dissolved in 350 c.c. of 80 per cent. alcohol, the solution is neutralised with alcoholic sodium hydroxide solution, and to the hot liquid (70°–80° C.) is added an equally hot solution of 6 grms. of dried magnesium chloride dissolved in 60 c.c. of 80 per cent. alcohol. After standing for 24 hours at 15° C. the magnesium salts of the azelaic glycerides are precipitated; the liquid is filtered, the filtrate containing the magnesium pelargonate, with a small and negligible quantity of the azelaic salts. The precipitate is washed with 100 c.c. of 95 per cent. alcohol, followed by water, and dried *in vacuo* over sulphuric acid. The azelaic acid value is determined on about 1 grm. of the mixed magnesium salts, the magnesium being determined as sulphate, and the weight of acid glycerides is calculated by means of the formula $(P-p)+\frac{p}{12}$, where P is the original weight taken, p that of the magnesium, and $p/12$ the weight of hydrogen corresponding with the magnesium. D. G. H.

Oil from the Seeds of Ganua (Bassia) Motleyana (Sap.). J. Zimmermann. (*Chem. Weekblad*, 1933, **30**, 657–658.)—Air-dry fresh seeds from Bangkok and old seeds from Palembang contained about 34 per cent. of shell and 66 per cent. of kernel, the latter yielding about 53 per cent. of oil on extraction with ether. The following data were obtained from the respective oils:—Colours, yellow and brown; sp.gr. (27·5°/27·5° C.), 0·913, 0·911; n_D^{40}, both 1·4611; acid value, 0·29, 0·5; saponification value, 191·1, 193·2; iodine value, 67·1, 66·5; Reichert–Meissl value, 1·52, 2·3; Polenske value, both 0·3; hydrocyanic acid, 0·14, 0·02 per cent.; total fatty acids 93·5 per cent. (equivalent weight 278·1; iodine value, 66·1); unsaponifiable matter (soluble in petroleum spirit), 2·1 per cent. Hydrocyanic acid was

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* The azelaic acid value is defined as the number of mgrms. of potassium hydroxide required to neutralise the acidity of 1 grm. of the mixture of insoluble acid glycerides derived from the oxidation of the fat by means of potassium permanganate.

determined by shaking 15 to 20 grms. of oil with 50 c.c. of *N* sodium hydroxide solution, followed, after 24 hours, by steam distillation of the mixture in the presence of a slight excess of phosphoric acid; the distillate was cooled in ice, and an aliquot portion was titrated by the silver nitrate and potassium thiocyanate method. The presence of benzaldehyde was established in both cases from the m.pt. of the semicarbazone formed from the material extracted on shaking with alcohol; previous workers have observed an odour resembling oil of bitter almonds, which has been attributed to deliberate perfuming. A solution of the unsaponifiable matter in petroleum spirit was evaporated, the residue was heated twice with acetic anhydride, an extract of the resulting product in a mixture of alcohol and water was filtered, and the acetate was obtained by crystallisation; its m.pt. (216·5 C.) and saponification value, and the m.pt. of the free alcohol indicated that it was derived from lupeol. A rubber-like substance, insoluble in petroleum spirit, was also obtained with the unsaponifiable matter. The fatty acids, which were examined by Twitchell's method, contained:—Stearic acid, 18·56; palmitic acid, 10·17; oleic acid, 68·77; linolic acid, 2·49 per cent.; small quantities of lower fatty acids were not taken into account. J. G.

Colour Reactions of Almond and Apricot-Kernel Oils. H. Mohler and H. Benz. (*Z. anal. Chem.*, 1933, **94**, 184–188.)—The smallest proportion of apricot kernel oil detectable in almond oil by Bieber's test is 20 per cent., whilst the nitric acid test of the German Pharmacopoeia will not detect less than 50 per cent., and the test of the Swiss Pharmacopoeia (shaking the oil for one minute with an equal volume of nitric acid of sp.gr. 1·4), 30 per cent. The following modification of the nitric acid reaction will detect at least 5 per cent.:—Four drops of the oil are shaken with 4 drops of chloroform in a test tube (about 15 mm. in diameter), and the mixture is then treated with 2 successive drops of fuming nitric acid, introduced, with an interval of 10 seconds between them, down the side of the tube, which is shaken after each addition. Apricot kernel oil gives immediately a blood-red colour, gradually changing to brownish-red; peach-kernel oil also gives a red colour within one minute; almond oil a light brown within 2 minutes. It is essential not to add the nitric acid too rapidly, or the red colour may be destroyed. The following table shows the results obtained with mixtures of the two oils in different proportions:

Apricot-kernel oil Per Cent.	Colour change: With fresh mixtures after	Colour change: With mixtures some weeks old after	Colour
5	5 minutes	15 minutes	Brilliant red
10	2 ,,	5 ,,	Brilliant dark red
20	30 seconds	1 ,,	Dull dark red
30	10 ,,	30 seconds	Dull dark red
40	10 ,,	20 ,,	Dull dark red
50	10 ,,	10 ,,	Blood red; brownish-red after 1 minute.

The yield of oil from peach kernels is so small that it is improbable that that oil is used as an adulterant of almond oil. In the German Pharmacopoeia peach-kernel oil is included under the name of *Oleum persicarum*, although, according to

Kogan (*J. Russ. Phys.-Chem. Ges.*, 1929, **61**, 8), the oil is unobtainable in Russia, Syria, Asia Minor, etc. Peach-kernels and peach-kernel oil are not commercial products.

Composition of Curcas Oil. M. T. François and S. Droit. (*Bull. Soc. Chim.*, 1933, **53–54**, 728–741.)—Two industrial curcas oils (*Jatropha curcas*) were examined, and 8 oils were extracted in the laboratory from seeds from various sources (Portugal, Dahomey, Cameroons, Madagascar, and Soudan); the values found for these extracted oils were remarkably constant. The weight of a hectolitre of seeds was between 44 and 49 kilos., the weight of one seed varying from 0·65 to 0·72 grm. The proportion of shell varied from 32 to 38 per cent., and that of the kernel from 62 to 68 per cent., and the decorticated seeds contained from 50 to 58 per cent. of oil. The chemical constants of six oils and the physical constants of two (in some cases five) oils were determined; they varied as follows:—Sp.gr. 15/15° C., 0·9168 to 0·9198; $n_D^{15°}$, 1·4720 to 1·4733; viscosity at 35° C., 0·277 to 0·360; at 100° C., 0·0623 to 0·0675*; solidifying point, −13° C. to −15° C.; saponification value, 176 to 180; iodine value (Hanus), 97 to 98; acetyl value (André), 4 to 8; acidity (as oleic acid), 0·4 to 0·6 per cent. (one sample 9·6). Investigation, described in detail, showed curcas oil to consist of the glycerides of stearic, palmitic, myristic, oleic and linolic acids, together with a small amount of a resin-sterol complex. D. G. H.

Oil from the Seeds of *Tectona Grandis* (Teak). S. V. Puntambekar and S. Krishna. (*J. Indian Chem. Soc.*, 1933, **10**, 401–403.)—Teak wood appears to contain no essential or fixed oil, but resinous and tarry matter. The teak seeds had hard tough shells, and each contained 4 to 5 kernels resembling sesame seeds. Twenty pounds of seeds yielded 50 grms. of kernels yielding about 41 per cent. of a bright red oil (0·23 per cent. on the seed). The oil had the following characteristics:—Sp.gr. at 20° C., 0·9213; n_D^{25}, 1·4655; saponification value, 194·5; iodine value (Hanus), 107·5; Hehner value, 93·2; acid value, 31·7; acetyl value, 146·1; unsaponifiable matter, 1·25 per cent. The mixed acids had a mean molecular equivalent of 278; iodine value (Hanus), 109; and contained 5 per cent. of resin acids (with iodine value 30·5, mol. equivalent 288) saturated fatty acids, 25 per cent. (with iodine value 3·8, mol. equivalent 275); and unsaturated acids 70 per cent. with iodine value 146. The solid acid portion appeared to consist mainly of stearic and palmitic acids, and the liquid of oleic and linolic acids, and no constituent was found, other than the resin acids, to which insecticidal value could be attributed. D. G. H.

Fat and Oil from the Seeds of *Actinodaphne Hookeri*, Meissn. An Indigenous Source of Lauric Acid. S. V. Puntambekar and S. Krishna. (*J. Indian Chem. Soc.*, 1933, **10**, 395–400.)—The kernels of *Actinodaphne* seeds (Nat. Ord. *Laurinaceae*), when separated from the endocarp, contained 75 per cent. of a hard, brittle, pale yellow crystalline fat (48·4 per cent. of the seed), of m.pt. 43°–44° C. The seed shells gave 25 per cent. (8·75 per cent. of the seeds) of a

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* Viscosity unit not stated.

reddish-brown oil depositing crystals of trilaurin when cooled to 20° C. This oil, described as "Pisitela" by Kirtikar and Basu ("Indian Medicinal Plants," Pt. II, p. 1103), is used as an external application for sprains. The general characteristics of (*a*) the fat, (*b*) the oil, respectively, were: Sp.gr. (*a*) at 25° C., 0·925, (*b*) at 20° C., 0·9163; n_D (*a*) at 30° C., 1·4490, (*b*) at 25° C., 1·4550; saponification value, (*a*) 255·5, (*b*) 199·6; Hanus iodine value, (*a*) 10·9, (*b*) 54·6; acetyl value, (*a*) 11·3, (*b*) 77·0; Hehner value, (*a*) 91·0, (*b*) 93·0; acid value, (*a*) 4·0, (*b*) 100·2; unsaponifiable matter, (*a*) 1·92, (*b*) 3·56 per cent. The fatty acids of the kernel fat were found to consist of lauric acid 96, and oleic acid 4 per cent., whilst the shell fat was found to consist of lauric acid, 33·00; oleic acid and its isomers, 48·78; isomeric dihydroxystearic acid, 7·02; and resin, 11·2 per cent. The kernel fat is regarded as a convenient source of lauric acid. In practice, the shells cannot be separated from the kernels very completely, but the hard fat is readily purified, and one crystallisation from alcohol gives pure trilaurin of iodine value 1.

D. G. H.

Caffeine-content of Maté. F. Krauss, E. Kleucker and A. Kollath. (*Z. Unters. Lebensm.*, 1933, **66**, 348–354.)—For the determination of caffeine in coffee or tea, Grossfeld and Steinhoff's modification (*ibid.*, 1931, **61**, 54) of Lendrich and Nottbohm's method (*ibid.*, 1909, **17**, 259) is recommended. When this method is applied to maté, it is advisable to retain the treatment with permanganate and to add paraffin to remove interfering oxidation products formed. The caffeine-content of maté (11 samples) was found to vary between 0·3 and 1·5 per cent. The product isolated was identified as caffeine by its composition and melting-point, but whether another alkaloid exists in small quantity in the maté is left undecided.

T. H. P.

Microscopic Identification of Heroin. G. D. Williams and C. C. Fulton. (*Amer. J. Pharm.*, 1933, **105**, 436–440.)—The most satisfactory microscopic tests for heroine, both as regards sensitivity and ready formation of characteristic crystals, are:—(i) precipitation with mercuric iodide in 10 per cent. hydrochloric acid. In the more concentrated solutions the crystals are mainly branching threads, and in more dilute solution (down to 1 in 3200) needles or blades; (ii) with gold chloride in concentrated hydrochloric acid, dark dense rosettes of needles and round yellow spherical crystals, suggestive of yeast cells, are formed down to a dilution of 1 in 3200; and (iii) with sodium picrate, brown rosettes of threads are formed in all solutions down to 1 in 1600. D. G. H.

Determination of Nicotine in Tobacco and Nicotine-containing Parasiticides. W. Mohr. (*Z. Unters. Lebensm.*, 1933, **66**, 362–365.)—The three procedures as yet suggested for this determination are subject to serious disadvantages. In the volumetric process, the crude nicotine extracted by solvents or separated by steam-distillation is contaminated with other basic substances, especially ammonia, and, in addition, the end-point of the titration is not sharp. The gravimetric process is long and requires strict adherence to definite conditions. The polarimetric method needs preliminary isolation of the nicotine, unmixed with other optically active substances, this being difficult to achieve, particularly with complex parasiticides. The following method gives, as product to be weighed, a crystalline substance of constant composition, and furnishes accurate results:

With tobacco rich (poor) in nicotine, 5 (10) grms. are taken, and with tobacco extracts of 8 to 10 per cent. strength, 5 grms. For tobacco, about 100 grms. are finely ground in a spice mill, and the required quantity weighed after mixing. This is treated with 100 c.c. of 5 per cent. sodium hydroxide solution and a little paraffin-wax in a 500-c.c. Kjeldahl flask and distilled in a current of steam. The flask is heated meanwhile with a small flame, and the volume of liquid in the flask is kept about constant. The distillate is allowed to flow into 100 c.c. of approximately saturated picric acid solution (12·5 grms. per litre of water) in a 500-c.c. Erlenmeyer flask, the condenser tube dipping into the reagent. The end of the distillation is determined by testing 5 to 10 c.c. of distillate separately in 5 c.c. of the picric acid solution; usually about 300 c.c. of distillate must be collected. The precipitate, which is nicotine picrate, $C_{10}H_{14}N_2, 2C_6H_3O_7N_3$, and contains 38·25 per cent. of the base, is collected on a tared Gooch crucible or Jena glass filter crucible, washed several times with one part of the picric acid reagent diluted with three parts of water, dried for 3 or 4 hours at 100° C., cooled in a desiccator, and weighed. With complex materials, especially those containing coal-tar products, the distillate contains volatile oils which contaminate the precipitate. In this case, the precipitate is collected on a filter-paper and afterwards dissolved in 5 per cent. sodium hydroxide solution, the process described above being then repeated on the solution thus obtained.

If the material contains pyridine, the presence of which is usually indicated by a low m.pt. for the picrate precipitate (normally 211° to 216° C.), the weighed sample for analysis is mixed with 10 c.c. of glacial acetic acid and 100 c.c. of water, and the mixture steam-distilled. The whole of the pyridine then passes over as acetate in the first 300 c.c. of distillate. The remaining contents of the distilling flask are neutralised with concentrated sodium hydroxide, 5 grms. of solid sodium hydroxide are added in addition, and the process is carried out as described above. Volatile tar products usually distil over completely with the pyridine. Ammonia does not interfere with the process, but may distil over in such quantity that the picric acid is neutralised and formation of the nicotine precipitate is prevented; more picric acid solution must then be added to the distillate. T. H. P.

Psyllium Seed. R. Hansche and E. U. Still. (*Amer. J. Pharm.*, 1933, **105**, 433–435.)—The seed of *Plantago psyllium*, commonly called psyllium seed, is included in the *United States Dispensatory* (21st Ed., p. 1440) as a mild laxative, and considerable quantities are now imported into U.S.A. Its action as a therapeutic agent has been discussed by Solis-Cohen and Githens (*Pharmacotherapeutics*, 1929, p. 996). Analyses were made of (*a*) blond seeds, ground and dried; (*b*) the separated hulls, also ground and dried; and (*c*) an aqueous extract of the hulls containing all the mucilage and water-soluble compounds dried *in vacuo* (gum). The mean percentage values from two series of analyses were as follows:—Moisture, (*a*) 7·5, (*b*) —, (*c*) —; ash, (*a*) 3·0, (*b*) 2·53, (*c*) 2·53; total nitrogen (nesslerisation method of Koch and McMeekin, ANALYST, 1924, **49**, 604), (*a*) 2·30, (*b*) 0·44, (*c*) 0·125; total phosphorus (Fiske and Subbarow method, ANALYST, 1926, **51**, 205), (*a*) 0·438, (*b*) 0·102, (*c*) 0·143; lipoids, (*a*) 8·36, (*b*) —, (*c*) —; total CHO as hexose (mgrms. per cent.), (*a*) 113·0, (*b*) 641, (*c*) 809; pentose, (*a*) 12·30, (*b*) 38·6, (*c*) 83·05. The gum, which is mainly composed of pentosans, is readily soluble in hot water, and sets to a fairly firm jell, and by filtering a hot aqueous extract of the hulls through cotton, a clear, almost colourless, preparation is obtained. D. G. H.

Iodine-content of Commercial Iodised Table Salt. G. Prange. (*Z. Unters. Lebensm.*, 1933, **66**, 369–374.)—The iodine-contents of 12 samples of iodised salt or "complete salt" (Vollsalz) were found to vary from 20·3 to 0·4 mgrms. (as potassium iodide) per kilo. The iodine-content did not diminish greatly during long storage of the material, most of the samples showing losses of 4 to 22 per cent. of the original iodide after 10 to 12 months; this change was the more marked with the salts poor in iodine. It is suggested that legislation should fix the proportion of potassium iodide in these salts at 5 mgrms. per kilo, with an allowable error of 10 per cent.

To determine the iodide, a faintly acid solution of 20 grms. of the salt is treated with chlorine water or bromine water and gently boiled for 10 minutes, with replacement of the evaporated water. The cooled (10° C.) liquid, after addition of phosphoric acid and potassium iodide, is kept in the dark for 3 minutes and then titrated with 0·004 *N* thiosulphate solution (*cf.* Gronover and Wohnlich, *ibid.*, 1931, **61**, 306). T. H. P.

Biochemical

Determination of Ammonia in Blood. D. D. Van Slyke and A. Hiller. (*J. Biol. Chem.*, 1933, **102**, 499–504.)—A method is described in which ammonia is removed from blood by the aeration procedure of Nash and Benedict (*J. Biol. Chem.*, 1921, **48**, 463), and is determined colorimetrically by Berthelot's reaction with hypochlorite and phenol. This reaction is more sensitive than Nessler's for small amounts of ammonia, and the coloured product does not flocculate. A dilution of 0·001 mgrm. of ammoniacal nitrogen in 5 c.c. of solution (the minimum obtained in blood analyses) is just beyond the limit at which Nessler's solution gives a perceptible colour, but with this dilution the phenol reagent still yields sufficient colour for approximate determination. The reagents required are: —(1) *Ammonia-free water*, obtained by distillation of dilute sulphuric acid and collection of the middle portion of the distillate. (2) 0·1 *N Hydrochloric acid.* (3) *Potassium oxalate and potassium carbonate solution* of Nash and Benedict, which contains 15 grms. of oxalate and 10 grms. of anhydrous carbonate in 100 c.c., and is prepared free of ammonia as described by Folin (*J. Biol. Chem.*, 1932, **97**, 141). (4) *Capryl alcohol.* (5) *Sodium phenate reagent*, for which 25 grms. of phenol are dissolved in a small amount of water, 50 c.c. of 40 per cent. sodium hydroxide solution are added, and the whole is diluted to 100 c.c. with ammonia-free water. (6) *Javelle water* containing 1 grm. of chlorine per 100 c.c; in about 500 c.c. of hot water 50 grms. of calcium hypochlorite having 56·5 per cent. of available chlorine are dissolved, the solution is mixed with one containing

50 grms. of anhydrous potassium carbonate in 200 c.c. of cold water, and the whole is made up to a volume of 1 litre. The clear solution is tested for excess of calcium with potassium carbonate, more of which is added, if necessary, to precipitate all the calcium. This reagent should be kept in the ice-box in a number of small bottles. The active chlorine is determined before use, and at intervals of a few weeks as follows: To 5 c.c. of the Javelle water are added 25 c.c. of water, 2·5 c.c. of 10 per cent. potassium iodide solution, and 2 c.c. of glacial acetic acid. The mixture is then titrated with 0·1 *N* thiosulphate solution (freshly standardised). (7) *Standard solutions,* made from a stock solution of ammonium sulphate (0·2358 grm. in 1000 c.c. of water). Each c.c. of solution contains 0·05 mgrm. of ammoniacal nitrogen. (8) *Potassium oxalate,* prepared ammonia-free as described by Folin (1932). A 30 per cent. solution of the neutral salt is used as an anticoagulant by drying it on the walls of the tube in which the blood is collected for analysis.

Procedure.—For the determination of ammonia in blood, 5 c.c. of blood, 1 c.c. of ammonia-free solution of potassium oxalate and potassium carbonate, and 1 drop of capryl alcohol are measured into the aeration tube, described by Nash and Benedict. Then 5 c.c. of ammonia-free water and 3 drops of 0·1 *N* hydrochloric acid are measured into the receiving tube, and aeration is carried out for 10 minutes at the rate of 6 litres of air per minute. Before it reaches the blood the air is washed through three wash bottles containing 5 per cent. sulphuric acid, and a fourth containing 1 per cent. hydrochloric acid. At the end of the aeration period, 1 c.c. of the sodium phenate solution and 0·5 c.c. of the Javelle water are added to the solution in each receiving tube, the contents are mixed, the tube is placed in a boiling water-bath for 3 minutes, and cooled with cold running water to room temperature, and the intensity of colour is compared with a standard in a colorimeter. A series of standards, chosen to cover the possible range of ammonia-content of the blood, is set up with each series of analyses. Each standard tube contains 5 c.c. of a standard solution, 3 drops of 0·1 *N* hydrochloric acid, and sodium phenate and Javelle water as for the blood-receiving tubes, and is heated and cooled as described above.

Calculation. $\frac{\text{Colorimeter reading of standard (usually 20 mm.)}}{\text{Colorimeter reading of unknown}} \times$ mgrms. of ammonia nitrogen per 100 c.c. of standard used = mgrms. of ammonia nitrogen per 100 c.c. of blood. Tables show the good recovery of ammonia by this method from standard solutions, and of the ammonia in whole blood from different blood vessels.

P. H. P.

Method for Determination and Detection of Carbon Monoxide in Blood. A. A. Christman and E. L. Randall. (*J. Biol. Chem.*, 1933, **102**, 595–609.)—A simple and accurate method requiring inexpensive apparatus is described for the determination of carbon monoxide in blood. For the qualitative detection of carbon monoxide in blood, only 15 to 30 minutes are required, but 1 to 2 hours are required for duplicate quantitative determinations. Amounts of carbon monoxide representing a 1 per cent. saturation of the haemoglobin can be determined. The method is based primarily on the fact that palladium chloride is reduced by carbon monoxide; the excess of palladium chloride present is determined by a colorimetric procedure. The blood gases, including carbon monoxide, are released from the blood under reduced pressure by the action of acid ferricyanide solution. This is prepared according to the directions of Van Slyke and Hiller (*J. Biol. Chem.*, 1928, **78**, 807). Potassium ferricyanide (32 grms.) is dissolved in water and made up to a volume of 100 c.c. For the acid solution 23 c.c. of the ferricyanide solution are combined with 2 c.c. of concentrated lactic acid of sp.gr. 1·2. The resulting gas mixture is passed into a bulb containing palladium chloride solution. For this 500 mgrms. of palladium chloride are placed in a 400-c.c. beaker and covered with 150 c.c. of distilled water, 2·5 c.c. of concentrated hydrochloric acid are added, and the mixture is heated until complete solution of the palladium chloride results. The solution is cooled, transferred to a 500-c.c. volumetric flask and made up to volume. The carbon monoxide reacts with the palladium chloride according to the following equation: $CO + PdCl_2 + H_2O = Pd + CO_2 + 2HCl$. After the reaction is complete the excess of palladium chloride is separated from the metallic palladium by filtration, and the palladium chloride is determined colorimetrically. The addition of potassium iodide in excess of that required to precipitate the palladium chloride as palladous iodide, re-dissolves the palladous iodide to give a red solution, which, in the presence of a protective colloid, such as gum ghatti, remains perfectly clear for at least 24 hours. The red colour is easily matched, and the intensity of the colour is almost exactly proportional to the amount of palladium chloride present over a wide range of concentrations. Christman (*J. Biol. Chem.*, 1932, **96**, 97) used the reduction of phosphomolybdic acid by the metallic palladium which results from the reaction of carbon monoxide with palladium chloride as a qualitative and semi-quantitative test for carbon monoxide in blood. This procedure was abandoned when it was discovered that the colour reaction of palladium chloride with potassium iodide could be readily applied to a quantitative determination. Von Fodor (*Deutsch. Vrtljschr. öff. Gsndhtspflg.*, 1880, **12**, 377) published methods for the determination of carbon monoxide in blood and air which involved the use of palladium chloride, but it is doubtful whether his method could be considered quantitative. Recently, numerous methods have been introduced for the determination of carbon monoxide in air, which depend upon the reaction with palladium chloride, but no recent attempts have been made to apply them to the determination of carbon monoxide in blood. The apparatus required is described and shown in a figure. Some results obtained are given. P. H. P.

Vitamin Values of Fermentation Vinegar and of Artificial "Essence" Vinegars. J. Křiženecký and M. Nevalonnyj. (*Z. Unters. Lebensm.*, 1933, **66**, 278–288.)—The results of experiments with pigeons, guinea-pigs and rats indicate that fermentation vinegar contains the vitamin B_1, and the water-soluble growth-promoting vitamin B_2. The presence of vitamin C in appreciable amount could not be detected. The activity of the vitamins and their detection are complicated and masked by the physiological effects of the acetic acid. Thus, when fermentation vinegar is administered over a long period, the effect of the vitamin B_1 becomes paralysed by the acidosis produced; the action of vitamin D

is affected similarly. In artificial vinegar (from synthetic glacial acetic acid) neither vitamin *B* nor vitamin *D* could be detected, but in the experiments on rats the acetic acid of this vinegar was far more toxic than that of fermentation vinegar.

To determine the nutrient values of different vinegars it appears, therefore, more important to study the influence of the acetic acid present than to evaluate the vitamin activity of the vinegars. Even though differences between the vitamin-contents of fermentation and artificial vinegars may exist, such differences play a subordinate part in the physiological action of vinegar as a constituent of a mixed diet. T. H. P.

Vitamin *A* in Fish Liver Oils. P. N. Chakravorty, H. C. Mookerjee and B. C. Guha. (*J. Indian Chem. Soc.*, 1933, **10**, 361–366.)—Values obtained by the Carr-Price colorimetric method are recorded for a number of liver oils from fish commonly consumed in Bengal. Results of biological tests are to follow. Since the values found were high, it became necessary to dilute the oils, but it is pointed out that the relation between dilution and intensity of colour produced is only approximately linear over a small range. The concentrations have been chosen to fall roughly within this range where the values are not too high for accurate matching. Blue values found for the liver oils were of the order; Rohit (*Labeo rohita*), 227; mrigal (*Cirrhina mrigala*), 174; katla (*Catla catla*), 109; vetki (*Lates calcarifer*), 284; hilsha (*Clupea ilisha*), 59. Two samples of commercial liver oil (used for comparison) gave blue values of 4·3 and 5·1, and halibut-liver oil gave a value of 614. The value for another sample of rohit-liver oil, collected at a different time, was 161, indicating that seasonal variations in vitamin *A*-content may be considerable. D. G. H.

Vitamin *C*-Content of Baldwin Apples and Apple Products. C. R. Fellers, M. M. Cleveland and J. A. Clague. (*J. Agric. Res.*, 1933, **46**, 1039–1045.)—In spite of the importance of the apple as a food, only a few investigations of its vitamin *C*-content have been reported, and at the present time the apple is not generally considered a particularly good source of vitamin *C*. The first results are now given of a study being made of the important apple varieties of the North-East of America and the effect of various factors upon the vitamin-content of apples and of products manufactured from apples. It is shown that the Baldwin apple grown in Massachusetts is a good source of vitamin *C*, about 4 grms. per 300 grms. of body-weight sufficing to maintain growth in guinea pigs and to protect them fully from scurvy. Nelson and Mottern (*Amer. J. Pub. Health*, 1932, **22**, 587) reported that lead arsenate sprays applied to orange trees considerably reduced the vitamin *C*-content of the fruit. Since apples are almost universally sprayed with arsenic compounds and other toxic substances for insect and fungus control, comparisons were made between sprayed and unsprayed Baldwin apples. In two tests, apples from completely sprayed trees contained as much, or possibly more, vitamin *C* than apples from unsprayed trees. The sprayed fruit was a deeper red and of better quality than the unsprayed. In 4 to 6 months of storage at 36° F. Baldwin apples lost about 20 per cent. of their vitamin *C*-content; in 8 to 10 months the loss was nearly 40 per cent. Freshly-expressed Baldwin apple juice was found to be nearly as rich in vitamin *C* as the fresh apple. Little loss occurred during the first 24 hours after extraction. Benzoated or pasteurised ciders over 48 hours old, however, failed to retain an appreciable quantity of vitamin *C*. Canned Baldwin apple sauce, either strained or unstrained, proved to be a poor source of vitamin *C*. The unstrained sauce was somewhat superior to the strained. P. H. P.

Vitamin-*D* Potency of Sun-Irradiated Dried Yeast. K. H. Coward. (*Lancet*, 1933, **225**, 920.)—Two samples of sun-irradiated dried yeast have been examined for vitamin-*D* potency by animal experiment. Certain rachitic rats were given the irradiated yeast as a percentage of their diet for 10 days, whilst others were given a daily dose of the international standard preparation of vitamin *D*, and the resultant healing was compared by the "live test" technique. The sample of yeast of known high ergosterol content proved to have been rendered very rich in vitamin *D* by sun-irradiation; the other was only slightly activated. Yeast I contained more than 5 units of vitamin *D* per grm., and yeast II contained less than 0·5 unit per grm. (Further tests would be necessary to show how much more or less was contained in each case.) It is concluded that yeast may be activated antirachitically by exposure to strong sunlight, and that different samples of dried yeast may differ greatly in the extent to which they may be thus activated.
P. H. P.

Decomposition of Timber under Industrial Conditions. E. A. Rudge. (*J. Soc. Chem. Ind.*, 1933, **52**, 283–285T.)—Examination of a greenheart pile embedded for forty years at Cardiff in a bank consisting of rubble, ships' ballast, clinker and ashes resting on a bed of river mud, showed that the pile had undergone limited external decomposition. The pile was about 20 ft. long and 18 in. square, and decomposition had occurred to a maximum depth of 3·5 in., being greatest in the middle of the pile and least at the ends; there was little sign of ground-line erosion. There was a sharp line of demarcation between the decayed and sound woods, but this line did not represent the difference between heartwood and sapwood. The decay of the wood was accompanied by the infiltration of calcium and aluminium compounds and the formation of much acidity. The acid was not identified, but was probably a product of the decomposition of the woody tissue. The process of decay was not a gradual one, the timber retaining its mechanical strength and chemical composition until such time as decay occurred.
W. P. S.

Toxicological and Forensic

Identification and Determination of Barbituric Principles in Urine. M. Paget and C. Desodt. (*J. Pharm. Chim.*, 1933, **18**, 207–214.)—A volume V (250 to 500 c.c.) of the urine is stirred vigorously with V/10 of 15 per cent. potassium ferrocyanide solution, V/10 of zinc acetate solution (112 grms. per litre) being then added and the stirring repeated. The liquid is filtered through a roughly-pleated paper resting on a loose layer of cotton wool in the stem of the funnel. The filtrate is measured, if necessary made acid with acetic acid, and transferred to a large separating funnel. After careful addition of 75 c.c. of ether, the two liquids are mixed, at first gently, and afterwards vigorously. After a few minutes the aqueous (urine) layer is slowly run into the cylinder in which the defecation was carried out, this having been rinsed out. The ethereal layer is poured from the top of the separator into a flask, which is at once corked. The extraction is repeated with four successive 75-c.c. quantities of ether. The combined ethereal solutions are shaken repeatedly, during 20 minutes, with anhydrous sodium sulphate, and are then filtered through cotton wool into a large Erlenmeyer flask, the cotton being afterwards pressed. The ether is distilled off on a water-bath, and the residue is boiled for a few minutes with 10 to 20 c.c. of alcohol. The alcoholic solution is filtered into a tared dish, from which the alcohol is evaporated on a water-bath. If the residue is white, the dish is weighed and the weight of barbituric compound per litre of urine calculated. A coloured residue is stirred seven or eight times, at intervals of two minutes, with 25 to 50 c.c. of boiling water and a few grms. of powdered animal charcoal (this must be previously tested to ensure that it does not absorb barbituric acid). The aqueous solution is evaporated after filtration. If necessary, the treatment with charcoal is repeated until a white residue is obtained on evaporation.

That the residue is a barbituric derivative may be shown by its reaction with cobalt nitrate and potassium cyanide. It may be identified by the various colour reactions of barbituric compounds. The different derivatives studied are all eliminated in the urine, but the percentage elimination varies, being very high for veronal, less for rutonal, still less for dial, and only about 25 for gardenal.

T. H. P.

Toxicological Detection of Veronal, Luminal and Phanodorm. J. Peltzer. (*Chem.-Ztg.*, **1933**, **57**, 816.)—The organs to be examined are passed through a mincing machine and mixed, 300 grms. being then ground with 200 c.c. of 20 per cent. trichloroacetic acid solution and the mixture heated for 15 minutes on a water-bath at 35° C. The resulting thin pulp is filtered by suction through a filter thimble, and the residue is washed with a 5 per cent. solution of the acid, and then extracted again with the same solution (5 per cent.). A slightly turbid filtrate (about 650 c.c.) is thus obtained, and this may be filtered, if necessary, through kieselguhr on a moistened filter. The liquid is then evaporated, best *in vacuo*, to about 75 c.c. and freed from deposit by filtration. The cooled solution is treated with 10 per cent. potassium hydroxide solution until it gives a yellow colour with methyl-red. In this way, the succinic acid often found, the hippuric acid appearing after luminal has been taken, and the bulk of the trichloroacetic acid are fixed. The barbitals, which contain no free carboxyl groups, are not neutralised even at p_H 6, and may be extracted by shaking the liquid with four quantities of ether. If, as seldom happens, an emulsion forms during the shaking, this is dehydrated by shaking it with gum tragacanth. In case the trichloroacetic acid extracts are highly coloured, they are shaken directly with ether without previous neutralisation. The ether takes up a large quantity of trichloroacetic acid, which tenaciously retains traces of ether. To avoid the tedious evaporation of these acid liquors, the acid layer initially not soluble in water on the water-bath is shaken with water until it dissolves. The solution is then cooled, neutralised (to methyl-red) with potassium hydroxide solution, and shaken with ether.

The residue left on evaporation of the ether usually appears moderately pure. If, however, luminal has been taken, its decomposition product phenylethylacetylurea will be present. This is removed by dissolving the residue in water, adding sodium hydroxide solution, and shaking with ether; after acidification with sulphuric acid the luminal can be separated pure.

To test the residue qualitatively, it is dissolved in 1 c.c. of anhydrous methanol and 0·1 c.c. of the solution is treated in a small test-tube with sufficient solid cobalt chloride to give a faint pink colour, and with a little saturated solution of barium oxide in methanol. With each of the three compounds, a blue colour, which gradually disappears, is formed. With veronal the colour is not influenced by a large excess of the barium methoxide, and the liquid ultimately becomes colourless, a blue precipitate being deposited. With luminal, the blue colour changes to yellow if excess of the reagent is added. The blue colour obtained with phanodorm disappears rapidly, even when little of the barium reagent is added.

For quantitative purposes the residue freed from methyl alcohol is dissolved in a few drops of hot pyridine, and water is added so long as no turbidity forms. The liquid is filtered and then treated with a few drops of a mixture of 4 c.c. of 10 per cent. copper sulphate solution, 1 c.c. of pyridine and 5 c.c. of water. Stirring with a glass rod leads to the separation of stout, violet-red cubes of the composition: 2 barbital + 2 pyridine + Cu. After remaining in the cold for a long time, the crystals are pumped off with the help of an Emich filter rod, washed with pyridine water, dried at 105° C. and weighed. The crystals have characteristic microscopic appearances in the three cases. Application of this method to a mixture of 300 grms. of organs with 20 mgrms. of luminal gave a recovery of 18 mgrms. of the luminal.

T. H. F.

Production of Toxic Gases from Ferrosilicons in the Presence of Water. H. Delomenie. (*J. Pharm. Chim.*, 1933, **125**, 289–292.)—The examination of a number of industrial ferrosilicons has confirmed previous findings that such alloys, containing about 50 per cent. of silicon, are able to disintegrate spontaneously in air, with the liberation of poisonous gases. It was found that ferrosilicons containing 20 to 30 per cent. of silicon, when submitted to the action of water *in vacuo* emitted, in certain cases, as much as 2 cub.m. of hydrogen per ton. When 30 to 60 per cent. of silicon was present, between 5 and 39 litres of arsine and 10 to 230 litres of phosphine per ton were liberated, but only a small amount of hydrogen. With alloys containing over 60 per cent. of silicon, much hydrogen was given off, but not more than 3 litres per ton of arsine or 3 to 60 litres of phosphine were liberated. The mode of production of the hydrogen appears to vary with the proportion of silicon. In alloys containing less than 30 per cent. of silicon it is probably produced electrolytically; with proportions between 30 and 60 per cent. there appears to be decomposition of calcium silicide (by the water) and of reducing gases; and with proportions exceeding 60 per cent. calcium silicide is decomposed by the water, and the resulting calcium hydroxide and the water act on the free silicon. Aluminium and calcium (present in small amounts as phosphides and arsenides) play a part in the production of the phosphine and arsine; aluminium phosphide and arsenide, in particular, are readily decomposed by water. D. G. H.

Effect on Fruit of Fumigation with Hydrocyanic Acid. F. Beran. (*Z. Unters. Lebensm.*, **1933, 66,** 317–321.)—Apples and pears are not damaged by fumigation with hydrocyanic acid if the concentration of the acid in the containing space does not exceed 0·5 per cent. by volume, and the treatment is restricted to 1 to 2 hours. Such treatment is sufficient to prevent attack by the San José pest (*Aspidiotus perniciosus* Comst.). If the fumigation is more prolonged, or if 1 per cent. of the acid is present, flecks first appear on the skin, and, later, the flesh becomes increasingly damaged. "Zyklon B," which is a product used for such fumigation, causes more injury than pure hydrocyanic acid of the same concentration acting for four times as long.

The fruit is not able to take up and retain any appreciable amount of the acid, the small quantities adsorbed superficially disappearing two or three hours after the fumigation. These results are directly contrary to those of Wille (*Gesundheits-Ingenieur*, 1920, No. 27), who, however, fumigated with 1 per cent. of hydrocyanic acid for 24 to 28 hours. The acid taken up may be determined by distilling the comminuted fruit (free from pips) in presence of tartaric acid and in a stream of carbon dioxide, and titrating the distillate with 0·01 *N* silver nitrate solution. This procedure gives results slightly in excess of the true values. Titration of the distillate with iodine solution, after addition of sodium bicarbonate, is inadmissible, as the fruit yields volatile substances which react with iodine.

T. H. P.

Silica-Content of Lungs. A. F. Sladden. (*Lancet*, 1933, **225**, 123–125.)—Analyses of the dried lung substance, 60 cases, including "hard-heading" workers in collieries, ordinary colliers, stone masons, coal-trimmers, potters and a gannister worker, were made by F. V. Tideswell and C. A. Seyler. The results are grouped in a series of six tables according to the proportions of silica found, and the amounts of fibrosis of the lungs, ascertained *post-mortem*, are classified under five headings ranging from none to intense. It has been shown by Cummins and Sladden (*J. Path. Bact.*, 1930, **33**, 1095) that advanced fibrosis is not uncommon among Welsh coal-miners, and research in America (*U.S. Public Health Service, Pub. Health Bull.*, 1929, "Health of Granite Workers") has shown that, of all industrial dusts, coal-dust in the mines is the most concentrated (8000 particles of less than 10μ diameter per c.c. of air). Coal dust in mines contains about 0·6 per cent. of siliceous matter per c.c., and a miner might inhale about 2 mgrms. of actual silica in a day's work; this is far below the "danger limit" of 200 particles per c.c., as agreed both in Australia and South Africa. Analyses of the lung substance of 24 representative coal-miners whose deaths were attributable to accident or illness, and not especially to lung fibrosis, showed an average of 4·1 grms. of total silica (1·15 per cent. of the dried lung). Five of the men were below the age of 50, and their silica totals averaged 2·0 grms.; for the remaining 19 men the average was 4·5 grms. Apart from the amount of silica retained by the lungs, the degree of fibrosis will also depend upon other factors, such as the chemical state (free or combined) of the siliceous particles influencing their solubility in the tissues, the size and conformation of the particles, the presence of any non-siliceous coating or matrix on the particles, and the period of time since the access of particles to the lungs. The results available lead to the conclusion that coal-dust alone cannot be regarded as a cause of serious fibrosis of lungs, and such fibrosis as does occur among coal-trimmers is attributable to the action of traces of siliceous dust. Collis and Gilchrist (*J. Indust. Hyg.*, 1928, **10**, 101) have shown that coal-trimmers, like coal-miners, have an excessive mortality rate from pneumonia and bronchitis, but that fibrosis of the lungs, comparable with that found in rock-drillers, or even in coal-miners, has not yet been demonstrated among them.

In cases in which lung fibrosis of important extent has been found accompanied by pulmonary infection, tuberculous or other, the author concludes that the fibrosis is clearly contributory to death when the silica-content of the dried lung substance exceeds 1·0 per cent. Quantities smaller than this, although not negligible, have seldom been associated with deaths attributable to pulmonary disease, except when tuberculosis has intervened. When the silica-content exceeds 1·6 per cent. the fibrosis is almost invariably very severe, and sufficient in itself to lead to death. The analyses suggest that a silica-content as high as 1·6 per cent. causes so much damage to the pulmonary drainage system that the accumulation of further inhaled dust is greatly accelerated; exposure to silica risk at this stage greatly enhances the danger to life.

Blue Dye as Evidence of the Age of Writing. C. E. Waters. (*Ind. Eng. Chem.*, 1933, **25**, 1034–1035.)—In a legal case in the United States the age of three letters was an important point at issue. It was found that the ink of the letters still contained sufficient blue dye to produce a stain on moist filter paper, and expert evidence was therefore given to the effect that the letters could not be 15 years old because the dye had not all disappeared. To test this assertion, tests were made of dated writing (1905 to 1910) in laboratory note books at the Bureau of Standards, Washington, all the samples being more than 15 years old. In each of the years, except 1906 and 1909, one or more bits of writing gave positive results (nine in all) in the tests for blue dye. Similar tests were applied to other authenticated specimens of writing more than 15 years old, a drop of distilled water being placed upon the ink, and white filter paper then pressed down upon it after 10 seconds. Blue dye was detected in 69 of the 137 specimens examined. The oldest writing in which blue dye was found was dated 1881. Still older writing (1851, 1852, 1865, 1872, and 1874) gave only dusty stains. The general conclusion drawn from the tests is that it is not possible to decide from the presence or absence of dye whether one specimen of writing is older or more recent than another.

Organic Analysis

Use of Agulhon's Reagent in the Analysis of Solvents. E. C. Craven. (*J. Soc. Chem. Ind.*, 1933, **52**, 242–244.)—The reagent may be used for the quantitative titration of certain solvents, and is preferably prepared by dissolving 4·9 grms. of potassium dichromate in 1 litre of nitric acid of sp.gr. 1·31. In most cases it is necessary to place equal amounts of the sample in a series of test-tubes; to add increasing amounts of the reagent; to allow the mixtures to stand in the cold for five minutes; and then to select the tube which has just failed to reach the full blue colour. With some solvents, the mixtures may be heated in a boiling water-bath for 5 minutes, cooled, and the tube then selected. Direct titration may be used for stable diluents, but some time is required; with acetone, the solvent itself undergoes slow oxidation, and it is better, therefore, to make up a series of test mixtures. Illustrations are given of the application of the method to methyl alcohol, formaldehyde, formic acid, ethyl alcohol in water, ethyl alcohol in acetone, etc. Normal results are obtained even with polymeric aldehydes, such as paraformaldehyde, metaldehyde, etc. (*Cf.* ANALYST, 1932, **57**, 372.) W. P. S.

Determination of Certain Phenols by Weighing their Aristols. M. François and L. Seguin. (*Bull. Soc. Chim.*, 1933, **53–54**, 711–723.)—A true aristol is defined as an insoluble phenolic derivative formed by the action of iodine on a phenol in the presence of alkali. Two groups are recognised; the first with thymol as a type and including carvacrol, guaiacol, α-naphthol and β-naphthol aristols, and the second represented solely by Lautemann's red—the aristol of phenol. The determination of a phenol in the form of its aristol involves effectual washing of the precipitates to free them from soluble mineral salts, and drying them at a temperature which will remove all moisture, but not the iodine, *i.e.* a temperature of 30° to 32° C. Preliminary washing by decantation, which is the best method for all heavy precipitates, is not possible with the very voluminous aristol precipitates. With porous glass filters (Schott, 3 G4, 4 cm. in dia. and with a capacity of 40 c.c.), only 4 to 5 washings are necessary. As soon as the liquid ceases to run through, the filter is filled up with water, not by a jet, but by pouring it gently from a flask, and the process is repeated for each washing. With guaiacol, about 0·3 grm. of the sample is weighed in a small tared conical flask, 5 c.c. of glycerin are added as solvent, and the mixture is warmed, with shaking, for 1 minute at 50° C. A mixture of 2 c.c. of sodium hydroxide solution and 18 c.c. of water is then added, and the whole is poured rapidly into a 250-c.c. conical flask containing 125 c.c. of a solution of 200 grms. of crystallised sodium sulphate in 1 litre of water, and 30 c.c. of a solution of iodine (iodine, 12·7 grms.; potassium iodide, 20 grms.; water to 100 c.c.). A brown precipitate of aristol is immediately formed and, after standing for 1 hour, is filtered off, washed 4 or 5 times as described above, and carefully dried; this last process takes some 4 days. To convert the weight of the precipitate into that of guaiacol the factor is 0·5535. Details are given for the determination of guaiacol carbonate (685 of the carbonate yield 1120 of aristol), α- or β-naphthol (factor 0·6834), benzonaphthol (factor 1·0909), and betol (factor 0·7674). Although there is a constant relation between the weight of the aristol and the weight of the phenol from which it is derived, yet the formulae and compositions of the aristols are not such as might be expected from the results obtained with thymol. D. G. H.

Molecular Compounds of Polyvalent Phenols with Aromatic Diamines used as Hair Dyes. C. Naegeli and H. Kaltman. (*Helv. Chimica Acta*, 1933, **16**, 980–988.)—A preliminary communication calling attention to the existence of hitherto unknown molecular compounds of pyrocatechol, resorcinol, hydroquinone, pyrogallol, or phloroglucinol with *m*- or *p*-phenylenediamine, *p*-toluylenediamine or 1,2-naphthylenediamine (the two *p*-amines do not react with phloroglucinol).

The application of the molecular compounds in the analysis of hair dyes will form the subject of a future paper. The compounds may be formed by fusion of mixtures of the components, mixing of their solutions in water, benzene, ether, chloroform, etc., or simultaneous sublimation *in vacuo*. All the compounds crystallise; in nearly every case the molecular ratio of amine to phenol is 1 : 1.

W. R. S.

Determination of Aromatic Nitro- and Nitroso-compounds. S. Ueno and H. Sekiguchi. (*J. Soc. Chem. Ind. Japan*, 1933, **36**, 410B.)—About 0·01 grm. molecule of the substance is dissolved or suspended in about 200 c.c. of water in a 500-c.c. conical flask containing 50 to 80 grms. of ice. Ten c.c. of concentrated hydrochloric acid are added, followed gradually by 10 grms. of zinc dust, with vigorous stirring. After 10 minutes a further 10 c.c. of acid are added, the stirring is continued for an hour at a low temperature, and the mixture is filtered. Any deposited reduction-product is dissolved by treatment with very dilute hydrochloric acid or ammonia water. The filtrate and washings (total volume 350 to 400 c.c.) are transferred to a 600-c.c. beaker and titrated with $N/2$ sodium nitrite solution in the presence of 10 c.c. of hydrochloric acid at a temperature not exceeding 5° C. With substances not immediately diazotised 5 grms. of potassium bromide are added before titration. The three nitrobenzoic acids gave results exceeding 99·7 per cent. by this method, and equally satisfactory results were obtained with 18 other compounds. Nitro-compounds not containing sulphonic, carboxyl or hydroxyl groups are best sulphonated before reduction. R. F. I.

Determination of Basic Dyestuffs by means of Silicotungstic Acid. E. B. Johnson. (*J. Soc. Chem. Ind.*, 1933, **52**, 354T.)—One hundred c.c. of a 0·1 per cent. solution of the dyestuff to be tested are treated with a slight excess of a 5 per cent. solution of silicotungstic acid containing a little hydrochloric acid. The mixture is heated on the water-bath until the precipitate coagulates and settles clear, after which the solution is filtered through a Gooch crucible and the precipitate is washed thoroughly with very dilute hydrochloric acid, dried to constant weight at not less than 110° C. and weighed. After this the contents of the crucible are ignited, and the residue of $SiO_2.12WO_3$ is weighed. An experiment with 0·0820 grm. of pure aminoazobenzene yielded 0·3444 grm. of dried precipitate, and 0·2622 grm. of ash, corresponding with 0·0822 grm. of base. With the hydrochloride of methylene blue SS, the result was 99·77 per cent. of that required by theory. If the factor between dyestuff and ash is established, there is no need to dry the precipitate and the analysis can be completed in half an hour. R. F. I.

Volumetric Determination of Free Sulphur in Rubber. J. A. Robertson and J. Young. (*J. Soc. Chem. Ind.*, 1933, **52**, 296T.)—The following combination of methods, suggested by J. G. Mackay (*id.*, 1930, **49**, 401T) and by Strebinger and von Zombory (*Z. anal. Chem.*, 1929, **79**, 1), gave results showing an error of ±0·05 per cent. compared with the (former) gravimetric method for 6 samples containing 0·05 to 0·52 per cent. of free sulphur:—The extract obtained by boiling 1 grm. of the finely-divided sample in acetone for 6 hours is evaporated, and the residue, after being dried at 90° C., is warmed gently with 10 c.c. of nitric acid (sp.gr. 1·50), 5 c.c. of perchloric acid (sp.gr. 1·54), and 0·5 c.c. of bromine for

30 minutes, and is finally boiled for 15 minutes until dissolved. The mixture is then evaporated with 1 grm. of sodium chloride, the residue being evaporated again with concentrated hydrochloric acid and baked for a few minutes. It is then boiled with 200 c.c. of water and 1 c.c. of *N* hydrochloric acid, and 25 c.c. of a standard (0·02 *N*) solution of barium chloride are added gradually. There should then be an excess of barium ions, as indicated by the red stain produced by placing a drop of the liquid on test-paper prepared by adding a drop of a 0·2 per cent. solution of sodium rhodizonate to a moist filter-paper, which is then placed for 30 seconds over the mouth of a flask containing 1 c.c. of ammonia (sp.gr. 0·88) and 1 c.c. of water; the sodium rhodizonate solution should be made freshly each day. The liquid is then titrated with a standard (0·02 *N*) solution of sodium sulphate, being boiled for 2 minutes near the end-point. An allowance should be made for any sulphur found in the reagents in a blank test. J. G.

Control of Sewage Chlorination. The Use and Value of the *o*-Tolidine Test. C. Lea. (*J. Soc. Chem. Ind.*, 1933, **52**, 245–250T.)—Owing to the variety of the impurities in sewage there is no clearly defined chlorine saturation point, and the *o*-tolidine test requires to be rigidly standardised before useful results can be obtained. A preliminary test should be made to determine the approximate chlorine requirement, and this is followed by the more exact determinations. For the preliminary test, 250 c.c. of the sample are placed in a 10-oz. bottle and chlorine water added (1 grm. of available chlorine per litre) in portions of 0·5 c.c. each; after each addition, the sample is agitated gently for five seconds, and 0·25 c.c. is then withdrawn and added to one drop of *o*-tolidine reagent on a spotting plate. The chlorine addition is continued until the spot-test shows a faint yellow colour. The quantity of chlorine required in this preliminary test is termed the "immediate chlorine demand" or "I.C.D." A further quantity of 250 c.c. of the sewage is then treated directly with an amount of chlorine a few parts per million in excess of the "I.C.D." and, after ten minutes, 11·5 c.c. of the mixture are treated with 1 c.c. of *o*-tolidine reagent in the cell of a colour comparator. The colour is allowed to develop for five minutes. The test is repeated on a number of 250-c.c. portions of the sewage until the exact amount of chlorine required is ascertained. It is essential that pure *o*-tolidine of m.pt. 129° C. be used in preparing the reagent; 1 grm. is dissolved in 1 litre of 18 per cent. hydrochloric acid (by volume), *i.e.* 180 c.c. of concentrated hydrochloric acid, sp.gr. 1·18, diluted to 1 litre.

W. P. S.

Inorganic Analysis

New Complex Cyanogen Compounds of Metals. J. Foucry. (*J. Pharm. Chim.*, 1933, **42**, 432–433.)—The addition of a mixture of equal parts of solutions of potassium thiocyanate and of antipyrine (10 per cent.) to a faintly acid solution of a ferric salt yields a red precipitate or coloration depending on the amount present; iron in amounts down to 5 mgrms. per litre may thus be detected. Under similar conditions, precipitates are given by various other metals, *e.g.* uranium, zinc, manganese (white), copper (greenish grey). The precipitates are complex and are unsuitable for quantitative purposes. S. G. C.

The Gutzeit Test for Arsenic. G. Lockemann and B. v. Bülow. (*Z. anal. Chem.*, 1933, **94**, 322–330.)—The authors give preference, on the score of sensitiveness, to silver nitrate over mercuric chloride or bromide. The silver solution must be made of two parts of silver nitrate and one part of water. A sensitiveness of one ten-millionth grm. is claimed. A very simple apparatus is described, consisting of a 100-c.c. round flask surmounted by a plain, empty bulb tube, the upper end of which carries a 5·5-cm. filter tied down by means of a rubber band.

W. R. S.

Precipitation of Tin as Stannous Sulphide. B. Linke and H. Preissecker. (*Z. anal. Chem.*, 1933, **94**, 238–239.)—The procedure is claimed to give a dense, black precipitate which is easily filtered off, and can be washed with water without risk of a cloudy filtrate. The chloride solution is neutralised with sodium carbonate against phenolphthalein, and treated in the cold with 10 c.c. of strong hydrochloric acid, and then gradually with about 0·1 grm. of pure magnesium. The flask is closed with a Bunsen valve and warmed, if necessary, for the complete solution of the magnesium. Hot water (350 c.c.) is added, and hydrogen sulphide is passed through the flask while it is immersed in a boiling water-bath. Precipitation is complete in about 10 minutes. The precipitate is collected, washed free from chloride, dried, and ignited to SnO_2. W. R. S.

Colorimetric Determination of Aluminium with Aurin Tricarboxylic Acid. P. S. Roller. (*J. Amer. Chem. Soc.*, 1933, **55**, 2437–2438.)—The addition of alkali in Yoe and Hill's quantitative application (*J. Amer. Chem. Soc.*, 1927, **49**, 2395) of Hammett and Sottery's aurin tricarboxylic acid test (ANALYST, 1925, **50**, 152) greatly reduces the sensitivity of the reaction and also masks the effect of chromium (Cr^{+++}). Since the yellow colour of the dye in alkaline solution and its red colour in acid solution practically neutralise each other at p_H 6·3 in buffered solution, a method in which the comparison is made at that p_H value has been devised:—The aluminium solution (x c.c.) at about p_H 6·3 is diluted with $(12-x)$ c.c. of water, and 5 c.c. of 4 M ammonium acetate acidified with hydrochloric acid as a buffer (p_H 6·3). The mixture is shaken, treated with a 0·1 per cent. solution of the dye (1 c.c. per 0·01 mgrm. of aluminium), and again thoroughly shaken. The maximum intensity of colour is reached in about 15 minutes, and the colour is stable for many hours. As, in alkaline solution, the intensity of colour is not linearly proportional to the amount of aluminium for amounts of aluminium below 0·002 mgrm., the comparison is made in Nessler tubes. Blank results were obtained with 10 mgrms. of barium, calcium, magnesium, zinc, lead; with 0·1 mgrm. of cobalt and copper; and with 5 mgrms. of phosphate (PO_4). Ferric iron interferes and must be removed (0·010 mgrm. of iron was colorimetrically equivalent to 0·005 mgrm. of aluminium). Chromium has only a slight interference: At room temperature 0·10 mgrm. of green Cr^{+++} gave, after 15 minutes, a colour equivalent to 0·0005 mgrm. of aluminium; after 30 minutes the colour was equivalent to 0·001 mgrm. of aluminium; and after 18 hours to 0·008 mgrm. By increasing the p_H of the solution the interference is prevented, but at the expense of the sensitivity. The reaction under these conditions is twenty times as sensitive as that reported by Yoe and Hill (*loc. cit.*); a faint pink is obtained with 0·0001 mgrm. of aluminium (*cf.* Lampitt and Sylvester, ANALYST, 1932, 57, 418).

Colour Reaction of [Magnesium and Beryllium], Zirconia, Thoria, and Rare Earths with Quinalizarin. A. S. Komarovsky and I. M. Korenman. (*Z. anal. Chem.*, 1933, **94**, 247–249.)—Quinalizarin (tetrahydroxy-anthraquinone) is used as a sensitive reagent for magnesium and beryllium (ANALYST, 1928, **53**, 303). The authors have proved that zirconia, thoria, and the ceria earths also give an intense blue colour when treated with an alkaline solution of the reagent, the suspension gradually depositing a blue flocculent precipitate. The behaviour of the blue lakes towards bromine is the same as that of the beryllium compound. Hence the colour-reaction is not specific for beryllium or magnesium.

W. R. S.

Precipitation and Determination of Magnesium Hydroxyquinolate in Presence of Calcium Oxalate and its Application to the Analysis of Portland Cement. J. C. Redmond. (*Bur. of Standards J. Res.*, 1933, **10**, 823–826.)—Redmond and Bright's method for the determination of magnesium in Portland cement by the use of 8-hydroxyquinoline (ANALYST, 1931, **56**, 339) may be shortened without sacrifice of accuracy by omitting the removal of the calcium oxalate, and precipitating and titrating the magnesium hydroxyquinolate in the presence of the calcium oxalate. The time required by an experienced operator for carrying out a determination is thus reduced to 1–1½ hours. S. G. C.

Study of Potassium Perchlorate. E. Kahane. (*Bull. Soc. Chim.*, 1933, **53**, 547–555.)—As a result of solubility measurements, it is concluded that the addition, at the ordinary temperature, of an equal volume of perchloric acid (60 per cent., sp.gr. 1·61) to the solution under examination, yields a precipitate when the amount of potassium exceeds 0·7 grm. per litre. The solubility of potassium perchlorate increases with decrease in the concentration of perchloric acid, rising to 5 grms. per litre in the presence of 10 per cent. of perchloric acid. Ammonium, rubidium, and caesium also give precipitates with perchloric acid, but otherwise the reaction is specific for potassium. S. G. C.

Conductometric Method of Titrating Potassium. J. H. Boulad. (*J. Soc. Chem. Ind.*, 1933, **52**, 270–272T.)—In the reaction

$$2KCl + Ca_2Fe(CN)_6 = K_2CaFe(CN)_6 + CaCl_2,$$

the net effect during precipitation of the potassium calcium ferrocyanide (in the presence of alcohol) is the replacement of $K^{\cdot}$ by $Ca^{\cdot\cdot}$ ions, with very little change in conductivity. When, however, precipitation is complete, there is a rise in conductivity due to $Ca^{\cdot\cdot}$ and $Fe(CN)_6''$ ions from the excess of reagent, and the graph obtained on plotting the conductivity (as ordinate) against the volume of reagent added is, therefore, an almost horizontal line intersecting an upward gradient at the end-point. The well-known Kohlrausch apparatus and technique were used, with the telephone as a null-point instrument. The solution should be just alkaline to phenolphthalein, or preferably neutral, and should contain about 36 and 50 per cent. by volume of alcohol for potassium-contents of 0·015 to 0·06 *N* and 0·004 *N* to 0·01, respectively. Precipitation is initiated and accelerated by "seeding" with 0·5 to 1 grm. of finely-powdered potassium calcium ferrocyanide for every 50 c.c. of solution before the titration, and for the lower concentrations by adding also one or more equivalents of $Ca^{\cdot\cdot}$ ions (*e.g.* in the form of the poorly-conducting acetate); from 1 to 3 minutes are required for completion of precipitation after each addition of reagent. The reagent, which should be 20 to 30 times as concentrated as the solution under examination, and be added in 0·1 to 0·5-c.c. portions, is standardised by titration with potassium permanganate solution in the presence of an excess of cold dilute sulphuric acid (*cf.* de Haen, *Ann. Chem. Pharm.*, **90**, 160), or by titration of a standard solution of potassium. Sodium, calcium or magnesium ions do not interfere if calcium acetate is added, but heavy metals and barium ions should be absent. Hydroxyl ions give low results, owing to the insolubility of calcium hydroxide in alcohol; sulphates slow down the reaction, unless precipitated by calcium acetate. (cf. *supra*); phosphates act similarly, but can be eliminated by addition of a slight excess of calcium acetate, followed by sufficient calcium hydroxide to produce alkalinity to phenolphthalein; a solution containing acetic acid at a concentration about 2 *N* gave an indefinite end-point. The error is normally ±1 to 2 per cent. J. G.

Picrolonic Acid as a Reagent for Sodium. Y. Volmar and M. Leber. (*J. Pharm. Chim.*, **1933, 17, 427–431.**)—Picrolonic acid cannot be used for the detection of sodium in presence of potassium owing to the solubilities of sodium and potassium picrolonates being closely similar. Potassium may be removed by precipitation as picrate or chloroplatinate, and sodium detected in the solution by the picrolonate method (ANALYST, **1933, 57,** 368). S. G. C.

Determination of Sodium Hydrosulphide in Sodium Sulphide Products. E. Benesch. (*Chem.-Ztg.*, **1933, 57, 562–564.**)—The method provides for the determination of sodium hydrosulphide in materials containing the following sodium salts: sulphide, hydrosulphide, sulphate, thiosulphate, sulphite, carbonate and chloride (cf. *Chem.-Ztg.*, 1924, **48,** 573). Three main titrations are necessary: (*a*) A suitable volume of the sample solution is titrated with 0·1 *N* iodine solution, with starch as indicator. From this iodine-titration value is deducted the iodine-titration value of the thiosulphate and sulphite present, which is determined on a separate portion of the sample solution after shaking it with an excess of cadmium nitrate solution, diluting to 200 c.c., filtering off the precipitated cadmium sulphide, and titrating an aliquot part of this filtrate with 0·1 *N* iodine solution. (*b*) A similar volume of the sample solution to that used in (*a*) is titrated with 0·2 *N* sulphuric acid, with phenolphthalein as indicator. (*c*) Another similar volume of sample solution is titrated with 0·2 *N* sulphuric acid, with methyl orange as indicator. From the number of c.c. of sulphuric acid required in this titration is deducted one quarter of the number of c.c. of 0·1 *N* iodine solution required for the titration of the sulphite-content, which is determined as follows:—To a separate similar volume of sample solution contained in a 500-c.c. graduated flask, and diluted to 300 c.c., are added an excess of cadmium nitrate solution, to precipitate the sulphide, and an excess of barium chloride solution, to precipitate the sulphite;

the liquid is filtered, and 250 c.c. of the filtrate, containing the thiosulphate, are titrated with 0·1 *N* iodine solution; this iodine-titration value is deducted from the iodine value of the thiosulphate and sulphite together as found above, giving the iodine equivalent of the sulphite. The amounts of sodium hydrosulphide and of other constituents are calculated with the aid of the following equations: $x+y=a$; $x/4 + z/2 = b$; $x/2 + y/4 + z = c$, where x is the 0·1 *N* iodine equivalent of the sodium sulphide, y is the 0·1 *N* iodine equivalent of the sodium hydrosulphide, z is the 0·2 *N* sulphuric acid equivalent of the sodium carbonate; a, b, and c are the results of the above titrations. (*Abstractor's Note.*—No provision is made for the possible presence of sodium hydrosulphite.) S. G. C.

Determination of Selenium in Pyrites. K. Brückner. (*Z. anal. Chem.*, 1933, **94**, 305–322.)—The usual methods for the determination of selenium in pyrites do not give satisfactory results. The present investigation proves that volatilisation is the cause of the discrepancies. Selenium is shown to be markedly volatile in boiling, strong hydrochloric acid solution; no loss occurs if the same solution is boiled under reflux. On evaporation to dryness, the loss is of the order of 60 per cent.; the residue, when heated in an oven to 100° C., undergoes further loss. No selenium loss occurs when a nitric acid solution is boiled, but evaporation to dryness and heating of the residue in an oven cause a certain loss. *Aqua regia* solutions behave like those in nitric acid: losses occur only as dryness is reached. The addition of sodium hydroxide to the *aqua regia* solution reduces, but does not prevent, the loss of selenium. The same solution can be evaporated with sulphuric acid to the appearance of white fumes without appreciable loss; incipient volatilisation is noticeable only after an hour's fuming. Selenium is quantitatively precipitated by sulphur dioxide or hydrazine from a solution containing from 10 to 40 volumes per cent. of sulphuric acid. It is advisable to add 10 c.c. of strong hydrochloric acid to the sulphuric solution.

The following improved procedure was worked out for the determination of selenium in pyrites:—Twenty grms. are dissolved in 125 c.c. of strong nitric acid (cold) in a tall beaker. The solution is heated to boiling, the flame is withdrawn, and strong sulphuric acid (60 c.c.) is added, when the mass solidifies. The beaker is transferred to a hot sand-bath and heated until white fumes appear. The cooled residue is treated with 300 c.c. of water, then with 20 c.c. of strong hydrochloric acid, and heated to effect solution. The solution is diluted to 400 c.c. and filtered. The filtrate is treated for 10 minutes at 60° C. with sulphur dioxide, then with 3 c.c. of 10 per cent. hydrazine hydrate, and left to cool. The impure selenium is collected, washed, and transferred (with the paper) to a small conical flask, in which it is dissolved in 10 c.c. of hydrochloric, and 5 drops of nitric, acid at water-bath temperature. The solution is diluted, filtered, and treated with 2 c.c. of the hydrazine solution. After cooling, the precipitate is collected in a tared porous crucible, washed with water, dried at 105° C., and weighed. The determination can be made in a day. W. R. S.

Argentometric Determination of Selenocyanate. R. Ripan. (*Z. anal. Chem.*, 1933, **94**, 331–334; 335–337.)—(i) The neutral solution is titrated with silver nitrate solution and the end-point is ascertained potentiometrically. The potential rapidly becomes constant, except in the neighbourhood of the end-point, where small irregularities (due to adsorption phenomena) occur; from 4 to 6 minutes must be allowed to elapse between the addition of silver solution and the potential measurement. The first drop in excess causes a decided change in potential, and the solution becomes clear.

(ii) The determination can be carried out with the help of an adsorption indicator (1 to 2 drops of 0·2 per cent. alcoholic fluorescein solution). A silky crystalline precipitate is formed at first; the red colour, which is produced by each addition of silver solution, becomes more and more permanent as the end-point is approached, the silky precipitate gradually becoming coarsely flocculent. The silver solution being added slowly during vigorous agitation, one drop suffices to produce a distinct colour-change. W. R. S.

Microchemical

Quantitative Determination of Unweighable Amounts of Material. F. Emich. (*Mikrochem.*, 1933, 13, 283–288.)—Suggestions are made as to the possibility of determining unweighable amounts of material by an "enlargement" process, by which the final factor for calculation may be increased. Suggested examples include (*a*) the determination of a trace of silver sulphate. If this is precipitated with barium chloride, both silver chloride and barium sulphate are precipitated. If the precipitate is separated and treated with sulphuric acid, the silver chloride is re-converted into sulphate. This is again precipitated with barium chloride, and the precipitate is washed and converted into sulphate again. The process can be repeated until a weighable precipitate is obtained. Ten precipitations give an "enlargement factor" of 8·39. Example (*b*) is a similar series of re-precipitations

$$MgSO_4 \xrightarrow{Ba(OH)_2} Mg(OH)_2+BaSO_4 \xrightarrow{H_2SO_4} MgSO_4+BaSO_4 \xrightarrow{Ba(OH)_2} Mg(OH)_2+2BaSO_4,$$

and so on. For ten repetitions the "enlargement factor" is 20·4. Example (*c*) suggests the alternate decomposition and formation of carbonate from an unweighable amount of calcium carbonate, the carbon dioxide being absorbed in the same absorbing apparatus each time, until the total amount becomes weighable. Other suggestions include determination of sulphur and carbon dioxide, and also determinations of metals by measurement, in single bubbles, of the hydrogen liberated. J. W. B.

Microchemical Tests. VIII. L. Rosenthaler. (*Mikrochem.*, **1933**, **13**, 83–85.)—(i) *Detection of sodium with uranyl acetate.*—Difficulties in using this test occur when the uranyl acetate forms sparingly soluble compounds with any anions present—such as phosphates, arsenites, arsenates, sulphides, tungstates, benzoates, and salicylates. Sodium nitrate gives, with uranyl acetate, long needles and pointed prisms, and the characteristic tetrahedra and octahedra of sodium uranyl acetate do not appear. The reaction may be due to the nitrate ion, as potassium nitrate gives the same crystals; but ammonium, barium and strontium nitrates do not form crystals; and silver nitrate gives small flattened leaves.

(ii) *Detection of Zinc and Cadmium with Iodisan* (*hexamethyl-diamino-iso-propanol-diiodide*).—A drop of a 0·1 per cent. solution of a cadmium salt (sulphate or bromide) gives, with a drop of iodisan (commercial solution), a crystalline precipitate of stars and needles. The limit of dilution is about 1 : 50,000, and the smallest amount detectable is 1γ Cd. When the cadmium salt is added in the solid form only an amorphous precipitate is produced. Even in a 10 per cent. solution zinc salts give no crystalline precipitate with iodisan; crystals are formed only on evaporation, or by adding a solid zinc salt to the reagent. Iodisan reacts with some other heavy metals, and the presence of free iodine or insoluble iodides interferes with the test. Ferric iron gives a yellow crystalline precipitate. Four photomicrographs are given. J. W. B.

Rapid Micro-Analysis of Pure Aluminium. I. Iron, Copper and Manganese. F. Pavelka and H. Month. (*Mikrochem.*, **1933, 13, 305–312.**)—Small traces of foreign substances of considerable importance in corrosion effect may be present in 99·8 or 99·9 per cent. pure aluminium. These substances are usually one or more of the following:—iron, copper, manganese, silicon, alkalis and alkaline earths. For the rapid determination of *iron*, *copper* and *manganese* a single sample of not more than 0·1 grm. of a 99·8 per cent. aluminium is taken. The sample is weighed into a 10-c.c. graduated flask and dissolved in 50 per cent. sulphuric acid on a boiling water-bath. A drop of hydrogen peroxide is added to oxidise the metals, and the contents of the flask are made up to the mark. For the *iron* determination, 1 to 3 c.c. (for 0·12 to 0·04 per cent. of iron) of the solution are placed in a 5-c.c. graduated flask, 1 c.c. of a 0·02 per cent. solution of potassium ferrocyanide is added, and the mixture is made up to the mark with water. After 10 minutes the colour developed is compared with a standard in an Autenrieth colorimeter. The presence of excess of aluminium does not affect the colour, and determinations on samples of known iron-content gave errors of less than 2 per cent.

Copper.—The copper is determined in 1 to 2 c.c. of the original solution. The time of decolorisation of a mixture of ferric thiocyanate and sodium thiosulphate by a copper-containing solution is used to determine the small amounts of copper. *Reagents*.—(i) A 2·5 per cent. solution of sodium thiosulphate. (ii) A 0·5 per cent. solution of pure crystallised iron thiocyanate. The standard copper solution contains 0·005 mgrm. of copper per c.c. A standardisation curve is made of the times of decolorisation, by taking 100 c.c. of the thiosulphate solution, adding a known amount of copper, and then adding rapidly 100 c.c. of the iron thiocyanate solution mixed with 5 c.c. of $N/5$ hydrochloric acid. The mixture is shaken, causing the colour to turn from red to yellow, and finally to green, when the time is taken. In the absence of copper the decolorisation time is 153 seconds; for 0·005 mgrm. of copper, 102 seconds; for 0·010 mgrm., 82·5 seconds; for 0·015 mgrm., 66 seconds; for 0·020 mgrm., 57·5 seconds; for 0·025 mgrm., 49·5 seconds; for 0·035 mgrm., 42 seconds; for 0·050 mgrm., 36 seconds. The time taken by the mixture containing the unknown amount of copper to become decolorised gives (from the calibration curve) the copper-content. The error is of the order of a few per cent. Of course the calibration curve is made for the temperature* at which the determinations are carried out.

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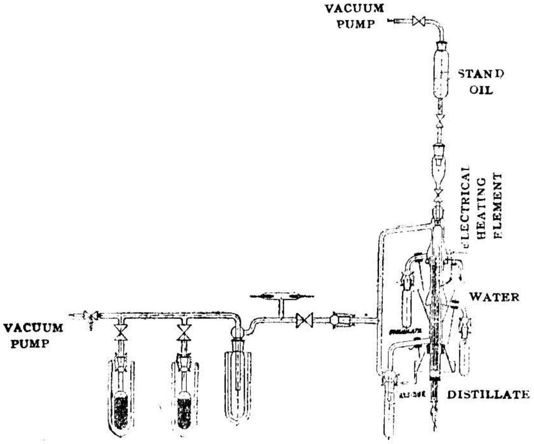
* Room temperature is implied, but not stated.—*Abstractor.*

Manganese is determined colorimetrically in 2 to 3 c.c. of the original solution by oxidation to permanganate. As the large excess of aluminium interferes, it is removed by adding 1 c.c. of a 1 per cent. solution of ferrous sulphate and 4 drops of saturated bromine water, followed by sufficient 10 per cent. sodium hydroxide solution to dissolve the precipitated aluminium hydroxide. The mixture is boiled and filtered through a micro-suction filter or filter stick with an asbestos filtering surface, and the precipitate is washed with hot water. It is then moistened with 3 drops of 3 per cent. hydrogen peroxide and dissolved by sucking through it about 2 c.c. of a 2 *N* sulphuric acid solution, followed by a little water, the solution being collected in a micro-beaker. The solution is evaporated to 2 c.c., oxidised with 0·5 c.c. of ammonium persulphate and 1 drop of 0·1 *N* silver nitrate solution, and heated for ten minutes over a boiling water-bath. The solution is cooled, transferred to a 5-c.c. graduated flask and made up to the mark, and the colour is compared with that given by a standard manganese solution. A blank determination should be made. The method is suitable for amounts of manganese varying from 3γ to 50γ, with an error of a few per cent. J. W. B.

Physical Methods, Apparatus, etc.

Polymerisation of Linseed Oil. H. I. Waterman and D. Oosterhof. (*Rec. Trav. Chim. Pays-Bas*, 1933, **52**, 895–900.)—The apparatus shown enables oils of high molecular weight to be distilled without decomposition or loss of material by minimising the period of heating and the distance between the surfaces of evaporation and condensation. The system is exhausted by means of mercury pumps attached to the top and extreme left-hand outlets, this process being aided when necessary by means of adsorbing carbon cooled in liquid air in the side-tube. The oil is allowed to drip from the top container on to an inner glass tube (with a frosted surface to ensure uniform distribution of the oil in a thin film), where it is heated electrically at 250° to 260° C. The volatile fractions are removed in about 4 seconds, and are condensed on the outer, water-cooled jacket. It was possible to distil part or all of a sample of linseed oil practically without decomposition,



as was demonstrated by the fact that a mixture of the distillate and residue gave the same refractive index, sp.gr. and acid value as the original oil. With a "stand-oil," 0·3 grm. of material, consisting mainly of fatty acid [Wijs iodine value, 128·8 (2 hours); n_D^{20}, 1·4735], was obtained before the hottest part of the tube was reached; in addition, the main distillate and residue gave the following results, respectively:—yield, 19·41 grms.; d_4^{20}, 0·9362, 0·9756; n_D^{20}, 1·4806, 1·4957; viscosity (20° C.), 1·51, 379 poises; molecular weight (benzene), 757, 3463; iodine value, 122·2, 120·2; acid value, 19·5, 1·5; saturated fatty acids, 14·7, 8·6 per cent.; hydrogen, 11·6, 11·0; carbon, 77·8, 77·3 per cent. The figures obtained for the re-constituted oil showed that there had been no material decomposition. The distillate became turbid after 1 day, but the residue remained clear for several months. The results suggest that the turbidity of a "stand-oil" is due not to "over-polymerisation" and flocculation of the polymers, but probably to crystallisation of the components of low molecular weight. The residue had better drying properties than the original oil, and the method, therefore, has commercial possibilities. The increase in viscosity appears to be related to the formation of turbidity; and, although the oil becomes clear after 1 hour at 80° C., the viscosity (at 20° C.) is then lower than the original value; it is, therefore, important to use only clear and unheated "stand-oils" for viscosity determinations.

J. G.

Reviews

INORGANIC COLLOID CHEMISTRY. By H. B. WEISER. Vol. I. COLLOIDAL ELEMENTS. Pp. xi+389. New York: J. Wiley & Sons; London: Chapman & Hall, Ltd. 1933. 28s.

This is the first volume of an intended series under the general title *Inorganic Colloid Chemistry*, by Professor H. B. Weiser, of the Rice Institute, his aim being to give "a critical survey of the colloidal behaviour of the elements and their inorganic compounds, with particular reference to the rôle they have played in the development of the theories and applications of colloid science." With the present emphasis on the biochemical and organic chemical aspects of colloid studies, this book is opportune in drawing attention to the part played by the study of inorganic chemistry in introducing the principles of colloid chemistry.

The present volume deals with the formation, properties and applications (including therapeutic) of sols of the elements, metals and non-metals, the order of discussion following, so far as practicable, the periodic table. Adequate and critical accounts are given of the various methods of the preparation of sols, the mechanism of sol formation, the composition and constitution of sols, their physical and chemical properties, and the nature of adsorption processes.

The fourteen chapters cover a wide range. Special mention may be given to the full discussions on colloidal gold, colloidal protection, coagulation of sols, contact catalysts, activated carbon, and colloidal sulphur—all very important subjects because of their theoretical or technical treatment. The account of

Lange's gold sol reaction with cerebrospinal fluid may be cited to illustrate biological applications.

The author's own views are given freely, and gaps in present knowledge are pointed out. The text is easy to read, and it will definitely repay careful study by all interested in the general field of colloidal phenomena. The printing and binding are excellent. Several minor misprints are noted: Page 26, line 9, *for* "greater" *read* "smaller"; page 313, line 22, *for* "island" *read* "Irish"; page 360, line 2, *for* "selenium" read "tellurium."

Full author- and subject-indexes are provided, in addition to nearly 1200 foot-references.

WILLIAM CLAYTON

ORGANIC SYNTHESES: AN ANNUAL PUBLICATION OF SATISFACTORY METHODS FOR THE PREPARATION OF ORGANIC CHEMICALS. Vol. XIII, pp. 119+vii. Editor: W. H. CAROTHERS. New York: John Wiley & Sons, Inc.; London: Chapman & Hall, Ltd. Price 10s. 6d. net.

The series to which this volume belongs is too well known to need any introduction. Volume XIII contains details of the preparation of thirty substances. Among those of particular interest may be mentioned the preparation of fluorobenzene and fluorobenzoic acid, for they involve the use of fluoroboric acid and an extension of the diazo reaction. The organic chemist will also find the preparation of *n*-butyl borate, butyroin and diphenyl-triketone worthy of attention. For the analyst the preparation of the "carbonyl" reagents—2:4-dinitro-phenyl-hydrazine and α-methyl-α-phenylhydrazine—is of special interest. An analytical method for checking the purity of benzoyl peroxide is also given.

In the compilation of the book the usual plan has been followed, and there is evidence that the authors have done their work with that attention to minute detail which is so characteristic of the series. The present volume contains an unusually interesting set of preparations, and is well worthy of a place alongside its predecessors.

HAROLD TOMS

METHODS OF CELLULOSE CHEMISTRY. By C. DORÉE, M.A., D.Sc., F.I.C. Pp. viii+499. With 67 illustrations and diagrams. London: Chapman & Hall, Ltd. 1933. Price 21s.

In the cellulose industries the number of variations in methods of examination is almost equal to the number of users. A certain amount of standardisation has been achieved by independent committees concerned with the determination of such factors as strength and cuprammonium viscosity, but the use of even these "official" methods has generally been confined to the countries of origin. Almost all the tests available are empirical, and some published analytical methods are expressly designed to give favourable results with materials from a particular source. The various methods for the determination of alpha-cellulose are typical examples of this practice.

In these circumstances it is very difficult for the scientific worker unacquainted with some section of cellulose chemistry to choose between the many methods scattered through a large number of scientific journals. This book by Dr. Dorée is of great value in this connection, since it gives selected methods, almost all of which have been independently tested. The inclusion in most instances of actual values obtained by the method described adds considerably to the usefulness of the book.

The subject has been divided into three sections. The first, "normal cellulose," includes discussion of oxy-cellulose, hydro-cellulose, and degradation products. The importance of viscosity measurement has been increasingly realised during recent years, and the chapter on the measurement of viscosity details the methods more generally used. Also in this section are very interesting chapters on the "measurable qualities of cellulose," and on "dispersed cellulose."

The second section covers what are described as "synthetic derivatives" of cellulose; that is, the nitrate, acetate, viscose, and other esters and ethers of cellulose.

The third section deals with the "compound celluloses"; finally, there is a short appendix, listing some of the relevant textbooks, and giving a few useful tables.

The wide range of subject-matter to be covered must make difficult the selection of material for inclusion. The present volume is well balanced in this respect, with the possible exception that the relatively large pulp and paper industries might have been allotted rather more space.

The *X*-ray investigation of cellulose "has been designedly omitted, as it is essential to gain experience of *X*-ray methods and their interpretation at first hand."

The views of many investigators are here for the first time assembled in accessible form, and can be read almost as a continuous narrative. This enables a comprehensive picture of the analytical side of cellulose chemistry easily to be visualised. This book, therefore, is much more than a mere descriptive list of methods to be employed, and will thus be of considerable assistance to chemists having contact with the cellulose industries, quite apart from its value as a source of reference.

L. HEBBS

Errata

Vol. LVIII, 1933.

P. 29, line 5 from bottom, *for* ' Chambers" *read* "Chalmers."
P. 374, line 8, *for* "30s." *read* "36s."
P. 430, second column of figures, *for* "27.10 and 10.13" *read* "24.10 and 27.13."

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