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 on Wednesday, October 6th, 1937, the President, Dr. G. Roche Lynch, in the chair.

Miss M. B. Elliott, M.B.E., was nominated as an honorary member of the Society.

Certificates were read in favour of Donald Colin Malcolm Adamson, A.I.C., B. Boas, B.Sc., Ernest Oddy, Gerard Lionel Ransome, B.Sc., A.I.C., and Reginald Frank Wright, B.Sc., A.R.C.S., F.I.C.

The following were elected members of the Society:—John Short Dunn, B.Sc., A.I.C., and Ernest Turner, B.Sc., F.I.C.

The following papers were read and discussed:—"Fluorine in Food Products," by H. C. Lockwood, B.Sc., F.I.C.; "Analysis of Coffee Infusions," by F. W. Edwards, F.I.C., and H. R. Nanji, Ph.D., D.I.C., F.I.C.; "The Determination of Tannins in Cacao Kernel," by D. W. Duthie, M.A., Ph.D., A.I.C.

Death

WITH deep regret we record the death on November 6th of Dr. J. Augustus Voelcker, C.I.E. An obituary notice will be published later.

Obituary

JOSEPH HAROLD TOTTON

BY the unexpected death of Joseph Harold Totton on September 7th, at the comparatively early age of fifty-seven, the Society loses one of its most distinguished members in Northern Ireland.

Totton received his early chemical training at Queen's College, Belfast, and took the degrees of B.A. and B.Sc. at the old Royal University of Ireland. Having decided to specialise in analytical chemistry he went to England and became an assistant to the late Colonel C. E. Cassall, Public Analyst for Kensington. In 1907 he passed the examination for the Associateship of the Institute of Chemistry, and later became a Fellow.

On his return to Ireland in 1907 Totton was appointed Public Analyst and Official Agricultural Analyst for County Armagh; later he was elected to the corresponding posts for the City of Belfast, for County Derry (1910) and for County Antrim (1923). As these were all part-time appointments, he was also able to engage in consulting work, and for many years, until his death, was senior partner in the firm of Totton and Hawthorne.

Notwithstanding the claims of his official work and private practice, Totton could still find time to promote the interests of his profession, and did much to improve the status of "chemist" in Northern Ireland. Thus he took an active part in the foundation of the Northern Ireland Section of the Institute of Chemistry and became its first chairman.

His sympathetic nature and sound common sense led his colleagues to seek his advice in their difficulties, and they never sought in vain. In the laboratory he was never ruffled, and he had a charm of manner that endeared him to all with whom he worked. Owing to his official position, he was unable to take any active part in public affairs, although he was prominent in masonic circles, and made very many friends, who found him a delightful companion, and, above all, valued him as one whom they could trust.

Totton was a man who will long be remembered. J. HAWTHORNE

Société de Chimie Industrielle

THE following Address was sent by the Society to the Société de Chimie Industrielle. Dr. L. H. Lampitt acted as representative of the Society and presented the Address.

The President has received from the Société de Chimie Industrielle a letter of thanks, together with a bronze plaque, struck to commemorate the occasion.

FROM
THE SOCIETY OF PUBLIC ANALYSTS
AND
OTHER ANALYTICAL CHEMISTS
TO
SOCIÉTÉ DE CHIMIE INDUSTRIELLE

On the occasion of the Commemoration, in Paris, on September 30th, 1937, of the Twentieth Anniversary of the Foundation of the Société de Chimie Industrielle, The President, Officers, Council and Members of the Society of Public Analysts and Other Analytical Chemists send Fraternal Greetings to the Officers and Members of the Société de Chimie Industrielle.

The Society sincerely welcomes this opportunity to express the friendship and goodwill of its members towards their French confrères and to offer best wishes for the continued prosperity of the Société de Chimie Industrielle.

(*Signed*) G. ROCHE LYNCH (*President*)
E. B. HUGHES (*Honorary Treasurer*)
LEWIS EYNON (*Honorary Secretary*)

Dated this Thirtieth Day of September,
Nineteen Hundred and Thirty Seven

Seal of the Society

Fluorine in Food Products

By H. C. LOCKWOOD, B.Sc., F.I.C.

(*Read at the Meeting, October* 6, 1937)

The presence of fluorides in water supplies of certain districts has caused some concern in medical and dental circles. It has been established by Churchill,[1] Smith, Lantz and Smith,[2,3] Ainsworth[4,5] and others, that even minute traces of fluorides produce fluorosis which manifests itself particularly as "mottled teeth" in growing children. The subject of fluorides in food products has received, however, comparatively little attention. Wichmann and Dahle[6] record the following results for produce sprayed with insecticides containing fluorides:—apples, 2 parts per million; cabbage, outside 33·6, inside 2·7; celery (two samples), 7·6 and 3·6 in the petioles, 77·1 and 135·3 in the leaves; preserved strawberry juice, 141 parts per million. Phillips, Hart and Bohstedt[7] found that adding sources of fluorine to cows' diet made no appreciable difference to the fluorine-content of the milk, which normally ranged from 0·05 to 0·25 p.p.m., with an average value of 0·14.

In 1936, Reid[8] published a series of results on Chinese food materials, dealing particularly with tea, in which he found appreciable amounts of fluorine. His results have been converted from parts per 100,000 to parts per million—for ease of comparison with other workers' figures—and are summarised in Table I. Reid used for his determinations Boruff and Abbott's[9] modification of Willard, Arbor and Winter's[10] distillation and titrimetric technique. His results on tea are particularly important, and he considered that the high fluorine-content may be the principal cause of mottled enamel of the teeth among the Chinese. It is interesting to note that foods grown near a fluorite mine show, generally, no increase in fluorine-content.

TABLE I

Material and source	No. of samples	Fluorine (as parts per million) Minimum	Maximum	Average
Chinese tea (various districts) ..	12	37·5	398·8	109·6
" (fluorite area) ..	3	15·0	1757·8	614·6
Imported tea	5	8·7	38·1	19·6
Coffee	4	0·2	1·6	0·9
Chinese wine	4	0·05	0·24	0·11
Lemon and orange juice	3	0·14	0·22	0·17
Chinese vegetables	8	1·2	8·5	4·4
Cereals	4	0·2	1·7	1·1
Soya bean	1	—	—	4·0
Foods grown near a fluorite mine—				
Cereals	3	0·2	2·0	1·0
Soya bean	1	—	—	6·7
Miscellaneous	3	1·4	4·3	2·5
Salted and dried mustard leaves	1	—	—	265·5
Salted and dried turnips ..	1	—	—	40·4

Examination of the foregoing table shows, for solid foodstuffs, figures ranging from 0·2 to 1757·8 p.p.m., which indicate, in my opinion, a greater degree of accuracy than could be attained by the method employed. Calculation shows that to obtain titrimetrically the result of 0·2 p.p.m., a kilogram of material would be required, giving a titration result of 0·5 ml. of 0·02 *N* thorium nitrate, which, considering the indefinite type of end-point, can be regarded as approaching the minimum reliable volume. In view of the statement that the material was ashed with sodium hydroxide, magnesium nitrate and calcium acetate, and the ash distilled with 50 ml. of conc. sulphuric acid, it does not seem likely that much more than 100 g. of a solid foodstuff could have been handled conveniently. My own work indicates that for the above-mentioned volumetric method to be applicable, solid material must contain at least 5 p.p.m. of fluorine.

Although several reactions have been formulated for the detection of traces of fluorine, that originated by de Boer[11] is generally used. He found that a mixture of solutions of zirconium nitrate and alizarin sulphonic acid in the presence of hydrochloric acid was capable of detecting 0·001 mg. of fluorine in 1 ml. of water. The reaction has since been used by Willard, Arbor and Winter[10] as an indicator for the titrimetric determination of fluorine, but the end-point is not very sharp. I have found that by substituting 25 ml. of glycerin for the 25 ml. of ethyl alcohol a decided improvement resulted. Boruff and Abbott[9] used the distillation technique of Willard *et al.*, but made the distillate slightly alkaline and evaporated it to a small volume. Subsequent titration of the concentrated solution would give enhanced accuracy but, as previously stated in connection with Reid's work, with a solid food at least 5 p.p.m. of fluorine must be present. Colorimetric methods based on the fading of de Boer's zirconium–alizarin reagent have been used by Thompson and Taylor,[12] Sanchis,[13] Elvove[14] and Barr and Thorogood[15] for the direct determination of fluorine in water supplies. For the determination of traces of fluorine in foodstuffs I have applied the same principle to the distillate obtained by a modification of the distillation technique of Willard and his collaborators.

Examination of a Typical Water Supply.—As a preliminary investigation I examined the Birmingham water supplied from the Elan Valley. The method of Barr and Thorogood[15] was tried but found inapplicable owing, first, to the small amount of fluoride present and, secondly, to the yellow colour of the residue when the water had been evaporated to concentrate the mineral matter. The distillation technique of Willard, Arbor and Winter[10] was adopted, bumping being prevented by the use of a slow stream of carbon dioxide. A suitable volume (2 litres) was made alkaline, concentrated to a small volume and distilled with perchloric acid. The distillate was tested by the Barr and Thorogood method; in several determinations made during September, 1935, less than 0·1 part per million was recorded.

Distillation of Fluorine as Hydrofluosilicic Acid.—Willard, Arbor and Winter[10] based their method on the distillation of the fluorine as hydrofluosilicic acid by heating the substance with perchloric or sulphuric acid to 135° C., and maintaining this temperature by the steady addition of water until sufficient distillate had been collected. To overcome bumping I used a slow

stream of carbon dioxide, but constant attention was still required, and a method of steam-distillation at 135° to 150° C. was therefore devised (*vide infra*).

Mention should be made of the method used by Gilkey, Rohs and Hansen,[16] in which the distillation flask is supported in a vessel containing boiling tetrachloroethane. The steam supply passes through a copper spiral in the vapour, and the superheated steam enters the flask down a glass tube. Owing to the violent oxidation of organic compounds which occurs with perchloric acid above 135° C., it is advocated that sulphuric acid should be used in the flask in case breakage should occur. Although bumping and splashing are prevented, the method is unnecessarily complicated and somewhat inconvenient to use.

Fluorine in Food Products

Apparatus.—The distillation principle used by Willard and his co-workers was adopted. The apparatus, of the design shown in the diagram (Fig. 1), had ground-glass joints. The distillation tube, 6 in. long and 1¼ in. wide, was fitted with a No. 4 I.S. joint and slightly distended at the closed end. A reducing

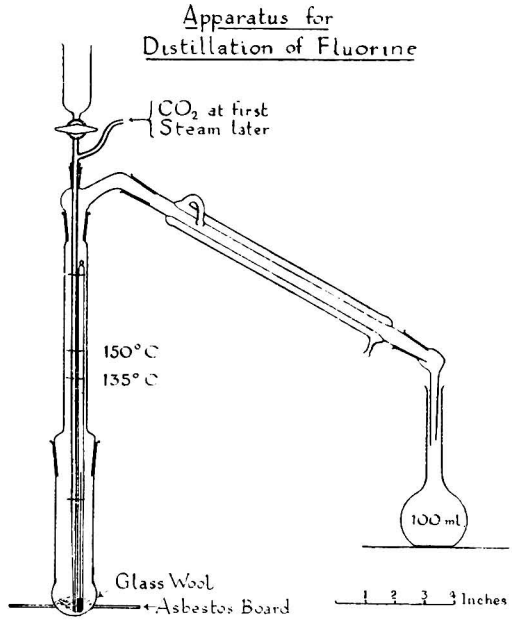

Fig. 1

socket carrying a tube, 0·65 in. internal diameter and 7 or 8 in. long, was attached, together with a still-head having a tubulure through which passed the extended stem of a small tap-funnel. The lower end of the stem was drawn out to a fine capillary, and reached practically to the bottom of the distillation tube. A 200° C. thermometer, having a half-inch bulb, was treated with alcohol and conc. nitric acid to remove the black pigment from the graduation marks, and well washed. The prepared thermometer was attached to the stem of the funnel by means of platinum wire, so that the bulb was level with the end of the capillary

tube. Owing to difficulty in reading the now faint graduation marks, it was found advantageous to have platinum wire supporting the thermometer at 135° and 150° C. marks, to make these conspicuous. Between the still-head and the funnel-tap a side tube was fitted for connection with a carbon dioxide generator, and later with the steam supply. This tube was suitably inclined to prevent liquid from the funnel flowing the wrong way. The side tube of the still-head was connected with one of the new-type single-surface condensers, having an inner tube with very thin walls for efficient cooling. In the distillation tube the glass beads used by Willard and his collaborators were replaced by a plug of glass wool, which proved more efficacious owing to the relatively greater surface exposed. The distillation tube rested on a piece of asbestos gauze or board, having a hole with a diameter slightly less than that of the tube.

PROCEDURE.—The material was prepared in a moderately fine state of division, and a weighed amount (generally 19 g., which would give direct parts per million) was placed in a platinum dish. Sufficient $N/10$ sodium hydroxide to give an alkaline reaction was added, and the contents of the dish were dried and ashed at a low temperature until ashing was as complete as possible. The dry ash was transferred to the distillation tube, which contained a small quantity of glass wool, and about 15 ml. of water containing a few drops of sulphuric acid were used for removing the residue to the tube from the dish. The apparatus was assembled, 8 to 10 ml. of conc. sulphuric acid were run down the tap-funnel, and the carbon dioxide supply was adjusted to give 3 or 4 small bubbles a second, although until boiling began a faster rate than this was advantageous, since it agitated insoluble matter on the bottom of the tube. The carbon dioxide prevented bumping until a temperature of 135° to 140° C. was attained, at which point the rubber tube was disconnected and replaced by that carrying the steam supply. The flame under the tube was reduced so that the temperature was maintained between 135° and 150° C., and the distillation with steam then proceeded smoothly, occasional adjustment of the burner being required. The distillate was received in a 100-ml. graduated flask, and afterwards a further 50 ml. were collected in a Nessler cylinder to ascertain if all the fluorine had distilled. The distillation of 150 ml. generally took about 45 minutes, and there was no trouble with bumping.

THE EFFECT OF CHLORINE IN THE DISTILLATE AND ITS ELIMINATION.—In tests on some ginger biscuits it was found that appreciable amounts of fluorine appeared to be present. This was proved to be due to traces of manganese, a natural constituent of powdered ginger. On ashing in the presence of sodium hydroxide a certain amount of permanganate was formed which, when heated with sulphuric acid, liberated chlorine from chlorides present. The distillate from powdered ginger caused the zirconium–alizarin indicator to fade rapidly, and gave a reading equivalent to 700 parts of fluorine, though later it was shown that only 1 part per million was present. Although the distillate from ginger made the indicator fade, yet, when the volumetric method of Willard[10] was tried, the colour of the indicator would not return to give the required end-point. In order to remove the chlorine without adversely affecting the zirconium–alizarin indicator, a number of substances were tried. The most satisfactory of these was sodium nitrite, which in half-an-hour removes every trace of chlorine.

As one would expect, other foodstuffs containing manganese are liable to behave in the same way as ginger. This has been confirmed with tea, a commodity in which Reid[8] reported very appreciable amounts of fluorine when using the titrimetric technique. Bodenstein[17] reported the presence of traces of manganese in pineapple, but in the sample of tinned pineapple that I examined no permanganate was formed on ashing, and the material was also free from fluorine.

Owing to the effect of free chlorine on the zirconium–alizarin reagent, it is evident that the technique of Barr and Thorogood[15] is somewhat inadequate, as in some districts appreciable amounts of chlorine are used in the purification of water supplies. Moreover, as conc. hydrochloric acid after storage is liable to contain traces of chlorine, it recommended that the Barr and Thorogood procedure for fluorine in water should be modified by including a treatment with sodium nitrite.

PREPARATION OF THE ZIRCONIUM–ALIZARIN REAGENT.—The indicator was prepared as described by Willard *et al.*,[10] the following solutions being used:—(*a*) 0·4 per cent. zirconium nitrate, (*b*) 1 g. of sodium alizarin sulphonate dissolved in 100 ml. of ethyl alcohol, and the solution filtered and made up to 250 ml. with alcohol. For use, 3 volumes of (*a*) were mixed with 2 volumes of (*b*) and diluted to 10 times the volume with water. The mixed indicator is serviceable for a week, after which a fresh supply should be prepared.

The solution thus prepared was preferred to that of Barr and Thorogood,[15] since it gives a brighter and more intense colour. The addition of hydrochloric acid which they recommended was found to be essential for making the reaction sensitive, and the optimum concentration was 2 ml. of the concentrated acid in 50 ml. of the test solution.

METHOD OF DETERMINATION.—Instead of preparing a number of standard tubes each time, as in Barr and Thorogood's method, it was found simpler and decidedly more effective to compare the fading produced by aliquot parts of the distillate with that in a tube containing a definite amount of fluoride and indicator. The standard fading test solution was therefore prepared with 10 ml. of 0·0001 *N* lithium fluoride, obtained, as required, by suitably diluting a 0·02 *N* solution. The lithium fluoride solution was pipetted into a Nessler tube, and 40 ml. of water, 2 ml. of conc. hydrochloric acid and 1 ml. of 10 per cent. sodium nitrite solution were added. The contents were well mixed and allowed to stand half-an-hour. A "blank" standard was also prepared with 50 ml. of water and the above-mentioned quantities of reagents. The preliminary testing of the first 100 ml. of distillate was generally carried out by treating 50 ml. in a manner identical with, and at the same time as, the standard fading test and blank solutions.

After half-an-hour any nitrous fumes in the upper parts of the cylinders were removed by means of a current of air, 1·5 ml. of the dilute zirconium–alizarin reagent was added to each, and the liquids were well mixed. The degree of fading was noted at intervals of approximately 30 minutes over a period of 2 hours or longer. It was quite practicable, and for the final matching preferable, to allow the fading to continue overnight, as judging in daylight was easier than by artificial illumination. The contents of the standard "fading" tube were of such

composition that the slightest indication of pink persisted for several hours; this condition is considered optimum for comparison. If preferred, a tube containing half the standard amount of fluorine can also be prepared, but generally this was not considered necessary.

As a rule, the first series of tests gave a good idea of the range required, so that for the final matching suitable volumes of distillate could be chosen accordingly and treated as described above; a standard "blank" and a standard "fading" tube for comparison were always prepared. The tube containing the 50 ml. of distillate collected after the first 100 ml. was tested at the same time, but never caused the indicator to fade to the extent of more than half the standard, except with tea containing appreciable amounts of fluorine. When dealing with materials having a fluorine-content of 1 part per million or less, it was necessary to concentrate the first 150 to 200 ml. of distillate to about 40 ml. after it had been made slightly alkaline with sodium hydroxide. The solution was then transferred to a Nessler tube, made up to 50 ml., and tested as prescribed; the estimation was made to 0·5 part per million.

Assuming that 19 g. of material were originally taken, the fluorine, as parts per million, equals the volume of distillate, *i.e.* 100 ml., divided by the volume of distillate required to fade the same amount as the standard tube. If the final 50 ml. showed half fading, 0·5 part per million was added to the result.

Recovery of Added Fluorine.—Consideration of the results given in Table II shows a fairly satisfactory recovery of added fluorine from several foodstuffs. The low result with biscuits was probably due to incomplete ashing of the material. The accuracy with which solutions can be matched diminishes with the increase in fluorine-content; at 5 parts per million and upwards the titrimetric method should be applied to the concentrated distillate.

Table II

Recovery of Added Fluorine (as parts per million)

Material	Fluorine added	Total fluorine indicated	Added fluorine recovered
Water	nil	nil	—
	4	4	4
Cocoa	nil	2	—
	5	7	5
Biscuits	nil	nil	—
	5	4	4
Milk chocolate	nil	2	—
	5	6·5	4·5
Beef	nil	2	—
	3	4·5	2·5

Fluorine in some Food Products.—The investigation was intended primarily for confectionery materials, but general foodstuffs have been included for comparison, and the results are given in Table III. It will be noticed that, with the exception of the five teas examined, the fluorine-content did not exceed 2 parts per million. It is generally recognised that fluorine in excess of 1·5 p.p.m. in water

constitutes a danger to health, but it may be suggested that foodstuffs may contain appreciably more than this before being regarded as unfit for human consumption.

TABLE III

FLUORINE IN FOOD PRODUCTS

Material	Fluorine (as parts per million)
Biscuit flour	nil
Gelatin	nil
Pineapple (tinned)	nil
Glucose	0·5
Plain chocolate	0·5
White bread	1
Potato	1
Honey	1
Powdered ginger	1
Malt (2 samples)	1, 1·5
Cocoa (3 samples)	0·5, 0·5, 2
Milk chocolate (3 samples)	0·5, 1, 2
Biscuits (5 samples)	nil, nil, 1, 2, 2
Ginger biscuits (3 samples)	2, 2, 2
Egg yolk	2
Beef	2

FLUORINE IN TEA.—Reid recorded the presence of 8·7 to 1757·8 p.p.m. of fluorine in the teas he examined, and found that 2 per cent. infusions after 5 minutes contained in solution 82 to 97 per cent. of the total fluorine. I have examined five teas, 300 to 400 ml. of distillate being collected in each instance, and the results are given in Table IV. With samples 3 and 5, 2·5 per cent. infusions were prepared and allowed to stand, with occasional mixing, for 5 minutes. The aqueous part was decanted and the residue was drained for half-a-minute. No further water was added, the object being to find the proportion which would be dissolved in normal practice. The results in Table IV show this to be approximately 75 per cent. of the total fluorine. Eight per cent. of the added water remained in the wet leaves, and calculation shows that with the China and Indian teas examined, 81 and 82 per cent. respectively of the total fluorine was in solution. The amounts of fluorine in the tea infusions and the residual leaves, determined for samples (3) and (5), agree well with the total amounts found in the teas directly.

TABLE IV

Material	Fluorine (as parts per million)	Dissolved in 2·5 per cent. infusion	Remaining in leaves
(1) Fine leaf tea	10	—	—
(2) China tea, A	40	—	—
(3) China tea, B	47	35	12
(4) Indian tea, A	60	—	—
(5) Indian tea, B	70	53	18
	73 (by titrimetric method)		

FLUORINE IN CHEMICAL INGREDIENTS.—As a matter of some importance, a number of chemical substances which may be used in connection with foodstuffs, *e.g.* baking powder, or in fireproofing material, were examined. The distillation method was not applicable to chlorides and was not used for carbonates. Solutions were prepared and filtered, before use, through well-washed filter-papers. Aliquot parts were then tested until a known amount faded to the same extent as the standard tube containing 10 ml. of 0·0001 *N* lithium fluoride solution. For carbonates, sufficient hydrochloric acid was added to neutralise the solution, followed by 2 ml. required for the test. Phosphates were distilled with sulphuric acid, and the distillate was concentrated and re-distilled with perchloric acid, as recommended by Churchill, Bridges and Rowley.[18] The results, given in Table V, range from 1 to 16 p.p.m.

TABLE V

Chemical substance	Fluorine (as parts per million)
Sodium chloride (*a*)	6
" (*b*)	6
Sodium carbonate (*a*)	3
" (*b*)	2
Sodium bicarbonate	1
Ammonium carbonate	3
Sodium di-hydrogen phosphate	6
Di-sodium hydrogen phosphate ($12H_2O$)	5
Ammonium phosphate	16

SUMMARY.—(1) The fluorine is steam-distilled at 135° to 150° C. in an apparatus of special design having ground-glass joints and containing glass wool and sulphuric acid; a slow stream of carbon dioxide is used to prevent bumping until a temperature of 140° C. has been attained. Distillation is complete in 45 minutes, and the fluorine-content is determined by "fading" zirconium–alizarin reagent in a similar way to that described by Barr and Thorogood.[15]

(2) The use of sodium nitrite is recommended for nullifying the bleaching effect of free chlorine, which is liable to be obtained with foodstuffs containing manganese, owing to the formation of permanganate on ashing.

(3) The volumetric method of Willard *et al.*[10] is discussed; it was found suitable for foodstuffs containing more than 5 p.p.m. of fluorine, but the use of 25 ml. of glycerin gave a much better end-point than 25 ml. of ethyl alcohol.

(4) Results are given showing that a satisfactory recovery of added fluorine is obtained by the method referred to in (1) and (2); the fluorine-contents of 17 different foodstuffs and 7 chemical substances are recorded.

(5) The fluorine-content of the foodstuffs examined did not, as a rule, exceed 2 p.p.m., but the important observation of Reid,[8] that tea contains considerably larger amounts of fluorine, is confirmed.

I wish to thank Mr. A. W. Knapp and Mr. J. R. Johnson for their interest and advice, and Messrs. Cadbury Brothers, Ltd., for permission to publish this work, which was carried out in their laboratories at Bournville.

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CHEMISTS' DEPARTMENT
BOURNVILLE
BIRMINGHAM

Discussion

The PRESIDENT remarked that in considering the possible toxic effects of fluorine it was important to distinguish between fluorine present in soluble and insoluble forms. The latter, even if in suspension in infusions, might not have any toxic effect.

Mr. R. W. SUTTON said that, in view of the occurrence of fluorspar in parts of Derbyshire, he had examined 30 to 40 water supplies in that district, using the method of Sanchis; in only one of the waters did he find more than one part per million.

Dr. B. S. EVANS, referring to the state of solubility of the fluorine, remarked that in teas, for example, it might possibly be in a colloidal state.

Mr. A. L. BACHARACH pointed out that many "toxic" substances (copper, for example) were harmless or even essential in minute quantities. It was therefore incorrect to speak of a substance as toxic or innocuous without reference to its concentration. It was not known at present if minute traces of fluorine were beneficial or at what concentration it became definitely injurious, and he welcomed the paper because the method described seemed to provide a means of obtaining light on these matters.

Mr. E. R. BOLTON recalled a time when relatively very large quantities of fluorine in foodstuffs were tolerated, and even thought by some to be beneficial.

Mr. E. HINKS also recalled an instance, years ago, of a food manufacturer advertising a product as bone-building on account of its fluorine-content.

Mr. C. E. SAGE asked if special precautions had been taken to eliminate the possibility of manganese causing high results. Was it only a coincidence that both tea and Cochin ginger, which contained notable amounts of manganese, gave such high figures for fluorine by the author's method?

Mr. LOCKWOOD, replying to questions in the discussion, said that numerous blank determinations had been carried out with the apparatus, but on no occasion was the presence of fluorine indicated, showing that the fluorine did not result from fluorides in the glass or reagents. This was supported by the number of commodities in which no fluorine was detected—water, biscuit flour, biscuits, gelatin and pineapple. With regard to the solubility of the fluorine in tea, he laid stress upon the fact that the decanted liquid was clear and bright while hot,

and the extracted fluorine was presumably in solution. In any event, a high proportion of the fluorine present was imbibed and was liable to be in a condition for ready absorption. The reason why more people did not suffer from mottled teeth was owing to the effect being generally restricted to growing children, who normally had very weak tea, the fluorine-content of which would not reach the danger mark of 1 part per million. In the United States of America it was proposed to fix a maximum limit of 1·4 part per million in foodstuffs. In reply to Mr. Sage, the author remarked that there seemed to be no connection between manganese-content and the amount of fluorine present; tea contained significant amounts of fluorine, whilst the powdered ginger examined contained only 1 part per million. In the presence of manganese, however, it was necessary to avoid the effect of free chlorine in the analysis; this was done by the addition of nitrite, as explained in the paper.

Characteristics of Some Reputed Cod-Liver Oils

By R. H. COMMON, B.Sc., Ph.D., A.I.C.

During December, 1936, and January and February, 1937, fourteen samples of "cod-liver oil," as sold for stock-feeding, were purchased in various towns in Northern Ireland, and subsequently examined in this laboratory. One suspicious sample (No. 5), one reliable guaranteed medicinal cod-liver oil (No. 16), and two reputable guaranteed stock-feeding cod-liver oils (Nos. 17 and 18) were also examined.

Unsaponifiable matter was determined by the ether extraction method of Archbutt and Deeley (*Lubrication and Lubricants*, 1926), the iodine value of the unsaponifiable matter by the method of Rosenmund and Kuhnhenn (*Z. Unters. Nahr. Genussm.*, 1923, **46**, 154; Analyst, 1924, **49**, 105), and the blue value on the lines of the B.P. 1932 method. Other characteristics were determined by the usual standard methods.

The presence of mineral oil is readily detected by intense bright blue fluorescence under the quartz mercury vapour lamp. Sperm oil gives a greenish-blue fluorescence, but this is masked by the bright grass-green fluorescence of shark-liver oil or cod-liver oil unless more than about 50 per cent. of sperm oil is present.

When the presence of shark-liver oil was suspected, 100 ml. of the oil was distilled *in vacuo* (3 to 4 mm. mercury), and the distillate was collected up to about 260° C. The distillate was dissolved in cold dry acetone saturated with hydrogen chloride, and dry hydrogen chloride was passed into the solution (Heilbron, Kamm and Owens, *J. Chem. Soc.*, 1926, 1630). The presence of the characteristic crystals of squalene hydrochloride after the solution had been in the refrigerator overnight formed a useful qualitative test for shark-liver oils of the squalene-containing group (Tsujimoto, *J. Soc. Chem. Ind.*, 1932, **51**, 317T).

The results of the examinations are summarised in the accompanying table. The medicinal cod-liver oil (No. 16) and the two guaranteed cod-liver oils (Nos. 17 and 18) had the characteristics of good cod-liver oil. No. 8 had the characteristics of cod-liver oil, although the iodine value of the unsaponifiable matter

TABLE I

CHARACTERISTICS OF REPUTED COD-LIVER OILS

Oil No.	Sp.gr. (15·5°/15·5° C.)	n_D^{40}	Free fatty acids (as oleic acid) Per Cent.	Saponification value	Iodine value (Wijs)	Blue value	Unsaponifiable matter Per Cent.	Iodine value of unsaponifiable matter	Nature of unsaponifiable matter	Fluorescence of oil in ultra-violet light
1	0·920	1·4673	0·54	177	139	13·5	10·4	145	Viscid pale yellow oil	Bright grass-green
2	0·916	1·4687	0·14	149	118	36·8	18·3	70	Greenish fatty solid	" "
3	0·914	1·4688	0·28	149	119	34·5	19·6	70	" " "	" "
4	0·917	1·4689	0·42	153	121	27·2	18·4	65	" " "	Bright grass-green, trace blue-green
5	0·908	1·4751	0·48	98	102	(0·7?)	51·0	10	Mobile pale oil	Intense bright blue
6	0·924	1·4684	0·71	186	140	nil	1·77	65	Viscid oil with small crystals	Bright grass-green
7	0·933	1·4730	0·65	192	182	1·5	0·83	63	Viscid pale yellow oil	Livid blue-green
8	0·928	1·4714	0·48	187	162	14·8	1·11	72	Yellow semi-crystalline solid	Grass-green, trace blue-green
9	0·899	1·4696	0·42	104	97	nil	44·1	20	Mobile pale oil	Intense bright blue
10	0·918	1·4694	0·71	163	146	29·6	9·1	278	Greenish fatty solid	Bright grass-green
11	0·919	1·4680	0·48	175	144	13·6	9·75	170	Very viscid pale yellow oil	" "
12	0·925	1·4678	1·40	188	147	5·6	0·93	92	Yellowish solid	" "
13	0·928	1·4713	0·73	188	167	3·6	1·62	70	" oil	Blue-green
14	0·918	1·4683	0·22	163	130	22·0	11·6	93	Greenish fatty solid	Bright grass-green
15	0·914	1·4718	0·45	134	116	3·1	25·9	19	Mobile pale yellow oil	Intense bright blue
16	0·927	1·4708	0·48	186	162	14·4	1·37	—	Viscid yellow oil	Bright grass-green
17	0·927	1·4720	0·45	190	162	10·4	1·15	117	Yellow semi-crystalline solid	" "
18	0·926	1·4711	0·25	176	162	12·4	0·96	108	Yellow semi-crystalline solid	" "

was rather low and the fluorescence slightly different from that of good cod-liver oil. No. 12 was apparently a rather poor sample of cod-liver oil, and No. 13 had, in the main, the characteristics of cod-liver oil, but the low iodine value of the unsaponifiable matter and the blue-green fluorescence were unsatisfactory features.

Nos. 5, 9 and 15 contained varying proportions of mineral oil. Nos. 2, 3, 4 and 14 were apparently mixtures of sperm oil with shark-liver oil, and Nos. 3, 4 and 14 gave small but definite yields of squalene hydrochloride. Nos. 1, 10 and 11 were apparently shark-liver oils, and gave significant yields of squalene hydrochloride (No. 11 gave a large yield). No. 6 gave Hoppenstedt's colour reaction for menhaden oil (*J. Soc. Chem. Ind.*, 1911, **30**, 36T) very strongly, none of the other oils responding to this test. No. 7 was not identified, but it does not appear to be cod-liver oil.

The results of the examination suggest that a fairly varied assortment of marine oils is still being offered for sale as cod-liver oils for stock-feeding.

CHEMICAL RESEARCH DIVISION
MINISTRY OF AGRICULTURE FOR NORTHERN IRELAND
BELFAST

The Use of Selenium in the Determination of Nitrogen in Potato Tubers

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(*Read at the Meeting of the Scottish Section, April* 16, 1937)

SINCE Lauro[1] drew attention to the value of selenium as a catalyst in the Kjeldahl digestion of cereal products, several investigations have been carried out to compare selenium, alone or mixed with compounds of mercury or copper, with the catalysts commonly used. In most instances the results have shown that with selenium a much shorter time suffices to obtain maximum figures for the nitrogen in various organic substances. Thus, Ashton[2] found that selenium was more efficient, in the analysis of grass, than copper sulphate in curtailing the time of heating by about 90 minutes, whilst Beet and Furzey,[3] using a mixture of selenium and mercuric sulphate, obtained, in the examination of various foodstuffs, maximum nitrogen results after 16 to 23 minutes' heating, or about one third of the time required by the method recommended in the Fertilisers and Feeding Stuffs Regulations.

There is, however, a considerable lack of agreement among the observations and conclusions of different investigators as to experimental details. It is generally accepted that digestion is not necessarily complete when the liquid has become clear, and that a further period of heating ("after-boil") is essential to complete the reaction. It is also obvious that the time of heating required depends on the composition of the catalyst mixture. Illarionow and Ssolowjewa[4] have examined the mechanism of the reaction, and point out that the selenium is oxidised to

selenious acid, which is the actual catalyst concerned, and that, although the catalytic effect is proportional to the amount of catalyst present when the amount is small, excess of selenium or prolonged heating leads to a loss of nitrogen, probably as a result of the decomposition of ammonium selenite with liberation of free nitrogen. In short, it would seem to be impossible to define the best conditions for the decomposition of a substance without a study of the reaction in question.

In the course of an investigation of the nitrogen in potato tubers, which do not appear to have been included in other investigations on foodstuffs, it was felt that a considerable saving of time in the digestion might result from using selenium instead of copper sulphate, and the following data were collected in the preliminary study of methods. A number of tubers, variety Majestic, were dried at 90° C. and ground up to supply material for examination, 1 g. being used for each determination. In order to determine the effect of "after-boil" on the results, a mixture containing 9·7 g. of potassium sulphate and 0·3 g. of selenium was added, together with 25 ml. of sulphuric acid. The heating was carried out in 500-ml. flasks over a series of Bunsen burners, and the arrangement of flasks and Bunsens was quite a random one. After digestion, the liquid was transferred to a distilling flask and made alkaline with caustic soda, and the ammonia was distilled into standard acid. The average figures from quadruplicate determinations are given in Table I.

TABLE I

EFFECT OF PERIOD OF "AFTER-BOIL" ON THE PERCENTAGE OF NITROGEN

Time of "after-boil" in min.	0	10	30	60	120
Mean per cent. of nitrogen ..	1·008	1·100	1·110	1·108	1·113
Standard error	0·005	0·008	0·007	0·008	0·012

The results agree with those obtained by Beet and Furzey, and it was decided that heating for 20 minutes after the liquid became clear was adequate in this instance, which meant that the digestion was complete in about 30 minutes. When 0·3 g. of copper sulphate was used instead of selenium, the liquid became clear after 35 to 40 minutes, and a subsequent heating of about 30 minutes was required to get maximum figures for nitrogen.

TABLE II

EFFECT OF DIFFERENT AMOUNTS OF SELENIUM ON PERCENTAGE OF NITROGEN

Weight of selenium in g. ..	0·15	0·30	0·45	0·70	1·00
Mean per cent. of nitrogen ..	1·048	1·100	1·023	0·950	0·950
Standard error	0·011	0·008	0·013	0·016	0·011

The effect of varying the amount of selenium in 10 g. of the catalyst mixture was also examined, the time of heating in each instance being 20 minutes after the solution had cleared. The results (Table II) indicate that the amount of selenium used is of considerable importance, and that, under the experimental conditions described, 0·3 g. is the most suitable quantity to take. This amount is of the same order as the amounts used by Beet and Furzey and by Ashton, but is nearly four times as much as that recommended by Illarionow and Ssolowjewa. The

last-mentioned chemists, however, worked with a simple organic compound, aniline sulphate, and used only 5 g. of potassium sulphate in each digestion.

The low results with the larger quantities of selenium, recorded in Table II, confirm those of other observers who found that large doses of the catalyst had this effect.

A further series of determinations, made on dried material and directly on fresh material from a single tuber, gave average results of 1·47 and 1·51 per cent. of nitrogen respectively, both calculated on the dry material; it is not known whether the discrepancy was due to the preliminary preparation of the dried samples or to the dilution of the digestion mixture by the 80 per cent. of water in the fresh samples.

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The Rapid Determination of Copper in Mild Steel

By T. P. HOAR, M.A., Ph.D., B.Sc.

The small amounts of copper found in mild steel may be rapidly and accurately determined colorimetrically by means of sodium diethyldithiocarbamate. The steel is taken up in dilute sulphuric acid, which leaves a residue containing almost all the copper, as noted by Reinhardt,[1] Koch[2] and Zinberg[3]; a little iron also remains undissolved, probably as carbide. The trace of dissolved copper is precipitated by addition of copper-free zinc, as suggested by Mohr[4] and others; Price[5] and Koepping[6] prefer aluminium for this purpose. The whole residue is then taken up in nitric acid. After suitable dilution, the copper is determined colorimetrically in the presence of the small remaining amounts of iron and zinc by the modified sodium diethyldithiocarbamate method previously described.[7]

The advantages of the method are:

(1) The main separation is effected by the initial process of solution; the precipitation of the trace of dissolved copper by zinc rather than by hydrogen sulphide avoids loss of copper (due to oxidation or peptisation of sulphide) during filtration.

(2) The colorimetric method using sodium diethyldithiocarbamate is at least as accurate as the electrolytic for such small amounts of copper, unless very special apparatus is available, and is very much quicker. It is much better than the iodimetric method, which requires either the removal of the small amount of iron still present as hydroxide, or the addition of fluoride, neither of which procedures is very suitable when very small amounts of copper are involved.

REAGENTS REQUIRED.—(i) Sulphuric acid: about 250 g. per litre (1 vol. of conc. sulphuric acid to 7 vols. of water); (ii) copper-free zinc (thin foil or filings); (iii) conc. nitric acid and the reagents listed in the preceding paper.[7]

PROCEDURE.—Dissolve 5 g. of steel in 80 ml. of sulphuric acid (250 g. per litre) in a 150-ml. beaker on the hot-plate until evolution of gas ceases (about 40 minutes). Dilute to 150 ml. and add 1 g. of copper-free zinc, stirring until it has almost all dissolved (5 minutes). Filter through a Gooch crucible or coarse glass frit fitted with one layer of filter-paper only (for rapidity, to avoid oxidation), wash the residue once with cold water and return it, with the filter-paper, to the beaker. Add 3 ml. of conc. nitric acid and return the beaker to the hot-plate. When the particles *in* the small filter-paper have dissolved (5 minutes), remove the paper, washing it well. Heat until the solution has a clear brown colour; a small brownish residue (probably SiO_2 and SnO_2) may be neglected. Add 0·5 g. of urea and boil for 2 minutes, to remove nitrous acid. Cool and make up to 200 ml. Take two 10-ml. or other convenient aliquot portions, and determine the copper by the citrate method of the preceding paper.[7]

Use half quantities if desired, making the solution up to 100 ml. Make a blank test to determine any traces of copper in the acids or the zinc. No filtration is necessary; add the nitric acid after the zinc has dissolved in the sulphuric acid. Add a considerable amount of ammonia or some sodium hydroxide to keep the zinc in solution.

Where the greatest accuracy is not essential, but rapidity is desired, omit the addition of zinc, and determine the copper by the pyrophosphate method of the preceding paper.[7]

TEST OF THE METHOD.—It was necessary to show that the residue after the zinc treatment contains all the copper originally present in the steel. The middle part of a sheet of dead-mild steel of the tinplate type was carefully cleaned with emery-paper and cut into small pieces, about 0·5 by 0·5 cm., which were thoroughly mixed. Copper was determined in 5-g. samples of this uniform material by the method as described. Precipitation by hydrogen sulphide or sodium thiosulphate, and determinations without precipitation, were also tried. In each instance the copper in the filtrate was determined by the method of Haddock and Evers,[8] the large excess of ferrous iron being oxidised with nitric acid and fixed with the appropriate excess of citric acid. The results are shown in Table I.

TABLE I

Steel taken g.	Precipitation method	Copper found in residue mg.	Copper found in filtrate mg.	Copper found (residue only) Per Cent.	Copper found (total) Per Cent.
5·02	Zinc	5·4	0·00	0·107	0·107
5·00	"	5·3	0·04	0·106	0·107
5·00	"	5·3	0·00	0·106	0·106
5·02	H_2S	5·2	0·06	0·104	0·105
5·00	"	$5·2_5$	0·14	0·105	0·108
5·01	Thiosulphate	5·2	0·16	0·104	0·107
5·03	"	5·2	0·09	0·103	0·105
5·03	None	$5·2_5$	0·13	0·104	0·107
5·02	"	5·2	0·09	0·104	0·105

Clearly the zinc precipitation is effective, whilst the sulphide precipitations offer no advantage over the omission of the precipitation altogether. Results obtained without the zinc precipitation may be expected to be systematically about 2 to 3 per cent. low.

The small amount of zinc which may be present if the liquid is filtered before the added zinc has entirely dissolved has no ill effect; any turbidity produced can be eliminated by making the solution more strongly ammoniacal.

To confirm the conclusion previously reached,[7] that the nitric acid used to take up the copper has no influence on the copper determination, several analyses of the steel referred to above were made, with and without the removal of excess nitric acid, by "fuming" with sulphuric acid, by the method previously described[7] without the zinc addition. The results were as follows (Table II):

TABLE II

Procedure ..	.. Nitric acid removed				Nitric acid not removed				
Copper found, per cent.	0·103	0·104	0·104	0·103	0·103	0·104	0·102	0·104	0·104

Evidently the nitric acid has no influence.

The only other ion likely to be present is a trace of arsenate derived from arsenic in the steel. As noted in the preceding paper,[7] this does not interfere.

This work, and that of the preceding paper, has been carried out for the International Tin Research and Development Council in connection with investigations of the steel-base of tinplate.

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Notes

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

THE DETERMINATION OF TOTAL ALKALOIDS IN COCOA, AND OF COCOA-MATTER IN FLOUR CONFECTIONERY

THE useful method of determining the total alkaloids, published by Moir and Hinks (ANALYST, 1935, **60**, 439), is tedious, owing to the low solubility of theobromine in chloroform. As stated, ten to twelve extractions of the final solution by chloroform may be necessary to ensure complete removal of the alkaloids, otherwise the results are uncertain.

In an endeavour to shorten and improve this part of the process, the use of several other solvents was tried, and one of them—chloroform and phenol, as recommended by Autenrieth (*Detection of Poisons*, 6th Ed., p. 624)—was found to give the best results. Chloroform and alcohol, as recommended by Nicholls (ANALYST, 1922, **47**, 506), for the extraction of morphia was also tried, but the results were not so satisfactory.

The following table gives the results, in grams, obtained by the use of various solvents. An aqueous solution containing 0·025 g. of theobromine was extracted four times with 40 ml., 30 ml., 20 ml. and 10 ml. respectively of the solvent:

	Chloroform plus phenol				Chloroform plus alcohol (50/50)
Chloroform	1%	2%	5%	10%	
g.	g.	g.	g.	g.	g.
0·008	0·012	0·015	0·025	0·025	0·015

A 5 per cent. solution of phenol in chloroform was afterwards adopted as the best solvent for the purpose.

The final solution of alkaloids obtained from a sample of chocolate cake by Moir and Hinks's method yielded 0·013 g. of mixed alkaloids to chloroform after 12 extractions, and 0·018 g. to a 5 per cent. solution of phenol in chloroform after 4 extractions, as previously detailed. The extracts may contain a trace of sugar, and their nitrogen-content should be determined.

It was found by experiment that 100 ml. of a solution of 5 per cent. of phenol in chloroform would, in 24 hours at ordinary temperature, take up 0·23 g. of pure theobromine, whilst 100 ml. of chloroform would take up 0·03 g., and 100 ml. of equal parts of chloroform and alcohol 0·11 g.

When a mixture of phenol and chloroform is distilled on a water-bath, the phenol is left behind; it can be removed conveniently by holding the flask in the fingers and rotating it over a small flame while sucking a gentle current of air through it with a filter-pump.

An alternative procedure recommended is the extraction of the final solution with chloroform in a Pregl type of extractor (Autenrieth, p. 467). In 9 hours, 0·024 g. of theobromine was extracted from a solution containing 0·025 g.

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IODINE VALUE OF SHELLAC

AN important investigation into the various factors affecting the iodine value of shellac, as determined by the Wijs–Langmuir method, was made by a Sub-Committee on Shellac Analysis of the Committee on Uniformity in Technical Analysis (*J. Amer. Chem. Soc.*, 1907, **29**, 1221). One of the most significant points established was the comparatively large effect of variation in the concentration of the acetic acid used in the test. This is shown by the results in the following table:

Strength of acetic acid Per Cent. (approx.)	Melting-point of acid	Iodine value
97	11·8° C.	14·67
99	14·95° C.	17·59
100	16·4° C.	20·48

These results have been confirmed in general by a number of research laboratories, including those of Messrs. Angelo Bros., Calcutta, the Indian Lac Research Institute, and the London Shellac Research Bureau.

Unfortunately, the details of the Wijs method for the determination of the iodine value of oils and resins given in various publications in this country (*e.g. Analysis of Resins*, by K. Dieterich, and *Oil and Colour Chemists' Handbook*) specify *glacial* acetic acid, without qualification as to strength and, thus, such specifications are unsuitable for shellac analysis. A specification (*A.S.T.M.*, 1930, p. 299), which has been found to give every satisfaction, aims at overcoming this difficulty by prescribing "glacial acetic acid, 99 per cent., having a melting point of 14·8° Centigrade and free from reducing impurities. If these requirements are not complied with, the result of the iodine number determination will be erratic."

In view of the fact that certain consumers of shellac have obtained high iodine values by using *glacial* acetic acid without dilution, it was felt advisable to draw attention to the earlier work on this test.

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Official Appointments

THE Minister of Health has approved the following appointments:

SAMUEL RUSSELL TROTMAN as a Public Analyst for the County Borough of Nottingham, in addition to William Wilder Taylor (May 28th, 1937).

Note:—Mr. S. R. Trotman retired on December 31st, 1936, on the appointment of Mr. W. W. Taylor as Public Analyst. The appointment of an Additional Public Analyst has become necessary owing to Mr. Taylor being away from business on account of illness.

HAROLD EDWARD MONK as a Public Analyst for Borough of Kidderminster, in place of C. C. Duncan, retired (June 10th, 1937).

RHYS PENDRILL CHARLES as a Public Analyst for the Borough of Penzance, in place of W. Partridge (deceased) and in addition to C. G. Moor (June 16th, 1937).

FREDERICK GRANT DUNCAN CHALMERS as a Public Analyst for the County Borough of West Bromwich, from July 1st, 1937, in place of Harry Silvester, resigned June 30th, 1937 (September 10th, 1937).

Notes from the Reports of Public Analysts

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

METROPOLITAN BOROUGH OF STEPNEY

ANNUAL REPORT OF THE BOROUGH ANALYST

OF the 1575 samples of food and drugs examined, 1025 were taken formally and 550 informally.

DETERMINATION OF HORTVET FIGURE BY ASSISTANT.—In one case objections were raised by the defence because the determination of the freezing-point had not been made by the Public Analyst himself. This involved the appearance of both my assistant and myself to give evidence. In the case of *Bakewell* v. *Davis*, 1894, in which the analysis had been done by an assistant under the supervision of the Public Analyst, the judges were of opinion that the Public Analyst had analysed the sample within the meaning of the Act (*cf.* ANALYST, 1881, **6**, 152; 1888, **13**, 143; 1930, **55**, 39).

SODA WATER.—Three samples of soda water were analysed. Two of these contained 7·3 and 8·1 grains of sodium bicarbonate per pint respectively; the remaining sample consisted of aerated tap water. There is no definite standard for sodium bicarbonate in soda water. It is generally admitted that a reasonable standard is 10 grains of sodium bicarbonate per pint of soda water, and many mineral water manufacturers conform fairly closely to that standard, the sodium bicarbonate varying from 5 to 10 grains per pint. The soda-free article is usually cheap and probably satisfies many purchasers, but it should be labelled "Aerated water," or the absence of sodium bicarbonate should be declared.

DOUGLAS HENVILLE

Department of Scientific and Industrial Research

REPORT OF THE BUILDING RESEARCH BOARD FOR THE YEAR 1936*

THE Annual Report of the Building Research Board for 1936 contains a survey by Dr. R. E. Stradling of the work of the Building Research Station since its establishment eleven years ago. It is mentioned *inter alia* that one investigation is likely to lead to specification testing of cement being placed upon a new footing, and that in another, methods have been evolved by which the variations in concrete, as produced on a job, can be reduced. Chemical studies have led to the discovery of substances from British sources which render concrete more resistant to chemical attack. The work has also resulted in new industries being started. For instance, the production of bricks by a steam-curing process from spent oil shale and lime has been investigated, and the commercial production of this type of brick has been started in Scotland. Commercial production of materials for making good light-weight concrete from blast-furnace slags has also been begun.

Studies have been made of the properties of building stones and the causes of their weathering and decay, and attention has also been devoted to the properties of bricks.

* H.M. Stationery Office, Adastral House, Kingsway, W.C.2. Price 4s. net.

It is now possible to examine samples of clay and indicate the best firing temperature for bricks made from it. It is also possible to estimate the risks involved if this temperature is not obtained. The importance of particular firing temperatures in eliminating the salts which by crystallising out may cause unsightly stains or even the disintegration of bricks has been clearly shown.

The work on plaster mortars and rendering has shown that the weaker porous mortars are more efficient in preventing damp penetrating into walls than the more impermeable cement renderings, since the shrinking of the latter causes cracks to form.

CLEANING BUILDINGS.—The work of the past year has shown that plain water is the most efficient cleaning agent. It has been found that by directing a fine spray of water on to the surface for a period depending on the nature and condition of the stone, the soot incrustations become so thoroughly softened that they can often be brushed off with a soft paint brush. A sample of Cotswold limestone, for instance, similar to stone in a building which had been cleaned, though not without difficulty, by the use of caustic soda, was completely cleaned in this way after spraying for no more than half-an-hour. Weathered Caen stone was easily cleaned, without the least damage to the tool marks, after spraying for one hour. A dirty sample of Portland stone required spraying for two hours before all the dirt could be removed with ease, but the stone could have been cleaned at an earlier stage by using stiffer brushes.

Laboratory experiments have also been carried out to imitate the effect of the sulphur pollution of the atmosphere on building stones. In the first series of tests, samples were placed in a vessel and the air was removed from the pores by means of a vacuum-pump. Sulphur dioxide was then admitted until atmospheric pressure was restored, and finally water was allowed to enter the vessel until the stones were covered. After standing for 5 hours the specimens were removed and dried in an oven. This cycle of operations was repeated a number of times.

On sandstones the effect of this treatment was to produce a type of flaking similar to that which occurs in buildings. Skin-formation, very similar to that observed at the original surface of the weathered sandstone, was produced after 13 cycles. After 18 cycles the skin flaked off and exposed the soft powdery surface underneath.

Similar experiments on samples of slate have indicated that it is possible to reproduce lamination in a slate of poor quality.

COLOURINGS FOR STUCCO.—At the present time there is an increasing demand for coloured stuccos and renderings. For the colouring of buff, yellow, green and red stuccos there exists a range of mineral pigments which are fast, both to light and lime, and can be used successfully. The production of a blue stucco has always been difficult. Ultramarine is not stable in contact with lime, although it can be used successfully in renderings, etc., exposed to the air. For use under water (as in swimming baths), where a blue rendering is often desired, it is not suitable. The use of crushed blue glass provides one method for obtaining a blue stucco, but does not fulfil all the requirements.

A method of colouring stuccos, which is of quite a novel character, has recently been tested at the Station. It consists in the use of sand, treated with a metallic oxide to produce a coloured skin, which is stated to be an integral part of the sand grains. The resistance of the coloured sand to alkalis and acids is such that it should be unaffected by the lime and alkalis in cement or by polluted town atmospheres, and there was no indication that the mechanical properties of cement with which it is used would be adversely affected.

Other subjects discussed in the Report are the structure and strength of materials, and the prevention of noise in buildings.

REPORT OF THE FOOD INVESTIGATION BOARD FOR THE YEAR 1936*

THE Report for 1936 contains the general summary of work and an account of the researches in progress by the Chairman, Sir Frank Smith. This is followed by the Report of the Director of Food Investigation (Mr. E. Barnard), in which is recorded the latest progress in those investigations which have not yet reached the stage at which full publication is feasible.

EXPEDITION TO THE DOMINIONS.—Steps are being taken to secure data on the performance of modern methods of gas-storage (*i.e.* in an atmosphere containing about 10 per cent. of carbon dioxide), and in co-operation with the Councils for Scientific and Industrial Research of Australia and New Zealand, an expedition is leaving for New Zealand and Australia in January, 1937. A large programme of work has been planned, embracing the study of conditions in spaces carrying frozen meat, chilled beef in gas-storage, cheese and fruit. The equipment to be carried by the expedition is considerable, and includes some 250 distant-reading electrical thermometers of special construction.

GAS-STORAGE OF PEARS.—The results of the investigations carried out at the Ditton Laboratory during the past few years with the Conference and William's Bon Chretien varieties indicate that the pear responds even more favourably than the apple to gas-storage. These two varieties, at least, can be gas-stored most successfully for several months. After removal from gas-storage the fruit ripens more slowly, and therefore allows more time for marketing, than fruit that has not been stored in this way.

STORAGE OF PLUMS.—The problem of successfully storing Victoria plums for a short period is also one of considerable importance to the grower and distributor. A comprehensive series of experiments, started in the past season, has shown that, at 32° to 34° F., a life of three to four weeks may be expected. Higher temperatures accelerate an abnormal softening of the fruit, and are only suitable for storage for a few days. Close attention must be paid to the degree of ripeness at picking if good quality is to be attained during ripening after storage. If the fruit is picked too green and hard, the flesh becomes soft and jellied at an early stage, especially at 40° F., and although a temperature of 32° to 34° F. holds the change in check to some extent, really good quality cannot be attained. Fruit that was sufficiently mature when picked ripens well after cold-storage at any temperature from 50° F. upwards; if the fruit was immature when picked, a great improvement in colour, and some improvement in quality, can be secured by ripening it at 70° F.

STORAGE OF GRAPES.—A special method that allows water to be supplied to the bunches during storage has been successfully applied in Belgium to the commercial cold-storage of hot-house grapes. Trials of this technique in the laboratory have given promising results with English Muscat grapes, and it is hoped that they may soon be extended on the commercial scale.

RELATIONSHIP BETWEEN PRODUCTION AND STORAGE RESEARCH.—The Board comments on the relationship between the problems of production and those of transport and storage and other post-production processes. Where the agricultural product is to be stored or to receive other special treatment, production and research on production must have that end in view. For instance, the storage of fruit and eggs and the manufacture of bacon from pork clearly introduce new considerations into production that may have an important effect on the course which production takes. Hence, the producers and those responsible for research on production require a specification towards which to work, and the preparation of that specification becomes a task of the Board.

In collaboration with the research institutions represented on the Pig Husbandry Committee of the Ministry of Agriculture and Fisheries, experiments are being carried out on the effect of the breed, growth and feeding of the pig on the quality of the carcase. In addition, investigations on a large scale are being

* H.M. Stationery Office, Adastral House, Kingsway, London, W.C.2. Price 3s. 6d. net.

carried out for the Bacon Development Board, on the effect of transport of the pig on the weight of the carcase, and for the Pigs Marketing Board of Northern Ireland, on the differences between the carcases of pigs killed on the farm and in the factory; both Boards are making substantial payments towards the cost of the work. In the course of the year over 800 carcases have been examined.

STORAGE OF EGGS AND POULTRY.—Among its other work on the storage of eggs, the quality of eggs stored at 0° C. in atmospheres containing different concentrations of carbon dioxide is being investigated by handling, by tests for palatability, and by the usual chemical and physical tests for the white and yolk. The results so far confirm the view that an atmosphere containing 2·5 per cent. of carbon dioxide improves the quality of the egg, notably in the yolk. Atmospheres containing high concentrations of carbon dioxide, in the range 60 to 100 per cent., affect the quality of eggs uniformly, the yolks being firmer and the whites markedly more liquid than in eggs stored in air. Eggs have been stored in saturated atmospheres within this range for eight months without spoilage by mould or bacteria.

It has been known for some years that eggs retain their fertility longest if they are stored at a temperature of 10° to 12° C. Experiments during the past year have indicated that, at 10° C., the period of fertility can be still further prolonged if the atmosphere contains 2·5 to 5 per cent. of carbon dioxide.

An interesting small trial is recorded in the report on the storage of frozen poultry packed in aluminium foil covered on both sides with waxed paper. Some Sussex cockerels were stored for five months at −10° C. in this way. The birds were thawed, cooked and eaten by several people, who without exception found them excellent in every way, and indistinguishable from freshly-cooked chickens.

STORAGE OF VEGETABLES.—An investigation of the conditions in clamps of potatoes has revealed considerable variation in temperature, sugar-content and sprouting, both between different points in a single clamp and between clamps of different construction. The evidence suggests that improved construction of the clamps may result in better storage of potatoes intended for the manufacture of crisps, and possibly also in improved quality in potatoes held for general domestic use.

Trials of the storage of broccoli have been made with two varieties. A temperature of 32° F. proved best, since higher temperatures reduced the life of the vegetable by accelerating the growth of moulds and yellowing of the leaves. A high humidity is necessary to prevent excessive wilting of the leaves, which would be detrimental to the appearance of broccoli in marketing. At 32° F., and with air of approximately 96 per cent. relative humidity freely circulating in the store, the broccoli kept for three weeks, with three to four days subsequently available for marketing before deterioration set in.

STORAGE OF FISH.—The Report states that herring cured with much less salt than is now employed keep well for six months at −3° C.—a temperature usual in the storage of salt-cured herring. They are very palatable, if a fatty fish is used, but do not develop the full flavour to which the trade is at present accustomed. A certain degree of preliminary ripening at ordinary temperature before the fish is put into cold-storage has been found to improve the flavour, and to bring it nearer to the normal.

Experimental evidence indicates that the rancidity that develops in herring's fat during cold-storage is due to certain enzymes. These are activated by common salt, and this explains the rapidity with which herrings that have been initially frozen in cold brine develop a rancid flavour during storage, particularly if left unglazed. Delay prior to storage has been found to be detrimental; under such conditions a stale flavour, rather than one of rancidity, develops after a few months' storage. It was found, however, that if quite fresh herring were brine-frozen and subsequently well washed, they showed only a slight indication of oxidation of the fat after six months' storage in boxes at −28° C.

Smoke-cured fish (*e.g.* kippers, finnans, fillets of white fish) are usually kept for some months in cold-store at temperatures ranging from −8° to −14° C., with a view to export. Tests at −20° and −28° C. have shown that the quality of the fish is improved by lowering the temperature. This improvement is the result of retarding deterioration, *e.g.* superficial drying, rancidity, development of cold-storage odour and flavour, loss of smoky odour and flavour and colloidal breakdown of the flesh.

Honey Producers' Association

POLLEN ANALYSIS

A MEETING of an informal character, held on May 5th, took the form of a discussion between the members present as to the value of pollen analysis, and in particular of the methods employed by Miss A. Betts and the Rev. Yates Allen, who make analyses for the Association.

Mr. Lindley, the Secretary of the Association, pointed out that admixture of foreign honey with English honey (the mixture being offered for sale as "English Honey") was of common occurrence during periods, such as 1936, when the crops of English honey were small; hence the desirability of the development of a means of detecting such admixture. The method of examination employed by the Association involved the separation of the pollen grains and the examination of their microscopical features in comparison with those of pollens of known origin, the standards consisting either of photomicrographs or of permanent mounts. It was suggested that the adoption of "pollen analysis" by Public Analysts would check the contravention of the Labelling Regulations. The following outline of the method was given:

METHOD OF EXAMINATION.—The honey is diluted with twice its volume of cold or tepid water, and the solution is placed in a tube drawn out at the bottom (such as a milk-sediment tube), and allowed to stand overnight. The pollen grains are removed from the bottom of the tube by means of a pipette and dropped on to microscope slides. The preparation is dried and mounted in either Canada balsam or liquid separated from crystallised honey.

Some doubt was expressed with regard to the efficiency of the sedimentation, as it was pointed out that some workers skim the pollen grains from the top surface of the solution; separation by centrifugal means was regarded as inferior to the sedimentation method.

Miss Betts suggested that the grains should be stained on the microscope stage with concentrated sulphuric acid. The colours acquired by the pollen were an aid to the recognition of the class of plant, if not of the actual plant.

The Rev. Yates Allen said that pollen analysis would enable deductions to be drawn; thus, for example, the recognition of characteristic grains would enable the time of year of the collection of the honey to be decided, and the presence of pollen derived from plants not in flower at that time of year would suggest admixture; this, of course, was only of value for unblended honey.

The general conclusion arrived at was that pollen analysis was of a rather indefinite character, but would be of value if grains characteristic of flowers not found in the stated country of origin were found; for example, grains characteristic of eucalyptus or ti-tree would suggest the presence of Australian or New Zealand honey respectively. Examination of pollen would be of no value in examining a honey labelled "Empire," as the possible sources of origin would embrace an extremely large field.

The examination would require considerable time and skill, as the number of standard pollens was great, and differences of opinion on the origin or presence of a particular pollen would, and did, occur.

Federated Malay States

ANNUAL REPORT OF THE INSTITUTE OF MEDICAL RESEARCH FOR THE YEAR 1936

THE Chemical Division of the Institute for Medical Research (until recently under the direction of the late Mr. R. W. Blair) does work for the Medical and Health services, and deals with exhibits from the Police and samples from other Government departments.

The total number of samples examined was 6564, of which 28 were samples of food submitted under the Sale of Food and Drugs Enactment, 1932.

SIGNATURES ON DOCUMENTS.—Of the 749 exhibits sent by the Police, a large number consisted of documents relating to 21 cases of suspected forgery, anonymous letter-writing, etc. The signatures on several land receipts were photographed under glass plates ruled in small squares. This immediately revealed the unnatural degree of coincidence among the various signatures.

ALGAL CONTROL OF WATER.—The cupri-chloramine treatment, *viz.* the addition of copper sulphate, ammonia and chlorine, has been tried at Gopeng and at the Impounding Reservoir, Kuala Lumpur, the final products being conveyed to *open* service reservoirs before passing into supply. In each instance the treatment caused a marked decrease in the algal growth.

TOXICOLOGICAL EXAMINATIONS.—Exhibits relating to 68 cases of poisoning were examined. No poisons were detected in 33 cases. The poisons found included sodium hydroxide in 11 cases, arsenic in 9, and mercuric chloride and morphine in 5 each. Unusual poisons detected were evipan, saponin and margosa oil (1 case each).

NATURE OF THE POWDER IN SHOOTING CASES.—A large number of cartridge cases for shot-guns, rifles and revolvers are now loaded with the so-called "smokeless" ammunition. When this type of cartridge is fired from a gun, it is impossible to say by an examination of the products of combustion found in the barrel how recently the gun was fired. It is frequently stated that the products of combustion of black powder are alkaline and that the products of combustion of smokeless powder are neutral or acid, but it must be remembered that a number of ammunition manufacturers now mix an alkaline substance with their smokeless powders; therefore the mere fact of finding alkaline products of decomposition is no longer an indication of the use of black powder.

CAROTENE-CONTENT OF MALAYAN PALM OIL.—An investigation to determine the variation, if any, in the carotene-content of samples of palm oil, obtained from fruit at different stages of maturity, was carried out by Dr. I. A. Simpson for the Department of Agriculture. The results show that oil of low acidity from fully ripened fruit appears to be richest in this pigment. Attempts to isolate carotene from palm oil have been made, and a satisfactory method has been devised (see *Bull. Institute for Medical Research*, F.M.S., No. 1, 1936).

Western Australia

ANNUAL REPORT OF THE CHEMICAL BRANCH, MINES DEPARTMENT, FOR THE YEAR 1936

THE Chemical Branch of the Mines Department is in control of the Government Laboratory for all the Government departments of Western Australia. The Laboratory is under the direction of Dr. E. S. Simpson.

HORTVET STANDARD.—The bovine milk supply is checked by the staff of the Co-operative Health Board's Laboratory and the Metropolitan Milk Board. In consequence, it is usually only referee or other special samples that are submitted to the Government Laboratory, which possesses the only Hortvet cryoscope in the State. The normal freezing-point for milk has been fixed by a regulation under the Health Act at −0·55° C.

EXCESS OF ALKALI IN TRIPE.—Proceedings were taken by the Department of Public Health for excess alkali in a number of samples of tripe (*p*H greater than 8·0). It was contended in one case by the defence that the moist tripe sample could have derived its alkalinity from the grease-proof paper in which it was wrapped by the inspector prior to being delivered to the analyst. The magistrate declined to record a conviction, although an officer of this branch showed in evidence that grease-proof paper similar to, and from the same supply as that which had been used, was slightly acid, which is the normal condition of most, if not all, of the grease-proof papers on the market.

WHEAT FLOUR IN JAM.—An unusual case of adulteration met with was the addition of starch, in the form of wheat flour, to jam. This was found in the jam of five different varieties made by one firm, evidently having been added for the purpose of thickening.

SULPHITE IN SAUSAGES.—In a case that was referred to the Government Laboratory the question was raised whether sausages containing 9 or 10 grains of sulphur dioxide (as sulphite) per lb. could lose this by decomposition during a period of 40 days in cold-storage. Since the authorities in the literature are divided on this point, experiments were made to determine it. Samples of sausages and sausage meat containing known amounts of a commercial sodium sulphite preservative and of potassium metabisulphite were wrapped in grease-proof paper and then in brown paper bags, sealed and stored for 40 days at about 30° F. The following results (grains per lb.) were obtained:

Preservative added	SO_2 added	SO_2 found immediately	SO_2 after 40 days' storage
Expt. 1—Sausages. Pot. metabisulphite..	9·0	7·38	6·15
" " ..	3·5	3·23	1·68
Expt. 2—Sausage meat " ..	9·0	7·83	7·06
Expt. 3—Sausages. Sodium sulphite ..	10·5	9·54	10·42
" " " ..	4·0	3·47	3·35

It was concluded that sausages and sausage meat stored for 40 days under the conditions described, retain the greater part of their original sulphur dioxide-content.

QUACK REMEDIES FOR CANCER.—Seven samples of quack remedies, consisting of powders and tablets given by an unauthorised practitioner to a person suffering from cancer, were examined for the Department of Public Health. They consisted mainly of powdered buchu leaves, some with and some without jalap or cascara sagrada. Judging by previous samples from the same source, the prescriber uses these materials as a sort of universal remedy.

"GINGIN DISEASE" OF SHEEP.—An investigation was made to ascertain the cause of "gingin disease" (enzootic ataxia) of sheep. As certain of the pathological symptoms pointed to the possibility of lead being the toxic agent, some work was done on the spectrographic determination of lead, but it was early recognised that the colorimetric dithizone method was more satisfactory. It was shown by this method that specimens from affected lambs and sheep did not contain appreciable quantities of lead in excess of the controls. Livers showed nil to 0·8 parts of lead per million (control *nil*), ribs 4 to 8 (control 2) brain nil to 0·6 (control *nil*). Certain evidence from spectroscopic examination of viscera and the ammonium chloride used in feeding experiments points to a deficiency of copper, and future work will be directed towards elucidating this point.

Spectrographic work to confirm the incidence of cobalt as the responsible factor in the Denmark wasting disease is at present being undertaken.

FRUIT OF THE "QUININE TREE."—A preliminary examination has been made of the fruit of the so-called quinine tree (*Petalostigma quadriloculare*), which is found in Kimberley, north of Wyndham. The fruits, which are orange-yellow when ripe and about one inch in diameter, are extremely bitter, and are said to be a useful medicine and vermifuge for horses. No quinine or other alkaloid was found in the fruits, the bitter taste being due to the presence of a glucoside or other bitter principle.

Physico-Chemical Symbols

REPORT OF A JOINT COMMITTEE OF THE CHEMICAL SOCIETY, THE FARADAY SOCIETY AND THE PHYSICAL SOCIETY

THE Chemical Society, having decided to revise the List of Physico-Chemical Symbols recommended for use in its Journal, invited the co-operation of the Physical Society and the Faraday Society in setting up a Joint Committee to correlate the views of physicists and chemists on symbols for "thermodynamical quantities" and to eliminate as far as possible the confusion which had arisen through the considerable diversity of usage in regard to such symbols.

The Councils of the three societies agreed to this proposal, and in March, 1936, a Joint Committee was set up consisting of: Mr. J. H. Awbery, Prof. F. G. Donnan, Prof. A. C. G. Egerton, Prof. A. Ferguson, Prof. G. I. Finch, Dr. C. F. Goodeve, Prof. C. N. Hinshelwood, Prof. J. R. Partington, Dr. H. J. T. Ellingham (*Hon. Secretary*) and Prof. E. K. Rideal (*Chairman*).

In June, 1936, the Joint Committee presented an Interim Report on the use of symbols for thermodynamical quantities, which, after slight amendment, was approved by the Councils of the three societies.

The Joint Committee was then invited to extend its work to symbols for other quantities of interest to both chemists and physicists, with a view to eliminating conflicts of usage and securing the greatest measure of agreement on the use of physico-chemical symbols.

The present Report to the Councils of the three societies embodies the agreed conclusions of the Joint Committee, including those already given in the Interim Report of June, 1936.

In forwarding this Report to the Councils of the Chemical Society, the Faraday Society and the Physical Society, the Joint Committee recommends:

(1) That the Report be referred also to the Royal Society and the British Standards Institution, so that the comments of these two bodies, together with those of the three co-operating societies, may be available before the Report is finally approved.

(2) That the Report as finally approved be utilised by the three co-operating societies as the basis of any lists of symbols and conventions which they may desire to issue as recommendations for practice in their publications.

In order to consolidate the present step towards uniformity among the three societies, and to facilitate future progress in this direction, it is suggested that:

(*a*) recommendations of the Joint Committee which are incorporated in the recommendations of one of the individual societies should be specially marked to indicate that they have been approved by both physicists and chemists;

(*b*) any other recommendations put forward by one of the individual societies should be such as will not conflict with any of the recommendations of the Joint Committee.

(3) That the Report as finally approved be printed (without this preamble), and copies sent for information to the principal societies and organisations in this country and abroad which are likely to be interested. This should help towards securing a greater measure of uniformity among a wider range of scientific and technical bodies in the future.

PART I

The objects of the Joint Committee have been:

(i) To correlate the views of chemists and physicists with regard to the use of symbols for quantities employed in thermodynamics, and to eliminate as far as possible the confusion that has arisen through the considerable diversity of usage in this field (discussed in Part II).

(ii) To deal similarly with symbols for other quantities which are of interest to both chemists and physicists (discussed in Part III).

The Joint Committee has examined the recommendations contained in the published reports of other bodies which have been concerned with symbols in recent years. In regard to symbols for thermodynamical quantities, particular attention was given to the Report on Symbols, Units and Nomenclature of the International Conference on Physics (1934); and in regard to symbols for physico-chemical quantities, to the old "List of Physico-Chemical Symbols" which the Chemical Society has decided to revise, and to a provisional draft of Standard Chemical Symbols and Abbreviations, drawn up recently by the British Standards Institution (largely on the basis of the old Chemical Society list), but held up pending the issue of the present Report.

Through the good offices of Mr. A. Sanford Moss, of the American Standards Association, the Interim Report of the Joint Committee was communicated informally to an "Informal International Conference on Letter Symbols for Heat and Thermodynamics," held in New York in September, 1936; and the Joint Committee has had access to an advance copy of a preliminary Report of this Conference. Consideration has also been given to a "Preliminary List of Abbreviations" recently circulated by the Royal Society.

The Joint Committee has given due consideration to usages adopted by authors of well-known text-books on relevant subjects; and to special lists of symbols in current use by workers in various fields, drawn up by individual members of the Committee.

In submitting this Report the Joint Committee wishes to draw attention to the fact that, although recommendations regarding the use of physico-chemical symbols have been put forward in the past by a number of Committees, these bodies have nearly always been representative of *either* physicists *or* chemists, and not as in the present instance of both.

In carrying out its work the Joint Committee has been guided by the following principles:

(*a*) Symbols and conventions regarding their use should be chosen from among those already widely adopted, unless there are definite objections to all current usages.

In choosing between possible alternative symbols and conventions preference should be given, in general, to the practice and needs of chemists and physicists, and to practice in English-speaking countries.

It is fully recognised that it would be highly advantageous to secure complete agreement with workers in fields other than chemistry and physics—especially with engineers in regard to thermodynamical symbols. It has to be realised, however, that this involves special difficulties, due not only to the strong feelings which workers in particular fields hold in favour of their accustomed symbols, but also to the limited number of alphabets and founts available for the representation of the large number of quantities to be referred to in the various branches of science and technology. Nevertheless, care has been taken to avoid as far as possible any direct conflict with established usage in related fields, and thus to leave open the way to a wider range of agreement in the future.

Where general agreement on preference for a particular symbol or convention is not reached, alternatives should be given; but one of such alternatives may be indicated as the first preference.

(*b*) Symbols for quantities constituting a well-defined class should, if possible, belong to the same alphabet, fount and case, which should be such as to permit of modification in accordance with a uniform scheme for the representation of any important series of corresponding derived quantities.

Methods of modifying symbols by means of subscripts, superscripts, etc., should be systematised as far as possible.

(*c*) There should be no departure from the standard practice of mathematicians regarding the use of signs, operator symbols, etc.

It is recognised that the detailed recommendations contained in Parts II and III of the Report will not cover all the requirements of workers in any particular field, but it is hoped that they will afford a common basis for practice in a wide range of specialised branches of science.

An important advance towards uniformity of usage will have been achieved if the quantities which appear in the lists below are represented whenever possible by the symbol recommended, or, when this is not possible, by an alternative symbol chosen in accordance with the principles set forth above. It is realised that owing to the limited number of available alphabets many of the symbols appearing in the following lists will have to be used in certain branches of science for quantities which are not referred to in this Report. There can be no objection to this provided that there is no danger of confusion with the quantities which the symbol represents in the lists (given in Parts II and III).

Part II deals with symbols for quantities employed in general thermodynamics, with conventions relating to their use. The lists of symbols recommended are also included in Part III.

Part III relates to symbols for physico-chemical quantities, with conventions relating to their use.

It opens with general recommendations regarding alphabets and founts.

It is recommended that certain important physical constants (*e.g.* Avogadro's number, $\boldsymbol{N}$; velocity of light in vacuo, $\boldsymbol{c}$; electronic charge, $\boldsymbol{e}$; Planck's constant, $\boldsymbol{h}$, gas constant per mol., $\boldsymbol{R}$) should be printed in black italic type.

To be Printed in Ordinary Italic, when not Greek

General Physics and Chemistry

Length	l
mean free path of molecules	l
height	h
diameter, distance	d
diameter of molecules	σ (sigma)
radius	r
Mass	m
molecular weight	M
atomic weight	A
gram-equivalent weight	Z, J
Time	t
time interval, especially half- or mean-life	τ (tau)
Velocity	v ; c (u, v, w)
of ions	u_+ and u_-
angular	ω (omega)
Acceleration	f.....a
due to gravity (as variable)	g
Force	F, (X, Y, Z)
Moment of inertia	I
Pressure	p, P
especially osmotic	Π (pi)
Volume	v, V
Density	ρ (rho).....d
Compressibility	κ (kappa).....K
Viscosity	η (eta)
Fluidity	ϕ (phi)
Surface area	A.....s
Angle of contact	θ (theta)
Surface tension	γ (gamma)..... σ (sigma)
Parachor	$[P]$
Surface concentration excess	Γ (gamma)

Number of mols	n
Concentration, mol fraction	N, x
in other terms	c, C
Solubility	s
Diffusion coefficient	D
Chemical equilibrium constant (products/reactants)	K
solubility product	K_s.....L
Velocity constant of chemical reaction	k
Number of molecular collisions per second	Z
Critical energy of activation	E_c
Partition function	f
Efficiency, of any process	η (eta)
Atomic number	Z
Wave function	ψ (psi)

Heat and Thermodynamics

Temperature, on absolute scale (°K)	T
on other scales	θ (theta).....t
Thermal conductivity	k
Energy (general symbol)	E
Work done by or on a system	w.....W
Heat entering a system	q
Specific heat	c_p and c_v
molecular heat	C_p and C_v
Latent heat, per g.	l
per mol	L
Intrinsic energy	U.....E
Enthalpy, total heat, or heat-content	H
Entropy	S
Free energy (Helmholtz)	A.....F
Thermodynamic potential, Gibbs function, free energy (G. N. Lewis)	G
Vapour pressure constant	i
Chemical potential	μ (mu)
Activity	a
coefficient (in terms of molar concentration)	f
Osmotic coefficient	g

Electrochemistry and Electricity

Van't Hoff's factor	i
Degree of electrolytic dissociation	α (alpha)
Valency, or number of charges on an ion	z
Ionic strength	I
Quantity of electricity	Q
especially electrostatic charge	e
Potential (difference)	V
Volta potential	V
electrokinetic potential	ζ (zeta)
especially electromotive force of voltaic cells	E
Potential gradient, in electric field	X
Electronic exit work function	ϕ (phi)
Current	I
Resistance	R
specific resistance	ρ (rho).....r
specific conductance	κ (kappa)..... σ (sigma)
Inductance, self	L
mutual	M
Electrostatic capacity	C
Dielectric constant	ϵ (epsilon)
Dipole moment	μ (mu)

Equivalent conductance	Λ (lambda)
equivalent ionic conductance, mobility	l_+ and l_-
Transport number	T_+ and T_-..... n_+ and n_-
Single electrode potential	e (with subscript) ..... E (with subscript)
Electrolytic polarisation, overvoltage	η (eta)..... π (pi)

Magnetism

Magnetic field strength	H
flux	ϕ (phi)
permeability	μ (mu)
susceptibility, volume	κ (kappa)
mass	χ (chi)
moment	M
induction	B

Optics

Wave-length	λ (lambda)
Frequency	ν (nu)
Intensity of light	I
Refractive index	n..... μ (mu) (with subscript)
specific refraction	r (with subscript)
molecular refraction	R (with subscript)
Molar extinction coefficient	ϵ (epsilon)
Angle of (optical) rotation	α (alpha)
specific rotation	$[\alpha]$ (alpha)
Specific magnetic rotation	ω (omega)

TO BE PRINTED IN ROMAN, WHEN NOT GREEK

(*a*) *Mathematical Constants or Operators*

Base of natural logarithms	e
Ratio of circumference to diameter	π (pi)
Differential	d
partial	∂
Increment	Δ (delta)
very small increment	δ (delta)
Summation	Σ (sigma)
Function of	f, ϕ (phi)

(*b*) *Examples of Single-Letter Abbreviations* (*cf.* provisional List of Abbreviations drawn up by the Royal Society)

*Ampère (in sub-units)	a.
Volt	V.
Ohm	Ω. (omega)
Watt	W.
Farad	F.
Henry	H.
Centigrade	C.
Fahrenheit	F.
Kelvin	K.
Ångstrom unit	A.
micron	μ. (mu)
metre	m.
gram	g.
litre	l.
Röntgen unit	r.
†Normal (concentration)	N.
†Molar (concentration)	M.

* *e.g.* "ma." for "milliampère"; but "amp." is preferred for "ampère."

† Separated by a hyphen (and no full stop) from a chemical formula which follows it.

SUBSCRIPTS AND OTHER MODIFYING SIGNS

(*a*) *Subscripts to Symbols for Quantities*

I, II ..... / 1, 2	*especially with symbols for thermodynamic functions*, referring to different systems or different states of a system.
A, B, ...	referring to molecular species A, B, etc.
i	,, a typical ionic species i.
+, −	referring to a positive or negative ion, or to a positive or negative electrode.
u	referring to an undissociated molecule.
p, *v*, *T*	indicating constant pressure, volume, and temperature respectively.
q	indicating adiabatic conditions.
w	indicating that no work is performed.
G, V, L, X	referring to gas, vapour, liquid, and crystalline states respectively.
f, e, s, t, d	referring to fusion, evaporation (vaporisation of liquid), sublimation, transition, and dissolution or dilution respectively.
c	referring to the critical state or indicating a critical value.
o	referring to a standard state, or indicating limiting value at infinite dilution.
p, *c*, *a*	*with symbol for an equilibrium constant*, indicating that it is expressed in terms of pressure, concentration, or activity.
C, D, F	*with symbols for optical properties*, referring to a particular wave-length.

Where a subscript has to be added to a symbol which already carries a subscript, the symbol with the first subscript may be enclosed in parentheses with the second subscript outside.

(*b*) *Other Modifying Signs*

o	*as right-hand superscript to symbol*, referring to a standard state.
[ ]	*enclosing formula of chemical substance*, indicating its molar concentration.

Numerals attached to a symbol for a chemical element in various positions have the following meanings:

upper left	mass number of atom.
lower left	nuclear charge of atom.
lower right	number of atoms in molecule.

e.g. $^{7}_{3}Li$; $^{2}_{1}H_{2}$ (= D_2).

British Standards Institution

HAEMACYTOMETER COUNTING CHAMBERS AND HAEMACYTOMETER DILUTION PIPETTES

THE British Standards Institution has just issued a Standard Specification (No. 748—1937) for Haemacytometer Counting Chambers and Haemacytometer Dilution Pipettes, which has been prepared with the co-operation of the medical profession. In framing the specification particular attention has been paid to the various tolerances specified. The accuracy of a blood count depends on the skill of the operator in obtaining a representative sample, in carrying out the dilution, and in the use of the counting chamber. It is also limited by the errors of the apparatus employed. In the preparation of this specification particular attention has been paid to the latter sources of error. The accuracy of the final count depends, for example, on the accuracy of a number of elements of the counting chamber, *e.g.* accuracy of ruling, accuracy of depth, departure of under-surface of cover glass from a plane, etc. In view of the number of possible sources of error, the tolerances on individual elements had of necessity to be kept small in order to avoid the possibility of a comparatively large error in the final count, due to an accumulation of small errors. The Committee, however, did not approach the question of tolerances in a purely theoretical manner. On the basis of measurements carried out at the National Physical Laboratory on samples submitted by manufacturers as representative of current production, the tolerances were also related to the degree of accuracy attainable in manufacture without unduly increasing the cost of production.

Copies of this specification may be obtained from the British Standards Institution, 28, Victoria Street, London, S.W.1, price 2s. 2d., post free.

DENSITY-COMPOSITION TABLES FOR SULPHURIC ACID

WHEN the "British Standard Specification for Density Hydrometers" (No. 718—1936) was published, the British Standards Institution announced that density-composition tables for various solutions of industrial importance were in course of preparation. The first of these "British Standard Density-Composition Tables for Aqueous Solutions of Sulphuric Acid for use in conjunction with British Standard Density Hydrometers" (No. 753—1937) has just been published. The tables are based on the International Critical Tables and are very comprehensive. They give percentage compositions (grams of H_2SO_4 in 100 grams of solution) and concentrations (grams of H_2SO_4 in 1 litre of solution) for densities progressing in steps of 0·001 g./ml. from 1·000 g./ml. to 1·846 g./ml. at temperatures progressing by steps of 2° C. from 10° C. to 40° C.

The tables are preceded by explanatory notes. Appendixes to the tables give details of the British Standard density hydrometers available for use in aqueous solutions of sulphuric acid, notes on the reading of British Standard density hydrometers in these solutions, examples of the use of the tables in conjunction with British Standard density hydrometers and details of corrections to hydrometer.

Hydrometer corrections are only necessary when the highest accuracy attainable with the hydrometer is desired. They may often be entirely ignored without prejudice to the degree of accuracy required. For example, over the temperature range 10° C. to 30° C. and for all strengths of acid, the error introduced by neglecting all corrections will not exceed $\pm$ 0·001 g./ml. when a British Standard density hydrometer subdivided in 0·0005 g./ml. intervals is used. When this degree of accuracy is adequate, the hydrometer reading may be taken as giving directly the density of the acid in g./ml. at the prevailing temperature of the solution, and the tables give directly the strength of the acid from the ascertained density. No temperature adjustment is necessary and no calculation is required. The use of British Standard density hydrometers in conjunction with the tables provides a concrete example of the simplicity of hydrometry based on density measurements.

Copies of this British Standard Specification (No. 753—1937) may be obtained from the British Standards Institution, Publications Department, 28, Victoria Street, London, S.W.1, price 2s. 2d., post free.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS

Food and Drugs

Detection of Decomposition Products in Butter and Cream. J. O. Clarke, J. H. Cannon, E. W. Coulter, M. S. Goodman, W. S. Greene, K. L. Milstead, R. L. Vandaveer and J. D. Wildman. (*J. Assoc. Off. Agr. Chem.*, **1937**, **20**, 475–505.)—The indole-content of butter-fat serves as a useful index of the state of decomposition of the cream from which it was prepared. Other useful indications are the acidity of the fat determined by titration of a solution of the fat in benzene with standard sodium ethylate solution and the mould-content determined by the method of Wildman (Abst., ANALYST, 1937, **62**, 402). For the determination of indole, the following reagents are required:— Phosphoric-aldehyde mixture made by dissolving 0·4 g. of purified *p*-dimethylaminobenzaldehyde in 5 ml. of acetic acid and mixing the solution with 92 ml. of phosphoric acid (85 per cent.) and 3 ml. of conc. hydrochloric acid; acetic acid and ether mixture made by mixing equal volumes of purified acetic acid and peroxide-free ether; 95 per cent. alcohol (limits 94 to 98 per cent.); dilute hydrochloric acid, by diluting 5 ml. of the conc. acid to 100 ml. with water; standard indole solution made by diluting 100-fold, with 95 per cent. alcohol, a stock solution (stable for 10 days) containing 20 mg. in 200 ml. of 95 per cent. alcohol. In the distillation apparatus all stoppers should be of synthetic rubber. Place 100 ml. of melted butter-fat at about 40° C. in a stoppered measuring cylinder and fill to 250 ml. with 95 per cent. alcohol. Shake the cylinder for two minutes, placing it in

water at 60° C. if the fat solidifies, and, when the layers have partly separated, shake for a further two minutes and then allow complete separation to occur. It is important that the fat should remain liquid during the separation. Since indole distributes itself between the two liquids in amounts proportional to their respective volumes, 125 ml. of the alcoholic layer will contain one-half of the indole present. Distil this quantity in steam, collecting 450 ml. of distillate in 90 minutes and maintaining the volume of the distilling liquid at about 70 ml. by the application of heat. Acidify the distillate in a separator with 5 ml. of dilute hydrochloric acid, and extract with 25 ml. of chloroform which has previously been used to rinse out the condenser and receiver. Repeat the extraction with 20 and 15 ml. of chloroform, wash the first two extracts (combined) with 400 ml. of water and extract the wash-water with the third extract. Filter the extract through cotton-wool into a dry 125-ml. separator. To the chloroform extract add 10 ml. of the phosphoric-aldehyde reagent, shake vigorously for two minutes with a frequency of 200 per minute, allow the separator to stand for exactly ten minutes, and separate the acid layer as completely as possible into a glass-stoppered 50-ml. cylinder. Allow the separator to stand for successive periods of two minutes until no more acid liquid can be drained away. The separated acid layer must not contain chloroform, and no attempt to drain acid from the bore of the tap should be made. Dilute the liquid in the cylinder with the mixture of acetic acid and ether first to about 40 ml. and, when cold, to 50 ml. Filter if necessary, using a covered funnel and, within 15 minutes, read the colour of the solution in a photometer fitted with a filter transmitting a narrow band around 500$m\mu$. Deduct the result of a blank determination made upon 125 ml. of alcohol in the same apparatus and convert the photometer reading to γ of indole by means of a standard curve constructed by treating the requisite volumes of standard indole solution, diluted to 125 ml. with alcohol, in the same manner as in the blank determination. A series of standards of 1, 2, 5, 7, 10, 15, 20 and 30γ of indole should be made, the blank being deducted from each one. Readings should be taken in cells of about 25, 50 and 100 mm. in length, a separate standard curve being constructed for each cell. The foregoing method is applicable in the presence of various artificial butter flavours and the butter colours used commercially in U.S.A. A more rapid method giving rather higher results may be used in absence of the butter colours Yellow AB and Yellow OB. The butter-fat is treated with alcohol as previously described, and 125 ml. of the alcoholic layer are mixed in a separator with 325 ml. of water, 5 ml. of dilute hydrochloric acid and 25 ml. of chloroform. The procedure is then as before. By another method, also inapplicable to butter coloured with Yellow AB or Yellow OB, and in which strict adherence to detail is necessary, 50 ml. of butter-fat are washed into a separator with 50 ml. of chloroform, 15 ml. of the phosphoric-aldehyde reagent are added, and the mixture is shaken vigorously as in the distillation method. Separation and drainage of the acid layer are carried out as previously described, and the liquid is diluted with the acetic acid and ether mixture first to about 80 ml. and finally to 100 ml. In this method the standard curve is constructed by means of volumes of the standard solution corresponding with 0 to 50γ of indole diluted to 100 ml. with chloroform.

It was found that lactic acid fermentation of cream with pure cultures caused

no increase in the indole-content and acidity of the butter made therefrom. Decomposition induced by inoculation of sterile cream with decomposing cream caused increased acidity of the fat, increased mould-content and sometimes increased indole-content. When commercial cream was exposed to an environment favourable to decomposition, the acidity, mould-content and indole-content increased both in the cream and the butter, the rate of increase being more rapid with higher temperature. No significant changes in these indications were noted in butter stored under conditions imitating commercial shipping or storage. The indole-content of commercial butter of good quality is of the order 4 to 6γ per 50 ml. of fat. A. O. J.

Hydrolysis of Potato Starch by Malt Amylase at Different Temperatures. J. L. Baker and H. F. E. Hulton. (*J. Inst. Brewing*, 1937, **43**, 301–307.)—Malt amylase was prepared by grinding 100 g. of a pale English malt (diastatic power, 25° Lintner) with 300 ml. of 20 per cent. alcohol, and precipitating the filtered extract with three times its volume of 95 per cent. alcohol, the resulting precipitate being separated in a centrifuge and dissolved with the amylase solution in 100 ml. of water. A 3 per cent. paste of pure potato starch farina was digested (at the rate of 0·5 ml. per g. of starch and at *p*H 6·5) for 1 or 12 hours at 15·5°, 50°, 65° and 74° C. The cleavage products were then evaluated, the sp.gr., dissolved solids (using the factor 3·93), $[\alpha]_D$ and copper-reducing power (Fehling's) being determined; in the last instance the results obtained by Lane and Eynon's volumetric method were expressed as a percentage of anhydrous maltose (*R*) on the solids found by means of the factor 3·93, and they are accurate to within 2 per cent. Fractionation experiments with 90 per cent. alcohol and fermentation tests using a pure culture of *S. cerevisiae* were also made. Maltose and probably one dextrin only (*R*, 10; $[\alpha]_D$, 185–190, approximately) are produced as the sole ultimate products of conversions made at temperatures up to 50° C., the whole of the "apparent maltose" being fermentable; above 50° C., and definitely at 65° and 74° C., maltodextrin is also present in addition to these substances. The evidence for the presence of the maltodextrin is that (*a*) part of the "apparent maltose" is unfermentable (and this corresponds with the maltose portion of the maltodextrin); (*b*) those fractions which are soluble in alcohol, or have been freed from maltose by fermentation, indicate the presence of a complex which is not maltose but which is soluble in alcohol and unfermentable, and the constants of which have the mean values *R* 35 and $[\alpha]_D$ 174, *i.e.* intermediate between those of maltose and dextrin. The extent of the formation and survival of this maltodextrin is dependent on the temperature of conversion, being negligible at 15·5° and 50° C., definite at 65° C., and very marked at 74° C. (when only 50 per cent. of the "apparent maltose" is free, *i.e.* fermentable, maltose). The well-known inhibiting effect of high temperatures on the saccharifying activity of malt amylase provides a probable explanation why maltodextrin is found only in starch conversions which have been carried out at such temperatures. Thus, although the maltodextrin is probably an intermediate compound which is formed in starch conversions at all temperatures, it is converted into maltose by amylolytic hydrolysis at 15·5° and 50° C.; at higher temperatures this cannot occur, and the maltodextrin, therefore, accumulates in the final products. J. G.

Seeds and Oil of *Rhus glabra*. G. H. McFadden and R. L. McMurray. (*Amer. J. Pharm.*, 1937, **109**, 397–406.)—The fruits of *Rhus glabra*, or Sumac berries, were collected in Ohio, shelled and air-dried, and the cleaned fruits were powdered. They contained 5·25 per cent. of moisture, and gave on extraction with ether a total extract of 17·27 per cent. They contained 31·86 per cent. of crude fibre, 21·61 per cent. of pentosans, 1·32 per cent. of total nitrogen; arsenic, none; total ash, 2·55 per cent. Petroleum spirit extracted from the cleaned ground fruits 12·42 per cent. of oil with the following characteristics:—sp.gr. at 20° C., 0·9227; n_D^{20}, 1·4719; $[\alpha]_D^{20}$, 0·00; saponification value, 168·17; iodine value, 87·17; ester value, 159·2; acid value, 8·9; unsaponifiable matter, 2·67 per cent. The percentage composition of the ash was: sand, 2·95; silica, 7·44; iron, 0·86; Al_2O_3, 4·67; CaO, 22·80; MgO, 7·24; Mn_3O_4, 0·147; copper, nil; zinc, 0·078; Na_2O, 4·16; K_2O, 38·15; Cl, 1·58; SO_3, 7·15; P_2O_5, 21·17. The pure oil obtained by steam-distillation of the crude oil, solution in petroleum spirit and washing with water, amounted to 94·6 per cent. of the crude product, and was blackish-brown with a green fluorescence; saponification value, 176·7; iodine value, 94·15. The aqueous fraction contained tannin and sugar. The sterol acetate obtained from the unsaponifiable matter had m.p. 117° to 118° C., and a hydrocarbon (hentriacontane), m.p. 68° C., was obtained from the alcoholic filtrate from the digitonin treatment. The solid and liquid fatty acids were separated, and the liquid acids identified were linolic and oleic acids. The methyl esters of the solid fatty acids were repeatedly fractionally distilled until 8 well-defined fractions were obtained. The acids identified were *n*-butyric (small quantity), palmitic, lignoceric and (probably) arachidic acids. D. G. H.

Fatty Oil from the Seeds of *Valerianella olitorea*, Poll. A. Steger and J. van Loon. (*J. Soc. Chem. Ind.*, 1937, **56**, 298–300T.)—The fruits of *Valerianella olitorea*, the corn salad, were crushed and extracted with ether, yielding 14 per cent. of oil. The kernels alone gave 38·7 per cent. of a nearly colourless oil, and the shells contained some 4 per cent. of a dark green oil. By running through a mill, sieving and winnowing, kernels with a very small quantity of shell may be isolated, yielding 23–24 per cent. of a light-coloured oil (B). Oil (A) was from hand-selected kernels and oil (C) from the hulls.

	Oil A	Oil B	Oil C
Sp.gr. at 78/4° C.	0·8830	0·8800	0·9200
n_D^{70}	1·4575	1·4582	1·4620
Saponification value ..	192·6	190·5	205·4
Iodine value (Wijs)	145·2	144·9	116·5
Thiocyanogen value	84·2	84·2	67·7
Acid value	1·2	3·7	48·1

Thin films of oils A and B dried in 2 days on glass plates at 45° C. in diffused daylight; oil C did not dry. The compositions of oils B and C (the fatty acids being calculated from the iodine and thiocyanogen values and proportion of saturated acids) were:—unsaponifiable matter, 1·2, 2·6; glyceryl (as C_3H_2), 4·2, 3·5; volatile and insoluble products, 1·8, 19·8; saturated acids, 11·5, 15·2; oleic acid, 18·0, 7·6;

linolic acid, 52·7, 47·9; linolenic acid, 10·6, 2·4 per cent. The extraction residues of the two oils contained, respectively, moisture, 10·0, 9·7; ash, 7·1, 6·8; nitrogen, 3·0, 1·15; protein, 18·8, 7·2; crude fibre, 25·9, 34·2; nitrogen-free extract, 38·2 and 42·1 per cent. The cake from oil B was found to be suitable for cattle feeding, that from oil C for manure. D. G. H.

Oil from the Seeds of *Blepharis edulis*. G. P. Pendse and J. B. Lal. (*J. Indian Chem. Soc.*, 1937, **14**, 362–366.)—A bitter glucoside (blepharin) and *dl*-allantoin have already been isolated from the seeds of *Blepharis edulis* (*J. Indian Chem. Soc.*, 1936, **13**, 109), and the composition of the oil has now been investigated. Extraction of the powdered seeds with benzene yielded 3·8 per cent. of a thick reddish-brown oil with the characteristic odour of the drug. The purified oil had the following characteristics:—sp.gr. at 28° C. 0·9332; $[\alpha]_D^{28}$, — 8·4; n_D^{30}, 1·4846; viscosity (*cf.* with rape oil), 8·35; solidifying pt., — 3° C.; saponification value, 186·5; iodine value, 90·8; Hehner value, 91·65; acetyl value, 11·54; acid value, 11·85; unsaponifiable matter (including phytosterol), 2·5 to 3·0 per cent. The fatty acids were separated by Twitchell's method into 12·38 per cent. of saturated (iodine value 2·6; mean molec. equiv. 276) and unsaturated acids, 87·62 (iodine value 104·7; mean molec. equiv. 270·2). The unsaturated acids consisted of 83·45 per cent. of oleic and 16·37 per cent. of linolic acid. The saturated acids were converted into the methyl esters, and 4 fractions were separated by fractional distillation. Palmitic, stearic and arachidic acids were identified. A light brown solid deposited from the oil was for the most part soluble in boiling alcohol, yielding a crystalline phytosterol which appeared to be identical with arnidiol described by Klobt (*Compt. rend.*, 1904, **138**, 763; **140**, 1700). D. G. H.

Oil from the Seeds of *Solanum nigrum*. G. P. Pendse. (*J. Indian Chem. Soc.*, 1937, **14**, 367–370.)—*Solanum nigrum* (Gurkamai and Choti Makoi in Hindustani) is cultivated in India for its medicinal properties, and is described by Dymock in *Pharmacographia Indica*, 1891, **2**, 549, and by Basu and Kirtikar, in *Indian Medicinal Plants*, 1918, **2**, 889. The active principle of the fruits is solanine. The fresh fruits yielded 30 per cent. of juice, 3·75 per cent. of dried husk and 9·5 per cent. of seeds. The powdered seeds on extraction with petroleum spirit gave about 2 per cent. of a greenish-yellow oil with the characteristic odour of the drug. The purified oil had the following characteristics:—sp.gr. at 30° C., 0·8964; $[\alpha]_D^{28}$ — 6·61; n_D^{30}, 1·4436; viscosity (compared with rape oil), 7·12; solidifying pt., — 7° C.; saponification value, 184·7; iodine value, 111·7; Hehner value, 93·10; acetyl value, 9·97; acid value, 2·4; unsaponifiable matter, 1·4 to 1·6 per cent. The fatty acids consisted of 5·88 per cent. of saturated (iodine value 3·6; mean molec. equiv. 180·4) and 94·12 per cent. of unsaturated acids (iodine value 114·8; mean molec. equiv. 176·6). The unsaturated fatty acids consisted of 73 per cent. of oleic and 27 per cent. of linolic acid. No linolenic acid was found. The saturated acids were converted into the methyl esters which were fractionally distilled; only palmitic and stearic acids were found. The unsaponifiable matter deposited from ether in silky flakes, m.p. 127° to 129° C., and the acetyl derivative of the sterol had m.p. 119° to 120° C., with $[\alpha]_D^{32}$, in alcoholic solution — 30·5°. D. G. H.

Colorimetric Determination of Morphine. D. C. Garratt. (*Pharm. J.*, **1937, 139**, 193.)—The accuracy of the B.P. colorimetric determination of morphine can be increased by "compensating" the blank solution, after it is made ammoniacal, with a quantity of the test solution equal to that used in the test. The necessity for this "compensation" is shown by the brown colour given by the extracted alkaloidal residue with ammonia without addition of nitrite. Except for aromatic powder of chalk and opium, for which it requires further modification, the method may be used for all galenicals, including mixtures of opium and ipecacuanha, for the latter does not interfere with the test. In aromatic powder of chalk and opium the phenolic constituents of the aromatics give a colour similar to that obtained with morphine, but these may be eliminated by extraction with ether *after* the lime solution has been made ammoniacal with ammonium sulphate; the morphine remains in the aqueous phase, and is extracted as usual with chloroform in the presence of alcohol. S. G. S.

Relationship between the Constitution of Tragacanth Gum and the Viscosity of its Mucilage. J. M. Rowson. (*Quart. J. Pharm.*, 1937, **10**, 161–176.)—The object of this work was to compare the bassorin and methoxyl contents and saponification values of a number of gums with the viscosities of the mucilages prepared from them. It is the insoluble bassorin in tragacanth which absorbs water and gives the mucilage its viscosity, the influence of the small quantity of soluble gum present in a pharmacopoeial mucilage being only slight. The direct determination of bassorin by physical methods (*e.g.* separation by filtration or centrifuging, and subsequent weighing) was unsuccessful, and Ogle's method (*Pharm. J.*, 1889, **20**, 3) was therefore modified so as to overcome the difficulty of the variable hydrolysis of the bassorin and the consequent production of soluble decomposition products; the amount of bassorin was found by subtracting the percentages of moisture and soluble gum (tragacanthin) from 100. A 0·1 per cent. mucilage was prepared by shaking a weighed quantity of gum with a little alcohol in a dry graduated flask, adding distilled water, and shaking well. After it had stood for 48 hours, with occasional shaking, it was filtered by suction through a double layer of filter-paper into a dry suction flask; 100 ml. of clear filtrate were then evaporated on the water-bath in a tared flask, and the residue was dried in a water-oven and weighed to obtain the tragacanthin-content. Variable results obtained in duplicate determinations on the same gums could not be traced to impurities in the solvents, or to variations in the areas or capacities of the funnel and vessels used, but appeared to occur when the time elapsing between the preparation of the mucilage and filtration exceeded 48 hours. Further experiments showed that it is therefore desirable to boil the mucilage for exactly 3 minutes, in order to attain the maximum particle size and degree of hydration and, therefore, the maximum viscosity and minimum soluble extract; if this period is exceeded, the bassorin is hydrolysed and forms soluble products, which increase the apparent tragacanthin-content. The use of freshly-boiled and cooled distilled water is also recommended. The moisture was determined at 100° C., and also in a vacuum desiccator over sulphuric acid; the latter procedure gave higher results and is preferable. The average tragacanthin

and bassorin contents of 12 powdered gums ranged from 9·52 and 75·47 to 12·62 and 75·57 per cent., respectively, but no direct mathematical ratio could be traced between the bassorin (when expressed as relative percentages of an arbitrary standard) and the corresponding viscosities (6·26 to 1064·0 poises for a 1·25 per cent. mucilage). There is evidence, however, for the generalisation that the mucilage from a gum with a low bassorin-content will have a low viscosity or suspending-power, and that, although the determination of the bassorin-content is of value for distinguishing between a good and a poor gum, the viscosity depends on the properties as well as on the quantity of the bassorin present. The B.P. (Zeisel) method for the determination of the methoxyl-content was used, 1 g. of the sample and 50 ml. of a 0·1 *N* solution of silver nitrate being taken; the excess of silver nitrate was titrated with a standard solution of ammonium thiocyanate in the presence of strong nitric acid. Replicate tests made in this way agreed well, and the average values found ranged from 0·763 to 4·426 per cent. (4·70 to 7·03, if expressed as a percentage of the bassorin-content). A close proportionality exists between the bassorin and methoxyl contents of the gums examined; the latter value is therefore a satisfactory measure of the former, and the conclusions as to the relationship of the methoxyl-content to the viscosity are the same as for the bassorin-content. The changes produced in a gum by the action of heat appear to be (*a*) partial demethylation of bassorin, which forms products which are insoluble in water, but do not swell readily in it, and therefore, yield mucilages of low viscosity; (*b*) complete demethylation of bassorin, the resulting products being soluble in water, but incapable of exerting much effect on the viscosity of a mucilage compared with that due to the methylated compounds. The saponification value was determined on 1 g. of sample, which was heated for 1 hour under a reflux condenser with 100 ml. of water and 25 ml. of 0·5 *N* potassium hydroxide solution; the cooled solution was titrated back with acid, 5 ml. of a solution of phenolphthalein being necessary on account of the colour of the liquid. The average results for 5 gums ranged from 222·0 to 256·5; they showed no relationship either to the bassorin or methoxyl contents or to the viscosities. It is suggested that the B.P. monograph on tragacanth should be modified to require a bassorin-content of not less than 60 per cent. when determined by the process described above, and a methoxyl-content (Zeisel method) of 3·75 per cent., and that a concentration of not more than 1·65 per cent. of gum should be required to produce a mucilage having a viscosity of 400 poises when measured by means of 5/32 in. steel spheres falling in a sphere viscometer at 20° C. The mucilage for this purpose should be prepared in a bulk of 600 ml. by the dry-flask process, and should be allowed to stand for 48 hours before the measurement is made. A description of the falling-sphere viscometer and the method of calculating viscosities by the Ladenburg formula should be inserted in an appendix to the Pharmacopoeia (*cf.* Brindle and Rowson, *Quart. J. Pharm.*, 1936, **9**, 161). J. G.

Estimation of Acriflavine and Related Compounds in Pharmaceutical Preparations and Surgical Dressings. G. F. Hall and A. D. Powell. (*Pharm. J.*, 1937, **139**, 195–196.)—The solution obtained by a preliminary treatment of the dressing or galenical, and containing between 0·02 and 0·2 g. of the

diaminoacridine derivative, is diluted to approximately 200 ml., and adjusted until the reaction is faintly acid to Congo red paper. One g. of sodium acetate crystals is added, followed by sufficient excess of $M/50$ potassium ferricyanide solution (19 ml. to 30 ml. according to the amount required for precipitation) with stirring during the addition. The mixture is allowed to stand for 30 minutes and is then filtered through a Buchner funnel. The precipitate is washed with three successive quantities of 10 ml. of water, and to the combined filtrate and washings are added 5 ml. of conc. hydrochloric acid, 1 g. of sodium chloride, 0·5 g. of potassium iodide and 5 ml. of a 30 per cent. solution of zinc sulphate, the contents of the beaker being well mixed after each addition. After 3 minutes the liberated iodine is titrated with $N/100$ sodium thiosulphate solution. A blank determination is also made. Each ml. of $M/100$ ferricyanide solution precipitated is equivalent to 0·00888 g. of acriflavine, 0·00779 g. of euflavine or 0·00921 g. of proflavine. For the assay of medicated gauze, 20 g. or other convenient quantity are extracted in a Soxhlet extractor with 95 per cent. alcohol slightly acidified with hydrochloric acid (250 ml. of alcohol and 2 ml. of 10 per cent. hydrochloric acid). After about 3 hours the extract is transferred to a beaker, 50 ml. of water are added, and the bulk of the alcohol is evaporated. To the hot liquid 25 ml. of chloroform are added and the whole is mixed thoroughly. After cooling, the mixture is transferred to a separator and allowed to separate, and the removal of the fats is completed by a further two extractions with 25 ml. of chloroform. The combined chloroform extracts are washed twice with 10 ml. of water which has been acidified with one or two drops of dilute hydrochloric acid. The united aqueous solution is evaporated to a small volume to remove the alcohol, then diluted to about 200 ml., made slightly acid to Congo red paper, and treated as described above.

Fatty and oily preparations are prepared for the assay by dissolving 25 g. to 50 g. in about 50 ml. of chloroform. This solution is extracted with 20 ml. of water containing 2 ml. of 10 per cent. hydrochloric acid, followed by two extractions with 10 ml. of water containing 0·5 ml. of the dilute hydrochloric acid. The combined aqueous extracts are washed with 25 ml. of chloroform; any medicament taken into the chloroform is extracted with 10 ml. of water containing one or two drops of hydrochloric acid solution. The combined aqueous extracts are evaporated sufficiently to remove alcohol if present, cooled, diluted to about 200 ml., treated with dilute sodium hydroxide solution until the reaction is only slightly acid to Congo red paper, and examined by the general method. Glycogelatin preparations present certain difficulties, but the following treatment was found satisfactory with glycogelatin pessaries. About 15 g. to 30 g. of the sample are dissolved in 100 ml. of warm water, and the gelatin is precipitated by the addition of 200 ml. of 95 per cent. alcohol. The mixture is cooled, the precipitate is coagulated by stirring, removed by centrifuging, re-dissolved in 50 ml. of water and re-precipitated with 100 ml. of alcohol. The clear alcoholic liquids are combined, evaporated to remove the alcohol, and diluted to 200 ml. with water. After the acidity has been adjusted the general method is followed. S. G. S.

Biochemical

Conversion of Stearic Acid into Palmitic Acid in the Organism. R. Schoenheimer and D. Rittenberg. (*J. Biol. Chem.*, 1937, **120**, 155–165.)—Palmitic acid was isolated from the carcases of mice which had been fed for 5 days with deuterostearic acid. The deuterium-content of the palmitic acid indicated that it had been derived from the stearic acid. The separation was obtained by fractional distillation of the methyl esters. This was done in a distillation apparatus which is described, and which is capable of separating the higher fatty acids from a mixture of 1 to 3 g. A method is also described for the removal of a contaminating deutero substance from another one. S. G. S.

Amino Acid Catabolism. The Fate of Certain Synthetic α-Amino Acids Administered by Subcutaneous Injection to the Normal Dog. J. A. Leighty and R. C. Corley. (*J. Biol. Chem.*, 1937, **120**, 331–334.)—When amino acids were injected into a normal dog under the experimental conditions described it was found that those acids having a straight carbon chain readily lost their nitrogen, which was excreted as urea. The presence of a methyl group on the same carbon acid as the nitrogen retarded the loss of nitrogen, while the presence of the methyl group on the carbon atom adjacent to the one with the amino group prevented deamination. This latter effect was probably due to spatial configuration. S. G. S.

Determination of Thiocyanate in Tissues. B. B. Brodie and M. M. Friedman. (*J. Biol. Chem.*, 1937, **120**, 511–516.)—Thiocyanate in tissues may be determined by placing 0·5 g. to 1·0 g. of finely hashed and thoroughly mixed wet tissue, or 100 mg. to 300 mg. of the dried pulverised tissue, in a 125-ml. Erlenmeyer flask and adding 20 ml. of alcoholic potash solution (40 g. KOH in 1 litre of 95 per cent. ethyl alcohol). The flask is attached to an air condenser, and the mixture is digested on a steam-bath for 30 minutes. The flask is then emptied into a 200-ml. evaporating dish, and rinsed out into the dish with alcohol, then with water, and finally again with alcohol. The dish is placed on the steam-bath and the alcohol is evaporated. The residue is washed with small amounts of water into a 25-ml. graduated cylinder. One drop of phenolphthalein solution is added, the volume is diluted to 20 ml., and the whole is transferred to a 125-ml. Erlenmeyer flask. The cylinder is washed with exactly 3 ml. of water, and this is added to the solution in the flask. The liquid is neutralised with 4 *N* nitric acid, the amount required being noted, and 0·5 ml. is added in excess. To this acid solution 1 ml. of 10 per cent. sodium tungstate solution is added dropwise, the flask being shaken during the addition. Sufficient water is added to give a total volume of 30 ml., but since the measurement of the total volume is difficult on account of frothing, the amount of the water required is ascertained by the careful measurement of the wash-water, the nitric acid and the sodium tungstate solutions. The flask is then stoppered, shaken vigorously, and left for 10 minutes. The mixture is filtered, and a 25-ml. portion of the

filtrate is transferred to a 100-ml. beaker; a turbid filtrate is not objectionable. The solution is neutralised with a 10 per cent. aqueous solution of caustic potash, and 0·5 ml. is added in excess. The pigments in solution are removed by adding 1 g. of Norit carbon and heating to boiling, with constant stirring. The mixture is filtered hot into a 100-ml. beaker, and the carbon is washed four times with 5-ml. portions of water. If the filtrate is still coloured, this treatment must be repeated. The colourless filtrate is evaporated to about 5 ml. and cooled, and to it is added 0·5 ml. of 4 *N* nitric acid. The solution is then quantitatively transferred to a 25-ml. glass-stoppered graduated flask, diluted to 20 ml., and the ferric thiocyanate colour is developed by the addition of 4 ml. of the ferric reagent. After thorough mixing, the resulting solution is compared with a standard in a colorimeter. Should a turbidity occur on acidification with the nitric acid, the ferric thiocyanate solution is filtered into the colorimeter cup. The standard is prepared by putting an amount of standard solution containing approximately the same amount of thiocyanate as occurs in the unknown into a graduated cylinder, adding 1·5 g. of potassium nitrate, 0·5 ml. of 4 *N* nitric acid, water to make up 20 ml., and then 4 ml. of the ferric reagent. If the volume of the standard differs from that of the unknown, the ferric reagent is added in the ratio of one volume of reagent to 5 volumes of solution, and the amount of potassium nitrate added is such that its concentration is equal to that in the unknown.

The ferric reagent is prepared by dissolving 100 g. of ferric nitrate, $Fe(NO_3)_3,9H_2O$, in 1 litre of water, adding 50 ml. of conc. nitric acid with shaking, and diluting to 2 litres. For the standard thiocyanate solution, 14 g. of sodium thiocyanate are dissolved in 1 litre of water. This solution is standardised against silver nitrate solution and is kept in a brown bottle in an ice-box. The solution changes slowly and occasional standardisations are necessary. Weaker solutions, which do not keep, are prepared from this. The sensitivity of the test is such that 75γ of thiocyanate per g. of wet tissue may be determined. The average error ranged from 8 per cent. on 75γ to 2 per cent. on 350γ. The thiocyanate-content of rat tissue varied from 0·56 mg. per g. of dry tissue for muscle to 4·26 mg. per g. for blood. S. G. S.

Use of Mercuric Salts and Nitrous Acid in the Colorimetric Determination of Tyrosine and Tryptophane present in Solution. J. W. H. Lugg. (*Biochem. J.*, **1937**, **31**, 1422–1433.)—The Folin and Ciocalteu method for estimating tyrosine, which is based on the Millon reaction, was found to be unsatisfactory when applied to the hydrolysates of plant leaf proteins, as the unknown colour solutions were cloudy. The difficulty has been overcome by changes of procedure, embracing mercuration of the tyrosine and precipitation in one step of any tryptophane present, and dilution of the reacting mixture with a solution approximating closely to it in composition. The liquids under investigation are separated from suspended solids by centrifuging for 10 minutes at 1500 times gravity being usually sufficient. A glass rod of 2-mm. diameter, slightly bent at the end, is used to stir the solutions and to suspend precipitates in them. Barely moistened with octyl alcohol it serves as a whisk to force solids at the air-liquid interface beneath the surface before centrifuging. It is rinsed down with

a few drops of the appropriate solution. The aliquot portion of test solution (up to about 3 ml.), together with sufficient 5 *N* sulphuric acid solution to bring it to *p*H 0·3 (found by the titration of a separate aliquot portion with the use of an indicator, such as brilliant cresyl blue), is diluted to 5 ml. with *N* sulphuric acid solution in a 15-ml. centrifuge tube. Five ml. of a mercury solution (75 g. $HgSO_4$, 55 g. $HgCl_2$, 70 g. Na_2SO_4 in 850 ml. of water; addition of 128 g. of 98 per cent. H_2SO_4 and dilution to 1 litre) are added, and the tube is maintained at 60° to 65° C. in a water-bath for 30 minutes. It is then cooled in a water-bath for 1 hour at 1° or 2° C. below room temperature, and, after centrifuging, the clear liquid is drained into a 25-ml. graduated cylinder. The precipitate in the centrifuge tube is treated with 10 ml. of a solution prepared by diluting the mercury solution with an equal volume of *N* sulphuric acid. Any precipitate is well stirred, and the tube is again centrifuged. The liquid is drained as before into the cylinder, the contents of which are diluted with the wash solution to 24·5 ml. in readiness for the tyrosine estimation. A standard is prepared simultaneously with the unknown solution in an entirely analogous manner. The precipitate remaining in the tube is used for the tryptophane estimation.

For the estimation of the tyrosine the contents of the graduated cylinders should be employed within an hour, as cloudiness may develop on long standing. Into each cylinder 0·5 ml. of a solution of sodium nitrite (*M*/1) is run slowly so as to float on top, and as soon as possible the cylinders are shaken simultaneously. Colorimetric comparison of the unknown with the standard should be made 3 minutes after the mixing. For the estimation of the tryptophane the mercury precipitate, which may be left moist in the tube for a day without detectable destruction, is well rubbed up with 10 ml. of a solution prepared by dissolving 12 g. of mercuric sulphate and 9 g. of mercuric chloride in 600 ml. of water plus 100 g. of 98 per cent. sulphuric acid and then adding a further 500 g. of sulphuric acid, with cooling, and diluting the mixture to 1 litre. The centrifuge tube is kept at 40° to 45° C. in a water-bath for 15 minutes, with occasional rubbing of any solid that separates. It is then cooled in a water-bath for 30 minutes at 1° or 2° C. below room temperature and, after centrifuging, the clear liquid is drained into a 25-ml. graduated cylinder. A further 10 ml. of the mercury solution is run into the tube, the contents are stirred, any separating solid is well rubbed for a few minutes, and, after centrifuging, the liquid is drained into the cylinder, and the volume is made up to 24·5 ml. with the same solution. Within about an hour 0·5 ml. of the sodium nitrite solution is run into each cylinder so as to float on the top, and as soon as possible both tubes are shaken simultaneously, and the colorimetric comparison is made with the least delay. The standard tyrosine solution contains 0·25 to 1·0 mg. per ml. in either 0·1 *N* sulphuric acid or 0·05 *N* caustic soda solution; it keeps for several months. The standard tryptophane solution is of the same strength, with water as the solvent; it deteriorates to the extent of about 1 per cent. at 20° C. in a week. The errors are less than 1 per cent. for tyrosine and less than 2 per cent. for tryptophane, and the recovery of added tyrosine and tryptophane was within these limits. S. G. S.

Bacteriological

Use of Brilliant Green—Eosin Agar and Sodium Tetrathionate Broth for the Isolation of Organisms of the Typhoid Group. E. R. Jones. (*J. Path. and Bact.*, 1936, **42**, 455–467.)—The brilliant green—eosin agar is made by the addition of 1 per cent. of lactose, 0·09 per cent. of eosin and about 0·006 per cent. of brilliant green to proteose agar with *p*H 7·4. As the combined inhibiting effect of eosin and brilliant green varies with different batches of these dyes, the optimum quantity of brilliant green for the suppression of *B. coli* and the growth of *B. typhosus* is determined experimentally as follows:—Five tubes of melted agar, each containing 20 ml., are set up in duplicate in a rack and maintained at 55° C. One ml. of a 20 per cent. solution of lactose and 0·6 ml. of a 3 per cent. aqueous solution of eosin are added to each, and finally 0·2, 0·4, 0·6, 0·8 and 1·0 ml. of a 0·1 per cent. aqueous solution of brilliant green severally to each of the five tubes. The plates are poured and allowed to set, and a weak suspension of *B. coli* is spread over one half and a similar suspension of *B. typhosus* over the other. That dilution is chosen which gives the best suppression of *B. coli* and growth of *B. typhosus*. When the number of typhoid bacilli is likely to be small, the use of this medium may be combined with a tetrathionate broth enrichment medium made as follows:—To 90 ml. of ordinary broth are added 2·5 g. of chalk previously autoclaved, 10 ml. of a 60 per cent. solution of sodium thiosulphate (sterilised by steaming for 30 minutes), and 2 ml. of a 30 per cent. solution of iodine (iodine 6 g., potassium iodide 5 g. and water to make 20 ml.). This medium is distributed in 5-ml. lots. For use, 0·5 g. of faeces is emulsified in 5 ml., from this a 1 : 10 dilution is made in the same broth, the two are incubated overnight at 37° C., and brilliant green—eosin agar plates are spread from each tube. It is claimed that this medium is suitable for the isolation of typhoid, paratyphoid and the Salmonella bacilli, and that it is at least as good as, if not better than, Wilson and Blair's bismuth sulphite—brilliant green—agar and free from the disadvantages of the latter, *viz.* complexity, loss of selective properties on keeping, and longer time required for incubation before colonies become characteristic. D. R. W.

A Satisfactory Method of Isolating Tetanus Organisms from Mixed Material. Eric C. Giller. (*Amer. J. Hyg.*, 1937, **26**, 394–400.)—The method of injecting mixed cultures into laboratory animals with a view to demonstrating characteristic spasm-producing toxin is criticised on the ground that other organisms interfere with the production of, or destroy such toxin, and the necessity for the isolation of *Cl. tetani* for the performance of the test with pure cultures is emphasised. For the separation of obligatory anaerobes from facultative anaerobes the method suggested by Hall (1919) of using a dye for the elimination of all aerobes was found to be entirely satisfactory, and for the isolation of tetanus organisms from a mixed culture of anaerobes the method of Fildes (1925), which takes advantage of the tendency of these organisms to grow in a thin film on the surface of peptic blood infusion agar, spreading to the very apex of the slant, was found to be of great value. The procedure finally adopted was briefly as follows:—The material (street dust of the city of Baltimore) suspended in normal saline was heated to

80° to 82° C. for 1 hour, transferred to Bengston's cooked meat medium (previously heated to 100° C. for 15 minutes), layered with vaseline, and incubated for 8 days. A portion was withdrawn, heated to 80° C. for 15 minutes, sown into glucose broth containing crystal violet in concentration 1 : 100,000 (previously heated to 100° C.), layered with vaseline and incubated for 18 hours at 37° C. Transfers were then made to aerobic agar slants, crystal violet dextrose broth and Holman's meat tubes. The aerobic slants show whether the aerobes are eliminated, the crystal violet broth provides for repetition if they are not, and the Holman's meat tubes provide a viable culture if they are. Sub-culturing from the dye-broth tube in the three separate media was repeated if necessary to three, four or five transfers (usually three sufficed) to eliminate aerobes. After elimination of all aerobes, peptic blood infusion agar (Fildes' influenza medium, 1920) slopes were inoculated in their condensation water and incubated for 3 to 4 days in a MacIntosh and Fildes' jar. The tubes were then carefully examined with a lens for film-like growth at the apex of the slopes. In 8 of the 63 samples from which tetanus bacilli were finally isolated they were isolated from the first slopes; in the remainder they were isolated only after re-sowing in the condensation water of another slope several times in succession. Aerobic control tests were also made. From the last series of tubes the uppermost film of growth was transferred to Holman's meat medium layered with vaseline and incubated for 3 days at 37° C. The strains isolated were identified as *Cl. tetani* by their morphology, cultural reactions, and the toxicity test for pathogenic action, which is described in another contribution (see next Abstract). D. R. W.

Study of the Biochemical Reactions of Strains of *Cl. tetani* isolated from Street Dust. E. C. Giles. (*Amer. J. Hyg.*, 1937, **26**, 401–415.)—Twelve strains of proved purity were studied. The characters used in their identification were their morphology, staining reactions, motility, biochemical reactions and pathogenicity. The biochemical tests were directed to the determination of the proteolytic and saccharolytic properties of the strains, the media employed being Hall's modification of Von Hibler's brain medium, Robertson's egg-cube broth and alkaline egg broth, litmus milk, gelatin, and plain broth containing 1 per cent. of one of the following substances—lactose, maltose, dextrose, xylose, mannitol and dulcitol. A detailed description of the action of the strains on these media is given. Variations in the reactions similar to those recorded by other observers occurred, so that hard and fast rules cannot be laid down. The general conclusion arrived at was that *Cl. tetani* has feeble proteolytic properties and does not ferment carbohydrates, the apparent gas production (without acid) observed with three strains being attributed to action on the broth medium. Even pathogenicity showed variation; nine strains produced a spasm-producing toxin neutralised by tetanus antitoxin, but three did not. One of the strains that produced no such toxin also failed to liquefy gelatin and produced acid and gas in glucose and maltose and was therefore regarded as *Cl. pseudo-tetanus*. For the pathogenicity test the strains were grown anaerobically in Holman's meat medium for 10 days at 37° C. and, after standing overnight, 0·3 ml. of the clear supernatant liquid was inoculated intramuscularly into the right thigh of white mice. Control tests were made with

white mice which had been given, one hour before receiving the same dose of culture fluid, 0·3 ml. of antitoxin containing 700 units per ml. Paralysis and death within 96 hours was the result recorded after injection with toxic strains without antitoxin. D. R. W.

Errata

September issue.

p. 671, 3rd line from bottom. In the sentence "toxins produced by members of the food-poisoning or dysentery groups" *omit* "toxins produced by."

October issue.

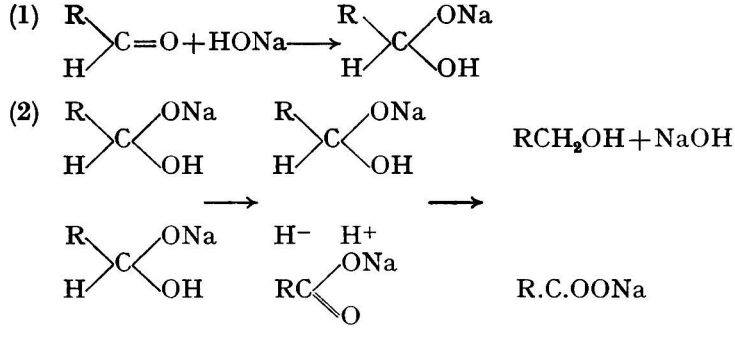
p. 753, line 3, *for* "50 to 90 bacteria per gram" *read* "50 to 90 millions."
,, ,, 5, *for* "both" *read* "broth."

Organic

Dehydration of Ether. N. Schoorl. (*Pharm. Weekblad.*, 1937, **74**, 1108–1109.)—Anhydrous sodium sulphate is not completely satisfactory for the dehydration of ether for extraction purposes. It is more efficient when the water is present in suspension as fine droplets (when it acquires 10 mol. of water of crystallisation) than when the water is present in solution (*cf. id.*, 1910, **47**, 963; and von Siebenrock, *Monatsh. f. Chem.*, 1909, **30**, 759). The use of ignited gypsum ($CaSO_4,\frac{1}{2}H_2O$), which is converted eventually into $CaSO_4,2H_2O$, has also been suggested, but the objection here is the formation of the intermediate hydrate $CaSO_4,1{\cdot}5H_2O$ which resists further absorption of water. Anhydrous magnesium sulphate is preferred, as it will remove about two-thirds of the water present. The inverse relative efficiencies of these three dehydrating agents, expressed in terms of the vapour tensions of the water in the respective hydrated salts, are approximately 0·76 : 0·94 : 0·21. Ether containing over 0·6 or over 0·35 per cent. of dissolved water gives a turbidity when shaken with twice its volume of carbon tetrachloride or carbon disulphide, respectively. These reagents give positive results with ether saturated with water (*i.e.* containing 1 per cent. of water) after it has been treated with sodium sulphate or calcium sulphate, but if magnesium sulphate has been used, no turbidity is produced when the amount of water has been reduced to about 0·35 per cent. J. G.

Catalysis of Cannizzaro's Reaction by Active Nickel and Platinum. Application to some Aldoses. M. Delépine and A. Horeau. (*Bull. Soc. Chim.*, 1937, **4**, 1524–34.)—Experiments with formaldehyde, butaldehyde, and benzaldehyde showed that these aldehydes, if shaken in solution with sodium hydroxide solution and Raney nickel, give Cannizzaro's reaction, and the corresponding acids and alcohols can be detected in the liquid. Extending the experiments to aldoses, the authors worked with galactose, glucose, and arabinose. In a typical experiment, 18 g. of galactose and 100 ml. of $N/2$ sodium hydroxide solution were shaken with 4·5 g. of nickel. The alkalinity of the liquid decreased rapidly, the volumes of $N/10$ sulphuric acid required to neutralise 1 ml. of the solution after a few seconds, 2 hours, 4 hours, and 6 hours being respectively

4·5 ml., 2·5 ml., 1·8 ml., and 1·25 ml. The solution gave 5 g. of dulcitol and (by treatment with cadmium chloride) 4·2 g. of cadmium galactonate. Glucose gave sorbitol and gluconic acid, and arabinose yielded arabitol and arabonic acid. When Vavon platinum was used as the catalyst, hydrogen was evolved, and the alcohol and acid corresponding to the sugar were formed. In one experiment, 18 g. of galactose, 1·5 g. of platinum, 100 g. of water, and 5 ml. of sodium hydroxide solution were shaken in an atmosphere of hydrogen; after 1 hour a further 5 ml. of alkali solution were added. One litre of hydrogen was evolved, and 14 g. of calcium galactonate and 2·2 g. of dulcitol were isolated from the liquid. If the quantity of platinum is reduced, a smaller volume of hydrogen is evolved and larger quantities of alcohol and acid are formed. The hydrogen formed during the reaction can be used to reduce an easily hydrogenisable substance added to the system, as was shown by shaking 18 g. of galactose, 7 g. of crotonic acid (as the sodium salt), 8 ml. of 10 *N* sodium hydroxide solution and sufficient water to bring the volume up to 250 ml., with 4·5 g. of nickel. After 8 hours 5 ml. of butyric acid and 14 g. of calcium galactonate were isolated. The following reactions are suggested in explanation of the phenomena.



If the hydrogen ions are not fixed with sufficient rapidity, molecular hydrogen is evolved. E. M. P.

Needle Shellac. E. Stoch. (*Färb.-Ztg.*, 1937, July 31st; *Chem. Trade J.*, 1937, **101**, 114.)—This product is prepared (*Belgian Pat.*, 413,775, 1936) by spinning the shellac into filaments, which are subsequently broken up into "needles." It contains only 2 per cent. of water, as compared with 25 and 5 per cent. in button- and in powder- or leaf-shellac, respectively, and it therefore keeps well without special precautions. Its large surface area per unit weight also ensures that it dissolves rapidly without agglomeration of the needle-clusters, even when very concentrated solutions are being made. The samples of ruby and bleached needle shellac examined gave a negative reaction for rosin and a positive Tschirch reaction; their analytical values were in close agreement with those obtained for the various grades in their usual commercial forms, the only exception being that the ash-contents of the needle shellacs were slightly higher. Examination of the fluorescence produced in ultra-violet light provides the best method of distinguishing needle shellac from the other grades, and the original paper (in the *Farb.-Ztg.*) gives the results so obtained with solutions in a wide range of organic solvents. J. G.

Polymerisation of Tobacco Seed Oil. M. Brambilla and G. Balbi. (*Chim. e Ind.*, 1937, **19**, 373–377.)—The recorded values for the composition of tobacco seed oil (*Nicotiana tabacum*) are as follows:—palmitic acid, 32 to 40 per cent.; oleic acid, 24·5 to 30 per cent.; linolic acid, 15 to 20 per cent.; unsaponifiable matter, 1 to 1·5 per cent. As in previous work on seed oils by the same authors (ANALYST, 1936, **61**, 716, 855) tobacco seed oil was polymerised at high temperatures (333° to 337° C.) and at lower temperatures (297° to 300° C.). The polymerisation products obtained at the higher temperatures are unsuitable for industrial application. The following table shows the effect of polymerisation at 297° to 300° C. on refined pressed and extracted oils:—

	Sp.gr. at 20° C.	n_D^{20}	Viscosity, η_{20}	Acid value	Iodine value	Colour (Hellige colorimeter)
Expressed oil	0·9220	1·4789	58·31	0·47	93·5	0·51
polymerised, 6 hours ..	0·9289	1·4842	1280·3	7·59	89·52	1·93
" 7 hours ..	0·9301	1·4864	2027·8	9·27	79·81	2·13
" 8 hours ..	0·9387	1·4883	3848·1	11·42	72·27	2·41
Extracted oil	0·9298	1·4782	53·29	0·88	94·5	0·68
polymerised, 6 hours ..	0·9349	1·4848	1213·4	7·33	91·14	2·33
" 7 hours ..	0·9370	1·4866	1990·0	9·04	80·74	5·50
" 8 hours ..	0·9403	1·4898	3793·1	11·33	74·31	7·44

The colour values were obtained by comparison with a standard potassium dichromate solution (3·863 g. in 1 litre of water); the figures in the table were calculated by dividing 100 by the thickness, in mm., of the oil layer.

Further experiments, in which acids and colloidal substances were added to the refined oils before polymerisation, showed that the colloidal substances present in crude oils are the cause of their comparatively smaller tendency to polymerise. The general conclusion drawn from the results of the experiments is that the polymerised oils obtained at the lower temperatures are semi-drying stand-oils suitable for use in varnishes; their low acidity is a particularly useful property.

E. M. P.

Colorimetric Determination of Carbon Disulphide in Gas or Motor Fuels. Chem. Dept. South Metropolitan Gas Co. (*J. Soc. Chem. Ind.*, 1937, **56**, 287–290T.)—A modification of the diethylamine test is based on the reaction between piperidine and carbon disulphide. Monochlorobenzene is substituted for alcohol as solvent, and cupric oleate for cupric acetate, since the latter is insoluble in chlorobenzene. The apparatus for measuring the colour consists of three compartments bored out of square oak blocks, the lowest containing a 6-volt 6-watt lamp mounted at the focus of a silver lacquered parabolic reflector to give an approximately parallel beam of light. A diaphragm between this chamber and the next restricts the beam, and on the diaphragm rests a crystallising dish, 7 cm. in diameter, in which a coloured salt may be used as a light-filter. Above this is an iris diaphragm on which the dish (with a flat base with an area of about 20 sq. cm.) containing the test liquid rests. Mounted in the hinged lid of the top compartment is the light-sensitive cell, connected with a 0–100 micro ammeter (resistance 100–150 ohms). Fifty ml. of copper sulphate solution containing 100 g.

of the pentahydrate per litre are placed in the filter-dish, and 25 ml. of heavy medicinal paraffin are floated on the surface to restrict evaporation. The coloured test solution is poured into its dish, and the same volume of a blank solution of the reagents free from carbon disulphide is put into a second dish of the same dimensions. The iris is closed and the lamp is lighted; after 3 minutes the dish and blank solution are placed centrally in the top compartment, and after 15 seconds the iris is adjusted to give a reading of exactly 100 microamperes. The test-dish is substituted for the blank and the reading is again taken after 15 seconds. The instrument is calibrated by a series of tests upon known weights of carbon disulphide from 0 to 1·5 mg. The solutions required are (1) piperidine in chlorobenzene: 20 ml. per litre; (2) cupric oleate in chlorobenzene: 2·5 g. per litre; (3) carbon disulphide in chlorobenzene: 0·2 g. per litre. Ten ml. of solution (1) are mixed with 5 ml. of solution (2) and a measured volume of solution (3) is added. The mixture is diluted to 25 ml. with chlorobenzene and the light transmission is measured. The colour obtained with a given weight of chlorobenzene should be measured within 30 minutes of its formation. A 1 per cent. solution of piperidine in chlorobenzene is satisfactory for the removal of carbon disulphide from gas, even when present in concentrations as low as 0·2 grain per 100 cb. ft., and gas containing an equivalent of 30 grains of sulphur per 100 cb. ft. when passed at a rate of 1 cb. ft. per hour through two piperidine washers in series gave practically no carbon disulphide in the second washer. In period tests the gas should be pre-dried with calcium chloride. Carbonyl sulphide gives a colour similar to that given by carbon disulphide, but it is much less stable; mercaptans do not seriously affect the test; thiophene is without effect, but hydrogen sulphide must be removed by a lead carbonate tower. Details for the determination of carbon disulphide in coal gas are given for a short period (15 minutes) test, and for long periods. For short period tests the mixture of solutions is placed in a dry gas washer of the boiling-tube type, the inlet tube terminating in a bulb with pinholes. The gas is passed through the apparatus at the rate of 1 cb. ft per hour and the amount of that leaving the outlet is recorded by a meter. To determine carbon disulphide in benzole and other hydrocarbon oils, a measured volume of oil is added to the mixed reagent (20 ml. of solution (1) and 5 ml. of solution (2)) and the colour is measured as described. D. G. H.

Colour Reaction of Woody Tissues with Chlorine and Sodium Sulphite. W. G. Campbell, S. A. Bryant and G. Swann. (*Biochem. J.*, 1937, **31**, 1285–1288.)—A qualitative study has been made of the well-known reaction of woody tissues with chlorine and sodium sulphite. The reaction, which is probably specific for phenolic bodies containing the 1 : 2 : 3-trihydroxybenzene nucleus such as tannic acid and pyrogallol, is given by hot and cold aqueous extracts of wood, by the acid filtrate from lignin determinations, and even by cold aqueous extracts of isolated sulphuric acid lignin itself. It is therefore probable that phenolic compounds, which may be either tannins or even a form of lignin, permeate the structure of certain hard woods and soft woods, and that these survive, in part at least, the conditions used in the isolation of lignin by means of conc. sulphuric acid. S. G. S.

Determination of Alkali in Wool. D. E. Stocker. (*J. Soc. Dyers and Col.*, 1937, **53**, 348–349.)—A weighed quantity of wool is dried and extracted continuously with absolute alcohol, any free caustic alkali being then determined by titrating the extract with 0·1 *N* hydrochloric acid, with phenolphthalein as indicator. The extract is then evaporated, and a solution of the residue in water is treated with acid, which decomposes any soap. The fatty acids from the soap may then be extracted with ether, the residue from the evaporation of this extract being dissolved in alcohol and the fatty acids determined by titration in the usual way. The extracted wool is soaked for several hours in a 1 per cent. solution of boric acid, and transferred with the solution to a small tap-funnel in which is a plug of glass wool. The stem of the funnel is inserted in the rubber bung of a suction filter-flask, and the liquid is drawn out. The tap of the funnel is closed, water is introduced, and, after thorough stirring, the tap is opened and again the liquid is drawn out. After ten such washings the united filtrates are titrated with 0·1 *N* hydrochloric acid, with methyl red or methyl orange as indicator. If the calcium soaps or dyestuffs are present, the boric acid solution and the wash-water should be saturated with salt, and the difficulty of titrating a coloured liquid does not arise. Other advantages are that boric acid has no action on the indicators used, that adsorption of the acid by the wool does not affect the results, and that sodium borate is easily removed from the wool by the washing process. The method is more easy to manipulate than the terephthalic acid method of Hirst and King (ANALYST, 1926, **51**, 212), and the necessity for a separate determination of calcium soaps, which are also decomposed by terephthalic acid and must therefore be allowed for, is eliminated. When 5·0 ml. of 0·1 *N* sodium carbonate solution were allowed to dry into 1·0 g. of purified wool, the quantities found (4 experiments) ranged from 4·7 to 4·9 ml. When purified wool was treated with 10 ml. of 0·2 *N* borax solution and extracted as described above, the amount recovered was equivalent to 9·9 ml. of 0·2 *N* hydrochloric acid. J. G.

Weighting of Indian Silk. C. R. N. Reddy and B. S. Srikantan. (*J. Indian Chem. Soc.*, 1937, **14**, 371–375.)—The conditions for weighting Indian silk (Eri silk and Mysore-Japanese varieties) have been ascertained. The weighed silks (0·5 g.) were immersed in 50 ml. of the weighting solution (lead acetate), washed, dried at 78° C. in an air-oven for 1 hour and weighed. The optimum *p*H of the bath was found to be 8·0; weighting occurs chiefly in the first half-hour, and prolonged treatment has a detrimental effect on the quality of the silk, rendering the appearance dull. Maximum weighting occurred when the lead acetate solution had sp.gr. 1·25 and a further increase in the concentration had no effect. The best results were obtained at 28·5° C., and higher temperatures affected both the tensile strength and appearance of the silk. Eri silk had a higher weighting capacity than Mysore-Japanese, but it acquired an undesirable deep colour and lost tensile strength. By washing after weighting and immersing for 5 minutes in a fixing bath of sodium hydrogen phosphate, washing again, and repeating the process until the fibres were adversely affected, it was found possible to weight a Mysore-Japanese silk to 105 per cent. D. G. H.

Inorganic

Colorimetric Determination of Copper as Ferrocyanide. H. Hahn, R. Juza and R. Langheim. (*Z. anal. Chem.*, 1937, **110**, 270–275.)—The flocculation of copper ferrocyanide is delayed by gelatin. The neutral copper solution is treated with 2 per cent. acetic acid (5 ml.), 10 per cent. ammonium acetate solution (5 ml.), 1 per cent. gelatin solution (10 ml.), and 0·2 per cent. potassium ferrocyanide solution (10 ml.), and diluted to 50 ml. The tint should be matched within an hour against water and the blue-green filter S50 of the Pulfrich photometer. Unduly large quantities of other electrolytes (alkali and ammonium salts) should not be present. A large excess of lead (1000 parts to 1 of copper) may be present without interfering, lead ferrocyanide being comparatively soluble in presence of excess of ammonium acetate. W. R. S.

Colorimetric Determination of Cadmium as Sulphide. R. Juza and R. Langheim. (*Z. anal. Chem.*, 1937, **110**, 262–270.)—The procedure utilises gelatin as a protective colloid in ammoniacal cyanide solution. The neutral solution is treated with 1 per cent. ammonia solution (1 ml.), 10 per cent. potassium cyanide solution (4 ml.), 10 per cent. ammonium sulphate solution (1 ml.) and 1 per cent. gelatin solution (1 ml.), diluted to 30 ml., and poured into a 50-ml. flask containing 5 ml. of saturated hydrogen sulphide water. The volume is adjusted, and the colour matched after 15 minutes' standing. This is done against water with a Zeiss photometer and the blue filter S43. A considerable amount of zinc in the solution (1000 parts to 1 of cadmium) does not interfere with the determination; the solution is titrated with the cyanide solution until the zinc precipitate re-dissolves. After addition of an excess of 4 ml. of cyanide solution, the determination is conducted as described above. If copper is present (maximum, 10 parts to 1 of cadmium), the ammoniacal solution is decolorised by cyanide, after which 4 ml. of cyanide solution are added. The determination can be made in presence of nickel (maximum, 100 parts) and cobalt (10 parts). An allowance may be necessary for the yellow tint of the nickel or cobalt cyanide solution containing the excess of cyanide prescribed above. W. R. S.

Determination of Platinum and Gold, and Detection of Platinum Metals. S. O. Thompson, F. E. Beamish and M. Scott. (*Ind. Eng. Chem., Anal. Ed.*, 1937, **9**, 420–422.)—The crystals formed by heating a slightly acid solution of platinum chloride with dimethylglyoxime on a steam-bath for several hours were found to be of fairly definite composition, corresponding closely with $PtC_8H_{14}N_4O_4$; the precipitation, however, was incomplete. The addition of an excess of dimethylglyoxime in alcoholic solution to a gold chloride solution acidified with hydrochloric acid gave a yellow precipitate. The mixture was boiled for 30 minutes, and the precipitate was filtered off, washed with water and finally ignited, giving metallic gold. The recovery of gold in this way was found to be quantitative. Various tests available for the detection of the platinum metals are described. New tests for osmium are as follows:—(1) An aqueous solution of pyrogallol produces a blue colour with a solution of sodium osmate. "If a drop of hydrochloric acid is added to the test solution followed by a drop of conc.

pyrogallol solution, a definite colour is obtained if the test solution contains 0·01 mg. of osmium per ml." Rhodium and iridium do not interfere, but palladium and platinum give brown colours, and gold gives a purple colour. (2) Ephedrine hydrochloride gives an orange colour with alkaline sodium osmate solution. The sensitiveness of the test is improved by extraction of the coloured compound with carbon tetrachloride; 0·01 mg. of osmium per ml. produces a faint colour. Platinum and rhodium give no colour. Palladium and gold give a very faint yellow colour, but the test is not sensitive for these metals, nor for iridium, which gives a faint green colour under the same conditions. S. G. C.

New Fluorescence Test for Aluminium. C. E. White and C. S. Lowe. (*Ind. Eng. Chem., Anal. Ed.*, 1937, **9**, 430–431.)—The orange-red fluorescence produced under ultra-violet light by Pontachrome Blue Black R dye (the zinc salt of 4-sulpho-2,2′-dihydroxy-azonaphthalene) with aluminium is a highly sensitive test and can be used to detect aluminium in the presence of beryllium. The solution to be tested should first be made alkaline with sodium hydroxide, any precipitate formed being filtered off and rejected. Sexavalent chromium must be reduced and fluorides must be removed. The solution is then acidified with acetic acid, and 0·5 ml. of a 0·1 per cent. solution of the dye is added. With solutions of aluminium as dilute as 1 p.p.m., half-an-hour should be allowed for the fluorescence to develop. The fluorescence is preferably observed in a dark room under a quartz mercury vapour lamp equipped with a Wood's glass filter. The maximum sensitiveness of the test under these conditions was found to be 0·2 p.p.m. of aluminium, but with this extreme dilution it was necessary to keep the solution overnight for the fluorescence to develop. Quartz containers for the test solution had no advantage over those of soft glass. With argon bulbs as a source of ultra-violet radiation, the sensitiveness of the test was much less, being 10 p.p.m. of aluminium. Highly coloured ions, such as copper, chromium, iron, nickel and cobalt, masked the fluorescence, but precipitation with sodium hydroxide as described above, removed the interference. Fluoride interferes, but may be removed satisfactorily by precipitation as calcium fluoride. The following ions (amounts not stated) did not interfere in the test: beryllium, gallium, indium, thallium, silver, mercury, lead, bismuth, cadmium, arsenic, antimony, tin, zinc, calcium, strontium, barium, magnesium, sodium, potassium, ammonium, lithium, rare earths, chloride, nitrate, sulphate, phosphate, tartrate. S. G. C.

Determination of Gallium in Germanite. F. Sebba and W. Pugh. (*J. Chem. Soc.*, 1937, 1371–1373.)—Previous analyses of germanite (*e.g.* ANALYST, 1925, **50**, 91) involved decomposition with acids and have given gallium results which the authors consider low. They decompose the finely powdered ore (5 g.) by fusion with sodium hydroxide (10 g.) in an iron crucible. The cooled melt is extracted with water, and the residue is collected, washed with water, and rejected. The filtrate contains the germanium, arsenic, and a little molybdenum as thio-compounds, as well as the whole of the gallium; it is treated with sufficient hydrochloric acid to produce 2 *N* acidity, and boiled for the coagulation of the sulphide precipitate with simultaneous solution of a little adsorbed gallium; there is sufficient sulphide in the fusion extract to remove all the arsenic at this stage. The precipitate

is collected, and the filtrate is neutralised to methyl red with sodium hydroxide. The precipitated gallium hydroxide is collected, dissolved in hydrochloric acid, and the solution is evaporated to dryness to eliminate traces of germanium and dehydrate silicic and tungstic acids. The filtered solution still contains a trace of lead and molybdenum which are removed by another treatment with alkali sulphide and acidification, with intervening filtration to eliminate lead sulphide. Filtration of the acidified liquid eliminates molybdenum sulphide. The gallium is precipitated in the filtrate by means of cupferron (*cf.* ANALYST, 1933, **58**, 111). The procedures described above gave concordant results of 1·2 per cent. Ga.

W. R. S.

Gravimetric Determination of Lithium as Aluminate. H. Grothe and W. Savelsberg. (*Z. anal. Chem.*, 1937, **110**, 81–94.)—The procedures based on precipitation as fluoride, phosphate, and lithium zinc uranyl acetate were re-investigated and found to be unreliable except in certain special cases, and hence not adapted for general work. A new method was worked out in which the lithium is precipitated as aluminate (solubility 0·09 g. per litre), which is ignited and weighed as $2Li_2O{\cdot}5Al_2O_3$. Lithium factor 0·0488. Dobbin and Sanders have proposed the use of the same compound for the determination of aluminium (*J. Amer. Chem. Soc.*, 1932, **54**, 178; Abst., ANALYST, 1932, **57**, 197).

The reagent is prepared from 50 g. of potash alum dissolved in 900 ml. of warm water. The cooled solution is stirred and treated with a strong solution of 20 g. of sodium hydroxide until the precipitate re-dissolves completely. After standing overnight the liquid is filtered and diluted to 1 litre, the *p*H having previously been adjusted to 12·6. This is ascertained electrometrically by measurement of the potential of an antimony electrode against a calomel electrode. A close adjustment of the *p*H concentration is necessary for the complete precipitation of the lithium without co-precipitation of aluminium hydroxide.

The lithium solution is adjusted to *p*H 3 with the aid of colour indicators, treated in the cold with 40 ml. of reagent per 10 mg. of lithium, re-adjusted electrometrically to *p*H = 12·6 by means of *N* sodium hydroxide, and filtered after a short time. The precipitate is washed with cold water, by decantation first, until the washings no longer redden phenolphthalein, ignited, and weighed. The results of six test analyses show errors varying from + 0·52 to — 1·04 per cent., with a mean error of — 0·27 per cent.

W. R. S.

Elimination of Phosphate in Qualitative Analysis. L. W. N. Godward and A. M. Ward. (*J. Chem. Soc.*, 1937, 1337–1338.)—The scheme is based on the precipitation of ammonium phosphomolybdate. The filtrate from the hydrogen-sulphide group is boiled with nitric acid, and a small portion is tested for phosphoric acid by the molybdate reaction. Another portion is treated with ammonia; if no precipitate is formed, or a precipitate soluble in excess, the solution is treated with ammonium sulphide for the zinc group. If a permanent precipitate is formed with ammonia, the bulk of the filtrate is treated with a little sulphuric acid and 1 g. of ammonium sulphate, and any sulphate precipitate is collected and tested for barium and strontium. The filtrate is diluted to 25 ml. and treated with 5 ml. of strong nitric acid and 5 g. of ammonium nitrate. An excess of solid

ammonium molybdate (1·7 g. for 0·1 g. P_2O_5) is stirred into the boiling solution, and boiling is continued for 5 minutes. • The solution is filtered, and the filtrate is tested for complete precipitation. After cooling, a very slight excess of strong ammonia is added. Any precipitate is collected and tested for iron, aluminium and chromium; the filtrate is tested with ammonium sulphide for the zinc group. It remains to test the filtrate from the zinc group for calcium and magnesium. The liquid is acidified and boiled, and the molybdenum sulphide is filtered off. The filtrate is boiled with sodium perborate and hydroxide until colourless, after which boiling is continued to the destruction of the perborate. The liquid is then acidified with hydrochloric acid, boiled, filtered, made ammoniacal, treated with 5 g. of ammonium acetate and 10 ml. of 6 per cent. ammonium molybdate solution in dilute ammonia, and boiled gently for 10 minutes. The white precipitate is calcium molybdate, which is confirmed by solution in dilute sulphuric acid and precipitation with ammonium oxalate. The filtrate from the calcium molybdate is treated with a solution of *p*-nitrobenzene-azoresorcinol, followed by sodium hydroxide until the colour of the dye changes; a blue gelatinous precipitate indicates magnesium. W. R. S.

Volumetric Determination of Selenium by the Norris and Fay Method. W. C. Coleman and C. R. McCrosky. (*Ind. Eng. Chem., Anal. Ed.*, 1937, **9**, 431–432.)—The method originally due to Norris and Fay (*Amer. Chem. J.*, 1896, **18**, 705; Abst., ANALYST, 1897, **22**, 82) involves the titration of selenious acid with sodium thiosulphate, the reaction proceeding according to the equation:

$$4Na_2S_2O_3 + H_2SeO_3 + 4HCl \rightarrow Na_2SeS_4O_6 + Na_2S_4O_6 + 4NaCl + 3H_2O.$$

The procedure, as slightly modified by the present authors, is as follows: To the selenious acid solution (150 ml.) containing the equivalent of 5 ml. of 6 *N* hydrochloric acid, some starch indicator solution is added, and the liquid is titrated with standard sodium thiosulphate solution (0·1 *N*) added in 5-ml. portions; a few drops of potassium iodide solution (2·5 per cent.) are added after each 5-ml. addition of thiosulphate solution, and the titration is stopped when the potassium iodide addition no longer produces a local starch-iodide blue. The solution is then overtitrated, an excess of not more than 5 ml. of thiosulphate being present. The excess of thiosulphate is back-titrated with standard iodine solution until a blue colour is produced. The thiosulphate solution may be standardised against elemental selenium as follows:—The selenium (0·35 to 0·45 g.) is dissolved in 5 ml. of 10 per cent. sodium cyanide solution by heating, the solution is diluted to 250 ml., and a 50-ml. aliquot portion is evaporated to 20 ml. Two ml. of a filtered 1 per cent. solution of gum arabic and 5 ml. of 5 *N* hydrobromic acid are added. A precipitate of red selenium is thus produced, and is kept colloidally dispersed by the gum arabic. A rapid stream of air is bubbled through the solution for 15 minutes to remove most of the hydrocyanic acid. Most of the selenium is oxidised by adding, drop by drop, a saturated solution of potassium bromate. When only a little red selenium remains, 0·1 *N* potassium bromate solution is added, the disappearance of the red colour marking the completion of the oxidation to selenious acid. A few drops of a saturated alcoholic solution of acetanilide are added to discharge any excess of bromate, and the liquid is heated to boiling.

After cooling, the solution is diluted to 150 ml., and the selenious acid is titrated as described above. The accuracy of the titration process on the basis of the stoichiometric reaction is within 1 part in 1000. The temperature of the solution titrated may vary from 0° to 20° C., but it is essential that the excess of thiosulphate used be limited in the manner indicated, as a large excess introduces an error, owing to its decomposition in the acid solution. Hydrobromic, sulphuric or perchloric acid may be used instead of hydrochloric acid in the titration liquid; nitric acid, acetic acid and phosphoric acid were found to be unsatisfactory. Small amounts of selenium may be determined by the use of 0·01 *N* or 0·001 *N* thiosulphate solution, the only modification being that a 25 per cent. solution of potassium iodide is employed instead of the 2·5 per cent. solution as indicator.

S. G. C.

Microchemical

Systematic Semi-micro Procedure for the Qualitative Analysis of the Commoner Cations. J. H. Winkley, L. K. Yanowski and W. A. Hynes. (*Mikrochem.*, 1936, **21**, 102–116.)—A scheme of analysis is given for the following cations: silver, lead, mercury, bismuth, copper, cadmium, arsenic, antimony, tin, iron, chromium, aluminium, zinc, manganese, calcium, barium, strontium, magnesium, potassium, sodium, lithium and ammonium. Spot tests are used directly, and not merely as confirmatory tests, but not for the members of the alkaline earth and alkali groups. The solutions used contained sufficient amounts of the nitrates and chlorides to furnish concentrations of 10 mg. of the cations per ml. The maximum initial volume of solution employed in any analysis was never more than 0·2 ml.; the amount of any cation present in the solution never exceeded 2 mg. A complete list of the concentrations of test substances and reagent solutions is given. The reagents are those found to give best results with relatively inexperienced workers.

J. W. M.

Micro-determination of Chloride in Biological Fluids with Solid Silver Iodate. J. Sendroy. (I) Gasometric Analysis. (*J. Biol. Chem.*, **1937, 120**, 335–403.)—Small amounts of chlorine in biological fluids may be determined by the following general procedure:—The sample is diluted in an acid solution (usually with 0·085 *M* phosphoric acid) to a *p*H between 2·0 and 3·0 and to a chloride concentration between 0·012 and 0·003 *M*. Solid silver iodate is then added, with vigorous shaking, which causes the precipitation of the chloride and the release of iodate into the solution. The precipitate of silver chloride and the excess of silver iodate are removed from the solution by centrifuging. The supernatant liquid is then analysed gasometrically for iodate by means of an alkaline hydrazine solution. For details of the method and for the calculation of the results, the original paper should be consulted.

(II) Titrimetric Analysis. (*Ibid.*, 405–417.)—The preparation of the sample is the same as that described above. An aliquot portion of the supernatant liquid, after centrifuging, is treated with potassium iodide, and the liberated iodine is titrated with 0·03 *N* sodium thiosulphate solution, starch paste being used as indicator.

S. G. S.

Tests on Photographic Paper. I. M. Korenman. (*Mikrochem.*, 1936, **21**, 17–20.)—Glossy or semi-matt silver-bromide photographic paper is treated with 10 per cent. sodium thiosulphate to remove the silver salts, and well washed, after which the moist paper is immersed in one of the following reagent solutions and dried. *Solutions.*—(1) For the detection of ferric iron and copper: 10 per cent. solution of potassium ferrocyanide. (2) For the detection of nickel: saturated alcoholic solution of dimethyl glyoxime. (3) For the detection of stannous tin: 1 per cent. gold chloride solution; the test-paper is coloured pale yellow. (4) For the detection of sulphur: saturated solution of lead acetate. (5) For the detection of gold: dilute stannous chloride and pyrogallol solution. (6) For the detection of nitrite: Griess's reagent. *Method.*—A drop of the test solution (0·25 c.mm.) is placed on the reagent paper, where it makes a stain 1 to 1·5 mm. in diameter. When the correct ion is present, the stain rapidly takes on the colour of the reaction product. Although such very small amounts of substance are used, no microscope is necessary, as with reactions in threads. The limits of identification and concentration compared with those of the same test on filter-paper are given below; the reagent solutions are those given above:—

		On filter-paper		On photographic paper	
Ion	Reagent	Limit of identification	Concentration limit	Limit of identification	Concentration limit
$Fe^{\cdots}$ ..	1	1γ	1 : 20000	0·0008γ	1 : 300000
$Cu^{\cdot\cdot}$ ..	1	2γ	1 : 10000	0·0025γ	1 : 100000
$Ni^{\cdot\cdot}$ ..	2	0·025γ	1 : 800000	0·0003γ	1 : 800000
S'' ..	4	1·8γ	1 : 11000	0·005γ	1 : 50000
NO_2' ..	6	0·015γ	1 : 1300000	0·0002γ	1 : 1300000
		In threads			
$Sn^{\cdot\cdot}$ ..	3	0·003γ (cotton)	—	0·007γ	1 : 36000
$Au^{\cdots}$ ..	5	0·002γ (silk)	—	0·016γ	1 : 15600

The nitrite test may be applied to detect nitrous oxide in the atmosphere; as little as 0·2γ per litre may be detected. J. W. M.

Micro-determination of Blood Sugar by Ceric Sulphate Titration. G. Giragossintz, C. Davidson and P. L. Kirk. (*Mikrochem.*, 1936, **21**, 21–34.)—Ferricyanide in alkaline solution is used to oxidise the sugar, and the resulting ferrocyanide is titrated in acid solution with standard ceric sulphate, alphazurine G.G. or phenanthroline ferrous complex being used as indicator. The excess of ferricyanide has no influence on the results and the titration of the ferrocyanide proceeds smoothly to a sharp and reproducible end-point. *Reagent Solutions.*—14 per cent. sodium carbonate solution, made from the anhydrous salt; 3–5 *M* sulphuric acid solution; 0·8 per cent. potassium ferricyanide solution. Standard ceric sulphate solution, preferably about 0·0025 *N*, prepared by the method of Willard and Young (*J. Amer. Chem. Soc.*, 1929, **51**, 149). It is standardised against a weighed sample of potassium ferrocyanide which has been recrystallised and dried. Standard ceric sulphate solution must be kept in a glass bottle, free from organic contamination. *Indicator.*—Either 0·4 per cent. Alphazurine G.G., or 0·025 *M* phenanthroline ferrous complex. *Method.*—The blood is deproteinised

in the usual way. Either 2 ml. of the blood filtrate which has been diluted to 1 : 10, or 1 ml. if the dilution was 1 : 5, is pipetted into a large test-tube. To this are added 2 ml. of the ferricyanide solution and 2 ml. of the sodium carbonate solution. A blank solution is also prepared with distilled water in place of the blood filtrate. The tubes are shaken, heated for about 5 minutes on the water-bath, and then cooled, 2 ml. of sulphuric acid are added, followed by a drop of indicator, and the solutions are titrated with the standard ceric sulphate to the end-point, which is a sharp change from yellow to brown with Alphazurine G.G., or from orange to green with phenanthroline. A 10-ml. burette calibrated in divisions of 0·02 ml. is convenient for the titration when 0·0025 *N* ceric sulphate solution is used. Over the entire range investigated, the same factor holds good, *viz.* 1 mg. of glucose ≡ 2·735 ml. of 0·01 *N* ceric sulphate solution. The method has been tested in comparison with other methods, and is claimed to be simpler, more readily reproducible and more rapid than those tried, and to give the same value for glucose. J. W. M.

Differentiation of Chromate and Dichromate Ions. M. G. Malko, L. K. Yanowski and W. A. Hynes. (*Mikrochem.*, 1936, **21**, 57–60.)—Hexamminocobaltic chloride will distinguish between chromate and dichromate ions microscopically in the absence of interfering ions, such as metavanadate, ferrocyanide, ferricyanide, tungstate, molybdate, tetrathionate, ortho- and pyro-phosphate ions. The reagent is a solution of the salt containing 28 g. per litre. Precipitation is carried out at room temperature by mixing one drop of the test solution with two drops of reagent on a slide. With the chromate ion alone, lemon-yellow needles are formed which attain the size of 1 cm. or more, whilst with the dichromate ion the crystals are orange-yellow and microscopic in size, showing a tree-like branching of prisms from a common stalk. Neither crystals resemble those formed on evaporating the reagent. The formula of the chromate precipitate is $(Co[NH_3]_6)(CrO_4)Cl$, and that of the dichromate compound $(Co[NH_3]_6)_2(Cr_2O_7)_3$. On recrystallisation the chlorochromate gives long golden-yellow needles, the dichromate light yellow six-sided or square platelets. With mixtures of chromate and dichromate ions there is a tendency for the precipitation to be retarded and for the chromate needles to be shorter. The proportion limits of the detection of the anions in the presence of each other is $20Cr_2O_7 : 1CrO_4$ and $2{\cdot}5CrO_4 : 1Cr_2O_7$. Two photomicrographs and 4 drawings are given. J. W. M.

Use of Complex Salts for the Detection of Anions. I. Hexamminocobaltic Chloride. W. A. Hynes and L. K. Yanowski. (*Mikrochem.*, 1937, **23**, 1–9.)—By the use of the reagent and procedure described in the preceding abstract the following ions were found to give characteristic crystalline reaction products when treated with hexammino-cobaltic chloride: bifluoride, bisulphate, bisulphite, chromate, cobaltinitrite, dichromate, dithionate, ferricyanide, ferrocyanide, fluosilicate, iodate, iodide, metavanadate, nitroprusside, orthovanadate, permanganate, persulphate, phosphomolybdate, phosphotungstate, pyrophosphate, sulphate, sulphosalicylate, tartrate, tellurite and thiosulphate. Of these, the following were found to give rapid qualitative tests: bisulphite, chromate, dichromate, dithionate, ferricyanide, ferrocyanide, iodate, permanganate, sulphate,

sulphosalicylate and thiosulphate. The cation present in the test substance apparently causes no difference in the crystalline form of the reaction product obtained. The anions react in much the same manner on treatment with the reagent, regardless of whether they are in the form of their salts or occur in solutions containing several anions. Twenty-four photomicrographs are given. J. W. M.

Colorimetric Micro-determination of Manganese. C. P. Sideris. (*Ind. Eng. Chem., Anal. Ed.*, 1937, **9**, 445–446.)—Small amounts of manganese (1 to 20γ in 10 ml.) may be determined by the formaldoxime reagent of Denigès (*Compt. rend.*, 1932, **194**, 895). The reagent is prepared by dissolving 20 g. of trioxymethylene and 47 g. of hydroxylamine sulphate in 100 ml. of water, by heating. To the slightly acid manganese solution (10 ml.) about 5 drops of 40 per cent. potassium hydroxide solution and 3 drops of the formaldoxime reagent (*cf.* ANALYST, 1934, **59**, 200) are added. A wine-red colour immediately develops, and may be compared colorimetrically with that given by a standard manganese solution treated similarly. The amount of manganese in the unknown and standard solutions should be closely similar for accurate results. The addition of a suitable colloid (0·5 ml. of a 5 per cent. solution of gum Ghatti) to the solution prior to the development of the colour is advantageous. Ferric iron, even in traces, interferes by producing a similar red colour. This colour may be prevented from forming by the addition of 0·5 ml. of 20 per cent. sodium cyanide solution to the liquid before adding the formaldoxime. This results, however, in a greenish-yellow colour being formed by the iron, which also interferes, but to a lesser degree. It is necessary, therefore, that the amount of iron present in association with the manganese be determined and added in equal amount to the standard comparison solution. S. G. C.

Reviews

COLLOID CHEMISTRY. By JEROME ALEXANDER, M.Sc. Pp. xviii + 505. London: Chapman & Hall, Ltd. 1937. Price 22s. net.

This is the fourth edition of Mr. Alexander's little book, which first appeared in 1918; it has since been greatly enlarged. The Preface prepares the reader for unusual features. "The author has adhered to the principles which governed the preparation of its predecessors: (1) assemblage of experimental data into naturally co-ordinated and interlocking groups, to form a broad mosaic which gives a coherent picture of nature; (2) breaking down the artificial mental barriers arising from scientific specialisation and its incidental babel of scientific jargon, so that the resources of many fields of investigation may be considered and both breadth and depth of mental focus acquired."

"The book is built around no one theory." It is so arranged that emphasis is placed on observable facts, the theoretical considerations being incidentally invoked for a better appreciation. The author claims that his book should appeal to the student and to the general reader. "The attempt is made to humanise

the bare skeleton of scientific principles by using numerous practical illustrations, so that interest may aid memory and knowledge be painlessly acquired."

There is the Sunday School anecdote (p. 110); the French at Verdun: "They shall not pass!" (p. 121); the church raffle (p. 123); collapsed saponin foam films remain after bubble collapse, "like the grin of the Cheshire cat" (p. 75); ten million cornets playing *fortissimo* emit one horse-power of sound (p. 43); "grandma's copper preserving kettle" (p. 50); the "peck of dirt" we acquire in our lifetime (p. 50); these, and many others, certainly give this book a peculiar interest. When also one realises that zip fasteners, relativity, quantum mechanics and chain reactions all meet with the same racy treatment, and the author speaks of "slow movies" or "water won't wet oil," the difficulty of properly assessing the merit and utility of the book becomes obvious.

The twenty-four chapters embrace an astonishing range of subjects, and references are frequent to the author's well-known four volumes on Colloid Chemistry. Indeed, in a sense, the present volume is an epitome of the larger volumes which Mr. Alexander edited. There is a chapter on genetics, one on hormones, another on physiology and pathology, and one on bio-electricity. Some sections, for example that on meteorology, are unusually arresting to the reader.

Among several errors noted are, on p. 213, the "British" Mayonnaise Manufacturers' Association should read "American"; "dynes per sq. cm." (p. 103) is unpardonable; Pbs (p. 80); Jordan and Falk's book is wrongly titled (p. 128).

The reviewer has read this book with very real pleasure and interest, but he cannot recommend it as a *text-book* for students. The treatment is far too cursory—in fact, scrappy, on the basic principles. To anyone already familiar with a modern text-book on the subject, the volume can be recommended as very entertaining reading, and as a guide to more thorough sources of information on the many aspects of knowledge which involve colloid science in their fuller development.

WILLIAM CLAYTON

REAGENT CHEMICALS AND STANDARDS. By JOSEPH ROSIN. Pp. x + 530. London: Chapman & Hall. 1937. Price 30s. net.

This book contains methods for testing and assaying 474 different reagent chemicals, together with standards and limits of impurities. The author is chief chemist to Merck & Co. in the United States, and is thus well qualified to write on this subject. It is certainly the most comprehensive work on chemical reagents that has yet been published. The monographs are arranged in alphabetical order, and under each heading are given the formula, molecular weight, theoretical percentages of the constituent elements or groups, appearance and solubility. Then follow the standards, methods of assaying, and of testing for impurities. All the descriptions are highly compressed, and everything has been done to save type. As an illustration of the style we may quote from potassium bromide. "Sulfate: Dissolve 4 g in 25 cc of H_2O, add 1 cc of 1 *N* HCl and 2 cc of $BaCl_2$. Any resulting turbidity is not more than in a blank to which 0·2 mg of SO_4 has been added."

The book is, in fact, a laboratory methods book containing the essential

details for carrying out the tests expressed in the shortest possible form. Its practical value is, of course, not less for this reason. One very useful feature is the inclusion of the logarithms beside all chemical factors.

Free use has been made of the tests and standards prescribed by the Committee on Analytical Reagents of the American Chemical Society, though these have been improved in many instances.

The mass of material contained in the book makes it impossible to criticise it in detail, but the following are a few points which have been noted. There is no test for fluorides in phosphates—a notable omission. There is no mention of lead as an impurity in iron salts, though it is often present in serious amount. It is not clear on what principle arsenic tests are included. There are none, for example, for citrates, tartrates, or any iron salts except ferric chloride. It is rather surprising to find that the tests for reagent chloroform are less stringent than those of the British Pharmacopoeia.

At the end of the book are 38 pages containing in considerable detail methods for the preparation of volumetric solutions, and a most comprehensive list of equivalents and their logarithms. There is also a useful section on pH determinations.

Fortunately in these days it is rarely necessary for the analyst to test his own reagents. Nevertheless there are occasions when it is essential to do so, and at such times this book will be found extremely useful. It is also of great value as an indication of what impurities are likely to occur in individual chemicals. The printing is admirable, and not a single misprint has been detected.

NORMAN EVERS

DAS WASSERSTOFFPEROXYD UND DIE PERVERBINDUNGEN. By W. MACHU. Pp. 408. Vienna: Julius Springer. 1937. Price RM.39.

In this volume are collected details of the preparation, properties, chemistry and uses of hydrogen peroxide, the metallic peroxides, the organic peroxides, the per-acids and the per-salts. These substances are finding increased application in industry, medicine and the household, and in this connection the voluminous data collected should be very valuable.

For over 100 years the only process for preparing hydrogen peroxide was from barium peroxide, the product being an impure solution with a maximum strength of about 5 per cent. This process is still carried on, but exists only because there is a market for the by-product, barium sulphate. At the present time 80 per cent. of the supply is made by three electrical processes, the usual strength of the product being 30 per cent. (100 volumes), but it may be higher. During the last twenty years the employment of distillation processes for concentrating dilute solutions of hydrogen peroxide up to the anhydrous article has yielded products of such strength and purity that the chemistry of the peroxides has been developed in a way impossible when hydrogen peroxide was only known as an impure solution. During the same period the development of electrochemical processes has brought the per-compounds from curiosities to household articles.

From many points of view molecular oxygen may be regarded as an

unsaturated unit capable of forming addition products. The —O—O— linkage may be saturated with hydrogen, with metals or with organic radicals. In the oxidation of organic substances with oxygen, peroxides are always formed as intermediate products, although they may not always be stable. This is important in determining the mechanism of biological processes involving oxidation. An example, which is perhaps outside the purview of this book, is the production of aroma in butter, which is due to peroxides; and whether or not the diacetyl and acetyl-methyl-carbinol are produced, as has been suggested, from citric acid, their formation appears to be closely associated with the presence of oleic peroxide. The use of anti-oxidants is increasing, and although many of them are of a non-reducing character, it is probable that they are capable of removing or decomposing peroxides under the particular conditions obtaining at the moment of their formation. Oxidases, peroxidases and catalases, which also have the power of decomposing peroxides, are widespread in nature.

Organic peroxides are important not only to oxidation processes in which they are essential intermediates, but also to many reactions promoted by their formation or decomposition. As a result of recent work, subsequent to the data here recorded, it has been shown that peroxides may control the velocity and orientation of reaction between hydrogen bromide and olefines, and the exceptions to Markownikoff's rules, which have been a mystery since 1870, are now largely explained. It has also recently been shown that in the absence of peroxides, benzaldehyde does not undergo the Cannizzaro reaction.

The book is well printed but, unfortunately, is uncut. Over 100 feet of paper have to be slit piecemeal before all the letterpress is available. A few minor misprints have been noted, such as "benzoperoxyd" (p. 80), "koovalenten" (p. 81), and "benzyl" for "benzoyl" on two occasions (p. 394); an error occurs in the lettering of the figure on p. 173. There are few English or American references since 1930, and as a result some recent work finds no place. The suggestion to use organic solvents for dissolving such organic peroxides as are insoluble in water prior to iodimetric determination is not of general application, for in some instances the solvent is oxidised.

The value of the book to certain technical specialists is undoubted. But the pure organic chemist or biochemist may find it of increasing and possibly unsuspected interest; for it may fairly be said that the part played by peroxides and allied compounds, either directly or indirectly, in many reactions is only beginning to be appreciated.

J. R. NICHOLLS