# THE ANALYST.

# PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

# ORDINARY MEETING, MARCH 3, 1920.

HELD at the Chemical Society's Rooms, Burlington House, Mr. Alfred Smetham, President, in the chair.

Certificates were read for the first time in favour of Mr. Laurence Harry Mills, B.Sc. (Birmingham), A.I.C., and Dr. Frederick Robertson Dodd, F.I.C.

Certificates were read for the second time in favour of Messrs. Henry Atlas, John Carmichael, F.I.C., Herbert John Evans, B.Sc., F.I.C., Cornelius Durham Garbutt, Douglas Henville, F.I.C., Maurice S. Hutchinson, B.Sc., Benjamin Richards James, F.I.C., Geoffrey Martin, D.Sc., Ph.D., F.I.C., Robert Leitch Morris, F.I.C., and James Wood, M.A., B.Sc., F.I.C.

Mr. William John Read, M.Sc., F.I.C., was elected a Member of the Society.

The following papers were read: "The Detection of Finger-prints on Documents," by C. Ainsworth Mitchell, M.A., F.I.C.; "Photo-micography with Simple Apparatus," by T. J. Ward; and "Note on the Solubilities of Theobromine," by Raymond V. Wadsworth.

**P P P P** 

#### ESTIMATION OF FIBRE IN FEEDING STUFFS.

A conference was held in London on December 8, 1919, at which representatives of the Agricultural Education Association and the Society of Public Analysts and other Analytical Chemists discussed the estimation of fibre in feeding stuffs. The Agricultural Education Association was represented by Messrs. J. Golding, H. T. Cranfield, C. Crowther, B. F. Davis, F. V. Dulton, W. Godden, H. A. D. Neville, and G. S. Robertson, and the Society of Public Analysts and other Analytical Chemists by Dr. S. Rideal (Chairman of the Conference), Messrs. E. R. Bolton, B. Dyer, J. Hughes, E. W. Voelcker, and J. A. Voelcker.

Although no definite decisions were arrived at yet the proceedings were not without value, and the matter is still open for further discussion. The Secretaries of the two bodies concerned are collaborating with the object of arranging a scheme of investigation which may be carried out under the ægis of the Society's "Analytical Investigation Scheme."

## THE DETECTION OF FINGER-PRINTS ON DOCUMENTS.

By C. AINSWORTH MITCHELL, M.A., F.I.C.

(Read at the Meeting, March 3, 1920.)

THE study of finger-prints, which began as a scientific investigation, has developed in practice into the most trustworthy method of identifying human beings. Although the general adoption of the system by the Police Authorities all over the world has had the effect of bringing the official methods of comparison, measurement, and classification of finger-prints to a high pitch of efficiency, it has, at the same time, tended to stereotype those methods, and to discourage experiment in other directions. The result has been that many questions connected with the subject have been left.



Fig. 1.—Prints of Sir William Herschel's Right Thumb in 1861 and 1914.

to be solved by independent workers, and this applies more particularly to the detection of latent prints on documents.

So much has been published upon the individuality of finger-prints and of the methods of comparing an unknown mark with a known finger-print, that it is hardly necessary at this stage to do more than refer to the bibliography at the end of this paper. I might, however, mention the accurate method of comparison devised by Mr. A. S. Osborn, of New York, in which the resemblances between two prints are clearly shown by means of photographs beneath little squares accurately ruled upon glass.

The Kew micrometer, as suggested by Dr. Faulds, affords an accurate means of measuring the lines and position of the pores in finger-prints, and also is a most useful instrument for many other purposes in the laboratory.

Another point which may be mentioned here is the more recent evidence of the

persistence of the characteristics of the papillary ridges on the fingers of an individual throughout a long life.

I am indebted to Sir William Herschel for permission to reproduce these prints (Fig. 1), one of which was made in 1861, and the other sent to me by him in 1914, with a repeat in 1916, not long before his death. Apart from the weathering of the ridges, due to age, the patterns of the two prints agree in all essential details.

It was Sir William Herschel who first introduced the use of finger-prints as a safeguard upon documents, but the first investigation of the subject in all its bearings, and especially of accidental finger-prints as a means of identifying an unknown individual, was made by Dr. Henry Faulds, whose work has been somewhat strangely neglected. His experience affords another instance of two men, unknown to each other, studying a subject from different points of view; and so when Faulds published his work he had the misfortune to find that the main idea had been anticipated.

In the course of his investigation Faulds paid considerable attention to the special branch of the subject, which forms the subject of this paper, and suggested the use of certain reagents, to which I shall refer presently.

The points upon which I found that there was little information available were: (1) Which is the most suitable method for detecting latent finger-prints upon paper, such as that of a book, under varying conditions? (2) After what period of time is it possible to detect such prints upon different qualities of paper by the different methods?

The nearly invisible marks which are made even by heavy pressure of fingers upon, say, the leaves of a book cannot, as a rule, be shown up directly by photography with oblique illumination, as is not infrequently the case with the prints made upon polished furniture or the like, and some method of development is therefore necessary.

#### MECHANICAL DEVELOPMENT WITH POWDERED SUBSTANCES.

The official English method of intensifying marks made upon various objects, such as glass bottles, window-panes, woodwork, etc., is to dust the surface very lightly with a light or dark powder, according to the conditions, and to remove the excess of powder by means of a soft brush. A mixture of chalk and mercury, the "grey powder" of the pharmacist, is used for dusting dark surfaces, whilst finely-powdered graphite is used for light-coloured surfaces (E. R. Henry, "Classification and Uses of Finger-Prints," p. 106).

Faulds found that a mixture of fine graphite and lampblack sold by Conté gave excellent results (H. Faulds, "Guide to Finger-Print Identification," p. 65). Other powdered substances used for the purpose are fine ferric oxide, magnesium carbonate, and aniline dyestuffs. For example, Stokis ("Annales Soc. Méd. Leg. Belge," 1906, p. 7), recommends the use of lycopodium powder with 10 per cent. of Soudan Red III., which is dusted on through a fine metal sieve, and then shaken off the paper.

Locard (*La Poroscopie*, p. 11) uses finely divided red lead, previously heated, for light surfaces, and lead carbonate for dark surfaces. He has also found lead

iodide to be effective, although its yellow colour is a drawback for photography. The advantage of heating the powder is that it will be thoroughly dry, and so not adhere to other parts of the surface, but only to the filmy impression.

For developing latent prints on paper I have found methylene blue to be an efficient agent, provided it is quite dry, and is lightly dusted over the surface and then blown off.

The results which this mechanical method is capable of yielding depend very largely upon the absorptive capacity of the paper. If the surface is very highly glazed, so as to be comparable with the surface of a japanned tin box, the prints seem to be quite permanent, and I have developed them quite sharply with methylene blue after a lapse of three years. But prints made on highly sized clayed paper, such as is used for book illustrations, although they could be brought out distinctly after several weeks, did not appear when treated in this way after three years. In the case of tracing-paper the marks were developed clearly after three weeks, but could not be brought up after a few months, whilst the prints on an ordinary card showed all their details for the course of a few days, but yielded only a faint outline after eighteen days. Similar impressions on white blotting-paper could be developed with sufficient detail for identification for an hour or two after being made, but showed only faint smudges, with no details, after about a week.

To protect the prints from being smudged after development, Stockis (loc. cit.) recommends coating the surface with a film of a solution of gum arabic, alum, and formaldehyde.

#### LIQUID CHEMICAL REAGENTS.

Forgeot (Archives de l'Anthropol. Criminelle, 1891, 6, 387) describes experiments made by Aubert with special papers saturated with various salts, such as silver nitrate and mercuric nitrate, the prints being subsequently developed with appropriate reagents. When the hand is pressed on white paper and subsequently developed with 10 per cent. sodium thiosulphate solution containing a few drops of alcohol, the imprints are frequently developed as oily stains. The success of these methods, however, depends upon the time during which the hand was in contact with the paper, and so they cannot be recommended as reagents for the detection of accidental finger-prints.

Osmic Acid.—Since imprints left by the finger invariably contain traces of organic matter, notably fatty compounds, the use of a solution of osmium tetroxide (osmic acid) suggests itself as a suitable reagent, and Faulds (Dactylography, p. 64) and Gross ("Handbuch der Untersuchungsrichter," p. 598) have both mentioned the possibility of using this reagent, but neither has given any particulars of the results which it gives.

From my experiments I find that, by brushing the ordinary 1 per cent. solution over a print and leaving the surface moist, preferably while exposed to sunlight, reduction takes place and the ridge becomes black through the reduced osmium, while the furrows remain uncoloured.

The method is capable of showing up prints distinctly on moderately sized paper after the lapse of several weeks, but is less trustworthy than some of the other

methods for long periods. Naturally it will not be effective in the case of prints on absorbent paper.

Ink Methods.—This objection also applies to the method of development by ink, which was first devised by Forgeot (loc. cit.), for the successful application of which a contact between the finger and paper of a few seconds at most is necessary. This method is based upon the principle that there is normally a slight secretion of oil on the fingers, and that in the impressions produced by them some of this oil will have been conveyed to those parts of the paper with which the ridges of the patterns have come in contact. Hence, when the place is subsequently treated with ink, the ridge markings will repel the liquid, whereas the intermediate furrows and the rest of the paper will be stained. In this way a negative finger-print will be produced as opposed to the positive prints given by adhering powder or osmic acid, in which the ridges receive most of the pigment and the furrows remain relatively uncoloured.

Forgeot found that the best method of applying the test was by means of a brush, and that there was no advantage in diluting the ink. In his experience developing the print in an ink-bath was fatal to good results, as it produced unequal staining.

Faulds (loc. cit., p. 67), however, discarded Forgeot's method of brushing for development in a bath, and also found that ink diluted with water gave much better prints than the ink by itself.

It is highly probable that the cause of these discrepancies lies in the fact that writing ink is not a definite chemical composition, but varies widely in the proportion of its constituents. For instance, I have shown (Analyst, 1908, 33, 8) that the amount of total solids in twenty-five different samples of commercial writing inks varied in their proportions from 1.89 to 7.94 per cent., whilst copying inks (with which Faulds also obtained good results) contain a still higher proportion of solid matter.

If comparable results are to be obtained by this method, it is necessary that a standard ink should be used, and the composition of this should be as simple as possible.

The drawback to the use of an ordinary iron-gall ink is that it is applied to the paper in an unoxidised condition, so that even those parts which appear very pale at first may eventually become much darker, with the result that it is difficult to control the final coloration. I have, therefore, made experiments with other ink compositions.

Schiff (Ann. Chem. Pharm., 1871, 169, 164) attributed the blue coloration given by ferric salts with gallic and gallotannic acids, phenol, etc., to the presence of hydroxyl groups, and concluded that the intensity of the coloration depended upon the number of free hydroxyl groups in the organic compound. Schiff's work was extended by Schlüttig and Neumann (Die Eisengallustinten, 1890, p. 16), who established the fact that to produce a permanent coloration with iron salts—i.e., an ink—a substance must contain three hydroxyl groups in juxtaposition. For example, hydroquinone with two hydroxyl groups does not yield an ink, whereas gallic acid and pyrogallol with three adjacent hydroxyl groups do form them. I have pointed

out (Analyst, 1903, 28, 146) that this law of atomic grouping also applies to the permanent colorations produced by ammonium vanadate with similar compounds, and I have also found that the same rule is applicable to the colour reactions of osmium tetroxide. The ordinary 1 per cent. solution of osmic acid produces inks with gallotannic acid and gallic acid, which have the advantage over iron-gall ink of attaining their maximum intensity of colour almost immediately after application to the paper. The gallic and gallotannic acids form violet-grey colorations, but the most suitable ink for developing finger-prints is obtained by adding pyrogallic acid to osmic acid solution. In very dilute solution the osmic pyrogallate is at first violet,



FIG. 2.—PRINT DEVELOPED AFTER THREE YEARS WITH "SWAN" INK.



Fig. 3.—Print Developed with Osmium Pyrogallate,

but almost immediately becomes greenish-blue, and when applied to paper gives a rich violet-black coloration.

This reaction may be used as a very sensitive test for osmium tetroxide, the distinctive blue coloration being obtained on adding a trace of solid pyrogallic acid to a solution containing a few drops of 1 per cent. osmic acid solution. Ink of a suitable dilution for treating finger-prints may be made by diluting 2 c.c. of the ordinary microscopic osmic acid reagent with the same quantity of water, and adding 0.05 grm. of pyrogallic acid. The liquid is ready for use immediately, and should be brushed over the paper with one broad sweep of a soft camel-hair brush.

These ink methods are applicable under varying conditions, and I have developed prints on highly sized paper after the lapse of three years both by means of ordinary "Swan" ink (total solids, 2.98 per cent.) and by means of osmic pyrogallate.

# REAGENTS IN THE FORM OF VAPOUR.

The fact that finger-prints may, and probably usually do, contain a certain amount of fat suggested the use of reagents which are absorbed by fat. After I had made experiments for some months with iodine vapour, I found that its use had already been suggested many years ago by Aubert and Coulier, although no details are given as to its effectiveness as a reagent.

On the whole, it is the most sensitive reagent of any I have tried. For example, a print made with light pressure on well-sized paper could not be developed with methylene blue a month later, but was shown up sharply by means of iodine vapour, and was again redeveloped after three and a quarter years. A print on parchment paper, however, could not be developed after a few months, whilst prints on porous paper only gave moderately good results after a few hours.

The drawbacks of the iodine method are that the prints are fugitive and, owing to their colour, are difficult to photograph. They may be fixed to some extent by treatment with solutions of silver salts, followed by thiosulphate, but lose a good deal of their sharpness in the process.

I have found that prints made with mercuric iodide vapour are considerably more stable. They are obtained by exposing the paper to vapour from a heated mixture of 4 parts of mercury and 5 parts of iodine. At first they are dark red, but the colour soon changes to yellow, probably through the formation of the yellow modification of mercuric iodide. An objection to this method is that the paper itself is sometimes stained a dark colour.

Bromine vapour only produces faint yellow prints, whilst iodine chloride vapour gives a much fainter coloration than iodine. Mercuric bromide is much less satisfactory than mercuric iodide as a reagent.

Since osmium tetroxide boils at about 100° C., it seemed probable that, applied in the form of vapour, it might prove a more sensitive reagent than when used as a solution. I found this to be the case, and obtained prints full of fine detail by holding the paper over a basin in which a little of the 1 per cent. solution was boiling. The method will give good permanent prints on moderately sized paper or a postcard, but is not so sensitive as the iodine method. It effected no development of a print on highly sized paper after a period of three years. The prints produced by osmium tetroxide vapour are positive, the ridges being grey, the furrows white, and the pores opening on the ridges dark grey to black.

Incidentally it may be mentioned that the method affords another test for osmium tetroxide. A slightly greasy finger-print is made on glazed paper, which is then exposed to the vapour of the boiling liquid. A permanent print of the type described indicates the presence of osmium tetroxide.

It is probable that ruthenium tetroxide or volatile ruthenium salts, which also darken on contact with organic substances, would also give good results in the development of latent finger-prints.

Faulds, in his experiments with the ink method, found that good results could often be obtained by adding creosote to the ink. This causes the reagent to run very rapidly over the surface of the paper, so that the period of contact is much

shorter than when ordinary ink is used. The differential staining is more pronounced when a finger-print is recent than after it has been made for some time, and Faulds was able in this way to make some estimate of the approximate age of a print.

It is obvious that any such deductions from the behaviour of a print would only be possible if all the conditions, such as the behaviour of different kinds of paper, duration of impression, and effect of temperature upon the permanency of the prints, were known.

The comparative results obtained with reagents in the different forms of powders,

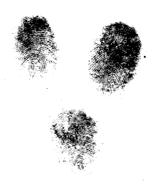


FIG. 4.—RECENT PRINTS DEVELOPED WITH OSMIUM TETROXIDE VAPOUR.

liquids and vapours, appears from the results obtained in my experiments to be more likely to afford a promising basis for estimating the relative age of finger-prints.

#### Examination of the Pores.

The osmic vapour method is particularly suitable for determining the relative position, number, sizes, and shapes of the pore openings in finger-prints. This recent development of the finger-print system is due to Dr. Locard, head of the Police Laboratory at Lyons, and I am indebted to him for the examples of the prints shown on the screen illustrating this new method of examination, which he terms "poroscopy," applicable to any part of the hand. In his communication on the subject (loc. cit.) he states that the pores are as persistent as the ridges themselves. In this connection it is interesting to compare the prints which were sent to me by Sir William Herschel. Making every allowance for defective printing in the later print, I found that although the ridges were obviously in agreement, it was difficult to trace correspondences between the pores except in one or two instances. The weathering of the skin on the fingers in old age thus appears to interfere with the pores, at all events in the case of prints made with printing ink, to a greater extent than it does with the ridges.

The chemical method which Mr. T. J. Ward has accidentally discovered offers many advantages over the old printing-ink method for the preparation of comparative prints.

In this method an ordinary slow gelatin photographic plate is exposed to light for a few seconds; the finger is then well moistened with any usual photographic developer, and pressed firmly and evenly upon the surface of the plate for about fifteen seconds. The plate is then left to develop spontaneously, and is finally fixed with sodium thiosulphate and washed in the usual way. The image obtained shows the ridges white and the furrows and pores dark, with the finest detail clearly defined.

The difficulty in preparing or developing finger-prints so as to show the pores effectively is that the pigment used, whether in the form of printing ink or of a powder or liquid, is liable to hide the distinctive features. Referring to this drawback, Wilder and Wentworth remark in a book recently published on the subject of "Personal Identification" (p. 309): "The authors look ahead to the time, not far distant, when all accidental impressions will be developed by chemical fumes, or by other equally delicate methods, thus reproducing the impressions exactly as they are, without the intervention of any kind of powders so much in use to-day."

I trust that the results of the experiments which I have brought before you this evening may contribute towards the solution of this problem.

The great assistance which Mr. Ward has given me in photographing various prints, developed by different methods, and in preparing lantern slides from them, has enabled me to demonstrate these results to you much more effectively than I could otherwise have done, and I take this opportunity of thanking him for the interest he has taken in this work.

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## PHOTOMICROGRAPHY WITH SIMPLE APPARATUS.

By THOMAS J. WARD.

(Read at the Meeting, March 3, 1920.)

RECORDS obtained by photomicrography are frequently of great service in many industries and scientific investigations at the present day, but it is a common misconception that in order to obtain satisfactory results it is necessary to employ expensive apparatus such as those figured in the catalogues of microscopical dealers. My purpose is to demonstrate that by means of simple and inexpensive apparatus it is possible to obtain satisfactory results with magnifications not exceeding about forty diameters.

The essential apparatus consists of a camera, a lens, a support for the object, and some source of illumination. The camera may be of any form, however simple, provided that it allows of extension from about 10 cm. to not less than 50 cm., and is so arranged that focussing may be achieved by altering the position of the back. Its size should allow the use of half plates if necessary. The lens may consist of any form of short focal length camera lens or a microscope objective, according to the magnification required. The micro-lenses I chiefly employ are achromatic objectives ranging in focal length from 76 to 8 mm., and costing ten shillings each in pre-war days. The object holder may be of any simple form and should be fitted with rubber bands or metal clips. Daylight in this country, at least, is too variable and uncertain to be of much use for this work, and some form of artificial illumination should be employed. I have at various times used the following: Oil lamp, batswing gas burner, incandescent gas (upright), acetylene (cycle lamp, flat flame), electric ½ watt 50 c.p., and magnesium ribbon. Of these, the two latter are the most satisfactory for constancy, high actinic power and convenience.

The relative actinic power of the above illuminants when using ordinary (not ortho- or pan-chromatic) plates is approximately as follows:

 Equivalent Exposures.

 Batswing burner
 ...
 ...
 40 seconds.

 Incandescent gas
 ...
 ...
 10
 ,,

 Acetylene (flat flame)
 ...
 ...
 6
 ,,

 Electric ½ watt 50 c.p.
 ...
 ...
 3
 ,,

 Magnesium ribbon
 ...
 ...
 1
 ,,
 (1·5 cm.)

PLATES.—In regard to the numerous makes upon the market there is but-little to choose between them for this work. On the whole, an "Ordinary" plate is slightly superior owing to its fineness of grain and ease of manipulation, but where economy of time is a consideration one requiring shorter exposures, such as a "Special Rapid," will be found satisfactory. On occasions when only a few copies are required and a negative image will serve the purpose equally as well as a positive, bromide paper may be used in the dark slide instead of the plate, this being much more economical. Although a half-plate camera is very suitable for

this work, any smaller sized plate may be employed by using adapters in the dark slide.

ARRANGEMENT OF APPARATUS: First Method.—The camera is fitted with a short focus (10 to 12 cm.) single meniscus photographic lens, and is then firmly fixed to the working bench. The object is placed in position and focussed roughly by altering its distance from the lens, the final focussing being done by adjusting the front or back of camera. By this method magnifications up to about  $\times$  4 may be attained.

Second Method.—The arrangements adopted in the first method are used with the addition of a large diameter lens on a separate support between the camera lens and the object. This will enable a magnification of about  $\times$  8 to be obtained before the optical qualities of the camera lens are upset sufficiently to produce poor images on the plate.

Third Method.—The general arrangements given above are adopted, but the camera lens is removed and replaced by a microscope objective fitted into a cork. According to the focal length of objective used (76 to 8 mm.) and the extension of camera, magnifications varying from  $\times$  5 to  $\times$  40 may be obtained without any difficulty in manipulation. It is possible with transparent objects to use an objective of only 4 mm. focal length and attain a magnification of about  $\times$  75, but the difficulty of focusing by means of the camera rackwork is great.

Fourth Method.—This method, which allows of magnification up to × 400, requires the use of a microscope and is therefore somewhat outside the scope of this paper. It will be sufficient to mention that the microscope is placed horizontally, and the camera without any lens is placed with its lens mount against the eyepiece. In order to keep the camera fairly rigid and at the same time allow of its ready removal for observation through the microscope, it is placed upon a pile of paper-covered magazines and has a wooden bar with weights suspended from each end laid across the base-board.

ILLUMINATION OF OBJECTS.—The method to be adopted for illumination depends chiefly upon whether the object is opaque or transparent, and secondly upon the detail required to be shown in the finished print. Transparent objects are usually photographed by transmitted light, and may be illuminated by (a) light passing through direct from the illuminant to the objective; (b) polarised light, by using a polariser and analyser before and behind the objective; and (c) black background obtained by the use of a 4-inch condenser having a central stop to prevent direct light from the illuminant reaching the objective, while the object is illuminated by an annular cone of rays from the periphery of the condenser.

When photographing opaque objects the light must be reflected by the object, which may be illuminated (a) by direct light from illuminant on the same side of the object as the objective; (b) by direct light with the use of a condenser between illuminant and object; (c) a Lieberkuhn reflector fitted to objective.

With objects which are not flat and require to be photographed with sharp detail in various planes, it will be necessary to reduce the aperture of the objective by placing black paper stops at the back which may be held in position by a small screw cap or a circular steel spring.

Since lenses of long focal length require a comparatively large even illumination when dealing with transparent objects, it is advisable to insert one or two pieces of ground glass between the illuminant and object. The increase in exposure necessitated by this addition is shown below:

One piece of ground glass ... ... ...  $\times$  1·3 Two pieces of ground glass ... ... ...  $\times$  1·5

EXPOSURE.—The determination of the correct exposure required is a difficult problem in photomicrography, and up to the present no exposure meter has been produced which is of any use for the purpose. The exposure depends upon a number of factors, among them being—the colour of the object, the method of mounting illumination (transmitted or reflected), actinic power of illuminant, aperture of lens used, magnification, speed of plate, etc.

By keeping a full record of the details of each exposure one soon acquires a series which is of great value in the determination of any exposure required for a new object. My own record contains spaces for twenty-one particulars in addition to a column for "remarks," but it is rarely necessary to fill up more than half of these.

The following table is an abbreviated form of my exposure record, and the figures given may be a guide to anyone commencing this work for the first time:

Object.	Mounting.	Magn.	Method.	Illumination.	Illuminant.	Objective.	Stop.	Plate.	Exposure
Coralline pebble	Dry	2	1	Direct, re-	Incandescent gas	111 mm.	<b>F.</b> 8	Imp. Ord	2 min.
Brome grass	i ,,	3	1	Dark back- ground	Incandescent gas	"	F. 22	Imp, N.F.	5 min.
Woven linen	 	9	2	Direct, re- flected.	Magnesium	111+240 mm.	F. 16	,, ,,	12 in Mg.
Ink and pencil marks	Xylol	11	3	Direct, trans- mitted.	½ watt, 50 c.p.	64 mm.	4 mm.	Imp. S.R.	6 in Mg.
Woven horsehair	Balsam	37	3	Polarised	Magnesium	8 mm.	٠,,	Thames	30 in Mg.
Globigerinia	Dry	19	3	Lieberkuhn	4 watt, 50 c.p.		1.5 mm.	Imp. Ord.	
Lions on half-a- crown	,,	10	3	Direct, re- flected	, , , , , , , , , , , , , , , , , , ,	76 mm.	4 mm.	Imp. S.R.	,,
Mercury globules from Reinsch test 0:0001 mg.	<b>,,</b>	320	4	Direct, trans- mitted	<b>21</b> 22	4 mm. +eye- piece ×5	5 mm, in Abbé con- denser	,, ,,	30 min,

THE STAG BREWERY, PIMLICO.

#### Discussion.

Mr. W. F. Reid remarked that if all schools taught their students to make the best of such simple means as illustrated by Mr. Ward, it would be of great help. He had fitted up several kinds of apparatus, using a vertical camera, the great advantage being that it was less subject to vibration.

With regard to illumination, the use of acetylene was to be recommended if a proper generator were available, as it worked very well. This method was adopted by the Italians in warfare. A great deal also depended upon the kind of light that was used, as it could be split up, and thus different results obtained. One of the

difficulties in photographing with invisible rays was the correct focusing of the apparatus.

In the case of photographing insects, different results were obtained with ultraviolet rays with the visible lights cut off, and an excellent photograph of a portion of the insect could be obtained straight away, without mounting, with the reflected light cut off.

Mr. Gilbard thought much better results in colour work could be obtained in different colour screens—that is, yellow, green and blue—were used. The latter especially was to be recommended, as this would sometimes bring out detail where yellow failed to do so.

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#### NOTE ON THE SOLUBILITIES OF THEOBROMINE.

BY RAYMOND V. WADSWORTH.

(Read at the Meeting, March 3, 1920.)

In examining the properties of a sample of pure theobromine it was found that the figures obtained for the solubilities were, in certain cases, notably lower than those published. The solubilities to be found in the literature on the subject vary so much amongst themselves that a revision is necessary. With this aim in view I have redetermined the solubility in most of the commoner solvents. The original error seems to have been due chiefly to Mitscherlich, who, working with impure theobromine, obtained high solubilities, which have been copied by many authorities. The impurity in Mitscherlich's alkaloid seems to have been caffein, for his high figures correspond with high solubilities for caffein. His figures are as follows:

			At 15.5° C.	At Boiling-point of Liquid.
			Per Cent.	Per Cent.
Chloroform	• • •	 		1.00
${f Ether}$		 •••	0.006	_
Water		 	0.160	1.81

Theobromine is only soluble with difficulty in most liquids, and precipitates very slowly. Consequently high figures for solubilities at low temperatures can be easily obtained if care is not taken to allow a considerable time for precipitation after the liquid has been heated. Also low results will be obtained if solution is performed in the cold, unless due precautions are taken. The following is the table of solubilities worked out. All the figures were estimated on the same sample, which I prepared and purified:

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Solvent.	Solubility of Theobromine in Grms. per 100 c.c. at—			
Solvent.	15·5° C.	Boiling-point of Liquid.		
	Per Cent.	Per Cent.		
Ether $(0.720)$	0.003	0.003		
Benzene	0.005	0.010		
Trichlorethylene	0.020	0.030		
Carbon tetrachloride	0.020	0.040		
Chloroform	0.060	0.070		
Alcohol (absolute)		0.100		
Alcohol (90 per cent.)		0.390		
Water	0.060	0.700		
Tetrachlorethane	0.090	0.870		
Anilina	0.650	8.000		
Ether petroleum	Insoluble.	Insoluble.		

The following are the chief examples of differences between my results and those of other chemists:

m.:.1.1			Per Cent.	
Trichlorethylene— At 15.5° C	•••	•••	Up to 7 0.02	Gowing-Scopes (Analyst, 1914, 39, 12). Wadsworth.
Chloroform— At boiling-point			1·00 0·07	B. P. C., Allen, Thorpe, Mitscherlich. Wadsworth.
Alcohol (90 per cent.)— At boiling-point	•••	, , , ,	2·13 0·39	B. P. C., Blyth, Thorpe. Wadsworth.
Water— At boiling-point	•••		1·81 0·70	B. P. C., Blyth, Mitscherlich. Wadsworth.

The work was carried out in Messrs. Cadbury Bros. Research Laboratory, under Mr. A. W. Knapp, and I wish to thank the firm for their permission to publish the results.

# ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

#### FOOD AND DRUGS ANALYSIS.

Formation of  $\beta$ -Methylumbelliferone as a Reaction of Acetylacetic Acids and its Esters. V. Arreguine and E. D. Garcia. (Ann. Chim. anal. appl., 1920, 2, 36-41.)—The condensation of the ethyl ester of acetylacetic acid with resorcinol in presence of hydrochloric acid yields  $\beta$ -methylumbelliferone, which is characterised by giving an intense blue fluorescence in extremely dilute alkaline solutions. From 0.1 to 0.2 grm. of resorcinol is mixed with 2 to 3 c.c. of the solution of the ethyl ester in concentrated hydrochloric acid, and the liquid heated for a few minutes up to boiling-point, then cooled, diluted with a little water, and rendered alkaline with ammonia solution. The test is capable of detecting 0.00002 grm. of acetylacetic acid or its derivatives in 1 c.c. of liquid, and the reaction is not given by acetone or  $\beta$ -hydroxybutyric acid. In applying the test to urine 50 c.c. of the sample are acidified with 2 or 3 drops of hydrochloric acid, and shaken with 5 c.c. of carbon tetrachloride or chloroform. The extraction is repeated with 3 c.c. of carbon tetrachloride, and the united extracts are evaporated to 2 to 3 c.c., and heated with 0.1 grm. of resorcinol and 2 c.c. of hydrochloric acid until the solution has evaporated. The liquid is then cooled, diluted, and rendered faintly alkaline with ammonia solution.

Rapid Method of Estimating Water in Crude Camphor. K. W. Lane and O. F. Lubatti. (J. Soc. Chem. Ind., 1920, 39, 50x.)—Five grms. of the crude sample in coarse granular fragments are placed in a centrifuge tube having a barrel of 25 mm. and a narrow tube of 4 mm. internal diameter, the latter being sealed at the lower end and graduated. Resting on the coned junction between the narrow and wide parts of the tube is a small funnel of nickel-wire gauze, on which is placed a piece of cotton-wool to act as a filter. Thirty c.c. of benzene, previously saturated with water at laboratory temperature and centrifuged before separating the water in a separating funnel, is then introduced into the tube containing the camphor, which it dissolves, and the whole is spun for a couple of minutes. water in the sample is found in the narrow tube, any insoluble impurities being retained by the cotton-wool. The percentage of water is read off direct, and any particles of water which may have been retained mechanically by the wool or on the walls of the tube are disturbed by carefully stirring with a glass rod and a second reading taken. If a sample is very pure the gauze filter may be dispensed with. H. F. E. H.

Cryscopic Method for the Determination of Cane Sugar. H. H. Dixon and T. G. Mason. (Scient. Proc. Roy. Dublin Soc., 1920, 16, No. 1, 1-8; through J. Soc. Chem. Ind., 1920, 39, 201a.)—A method for the determination of cane sugar

in small volumes of liquid is based on the fact that after inversion the depression of freezing-point of a cane-sugar solution is approximately doubled. Invertase is added to the sap or other liquid in the cold and the freezing-point determined, after which the mixture is incubated at 30° C. for forty-eight hours and a second observation made. Advantages of the proposed method are that no treatment to eliminate proteins and other colloids is necessary, and that only a small volume of liquid—viz., about 2.5 c.c.—is required. Readings of temperature may be obtained with an error of ±0.003°, and 0.01° corresponds to 0.2 per cent. of cane sugar.

Occurrence of Melecitose in Honey. C. S. Hudson and S. F. Sherwood. (J. Amer. Chem. Soc., 1920, 42, 116-125.)—It has long been known that bees collect manna during droughts when floral nectar is not abundant. In some mannas the rare trisaccharide melecitose has been found, and one sample from the Douglas fir of British Columbia contained as much as 70 per cent. of this sugar. therefore, likely that some honeys should be found to contain melecitose, and this has actually proved to be the case. A sample of comb honey of the honey-dew type coming from Port Royal, Pennsylvania, crystallised solidly in nearly all the cells, the crystals proving to be pure melecitose, while two other samples of honeydew honey from Maryland also contained melecitose crystals. The bees were found to collect a sweet fluid deposited on the young twigs of the Virginia pine (Pinus virginiana) by a soft scale insect or aphis, and this fluid itself contains melecitose. Since melecitose has already been identified in the manna of the European larch, the Douglas fir, and the scrub pine, it may probably be present in the sap of most coniferous trees, it being assumed that the insects producing these mannas and exudations do not themselves synthesise the carbohydrate, although this remains to In the analysis of honeys suspected to contain melecitose, the cane sugar present, when estimated by the Clerget process, must be inverted by means of invertase and not acid, since invertase is without action on the trisaccharide, while acid hydrolyses it. In the future, when a honey is found by the analyst to show a large increase in reducing sugars after acid inversion, but a much smaller increase when using invertase, the presence of melecitose should be suspected. honey should be condemned as being adulterated with cane sugar on the sole basis of an increase in reducing sugars by acid hydrolysis, since the presence of the trisaccharide can equally well cause such an increase (see Hudson, J. Ind. and Eng. Chem., 1910, 2, 143-6). Melecitose, after recrystallisation from hot aqueous alcohol and drying at 70° C., shows an  $[a]_{D}^{20} + 88.2$  and a melting-point of 148° C. hydrolysis, 100 grms. of the sugar has the same reducing power as 69 grms. of d-glucose (J. Amer. Chem. Soc., 1918, 40, 1456-60). From these data the percentage of melecitose in honey may be calculated. H. F. E. H.

Rapid Method of Estimating Lead in Cassia Oil. O. F. Lubatti. (J. Soc. Chem. Ind., 1920, 39, 35-36r.)—Cassia oil exported from China is contaminated with lead derived from the leaden vessels in which it is sold. For the rapid colorimetric estimation of this lead by means of ammonium sulphide 5 c.c. of the oil (or 2.5 c.c. if a preliminary test has indicated the presence of more than

0.025 per cent. of lead) are diluted to about 23 c.c. with 90 per cent. alcohol in a Nessler cylinder of narrow diameter (2.5 cm.). The same amount of cassia oil free from lead is diluted in the same way in a second tube, 1 c.c. of ammonium sulphide solution added to each liquid, and a standard solution of lead in 90 per cent. alcohol (1 c.c. = 0.0001 grm. Pb) is added to the blank until it matches the brown coloration of the sample under examination. The liquid is stirred three times after each addition of lead solution. The results thus obtained are slightly high, the average excess being 0.00018 grm. The amount of lead usually present in commercial samples ranges from 0.04 to 0.06 per cent. In a test experiment, in which a pure oil was left in contact with bright lead in closed tubes, which were shaken at intervals, the maximum absorption of lead was 0.074 per cent., and was reached after one month.

Factors affecting the Oxygen Absorption of Linseed Oil. A. de Waele. (J. Soc. Chem. Ind., 1920, 39, 48-50 t.)—Since linseed oil on exposure to air not only takes up oxygen, but loses some as volatile products, it is to be expected that equilibrium conditions of the oxidised product with the surrounding atmosphere will, to some extent, be determined by the actual proportion of decomposition products present in the latter. A further factor which has to be taken into account is the probable formation of peroxides stable as such in the absence of water, decomposition taking place in the presence of water vapour. A series of determinations were carried out and described by the author, in which variation in weight on exposure under conditions of light and darkness were compared with the figures recorded for variation in atmospheric conditions, results being shown on curves for the differently treated oils. Circular filter papers 11 cm. in diameter were spotted with raw and variously treated linseed oils in such a way as to secure the minimum thickness of distribution by allowing the maximum of spreading to take place. The papers were then suspended by glass hooks and exposed to the air. It is to be noted, however, that the first series received exposure to light only for about eight out of the twentyfour hours, as the experiments were carried out in mid-winter. Weighings were performed every twenty-four hours, and the variations in weight returned as percentages on the weights of oil spotted on the papers. In the curves representing siccatised oils it should be noted that the drier was introduced in such a way as to ensure the minimum of polymerisation and oxidation. The results tend to show that the presence and nature of the drier does not affect the value of the periodic variation in weight after the first period of ten days in the presence of light, while the accelerating effect of daylight is clearly shown. It would also appear that the inverse reading of the pressure of water vapour in the atmosphere has for its main effect the influencing of the "light" curves. Variation in the "darkness" curves seems to be less dependent on the effect of the pressure of water vapour than on that of the relative humidity, while the relative stability of polymerised oil is clearly shown. The author is of the opinion that the liberation of free fatty acid on oxidation is improbable, the high acid values observed in oxidised oils being more probably due to groupings which under suitable conditions of hydrolysis, such as in neutralisation, would function as carboxyl or hydroxyl groups. Such hydroxyl groups attached to ring nuclei formed by inner polymerisation subsequent to oxidation would function in the same way as phenolic hydroxyl groups, the existence of which are strongly indicated by the high acetyl values of oxidised oils. The samples used for the experiments were raw linseed oil (no drier), raw linseed oil containing 0·1 per cent. Mn, raw linseed oil containing 0·5 per cent. Pb, raw linseed oil containing 0·1 per cent. Pb and 0·0023 per cent. Mn, and, finally, polymerised linseed oil free from oxidised oil or drier.

H. F. E. H.

Hours of Labour as affecting Composition of Milk. G. C. Jones. (Dairyman, 1920, 42, 232.)—The author publishes the following table showing the average composition of milk passing through his hands during the ten years ending with 1918. They agree within about one in the second decimal place with those published in the Analyst by Richmond over a period of ten years:

Composition	OF	MIT.R
COMPUSITION	OF.	WILLIAM.

		M	orning.	Afternoon.			
-	Fat.	Non-fatty solids.	Fat.	Non-fatty solids.			
January		 3.62	8.95	3.91	8:95		
February	•••	 3.54	8.95	3.84	8.94		
March	• • •	 3.49	8.93	3.80	8.93		
April		 3.43	8.89	3.76	8.87		
May		 3.31	8.92	3.76	8.91		
June		 3.26	8.91	3.74	8.87		
July	•••	 3.40	8.81	3.80	8.75		
August		 3.49	8.79	3.94	8.74		
September		 3.61	8.86	4.05	8.84		
October	•••	 3.71	8.93	4.11	8.92		
November		3.82	8.96	4.14	8.95		
December		3.77	8.94	4.02	8.94		

The purpose of the paper is to show that recent alterations in the hours of agricultural labour tend to exaggerate the difference in composition of morning and afternoon milk, due to the fact that the interval between morning and afternoon milking is shorter, and that between afternoon and morning milking longer. That anequal milking periods exaggerate the difference in composition between morning and afternoon milk has long been known, Monday morning's milk being notoriously poor, due to early milking on Sunday afternoons.

That the shortened hours of agricultural labour would have the effect described was to be anticipated, therefore, and the author states that he has for some months had evidence that they were operating to an appreciable extent, but whilst milk was at its best in mid-winter even morning milk was of fair quality. In February, however, the author's morning samples averaged only 3.32 per cent. of fat, or 0.22 per cent.

below the decennial average; his afternoon ones 4.06 per cent., or 0.22 per cent. above the decennial average. If these results prove typical, this would appear to demonstrate that the average composition of milk has undergone no change, but that morning milk is poorer than formerly.

Carbonic Acid and Carbonates in Cow's Milk. L. L. Van Slyke and J. C. Baker. (J. Biol. Chem., 1919, 40, 335-344; through J. Soc. Chem. Ind., 1920, 39, 130A.)—The carbon dioxide of milk may be estimated by the process originally proposed for the estimation of carbon dioxide in blood plasma (Van Slyke, J. Soc. Chem. Ind., 1917, 36, 944). Milk is drawn from the cow's udder into a 100 c.c. cylinder so as to fill the cylinder from the bottom upward, thus avoiding mixture with air or loss of carbon dioxide. Two c.c. of this milk is transferred with precautions against loss of carbon dioxide into the Van Slyke carbon dioxide apparatus (loc. cit.) and treated with a 20 per cent. solution of lactic acid. This is found a more satisfactory method of liberating the carbon dioxide than by employing sulphuric acid, which clots the milk into large lumps. The carbon dioxide content of milk varies over a wide range; a normal value is regarded as about 10 per cent. by volume. The degree of acidity tends to decrease with an increase of carbon dioxide. The carbon dioxide may be completely removed by exhaustion from a thin film of milk which is kept in motion. After pasteurisation the carbon dioxide content of the milk falls, but the hydrogen in concentration remains the same. The suggestion is advanced that the carbon dioxide content of milk might afford a means of distinguishing heated from normal Probably the carbon dioxide of milk is present as a mixture of 1 part of carbonic acid to 2 of sodium bicarbonate.

Conditions causing Variation in Reaction of Freshly Drawn Milk. L. Van Slyke and J. C. Baker. (J. Biol. Chem., 1919, 40, 345-355; through J. Soc. Chem. Ind., 1920, 39, 130A.)—In over 300 samples of fresh milk the value of PH varied from 6.50 to 7.20, being under 6.76 in 80 per cent. of the samples. In the case of twenty cows it was found that the reaction of the milk from different quarters of the udder varied greatly, but in most cases the variations were not large. In general a decrease of acidity is associated with a marked tendency towards a decrease in specific gravity, and in percentage of fat, total solids, solids-not-fat, caseinogen, and lactose, but an increase in proteins other than caseinogen and in ash and chlorine. These changes are such as would be expected if blood serum or lymph were added to normal fresh milk. Abnormal conditions of the udder might cause such additions. Examination of milks of low acidity, PH above 6.80, indicates that the reaction is accompanied by the presence of large numbers of leucocytes, though the reaction in such cases may be neutralised by the presence of large numbers of acid-producing streptococci.

Detection of Abnormal Milk based on Hydrogen Ion Concentration. J. C. Baker and L. L. Van Slyke. (J. Biol. Chem., 1919, 40, 357-371; through J. Soc. Chem. Ind., 1920, 39, 130a.)—One drop of a saturated solution of dibromo-o-cresol-sulphon-phthalein (bromo-cresol purple) in water is added to 3 c.c. of milk

and the colour observed. Normal fresh milk gives a greyish-blue colour. The production of a darker or lighter colour seems to awaken suspicion in regard to the normal character of the milk. The colour is made lighter by acids and formal-dehyde, and by heating above the usual point of pasteurisation. The colour is of deeper blue in the case of milk from diseased udders, watered milk, skimmed milk, and milk containing alkaline salts. The method has been applied to 570 samples of market milk and has given trustworthy results. Colour standards can be prepared.

Determination of keeping Quality of Milk. J. C. Baker and L. L. Van Slyke. (J. Biol. Chem., 1919, 40, 373-382; through J. Soc. Chem. Ind., 1920, 39, 130A.)—Bromo-cresol purple (see preceding abstract) can be used to measure approximately and relatively the keeping quality of milk. The test is applied as given in the preceding paper, with the modification that the pipettes and tubes employed must be sterilised before use, and that the samples under test must be incubated for a given time at 18° to 20° C. The milk is examined for change of colour at twenty-four hour intervals. The main factor shown by this test as related to keeping quality is production of acid, but additional factors to be observed are coagulation of caseinogen, digestion of caseinogen, production of alkali and gas, and development of abnormal odour or taste. In the development of acidity four stages of progress are distinguishable through change of colour. In comparing this test with the bacterial count, it is found that in general large numbers of bacteria and increase of acidity are in fair correlation.

Composition of Hubbard Squash Seed Oil. W. F. Baughman and G. S. Jamieson. (J. Amer. Chem. Soc., 1920, 42, 152-157.)—Hubbard squash (Cucurbita maxima) belongs to the pumpkin and watermelon family; the seeds yield about 36 per cent. of oil, having the following physical and chemical properties:

Sp. gr. at 25°/25° C			•••		•••	0.9179
$\begin{bmatrix} n \end{bmatrix}_{ ext{D} \ 25^{\circ} \  ext{C.}} \dots$	•••			• • •		1.4714
			• • •			0.50
Saponification value		•••		• • •		191.5
Iodine value (Hanů				• • •		121.0
Reichert-Meissl val	ue	•••	• • •	•••		0.37
Polenske value					•••	0.39
Acetyl value		•••		• • •		27.8
Unsaponifiable mat	$\operatorname{ter}$	• • •				1.06 per cent.
Soluble acids			• • •	• • •	•••	0.33 ,, ,,
Insoluble acids	• • •			• • •		94.66 ,, ,,
Unsaturated acids	• • •	• • •		• • •	•••	76.45 ,, ,,
Saturated acids	•••		•••			18.37 ,, ,,

The amounts of the different fatty acids present were as follows, calculated as percentages of the oil: Palmitic acid, 12·12; stearic acid, 5·86; arachidic acid, 0·02; oleic acid, 34·09; linolic acid, 43·34.

W. P. S.

Okra Seed Oil. G. S. Jamieson and W. F. Baughman. (J. Amer. Chem. Soc., 1920, 42, 166-170.)—A sample of okra seed (Abelmoschus esculentus) obtained from Avery Island, La., yielded 15-60 per cent. of greenish yellow oil, having the following physical and chemical properties:

Sp. gr. at 25°/25° C					•••	0.9172
$[n]_{\mathrm{D},25^{\circ}}$			•••			1.4702
Acid value	•••	• • •				1.42
Saponification value	e	•••		•••		195.2
Iodine value (Hanu				•••		95.2
Reichert-Meissl val						0.26
Polenske value	• • •			•••		0.23
Acetyl value						21.4
Unsaponifiable mat	ter		• • •			~0.37 per cent.
Soluble acids					•••	0.14, ,,
Insoluble acids		•••	•••			96.2 ,, ,,
Unsaturated acids						67.33 ,, ,,
Saturated acids			•••	• • •		29.22 ,, ,,

The quantities of the different fatty acids, calculated as percentages of the original oil, were as follows: Palmitic acid, 25.82; stearic acid, 2.62; arachidic acid, 0.05; oleic acid, 41.86; linolic acid, 25.47. Specimens of oil obtained from three other samples of the seeds yielded similar results. The oil gives a coloration with Halphen's reagent.

W. P. S.

Estimation of Small Quantities of Nitrous Acid, especially in Salt Meat. F. Auerbach and G. Riess. (Arbeiten a. d. Reichgesundheitsamte, 1919, 51, 532; through Chem. Zeit. Übersicht, 1920, 44, 17.)—The quantitative colorimetric estimation of small quantities of nitrites with m-phenylenediamine is most successful in presence of sodium acetate and acetic acid in known concentration. For the estimation of nitrites in salted meat, the carefully prepared extract must be freed from dissolved albumen and coloured substances by treatment with a colloidal solution of ferric hydroxide. The clear filtrate contains 95 per cent. of any nitrite which may have been present in the meat. Meat pickled for many weeks contains only minute traces of nitrite. If more than 15 mg. per 100 grm. should be found, there is ground for suspicion that the meat has been treated with nitrites.

G. C. J.

# BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Testing the Amylolytic Action of the Diastase of Aspergillus Oryzæ (Taka-Diastase). S. A. Waksman. (J. Amer. Chem. Soc., 1920, 42, 293-299.)—
The various diastatic enzymes differ in their mode of action towards starch, particularly as regards the relative quantities and rates of formation of the intermediate and final products. In this sense it is convenient to differentiate between the amylolytic (liquefying) and the saccharifying powers of the commercial diastatic preparations. The measurement of saccharifying power has formed the subject of many exact researches, but the measurement of amylolytic power leaves much to

be desired in the way of refinement, and this property is the essential one in the valuation of diastatic enzymes applied to the textile industry. The Lintner method for measuring the saccharogenic action should not be used for comparative studies of different enzymes, since the end products are not the same in all cases. Most of the methods hitherto employed for estimating the starch liquefying power depend on the use of iodine for determining the end point, and the indications are somewhat arbitrary. In the method now described the end point taken to indicate the complete destruction of the starch is the change of the opaque starch paste into a clear To facilitate the recognition of this point it is found to be convenientto dye the starch with neutral red. About 50 to 100 grms. of dry potato starch in a large porcelain dish are wetted with 100 c.c. of a 0.5 per cent. solution of neutral red; the starch is allowed to absorb all the colour, and then washed repeatedly with water until the supernatant liquor remains almost clear. The dyed starch is then dried. For the test a 2 per cent. starch paste is made by stirring the coloured starch with a little cold water, gelatinising with boiling water, boiling for ten minutes, and making up to the required volume. The paste is introduced in portions of 10 c.c. each into large test-tubes, which are then placed in a thermostat at 40° C. the correct temperature is reached, increasing quantities of the diastase solution are added to the tubes, and these are well shaken and replaced in the thermostat. end of the reaction, indicated by the clearing up of the colour, is best observed by comparing the liquefied and unliquefied tubes, holding the tubes in the light. The times (T) at which the various tubes become clear are recorded in conjunction with the quantities of enzyme solution (E) present, and it is found that  $E \times T = a$ . Then if F = enzyme value at 40° C.; D = dilution multiple of the original enzyme solution; t = standard time (30 minutes); E = quantity of dilutedenzyme solution used; and T = the corresponding time of liquefaction,

$$F_{400}^{30 \text{ min.}} = \frac{D.t}{E.T}$$
 or  $\frac{D.t}{K.}$ 

In this way the results may be checked by taking the average of several tubes containing different amounts of enzyme. The method is particularly suitable for the study of enzymes which have a starch liquefying power rather large in comparison with the saccharifying power, as, for instance, "Taka-diastase."

J. F. B.

Guanidine Content of Fæces in Idiopathic Tetany. J. S. Sharpe. (Biochem. J., 1920, 14, 46.)—The work of Paton and Findlay (Quart. J. Exp. Physiol., 1917, 10, 3) has made it probable that the condition of tetany may be due to gastro-intestinal disturbances associated with the absorption of guanidine from the intestinal tract. The author has examined the fæces of normal children and of children suffering from tetany, with the result that he finds the average percentage of this base in the four tetany cases was 0.075 per cent. of the moist fæces, whilst in the six normals only faint traces, averaging 0.007 per cent., were detected. The average daily excretion of guanidine by the bowel in children suffering from tetany was 0.018 grm. as dimethyl guanidine. The following method was employed: 30 to 50 grms. of the moist fæces were mixed in a mortar with 100 c.c.

of water, a little chloroform and thymol added, and the whole dialysed with a parchment membrane for three days surrounded by 200 c.c. of water. The dialysate is then removed, and more water added for a further two days, the mixed dialysates being united and evaporated to a syrup. Ammonium, potassium, and excess of sodium were removed with chloroplatinic acid, and the platinum removed with sulphuretted hydrogen, which is evaporated off, and the bases then precipitated with saturated alcoholic picric acid. Dimethylguanidine crystals separate almost immediately as small bunches of yellow needle-shaped crystals, which are very insoluble in water, melt at 230° C., and contain 25 per cent. of nitrogen. A loss of about 5 per cent. is incurred by this method, as found by adding known amounts of a guanidine salt to normal fæces, and may be allowed for. An advantage of the method is the elimination of all suspended matter without the use of precipitants.

H. F. E. H.

Detection of Indican in Urine and Blood. A. Jolles. (Med. Klinik, 1919 15, 814; through Chem. Zeit. Übersicht, 1920, 44, 37.)—Instead of converting the indican into indigo according to the methods of Obermayer and Jaffé, the author recommends the following method. The indican of urine consists principally of potassium indoxylsulphate, which when submitted together with thymol to oxidation by means of ferric chloride yields 4-cymol-2 indolindolignon. This substance forms with one molecule of hydrochloric acid a deep violet dye, which is produced when the chloroform extract of the urine or blood is treated with thymol, fuming hydrochloric acid and ferric chloride.

G. C. J.

Analysis of the Swimming Bladder of the Red Mullet. L. and D. Leroux. (Ann. Chim. anal. Appl., 1920, 2, 47-48.)—The swimming bladder of a specimen of red mullet (Trigta pini) had the following composition: Water, 41.65; nitrogen, 4.14; fat, 30.36; and mineral matter, 1.05 per cent. The ash contained 22.2 per cent. of phosphoric acid ( $P_2O_5$ ), 44.7 per cent. of potassium oxide, and 20.8 per cent. of sodium oxide.

#### ORGANIC ANALYSIS.

Analysis of Acetone by Messinger's Method. L. F. Goodwin. (J. Amer. Chem. Soc., 1920, 42, 39-45.)—The following procedure, which is practically the standard recommended generally, gives trustworthy and concordant results in the estimation of acetone by this method. A quantity of aqueous acetone solution, containing from 30 to 40 mgrms. of acetone, is added to 50 c.c. of  $\frac{N}{1}$  sodium hydroxide solution contained in a stoppered bottle. After five minutes, about 20 per cent. excess of  $\frac{N}{10}$  iodine solution is added while the mixture is shaken continuously. The bottle is then stoppered, and, after a further twenty minutes, 25 c.c. of 2N sulphuric acid are added to neutralise the sodium hydroxide, an excess of 0.3 to 0.4 c.c. of the acid is then introduced, and the iodine is titrated with  $\frac{N}{20}$  thiosulphate solution. Each c.c. of  $\frac{N}{10}$  iodine solution is equivalent to 0.96747 mgrm. of acetone. Methyl and ethyl alcohols, if present, combine with a certain amount of

iodine. Rakshit (Analyst, 1916, 41, 246) has shown that a correction of 1.6 c.c. of  $\frac{N}{10}$  iodine solution must be made for each c.c. of ethyl alcohol, and the author finds the correction for each c.c. of methyl alcohol is 1 c.c. of  $\frac{N}{10}$  iodine solution.

W. P. S.

Micro-Method for the Estimation of Acetone. M. Richter-Quittner. (Biochem. Zeitsch., 1919, 93, 163-172; through J. Soc. Chem. Ind., 1920, 39, 206A.) —A micro-method in which 1 to 2 c.c. of urine and 1.5 to 3 c.c. of alkali need only be used. The urine is distilled once with steam in the presence of acetic acid and a second time with dilute sulphuric acid. Blood or plasma need only be distilled once, and instead of the steam, air is passed through the heated flask. The titration of the distilled acetone is carried out with  $\frac{N}{100}$  iodine and  $\frac{N}{100}$  sodium thiosulphate; 0.1 mgrm. of acetone in 100 c.c. can be estimated with accuracy by this method. The quantity of urine and blood used must not contain less than 0.04 mgrm. of acetone.

S. P. H.

Determination of Amino Acids by Means of the Hydrogen Electrode. E. L. Tague. (J. Amer. Chem. Soc., 1920, 42, 173-184.)—The colorimetric methods for the estimation of hydrogen ions do not differentiate between the excess of alkali necessary to overcome hydration and the amount of alkali necessary to neutralise Strictly speaking, neutralisation consists in bringing the concentration of hydrogen ions to  $1 \times 10^{-7}$  grm. per litre, or  $P_{II} = 7.00$ . Amino acids, however, require the presence of an excess of hydroxyl ions before they will show their maximum acid characteristics, or an excess of hydrogen ions before they will show their maximum basic nature. In the titration of amino acids, even in the presence of formaldehyde, a certain proportion of the added hydroxyl ions are used up in bringing the solvent itself to the hydroxyl ion concentration required for complete neutralisation of the solute. The hydrogen electrode furnishes a means for the accurate determination of this factor. The ionisation equilibria of an amino acid are produced by its functions as an acid, as a base, and as an inner salt, and the inner salt tends to hydrate as the solution approaches the point of neutrality. As soon as the concentration of the hydroxyl ion is sufficient to reduce the concentration of the hydrogen and hydroxyl ions, resulting from the dissociation of the inner salt, to the same value as that of the ions in pure water, the solute has been quantitatively neutralised. The investigation of the amino acids resolves itself into a study of three factors: the behaviour of (1) the solute plus solvent, (2) the solvent alone, (3) the solute alone, the value of the third being calculated from the difference between the other An hydroxyl ion concentration of about  $2 \times 10^{-2}$  ( $P_H = 12.5$ ) will suppress to a negligible quantity the basic ionisation of the sodium salts of the amino acids and thus make possible a more exact quantitative determination of the diamino acids, as well as of others containing strongly negative groups. Sufficient standard alkali is added to a definite volume of the aqueous solution of the amino acid to give it a  $P_{H}$ value of about 12.5. Then to an equal volume of water the same standard alkali solution is added in an amount sufficient to give it the same P<sub>H</sub> value, care being taken to add sufficient water to the blank solution to make its final volume the same as that of the amino acid solution containing the alkali. The difference between the c.c. of alkali used in the two liquids adjusted to the same P<sub>II</sub> value gives the quantity of alkali required to neutralise the amino acid. The apparatus used comprised a Kohlrausch slide wire bridge, a type B (No. 2,500) Leeds and Northrup galvanometer, a Weston millivoltmeter and multiplier, Edison storage cells and the hydrogen and normal calomel electrodes made according to the directions of Hildebrand. Decinormal solutions of the amino acids and sodium hydroxide were employed and the initial volume of liquid was in all cases made up to 100 c.c. The electrode vessel was protected from the carbon dioxide of the air by a rubber stopper through which the electrodes and the tips of the burettes were inserted. Hydrogen gas was passed through the liquid in the electrode vessel at a constant temperature of 18° C. until equilibrium was attained and the constant potential was noted. This required 30-60 minutes, the vessel being continuously shaken at the rate of 50-60 strokes per minute.

Reagents from n-Butyl Alcohol. R. Adams and C. S. Marvel. (J. Amer. Chem. Soc., 1920, 42, 310-320.)—n-Butyl alcohol is now available in commercial quantities as a product of the fermentation of starch in the manufacture of acetone and a number of organic chemical reagents may be prepared, starting from n-butyl alcohol as the raw material. For instance, butyl bromide is obtainable in almost quantitative yields and is easily converted into butyl cyanide. This product by reduction readily gives n-amylamine, or by hydrolysis with alkali, or by treatment with sulphuric acid and an alcohol it will give n-valeric acid or its esters respectively, compounds which have always been difficult to obtain in the pure state. By the reduction of ethyl n-valerate, n-amyl alcohol can be produced in good yields. Thus it is possible to obtain directly or indirectly the various n-amyl derivatives which have only rarely been available to the organic chemist. Another group of compounds most readily synthesised by means of n-butyl alcohol are n-caproic acid and its derivatives. Caproic acid is interesting on account of its wide distribution in many fats and oils. Its a-amino derivative, known as nor-leucine, occurs among the hydrolytic products of brain protein. Pure caproic acid can be very readily prepared by condensing n-butyl bromide with ethyl malonate to form n-butylmalonic ester, then saponifying and decomposing this latter compound. The a-bromocaproic acid is best obtained by the bromination of n-butylmalonic acid and subsequent loss of carbon dioxide. The a-amino derivative (nor-leucine) is made directly from the bromocaproic acid. Detailed directions for the preparation of the compounds enumerated above are given in the paper.

Deterioration of Cotton on Wet Storage. N. Fleming and A. C. Thaysen. (Biochem. J., 1920, 14, 25-28.)—Exposure to damp renders cotton brittle and short fibred and increases its alkali solubility. Loss amounting to 10 or 15 per cent. is often thus entailed in the shape of exceptional short fibres, the so-called "fly." Bacteria are responsible for this deterioration, which can be prevented if the moisture content of the cotton is reduced below 9 per cent. The present paper deals with the results obtained in the investigation of the bacterial changes and with a method for the quantitative determination of such deterioration. Direct microscopic examination, even with an oil-immersion lens, was found impracticable owing to the minute size of the attacked fibres, and it was therefore found necessary to make use of Cross and Bevan's viscose process for the preliminary treatment of the material as employed by Balls for the demonstration of the daily layers of growth in the cotton hair-cell wall; so treated the cotton fibre swells to about five times its normal size. From 0.1 to 0.3 grm. of the sample is boiled for a short time in 1 per cent. caustic soda solution, washed in running water, and then dipped in 1 per cent. acetic acid, and again washed. After squeezing out excess water, the sample is treated in a small, wide-mouthed, glass-stoppered bottle with 1.5 c.c. of 15 per cent. caustic soda and 1.5 c.c. of carbon bisulphide. In this mixture the fibres are left for fifteen to forty-five minutes and are then ready for microscopic examination, a drop of water being allowed to diffuse under the cover-glass after mounting the fibres. Photographs are appended showing the appearance of the cotton. The method may be made quantitative as regards detection of admixed damaged fibres by treating as above described and following the average of ten counts from each preparation under the microscope. Mixtures of known composition were thus examined, and extremely close results obtained. Further details of the technique will be published later.

H. F. E. H.

Quantitative Estimation of Traces of Dichloroethyl Sulphide (Mustard Gas) in Air. M. Yablick, G. St. J. Perrott, and N. H. Furman. (J. Amer. Chem. Soc., 1920, 42, 266-274.)—The method consists in reducing a 1 per cent. solution of selenious acid in diluted sulphuric acid (1:1 by volume) by means of the dichloroethyl sulphide vapour to an orange-red suspension of selenium, the solution being heated to about 85° C. to facilitate the reaction. By means of the nephelometer the mustard gas can be detected down to the limit of 0.005 mgrm., and amounts from 0.01 mgrm. to 0.001 mgrm. can be estimated with a fair degree of accuracy. The standard solutions of dichloroethyl sulphide in 1:1 sulphuric acid contained 0.0078 and 0.00078 grm. per 100 c.c. respectively. To 5.8 c.c. of the standard solution were added 4.2 c.c. of sulphuric acid 1:1, and 10 c.c. of selenious acid solution containing 1.0 grm. of selenium dioxide per 100 c.c. of 1:1 sulphuric acid. The mixture was heated at 85° C. for ten minutes, then cooled and examined in the nephelometer. For the absorption of mustard gas from air, the bubbling apparatus was designed to pass the air at the rate of 1 litre per minute. Ten c.c. of the absorbing agent was placed in the bubbler and the air mixture passed through it for ten minutes. The liquid from the bubbler was then made up with sulphuric acid so as to yield 20 c.c. of a 1 per cent. solution of selenious acid in sulphuric acid 1:1 by volume. When comparing the efficiencies of various absorption reagents a 1 per cent. of selenious acid in water was first adopted, but it was found that a 1 per cent. solution of selenious acid in 1:1 sulphuric acid gave more consistent results as an absorbing medium. The selenious acid solution should not be used until twenty-four hours after making up, but it is then stable for at least two weeks.

Estimation of Cyanides, Cyanates, and Bromides in the Presence of Each Other. G. Velardi. (Gazz. Chim. Ital., 1919, 49, 317.)—Cyanides are estimated in an aliquot part of the alkaline solution by the ordinary method of Liebig. Another portion is exactly neutralised with acetic acid, and the total volume of silver nitrate solution required by the bromides, cyanides, and cyanates together is determined by Mchr's method. A third portion is acidified with nitric acid, and the cyanides and bromides estimated by titration with silver nitrate by Volhard's method. Cyanates are not precipitated by silver nitrate from an acid solution. The amount of bromides is then found by difference.

Estimation of Organic Chlorine or Bromine by the Chromic Acid Method. P. W. Robertson. (Chem. News, 1920, 120, 54.)—The author has previously described a method (Analyst, 1915, 40, 413) depending on heating the substance with chromic anhydride and concentrated sulphuric acid in a simple apparatus, absorbing the volatile halogen compounds thus produced in a solution of sodium hydroxide containing hydrogen peroxide and then titrating with  $\frac{N}{10}$  silver nitrate. The chromic acid may be replaced by potassium dichromate. Whereas perhydrol was formerly recommended because few other commercial brands of hydrogen peroxide were chlorine-free, the author now recommends the use of a cold, freshly prepared solution of sodium peroxide, which is almost invariably halogen-free. Finally,  $\frac{N}{20}$  solutions of silver and of thiocyanate are recommended in place of  $\frac{N}{10}$  solutions. Trouble has sometimes been found with liquid substances, which are apt to be oxidised with explosive violence. This can be avoided by covering the substance with a rather large amount of dichromate and running in the cooled acid very quickly, cooling the reaction vessel in melting ice.

G. C. J.

Estimation of Fluorine in Organic Substances. E. Paternò. (Gazz. Chim. Ital., 1919, 49, 371.)—The combustion of organic substances containing fluorine in Berthelot's calorimetric bomb with oxygen under a pressure of 25 atmospheres does not give good results in the presence of water or of alkaline solutions. If, however, the combustion is made in presence of a mixture of potassium iodide and iodate, and the liberated iodine titrated with sodium thiosulphate solution an accurate estimation of the fluorine is possible.

C. A. M.

Estimation of Glycerol in Presence of Trimethyleneglycol. C. A. Rojahn. (Ber., 1919, 52, 1454; through Chem. Techn. Übersicht, 1920, 44, 4.)—Glycerol obtained by the fermentation process may contain trimethyleneglycol, and the crude liquors obtained in the process are likely to contain it in appreciable amount. In such glycerol and such liquor, therefore, it is useless to attempt to estimate the glycerol by Zeisel's method, because trimethyleneglycol is converted almost quantitatively into di-iodopropane, so that the presence of 1 per cent. of trimethyleneglycol will cause glycerol to be overestimated by 2.42 per cent. The oxidation method may be employed for the analysis of such mixtures, but the most important estimation is that of distillable glycerol.

G. C. J.

Estimation of Paraffins in Commercial Toluene. E. Lewis. (J. Soc. Chem. Ind., 1920, 39, 50x.)—The author refers to the paper by H. G. Evans (Analyst, 1920, 54), and describes a method in use in many of the T.N.T. factories during war time. The estimation was actually carried out on the mononitrotoluene, and the paraffin content calculated back to the original toluene. One litre of the mononitro compound was submitted to fractional distillation in a three-section Young's evaporator column, collecting that portion which distilled below 160° C. The distillate consisted of a small amount of the mononitro compound, together with the whole of the unnitrated toluene and paraffin. Extraction with 90 per cent. sulphuric acid removes the nitrotoluene, while sulphonation with 20 per cent. oleum and subsequent extraction with 90 per cent. sulphuric acid removes the toluene, the residual paraffins being weighed, and a solubility correction applied. Sulphonation of a large amount of toluene, which is difficult to regulate, is thus avoided, and, if required, the unnitrated toluene may be estimated by weighing before and after sulphonation. The paraffin present in toluene supplied by the Gas, Light, and Coke Co., Beckton, was found to consist almost entirely of isomeric octanes, which, in spite of surviving the initial nitration, were decomposed during the more vigorous final nitration of the toluene into trinitrotoluene. H. F. E. H.

Estimation of Paraffins in Commercial Toluene. H. G. Colman. (J. Soc. Chem. Ind., 1920, 39, 50x.)—The method described above (see preceding abstract), whilst giving a fairly accurate estimation of the true paraffin hydrocarbons  $(C_nH_{2n+2})$  series, in commercial toluene does not give a correct figure for the total percentage of saturated aliphatic hydrocarbons in the original toluene. Since these consist not only of true paraffins, but also to a considerable extent of cyclo-paraffins, these are to a very material extent attacked and removed, both in the original nitration of the toluene, and in the subsequent treatment with 20 per cent. oleum, described by Lewis.

H. F. E. H.

#### INORGANIC ANALYSIS.

Formation and Nature of Black Antimony Sulphide. F. de Bacho. (Annali Chim. Applic., 1919, 12, 143-152.)—The transformation of red antimony sulphide into the black modification takes place under varying conditions. When it is dried in a current of carbon dioxide or hydrogen sulphide, the change takes place at about 207° to 212° C. The sulphide loses about 1 per cent. in weight (about the same as in vacuo over sulphuric acid), and there is no material difference in the loss at 150° C. or 300° C. The presence of 50 per cent. of antimony chloride lowers the temperature of transformation to 115° C. Similarly, in the preparation of the black sulphide by precipitation, the process is promoted by the presence of antimony chloride, whether added as such, or formed by adding hydrochloric acid or ammoniun chloride. In this case a reaction takes place between the red antimony sulphide and the antimony chloride, which is probably as follows:

$$2\mathbf{Sb}_2\mathbf{S}_3 + 2\mathbf{SbCl_3} \mathop{\longrightarrow} 6\mathbf{Sb} + 3\mathbf{S}_2\mathbf{Cl_2}.$$

In the formation of the black modification by the dry method, in the absence of antimony chloride there is slight dissociation, with the liberation of metallic antimony, but this is not capable of gravimetric estimation by the ordinary analytical methods. This liberated antimony dissolves in colloidal form as a solid solution in the antimony sulphide, and thus forms the black modification. There is no pronounced difference in the sp. gr. of the two modifications. The sp. gr. gradually rises with the temperature of heating, but there is no sudden rise when the change into the black modification occurs.

C. A. M.

Volumetric Reduction Method with Arsenious Acid. F. de Bacho. (Annali Chim. Applic., 1919, 12, 153-174.)—A new method of estimating oxidising substances is based upon their reduction by means of arsenious acid in the presence of hydrochloric acid, and direct titration of the excess of the reagent by means of potassium bromate—

$$2KBrO_3 + 2HCl + 3As_2O_3 \rightarrow 2KCl + 2HBr + 3As_2O_5$$
.

Methyl orange, which is decolorised by a slight excess of free bromine, is used as indicator. To prevent loss of arsenic chloride by volatilisation the amount of free hydrochloric acid in the solution must not exceed 12.8 per cent. At 15 per cent. the loss was 0.44 per cent., and at 19.4 per cent. it was 48.48 per cent. For the estimation of permanganates the concentration of the acid may vary between 5 and 14 per cent., but a large excess of the arsenious solution must be used. Oxidation is complete in five to ten minutes at the ordinary temperature, and the liquid is then diluted and titrated with N potassium bromate solution until a yellow coloration appears. It is then titrated with the arsenious solution until the colour disappears, and finally titrated back with the bromate solution, after the addition of 2 drops of 0.1 per cent. methyl orange solution. In estimating manganese dioxide in pyrolusite by this method 0.1 grm. of the finely powdered sample is treated with 40 c.c. of N arsenious acid solution, 15 c.c. of water, and 25 c.c. of concentrated hydrochloric acid, and boiled under a reflux condenser until solution is complete, after which the liquid is reduced and titrated as in the case of permanganates. In the case of barium peroxide the reduction is effected slowly but completely at the ordinary temperature, but much more rapidly in a boiling solution. The concentration of the hydrochloric acid may vary from 6 to 12 per cent. without affecting the results. Chlorates are estimated by treating 15 c.c. of the solution of about  $\frac{N}{10}$ strength with 40 c.c. of  $\frac{N}{10}$  arsenious acid solution and 25 c.c. of concentrated hydrochloric acid, boiling the liquid for at least fifteen minutes beneath a reflux condenser, cooling it for ten minutes still beneath the condenser, diluting it with 50 to 60 c.c. of water and titrating it with the bromate solution. chlorine in bleaching powder or hypochlorites may be estimated in the presence of chlorates by keeping the concentration of the hydrochloric acid at 5 per cent., and effecting the reduction in the cold, whilst by increasing the acid concentration to 10 to 12 per cent., and effecting the reduction at boiling temperature, both hypochlorites and chlorates are estimated. Perchlorates do not interfere with the estimation. The method also gives good results with chromates, including lead chromate. C. A. M.

Use of Arsenious Acid in the Volumetric Method of Namias. F. de (Annali Chim. Applic., 1919, 12, 136-142.)—The volumetric method of Namias (Gazz. Chim. Ital., 1892, 22, 508) consists in treating any given oxidising agent with an excess of arsenious anhydride in the presence of ammonium acetate, effecting the reduction in boiling solution, and titrating the excess of arsenious acid with iodine in the presence of acetic acid. It is shown that this method gives inaccurate and inconcordant results, and that the use of starch paste as an indicator is inapplicable in a boiling solution, no blue coloration being obtained at 60° C. to 70° C. The presence of ammonium chloride, which is formed when the original solution contained hydrochloric acid, has also an influence on the results. estimation of chlorates and manganese peroxide in a boiling solution containing excess of hydrochloric acid is liable to result in a loss of arsenic as arsenic chloride by volatilisation. Contrary to the assertion of Namias, the reduction of chromic acid by arsenious acid in the presence of hydrochloric acid is not complete in a few minutes at the ordinary temperature, but requires prolonged action. On the other hand, the results thus obtained are too high, and not constant. C. A. M.

Determination of Chlorine with the Nephelometer. A. B. Lamb, P. W. Carleton, and W. B. Meldrum. (J. Amer. Chem. Soc., 1920, 42, 251-259.)—The nephelometric method has been applied to the estimation of toxic "war gases" in air, including mustard gas, which necessitated the use of alcoholic solutions for absorbing the gas. The Kober nephelometer was employed and several details in connection with the manipulation of this instrument, with a view to avoiding appreciable errors, are described. These details were of special importance in the present work, because the extremely minute quantities of chlorine to be determined required the utmost refinement of the method. The standardisation readings were made by adding sufficient alcohol to appropriate quantities of very dilute sodium chloride solution to give a volume of 20 c.c., then adding 10 c.c. of  $\frac{N}{10}$  nitric acid, followed by 10 c.c. of  $\frac{N}{200}$  silver nitrate, making a total volume of 40 c.c., consisting approximately of 50 per cent. alcohol. The opalescence of a silver chloride suspension changes with time at different rates according to the concentration and other conditions; a sensible time is required for the development of the maximum effect. and this effect then decreases owing to the coalescence of the particles. of constant opalescence may be conveniently prepared by shaking finely-ground kieselguhr with water, allowing it to stand for seventy-two hours and drawing off the supernatant liquor. This suspension had a value approximately equivalent to  $N/4.5 \times 10^{-5}$  silver chloride. Using this standard suspension as a constant, the rates of development and decrease of the opalescence of silver chloride were studied at various temperatures and concentrations. As the result of these observations the procedure adopted in the routine analysis was to place the sample, immediately after precipitation, in a water-bath at 40° C. for at least thirty minutes. It was cooled rapidly to room temperature and compared within thirty minutes with standard silver chloride suspensions similarly prepared. Using this method, chlorides could be estimated with an average deviation of about 3 to 4 per cent. over concentrations ranging between 4 and  $300 \times 10^{-6}$  grm.-mol. per litre. Experiments on the effect of light upon the permanence of the opalescence showed that the rate of decrease of opalescence was more rapid when the suspensions were exposed to diffused daylight than when they were kept in the dark, particularly in the more concentrated solutions. Further, it was established that the opalescence of the same quantity of silver chloride in water was about 15 per cent. greater than in 50 per cent. alcohol or 50 per cent. acetic acid.

J. F. B.

Copper Flame Test for Halogens in Air. A. B. Lamb, P. W. Carleton, W. S. Hughes, and L. W. Nichols. (J. Amer. Chem. Soc., 1920, 42, 78-84.)—The apparatus used consists of a roll of oxidised copper gauze, about 10 cm. in length, contained in a quartz tube about 1 cm. in diameter. To make the test, the air to be tested is drawn through the tube while the end of the copper gauze near the inlet is heated to dull redness. After a suitable time, the gauze is removed from the tube and inserted into a non-luminous flame. A green-coloured flame is observed with as little as 0.1 part of chlorine per million of air if the air is passed over the copper at the rate of 1.5 litres per minute for six minutes. Smaller amounts of chlorine may be detected by prolonging the time for the accumulation of the halogen on the gauze.

W. P. S.

Sensitive Reaction of Cobalt. J. Bellucci. (Gazz. Chim. Ital., 1919, 49 294-298.)—The colour reaction given by cobalt with  $\beta$ -nitroso- $\alpha$ -naphthol is capable of detecting 1 mgrm. of the metal in 17 litres of water, and is therefore much more sensitive than the corresponding reaction with  $\alpha$ -nitroso- $\beta$ -naphthol (Analyst, 1918, 43, 318), the limit of the sensitiveness for which is 1 mgrm. in 1 to 2 litres of water. The coloration ranges from pink to garnet red according to the amount of cobalt, and in some cases an amorphous red precipitate is formed after the liquid has stood for some hours.

C. A. M.

Volumetric Estimation of Iron. J. Houben. (Ber., 1919, 52, 2,072; through Chem. Zeit. Ubersicht, 1920, 44, 41.)—In solutions of ferric and ferrous salts containing free mineral acids, the ferric iron and free acid may be estimated by reducing ferric iron by means of hydrogen sulphide and then titrating with  $\frac{N}{1}$  alkali hydroxide, making use of ferrous sulphide as indicator.

G. C. J.

Estimation of Ferrous Iron. J. M. Kolthoff. (Pharm. Weekblad, 1919, 56, 1,565; through Chem. Zeit. Übersicht, 1920, 44, 41.)—A disadvantage of the iodimetric estimation of ferrous iron is the necessity for working with air-free solutions. The author has worked out a method free from this objection. The ferrous solution contained in a stoppered flask is, after acidification, treated with 25 c.c.  $\frac{N}{10}$  potassium bromate, and 10 c.c. 25 per cent. phosphoric acid, then with 5 c.c. of potassium iodide solution (10 per cent.), and two drops of molybdate solution. After five minutes the separated iodine is titrated with  $\frac{N}{10}$  thiosulphate. The molybdate is added to accelerate the reaction between bromate and iodide.

Action of Water on Lead. J. F. Liverseege and A. W. Knapp. (J. Soc. Chem. Ind., 1920, 39, 27-32T.)—Experiments have been made with a slightly alkaline natural water, which, like pure rain water, has the power of eroding lead so that the scales first formed on the metal fall off, leaving a bright surface exposed to further action. This water, from the Caban reservoir, Wales, has an alkalinity, due to bicarbonates, etc., equal to about 0.3 part CaCO, per 100,000. Erosion has been found to be due to the action of oxygen in the presence of water, but the variations in the natural proportion of oxygen in water do not appear to influence the amount of erosion. One day is the most suitable period for applying the erosion test, the results of which are influenced by exposure of the water to glass, and by the depth at which the lead is immersed; the greater the depth, the slower being the erosive action. The velocity of erosion falls with the duration of the test. The amount of lead eroded varies directly with the area of the metal exposed, but variations in the volume of water or small changes in the area of water surface have no appreciable effects upon the results. Erosion occurs readily in waters containing no carbon dioxide, but slight variations in the percentages of carbon dioxide in a water do not materially affect the results. The presence of 1 to 2 per cent. of carbon dioxide causes a sudden change from "erosion" to "plumbo-solvency," and the solution of lead takes place more rapidly when oxygen is present in addition to carbon dioxide. The alkalinity of the water is the chief factor, after the presence of oxygen, in determining the amount and the kind of erosion. The addition of small quantities of calcium hydroxide, carbonate and bicarbonate, or of potassium permanganate, decreases erosion, the most effective agent being calcium bicarbonate, 2 parts of which per 100,000 is usually sufficient for the purpose. In the case of calcium hydroxide, the erosion falls to a minimum when about 7 to 10 parts per 100,000 are present. Increasing the calcium hydroxide above this point causes an increase in the erosion, which gradually reaches its original amount. The addition of 5 parts of potassium permanganate per 100,000 was found to form a protective coating, which prevented erosion. Erosion takes place in the absence of bacteria. On the other hand, the bacteria in water are not all destroyed by exposure to the action of lead for six hours. C. A. M.

Separation and Estimation of Lead and Bismuth. G. Luff. (Chem. Zeit., 1920, 44, 71.)—The author agrees that in experienced hands the methods of Löwe and Jannasch are the best. In any but experienced hands, however, they lead to very poor results. The following method is said to give good results in the hands of any chemist who will follow the directions given. To the nitric acid solution of lead and bismuth, ammonia is added drop by drop until a faint permanent turbidity results. This is destroyed by the addition of the least possible quantity of nitric acid. Sufficient saturated ammonium nitrite of this solution is added to the lead-bismuth solution, which is diluted to 200 c.c., covered with a clock glass, and heated to boiling. When nitrogen ceases to be evolved, the solution is filtered, and the precipitate of bismuth hydroxide or basic nitrate washed with hot water, and then dissolved in dilute (1:1) nitric acid. The filtrate is evaporated to dryness, and the residue ignited and weighed as Bi<sub>2</sub>O<sub>3</sub>. It may contain as much as 5 per cent.

of silica and 5 per cent. of alkali. The former is expelled by treatment with nitric and hydrofluoric acids, after which the alkali can be readily washed out. The filtrate from the bismuth is acidified with acetic acid, treated with potassium dichromate, and the lead chromate is collected in a Gooch crucible, washed with hot water, dried at 120° C., and weighed.

G. C. J.

Volumetric Estimation of Sulphates by Oxidation of Benzidine Sulphate with Potassium Permanganate. P. L. Hibbard. (Soil Sci., 1919, 8, 61-65; through J. Soc. Chem. Ind., 1920, 39, 188A.)—A modification of the method of Raiziss and Dubin (J. Biol. Chem. 1914, 18, 297). The solution for the precipitation of benzidine sulphate should be free from iron, heavy metals, nitrates, phosphates, and organic matter, and should only contain between 0.5 and 4.0 mgrms. "SO4." The liquid (10-25 c.c.) is treated with one drop of phenolphthalein and then with dilute sodium hydroxide till just alkaline. It is then neutralised with N hydrochloric acid and 0.1 c.c. added in excess for each 5 c.c. of solution. Keeping the liquid quite cold, 5 c.c. of 0.8 per cent. benzedine hydrochloride solution is added, and after 15-30 minutes the benzidine sulphate is collected in a Gooch crucible, washed with the minimum of cold water, transferred with the asbestos pad to the precipitation flask, and heated with 1 c.c. of 10 per cent. sodium hydroxide solution in a boiling-water bath for a few minutes. Then 10 c.c. of dilute sulphuric acid (1 acid: 1 water) is added and hot water to bring the volume to 100 c.c. No potassium permanganate is run in until there is 5 c.c. in excess and the solution heated on the water bath for exactly ten minutes. Then 10 c.c. of  $\frac{N}{20}$  oxalic acid is added and the titration completed with N-permanganate. The factor for the conversion of c.c.s of permanganate solution into mgrms. of "SO<sub>4</sub>" is 0.12. It is advisable to make a blank determination.

Iodimetric Estimation of Sulphides. J. M. Kolthoff. (Pharm. Weekblad, 1919, 56, 1413; through Chem. Zeit. Übersicht, 1920, 44, 41.)—Contrary to the statement of Treadwell, the author finds that, in the iodimetric estimation of hydrogen sulphide, the resulting sulphur occludes no iodine.

G. C. J.

Separation of Zirconium and Titanium as Phosphates. J. Brown and H. T. Madden. (J. Amer. Chem. Soc., 1920, 42, 36-39.)—A method is described for the gravimetris estimation of titanium in the filtrate from the zirconium phosphate precipitate (cf. Analyst, 1918, 43, 42). The solution containing zirconium and titanium sulphates is acidified with 1 to 2 per cent. (by volume) of sulphuric acid, and excess of hydrogen peroxide is added, followed by an excess of disodium or diammonium phosphate. After about eighteen hours the zirconium phosphate is collected on a filter, washed with water containing a few drops of sulphuric acid and hydrogen peroxide, dried, fused with sodium carbonate, the melt extracted with water, and the insoluble portion fused with sodium hydrogen sulphate and dissolved in dilute sulphuric acid. The zirconium in this solution is then reprecipitated by the

addition of hydrogen peroxide, and phosphate as described, the precipitate collected, dried, ignited, fused with sodium carbonate, dissolved in sulphuric acid, and the zirconium precipitated with ammonia and weighed as  $ZrO_2$  after ignition. If the zirconium phosphate after the second precipitation is still coloured yellow owing to the presence of a remaining trace of titanium, it must be dissolved and precipitated a third time. The combined filtrates from the zirconium phosphate precipitates are heated to destroy hydrogen peroxide, the volume of the solution adjusted so that it contains 2 per cent. (by volume) of sulphuric acid, an excess of disodium or diammonium phosphate is added, the precipitated titanium phosphate collected after one hour, washed, converted into the dioxide as in the case of the zirconium, and weighed.

W. P. S.

#### APPARATUS, ETC.

Volumetric Testing of Scientific Glassware at the National Physical Laboratory. J. G. Petavel. (Report, National Physical Laboratory, November, 1919.\*)-Volumetric glassware has been tested on a small scale at the National Physical Laboratory for the past fifteen years, although its actual manufacture is an industry which was practically non-existent in this country before the war. Latterly, however, the industry has largely developed in this country, and it is desirable that it should remain permanently British. It is quite evident that British-made graduated apparatus should be of reliable accuracy, and many will feel that a reputation for such accuracy could best be guaranteed by an impartial institution such as the metrology department of the National Physical Laboratory. co-operation with manufacturers and users of scientific glassware this department issued in July, 1918, a pamphlet containing regulations relating to Class A tests -i.e., tests on apparatus required to be of the highest accuracy. A new building has just been completed at the laboratory specially designed for dealing with this class of work on a large scale, and they are now prepared to undertake Class B tests. pending the settlement of a permanent scheme for commercial testing by State or State-approved institutions—a matter which is now under the consideration of the Government. As regards Class B tests, the limits of error assigned are such as all graduated apparatus of good commercial quality should comply with as necessary for the attainment of satisfactory results in ordinary routine analysis. Apparatus graduated under the Class A and Class B limits are etched respectively with the date and monogram of the National Physical Laboratory. Vessels of ordinary type, such as used in volumetric analysis and other customary operations of the chemical or physical laboratory, and measuring vessels for gas analysis, are admitted to the Class A tests. The Class B tests are also available for these types of apparatus, for certain similar types fulfilling somewhat less stringent requirements, and for rain gauges and measuring vessels for milk analysis. The tolerances allowed in a few typical cases are:

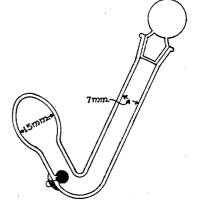
<sup>\*</sup> Copies of this report may be obtained gratis from the Director of the National Physical Laboratory, Teddington.

				•		Class A Limits.	Class B Limits.
2 c.c. Pipette			• • •	• • •	•••	0.006 c.c.	0·012 c.c.
100 c.c. ,,						0.05 c.c.	0.08 c.c.
100 c.c. Flask						0.05 c.c.	0·10 c.c.
1,000 c.c. ,,						0.20 c.c.	0.30 c.c.
50 c.c. Burette	•••	• • •		•••		0.04 c.c.	0.07 c.c.

The fees for testing flasks and pipettes range from 6d. to 1s. per instrument. burette is tested at five points for 2s. 6d. A charge of 6d. extra per instrument is made when certificates are issued. The highest fee charged is 5s. for a gas burette. including certificate. The above particulars as to limits and fees give a brief outline relating to a few special cases only. They are intended to give some idea of the accuracy required and the charges made. A full account of the tolerances allowed, methods of test, details of constructions, test fees, etc., is given in this report which was drawn up in co-operation with both manufacturers and users of volumetric apparatus. Users may send apparatus direct to the laboratory for test, or may specify when placing orders for apparatus that it is to be supplied with the National Physical Laboratory Class A or Class B mark, as required. If a certificate of corrections is desired with a Class A vessel this also should be specified. It is hoped that in the near future dealers will be in a position to supply verified ware from stock. In the event of users of apparatus requiring tests other than those provided for in the pamphlet, the laboratory is always prepared to make special arrangements to meet the particular requirements of any special work. H. F. E. H.

Simple Hydrogen Electrode. C. H. Bailey. (J. Amer. Chem. Soc., 1920, 42, 45-48.)—An electrode which is simple in construction and operation, has a short interval between the platinised electrode and the potassium chloride solution and in

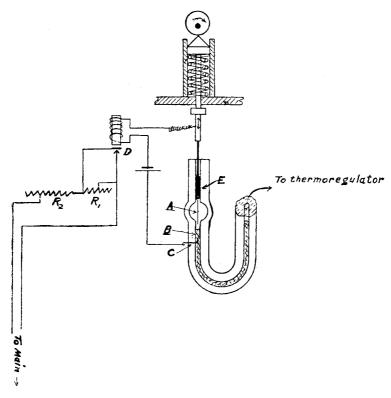
which the platinised electrode is immersed completely, is shown in the figure. The metal electrode consists of a disc of thin gold plate 5 mm. in diameter and is platinised in the usual way. To use the apparatus, the liquid under examination is introduced so as to fill the bulb arm completely, and the open arm to a depth of about 10 mm. above the top of the bend; hydrogen is now admitted through a thin glass tube until the bulb arm is full of gas and the liquid displaced into the open arm. If the latter is not full, more of the liquid is added, the stopper inserted so that excess of liquid is expelled and air excluded, and the apparatus shaken for two minutes. It is



then tilted so that enough liquid flows into the bulb arm to just cover the disc electrode, the stopper is removed, and part of the liquid in the open arm is drawn off. To complete the circuit, the disc electrode is connected with the potentiometer, and a thin siphon tube filled with saturated potassium chloride solution is dipped into the open arm. The end of the siphon in the open arm is drawn out to a fine point, and pieces of filter-paper soaked in potassium chloride solution are forced into the tip by means of a needle; this filter-paper prevents appreciable siphoning action while the readings are taken. The other end of the siphon dips into a vessel filled with saturated potassium chloride solution which in turn is connected with the calomel electrode. The tip of the siphon can be brought very close to the platinised electrode, thus reducing the resistance of the cell.

W. P. S.

Gouy Thermoregulator. T. S. Sligh, jun. (J. Amer. Chem. Soc., 1920, 42, 60-68.)—The working part of the apparatus is shown in the figure; a rise in temperature of the thermoregulator bulb (not shown) causes the meniscus B of the mercury in the U-tube to make contact with the platinum wire A, whereupon current flows



through the relay circuits opening a contact at D, thus introducing an additional resistance R in the heating circuit or opening this circuit entirely. The platinum wire A is fitted to the guide-plug E, and this is given a periodic oscillating motion along the axis of the capillary tube by means of the cam and spring mechanism shown. The range of this motion may be conveniently 0.1 cm., and the period of the

motion one second. Such a regulator will reduce the variations of the bath temperature and the erratic variations due to variations of the mercury surface to a fraction of the values obtained with the usual form of fixed wire thermoregulator. Regulation to within 0.0001° C. may be obtained. In addition, variations of mean bath temperature due to variations in external conditions are reduced to a very considerable extent.

W. P. S.



#### REVIEWS.

THE CHEMISTRY AND TECHNOLOGY OF THE DIAZO-COMPOUNDS. By JOHN CANNELL CAIN. Second Edition. Pp. xi+199. 1920. London: Edward Arnold. Price 12s. 6d. net.

Twelve years have passed since the first edition of this volume was published, and the scope has been somewhat enlarged in the present issue by including many references to the technical literature in order to render it more useful to industrial chemists. In its new form it makes a much wider appeal, and should be acceptable not only to all students of chemistry, but also to those who are occupied with the technology of dye-manufacture.

From the standpoint of systematic nomenclature, adherence to the expressions "diazo-salt," "diazobenzene sulphate," etc., until the concluding chapters is perhaps to be regretted. The word "diazonium" first occurs on page 156, and since it has now been the practice, for many years, to present the subject to students in the light of the diazonium theory, it would increase the value of the work as a class-companion if the introductory chapter, which surveys events from 1858 to 1916, included also a reference to this fundamental principle, and if thereafter the denomination of the respective compounds were harmonised therewith. An added reason for this procedure is offered by the existence of a large class comprising genuine diazo-compounds—namely, those belonging to the aliphatic series; since their inability to form salts distinguishes them sharply from the diazonium compounds, the two names, when systematically used, provide an excellent means of classification.

A minor criticism suggests itself in connection with the section describing the aromatic azoimides. The action of ammonia on diazonium perbromides and of hydrazine on diazonium salts does not involve "replacement of the diazo-group by the azoimino-group" in the sense that this occurs when diazonium salts are mixed with sodium azide, because in the former cases nitrogen is added instead of being eliminated; yet the three reactions are grouped under the heading above quoted. Moreover, the author does not mention the use of the word "triazo" in place of "azoimino," although it is more convenient and more accurately descriptive, since "azoimino" represents literally the arrangement

The maze of conflicting theories regarding the constitution of diazonium salts and of the metallic derivatives from the respective bases, associated more particularly

with the names of Hantzsch and Bamberger, has been carefully threaded, and a useful summary of heterocyclic types capable of undergoing diazotisation has been given. The chapter on aliphatic diazo-compounds, however, is not quite commensurate with the interest and importance of the subject, and might very profitably be extended in a subsequent issue. As one instance only, phenylbenzoyldiazomethane (diazodesoxybenzoin) deserves notice in connection with Schroeter's remarkable transformation of that substance into diphenylketene, since it offers the most convenient method of preparing the latter.

Having modified the title of the book suitably to the wider field which it now covers, the author might consider the desirability of reviewing a few typical analytical methods, such as the use of titanous chloride by Knecht. The subject of diazo-chemistry is one of such vast importance, historical, theoretical, and industrial, that it fully justifies a substantial volume being devoted to itself, and the foregoing remarks are offered solely in the hope that this excellent work, when it appears in the third edition, may be improved still further.

M. O. Forster.

POPULAR CHEMICAL DICTIONARY. By C. T. KINGZETT, F.I.C. Demy 8vo.; Pp. vi + 368, with seventy-eight figures in text. 1919. London: Baillière, Tindall and Cox. Price 15s. net.

This book embodies a desire to afford the general reader a true conception of the meaning of words that either are peculiar to chemistry or are used in some special sense in the science. The numbers of articles and phenomena that have been defined without use of a scientific word are surprising. Where this has not been achieved, the meaning of the intruding expression is certain of explanation in its alphabetical position, and the reader has not to waste time before his original enquiry is satisfied.

Usually the definitions are simple, pithy, and happy in expression. Sometimes, however, the words are such as might be spoken by one suddenly asked for information, and the result is long, involved, and far too dependent on commas. The book is well-balanced and comprehensive. Except for the oxide, the ferric salts have been overlooked. The treatment of volumetric analysis is weak, the only indicators mentioned being litmus, turmeric, and phenolphthalëin. The spectroscope is only applied to inorganic bodies. On the other hand, two monographs on the same subject appear under "India-rubber" and "Rubber."

Actual errors are few, and generally trivial: Cascara sagrada is defined as an extract, *Cocculus indicus* is said to be synonymous with "grains of Paradise," bloodheat is given as 36.6° C., acetone is credited with a pungent odour, *Cacao*-fat is said to be used in making soap and candles, basic-slag is given as a source of potash, pumice-stone is described as "molten," and a factor of 0.648 for the conversion of grammes to grains is given.

Misspelt words include cajeput, saccharine (for saccharin), saccharometers, conchinine (probably for quinidine), Aleuritis moluccana, Bacillus butylicus, proprionic acid, glycolic acid, wolfram (for wolframium), and Mycodermi aceti.

Though there can be no pus without them, leucocytes are not mentioned in

explaining it. It is exaggeration to say that bacteria "require to be magnified many thousands of times to become visible."

Taraxacum is said to be a diuretic; it is generally used as a laxative. It is difficult to see why liquid paraffin should be "a mild substitute for cod-liver oil." The mercury barometer is described without mentioning the vacuum.

Mr. Kingzett is correct in his scientific phraseology, and usually manages to avoid the weird and barbarous words which chemistry has collected to itself through the centuries. A good part of the text has been carried forward from "Chemistry for Beginners," so have most of the illustrations, the latter generally from blocks lent by dealers in apparatus, with the well-known names in the pictures.

WILLIAM PARTRIDGE.

CHEMICAL CALCULATION TABLES FOR LABORATORY USE. By Horace L. Wells Second Edition, Revised. 43 pp. 1919. New York: John Wiley; London: Chapman and Hall. Price 6s. 6d. net.

The present work is a revision and modification of the author's "Tables for Chemical Calculations," published sixteen years ago, and now out of print. A new table for facilitating the calculation of percentage composition of organic compounds has been included, while the five-place logarithmic table is provided with a novel double thumb-index, enabling the reader to turn backwards or forwards from any place in the table to any other reference by a single motion, "since all indices are visible from any part of the table." In the copy before the reviewer this is, unfortunately, not quite true, since the registering of some of the marginal cuts is not sufficiently accurate to ensure visibility of all figures. In other respects the work is well printed, and appears to have been compiled with care and accuracy. The tables included deal with atomic weights (1919), gravimetric factors, formula weights, reduction of gas volumes to 0° and 760 mm., calculation of percentage of nitrogen from gas volumes, barometer corrections for temperature, constants for molecular-weight determinations, weights and measures, and the logarithmic tables above alluded to. H. F. E. HULTON.

CHEMISTS' MANUAL OF NON-FERROUS ALLOYS. By JAMES R. DOWNIE. Pp. 168. 1920. London: E. and F. N. Spon, Ltd. Price 10s. net.

This book describes the analysis of metals and alloys, and its object, according to the preface, is to present a "reliable guide" to chemists interested in non-ferrous alloys, and "to give in as concise a form as possible information which hitherto has been scattered throughout various textbooks." The author would have been well advised had he given a full list of such textbooks; as it is, the manual does not contain a single reference to literature.

From the point of view of reliability the work can only be pronounced a complete failure. It is impossible within the limited space at the reviewer's disposal to enumerate all its shortcomings; sufficient to give some of the more startling statements found in Chapter IX., headed "Refraction of Stock Metals." For the determination of sulphur in copper (p. 105) and in nickel (p. 118), the author recommends

solution in nitric acid, neutralisation with ammonia, and addition of hydrochloric acid before precipitating with barium chloride. This looks suspiciously like an attempt at converting the nitric into a hydrochloric acid solution. In the determination of phosphorus in copper (p. 105), the nitric acid solution of the metal is evaporated twice with hydrochloric acid, presumably with intent to volatilise arsenic (in its higher state of oxidation), for the chloride solution is next treated with molybdate or magnesia mixture. Arsenic in copper is determined (p. 106) by neutralising the nitrate solution with caustic soda, adding 1 grm. of sodium carbonate, re-dissolving the precipitate, and precipitating the solution thus obtained with magnesia mixture. One would like to know what prevents phosphorus from interfering in this determination. In the analysis of tin (p. 107), antimony is titrated in hydrochloric acid solution with thiosulphate after addition of potassium iodide, in presence of iron, copper, and arsenic, while phosphorus is determined (p. 109) with magnesia mixture in the filtrate from the ferric hydroxide precipated by ammonia. In the determination of cobalt in nickel bullion (p. 118), the nickel from a 10-grm. sample is first eliminated by precipitation with dimethylglyoxime the precipitate having to be filtered off and thoroughly washed. The diameter of the filter required for this operation is not mentioned.

A list of "typical analyses" of metals and alloys is appended. Here the author has clearly committed a double error of judgment; not only has he estimated the principal constituent direct instead of by difference (for none of the complete analyses sum up to a round 100), but, though such direct determinations are hardly accurate to 0.2 per cent., he has calculated the results to the third decimal place, thus: Al 99.014, Sn 0.063, Fe 0.614, Cu 0.048, Si 0.164, Total 99.904 (p. 159). The author does not appear to be over-anxious as to the correctness of chemical formulæ; cobalt pyrophosphate is  $\text{Co}_2\text{P}_2\text{O}_4$  (pp. 44, 52, 151), but the correct cobalt factor is given. Bismuth chromate, with the puzzling bismuth factor 0.62561, is given the formula  $\text{BiO}_3$  ( $\text{Cr}_2\text{O}_3$ )<sub>2</sub> (pp. 57, 151). Sodium uranate is  $\text{NaO}_2\text{Ur}_2\text{O}_3$ , with a uranium factor of 0.876 (pp. 45, 151).

The preface also states that "the processes are given for the sake of clearness in as direct a statement as possible without regard to literary style." The disregard of style is manifest, with impaired clearness as an inevitable result. Numerous misprints, some serious, occur in the text. The book is intended also for the use of beginners, but a less trustworthy counsellor for such it is difficult to conceive.

W. R. Schoeller.

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#### THE INSTITUTE OF CHEMISTRY OF GREAT BRITAIN AND IRELAND.

AT the 42nd Annual General Meeting of the Institute of Chemistry held at 30, Russell Square on March 1, Sir Robert Robertson, Vice-President, occupied the Chair in the place of Sir Herbert Jackson, the President, who was absent through illness. In moving the adoption of the Report of Council, Sir Robert read the President's address, in which reference was made to the position of professional men

under prevailing economic conditions. Notwithstanding the limited number of appointments carrying reasonably high salaries and the difficulty of rising above the standard of mediocrity, most of the professions were attracting such a high proportion of the educated youth of the country that they were likely to be overcrowded. On the other hand, the Government and the industries of the country were becoming more and more alive to the necessity and the wisdom of making appointments for chemists more attractive to the best qualified. In order to secure better remuneration and conditions of appointment chemists were urged to do all in their power to render themselves prepared for such opportunities as came their way. The Institute was ready to help them to secure suitable appointments, and endeavoured to encourage the employment of the competent. The situation was far more promising than at the time of the Armistice. Over 530 chemists whose names had been on the Appointments Register had now no further need of this assistance.

The Institute had been in communication with several of the Whitley Industrial Councils for industries on which chemistry had a bearing, and although under the constitution of the Whitley Councils no provision was made for the direct representation of chemists, several were acting as employers' representatives, and the Councils were prepared, where occasion arose, to turn to the Institute for assistance. The roll of the Institute was steadily increasing, numbering nearly 3,000 Fellows and Associates, and over 500 Registered Students. The Council were engaged in work of reconstruction—remodelling the By-laws, providing for district representation on the Council, and revising the regulations for admission to membership. had taken up the question of securing representation of chemistry in the Ministry of Health, with the satisfaction of seeing Sir William Tilden appointed a member of the Council on Medical and Allied Services, and Dr. J. F. Tocher appointed Chemist to the Scottish Board of Health. Attention was being given to the continued production by British manufactures of glass apparatus, chemical reagents, and other laboratory requirements which hitherto were obtained mainly from Germany and Austria.

Jointly with the Institute of Metals, a committee was engaged on questions affecting the status and organisation of chemists and metallurgists with the Navy, Army, and Air Force. Mention was made of the activity of the local sections and of the help rendered by them and the Honorary Corresponding Secretaries Overseas in the work of the Institute.

The Officers and Members of Council for the year 1920-21 were elected as follows:

President: Sir Herbert Jackson, K.B.E., F.R.S.

Vice-Presidents: Horatio Ballantyne, Sir James Johnston Dobbie, LL.D., F.R.S., Ernest Mostyn Hawkins, Gilbert Thomas Morgan, D.Sc., F.R.S., Sir Robert Robertson, K.B.E., F.R.S., George Stubbs, O.B.E.

Hon. Treasurer: Edward William Voelcker, A.R.S.M.

Members of Council: Walter Ernest Adeney, D.Sc., A.R.C.S.I., William Bacon, B.Sc., Edward Charles Cyril Baly, O.B.E., F.R.S., Oscar Lisle Brady, D.Sc., Francis Howard Carr, Alfred Chaston Chapman, Allin Cottrell, M.Sc., Alexander Charles Cumming, O.B.E., D.Sc., John Thomas Dunn, D.Sc., Lewis Eynon, B.Sc.,

Alexander Findlay, M.A., D.Sc., Ph.D., George Watson Gray, Frank William Harbord, C.B.E., A.R.S.M., Charles Alexander Hill, B.Sc., Patrick Henry Kirkaldy, Joseph Henry Lester, M.Sc., William Macnab, Samuel Ernest Melling, Gordon Wickham Monier-Williams, O.B.E., M.C., M.A., Ph.D., Andrew More, A.R.C.S., Frederick Mollwo Perkin, Ph.D., George Henry Perry, M.B.E., B.Sc., A.R.C.S., Benjamin Dawson Porritt, M.Sc., Francis Martin Potter, M.B.E., B.Sc., A.R.C.S., John Rogers, O.B.E., Ernest Woodhouse Smith, D.Sc., William Maurice Gathorne Young.

#### PROFESSIONAL FEES.

THE Council of the Institute of Chemistry have received many communications relating to the fees for analyses and investigations at present charged by private practitioners.

Having regard to the facts that, in addition to the increased cost of living, practising chemists have to pay higher salaries to their assistants and to bear the enhanced cost of apparatus and materials, the Council are of opinion that Fellows and Associates are justified in making a corresponding increase in their fees for professional services.