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THE ANALYST.

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

ORDINARY MEETING, APRIL 6, 1921.

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 Messrs. William Ellard Woolcott and Thomas Henry Pope, B.Sc., F.I.C. Certificates were read for the second time in favour of Messrs. Percy N. Mould and W. J. Wright, F.I.C.

The following were elected Members of the Society: Messrs. Jules Cofman-Nicoresti, Walter K. Fletcher, William Singleton, James Darnell Granger, Ph.D. (Berlin), F.I.C., Edward Bradford Maxted, Ph.D. (Berlin), B.Sc. (Lond.), Russell George Pelly, F.I.C., and Francis George Henry Tate.

The following papers were read: "The Estimation of Strychnine in Scale Preparations containing Quinine and other Cinchona Alkaloids," by T. F. Harvey, F.I.C., and S. Back; "The Action of Water on Lead," by J. C. Thresh, D.Sc., M.D., F.I.C.; "A Colour Reaction for Aconite," by S. Mallanneh, M.D., D.P.H.; and "A Method for the Determination of the Acidity of Coloured Solutions," by J. L. Lizius, B.Sc., A.I.C.

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OBITUARY NOTICES.

CHARLES ALEXANDER CAMERON.

ALTHOUGH Sir Charles Cameron was personally known to but a small proportion of our present members, his name has been so long and so closely connected with the Society that his death, which took place on February 27, will be felt as a loss by all of us.

Cameron, who was born in 1830, was the son of an officer who served in the Peninsular War and in the war against the United States. He was educated in Dublin and in Guernsey, where his parents lived from 1844 to 1846. On the death of his father he returned to Dublin, and entered the laboratory of Messrs. Bewley and Evans, apothecaries. Here he acquired a practical knowledge of pharmaceutical chemistry, meanwhile attending lectures on chemistry, physics, and medicine at the Royal Dublin Society.

In 1852 the Dublin Chemical Society was founded, and Cameron was elected its Professor of Chemistry; and this appointment soon led to the Professorship of Chemistry and Natural Philosophy in the Dublin School of Medicine, to which he was appointed in 1856. Soon afterwards he became Professor in the Royal College of Surgeons, Ireland.

In 1860 the first Food and Drugs Act was passed, but, as the appointment was made permissive, few authorities troubled to appoint Public Analysts, and, in consequence, the Act soon became a dead letter. Dublin, however, was one of the few towns to make use of the Act, and, in 1862, Cameron was elected Public Analyst for that city—a post which he retained until his death. He was the third Public Analyst in the United Kingdom to be appointed under the Act. Subsequently, when the Act of 1870 was passed, rendering the appointment of Public Analyst obligatory, Cameron was chosen by no fewer than thirty-three counties and boroughs as their official analyst. In fact, from the number of these appointments he was often humorously referred to as "The Public Analyst for Ireland."

In the early days of our Society, Cameron contributed numerous papers on the analysis of milk, water, and drugs, which were published in THE ANALYST. He served on the Council in 1878-81, became Vice-President in 1882-3 and again in 1889-90, and held the office of President in 1893 and 1894. He was also a Vice-President of the Institute of Chemistry. In addition to his chemical posts, he held numerous medical appointments, being Medical Officer as well as Public Analyst for Dublin, and he served as President of the Royal College of Surgeons of Ireland.

He was an honorary M.D. of the Royal and National Universities of Ireland and an honorary Fellow of the Royal College of Physicians of Ireland. In 1885 he was knighted in recognition of his efforts to improve the dwellings of the working classes, and in 1898 was appointed a C.B. in recognition of his long services in his important public posts.

Of forensic medicine and toxicology Cameron was long the acknowledged chief exponent and practitioner in Ireland, and his versatility was such that he for many years occupied the leading position in the country as both teacher of and practitioner in agricultural chemistry. His re-written edition of Johnston's earlier well-known work on this subject long occupied the position of a leading textbook. Among his many other contributions to scientific literature was a history of the Irish Royal College of Surgeons, his intimate association with which has been already indicated.

Few men can have had a larger circle of friends than Cameron or were more popular in Dublin society. He was delightful as a host and as a guest, and a charming talker with an ever-flowing fund of wit and humour, never exercised, however, at the expense of pain to others, for his kindliness was even greater than his wit.

Apart from his numerous professional occupations, one of the main delights of his less strenuous hours was in Freemasonry, of which he was one of the most ardent and most honoured supporters in his native country.

But not the least of his achievements was the retention of his full mental faculties and his physical working powers beyond his ninetieth year.

BERNARD DYER.

ALEXANDER WYNTER BLYTH.

We deeply regret to record the loss of one of the original members of our Society, Dr. Alexander Wynter Blyth, whose death took place suddenly on March 31.

Wynter Blyth was the son of a medical practitioner, and was born in 1846 at Woolwich. He was educated for the medical profession at King's College, London, and became qualified in 1870. At a later period he studied law, and was called to the Bar as a member of Lincoln's Inn.

In 1878 he was appointed Public Analyst for Totnes, and subsequently for the County of Devon, in 1879 for Tiverton, and in 1882 Medical Officer of Health and Public Analyst for St. Marylebone, posts which he held until his death.

In the early days of the Food and Drugs Acts the appointments of Medical Officer and Public Analyst were frequently held by the same individual, and it was quite a common practice for medical men to spend a few months in the laboratory of a chemist to acquire some insight into the duties of what was then regarded as a subsidiary appointment.

Under such conditions it is not surprising that many of the early medical Public Analysts were not competent chemists, or that few improvements in the methods of chemical analysis originated with them.

Wynter Blyth was a striking exception, for having been trained as a chemist, he took an active interest in the affairs of the Society of Public Analysts from its inception.

A paper of his upon milk was published in 1876 in the *Proceedings*, which was the forerunner of the Analyst. Numerous other contributions upon the analysis of foods and drugs will also be found in subsequent volumes of the journal. He served on the Council of the Society in 1877 and on three other occasions, and was thrice elected Vice-President, the last occasion being in 1895.

He also contributed to the *Transactions* of the Chemical Society, but he will be best remembered by his books on foods and poisons. Originally these formed parts of one volume, but in later editions the subjects were divided and published as separate works. The section on Foods was a comprehensive survey of the existing microscopical and chemical knowledge of the subject, and was one of the earliest attempts to systematise the modern methods of analysis. Its sixth edition in separate form was published in 1909 and reviewed in the Analyst of that year. The second portion of the book included much information which was not readily accessible elsewhere, and as a manual of historical toxicology filled a distinct gap. Its fourth edition as a separate book was reviewed in the Analyst for 1907, and a new edition has just been published.

As an instance of the unexpected detail to be found in this work, it may be recalled that in one of the actions arising out of the epidemic of arsenic poisoning of twenty years ago, it was pointed out that the possibility of glucose being a source of arsenical poisoning had there been mentioned.

It is interesting to note in this connection that Wynter Blyth was one of the earliest chemical investigators of cobra poison, and in 1878 received a Government grant in aid of his research.

He was elected a Fellow of the Institute of Chemistry in 1887 and served on its Council on two occasions. He was also Past President of the Incorporated Society of Medical Officers and Registrar of the Sanitary Institute. EDITOR.

SOREN HOY BLICHFELDT.

MR. SOREN HOY BLICHFELDT died on March 3, after a very short illness, at the age of forty-four.

Blichfeldt was born in Denmark, and received his chemical training at the Royal Pharmaceutical College in Copenhagen. His inclination towards fermentation problems then led him to the world-famous fermentation laboratories in Copenhagen, where he became one of Alfred Jörgensen's most valued assistants. Here he worked for several years on questions connected with the application of pure culture methods in the brewing and dairy industries, and while here he wrote a well-known Danish textbook on bacteriology.

In 1906 he joined the staff of the Maypole Margarine Works (then Messrs. Otto Monsted, Ltd.), and, as its chief technologist, was responsible for much of the development of the improved methods of margarine manufacture now in use. He remained with this firm until his death, at which time he was Assistant Managing Director.

He was always more interested in the application of science to industry than in pure science; hence his contributions to scientific literature lie mainly in the records of the Patent Office.

He was the originator of a distillation method for the estimation of mixtures of butterfat, coconut oil, and palm kernel oil, and a few days before his death his method for the determination of melting-points of fats was read before this Society.

He possessed a remarkable insight into the scientific problems underlying the industry with which he was connected, and his keenness and inexhaustible energy led to their rapid solution. His kindly tolerance and his generous spirit inspired a rare loyalty and enthusiasm in his staff, and his joyous disposition and entire freedom from affectation makes his loss all the greater.

T. THORNLEY.

LIQUID EXTRACT OF RED SQUILL (*SCILLA MARITIMA*) AS A RAT POISON.

BY F. W. SMITH, B.Sc., A.I.C.

(*Read at the Meeting, February* 2, 1921.)

MR. BOULENGER, of the Royal Zoological Society, stated in an official pamphlet published in 1919, that an extract prepared from the bulb of the squill formed a most effective rat poison. It had the great advantage of being practically harmless to domestic animals. Opinions at the time appeared to be much divided as to the efficiency of the squill extract as a rat poison. It was decided, therefore, that

experiments should be undertaken in order to determine the toxic effect upon rats of various preparations of squill.

Samples of the various squill rat poisons on the market were found to differ to a very great degree. Their extractives varied from 2 per cent. to 15 per cent.

The results obtained with these preparations were most unconvincing, for out of five extracts considered, only one caused the death of a rat.

An aqueous extract (100 grms. drug—200 c.c. water) was prepared from the dried official squill (*Scilla urginea*). The toxic effect of this extract upon rats appeared to be nil. It was noticed that this extract was not very bitter.

Kopaczewski (*Comptes rend.*, 1914) discovered that the toxic principle of squill was an amorphous glucoside, which he called " scillitin," and to which he assigned the formula $C_{17}H_{25}O_6$. This glucoside is sparingly soluble in water, but is quite soluble in alcohol. It is easily hydrolysed, yielding dextrose and amorphous products. It is also intensely bitter. The aqueous extract prepared was not bitter. In view of Kopaczewski's observation it would appear that scillitin was absent from the extract, particularly as scillitin is almost insoluble in water.

An aqueous extract prepared from the fresh white squill gave negative results.

From the results obtained with aqueous, alcoholic, and acetic acid extracts, it would appear that the glucoside, scillitin, which Kopaczewski said was most lethal to rats, was present in greater quantities in the red squill. The extract prepared from the dry red squill by means of alcohol also proved very effective ; it was used three times, and proved fatal to rats in each case.

Other results suggest that the convenient method of preserving extracts by means of salicylic acid would not destroy the toxic principle of the squill, and could be adopted, without fear of hydrolysing the glucoside.

In the preparation of the extract, alcohol should be employed, otherwise the toxic glucoside would not be readily extracted. In the final form the glucoside appears to exist in solution in the aqueous liquid, as the alcohol is driven off.

During the first campaign against rats, the red squill was most difficult to obtain. Samples of alleged dried and fresh red squill were collected, and on three occasions proved to be white squill. They were easily identified by the absence of the deep red colour. It was observed that a section of red squill showed, under the microscope, the presence of isolated cells, at irregular intervals, filled with red colouring matter. These cells were not discovered in the white squill. A section of dried white squill coloured with vegetable dye did not show these isolated cells, but a continuous strip of colour. It is apparently impossible to disguise the white squill.

The non-toxic effect of some extracts and their unusual colour suggested the presence of artificial colouring matter in the white squill extract. Experiments were carried out to determine whether the addition of artificial colouring matter could be detected. It was observed that the extract of red squill gave, on evaporation to dryness, a residue which was of a homogeneous brown or brownish-red colour.

Extract of white squill, coloured with vegetable dyes, invariably yielded, on evaporation to dryness, a residue which was not homogeneous in colour, but showed bands of yellow round its outer edges. On spraying the residue with distilled water these yellow bands were more pronounced, owing to the greater solubility of the dye.

When coloured with aniline dye, this effect was not so marked, but on washing the residue, the distinctive yellow colour made its appearance.

In conclusion, I wish to thank Mr. F. J. C. Bird, of Messrs. C. R. Harker, Stagg and Morgan, Limited, in whose laboratory the initial experiments were conducted, and also Mr. T. J. Drakeley, M.Sc., for his kind encouragement and advice.

CHEMICAL DEPARTMENT,
NORTHERN POLYTECHNIC INSTITUTE, HOLLOWAY.

DISCUSSION.

Mr. E. C. READ (*Technical Adviser, Ministry of Agriculture and Fisheries*) remarked that it might be assumed that scillitin was not, as Kobert had stated in his *Lehrbuch der Intoxicationen*, the only active principle in red squill. Preparations from the raw bulb soon lost their toxicity, especially in warm weather. The Pasteur Institute was originally responsible for the introduction of commercial preparations of red squill into this country, and at first they were intended to be used as supplementary in cases where rats and mice proved immune to the bacteriological "viruses."

Mr. C. L. CLAREMONT (*Ministry of Agriculture and Fisheries*) considered that Mr. Smith's experiments with aqueous extracts were unconvincing, since such preparations required diluting with milk and gruel to render them attractive. The whole of the toxic principle was not removed by extraction with alcohol, the residual powder left after extraction with 90 per cent. alcohol being more toxic than the alcoholic extract. It was possible that certain red squills on the market contained bulbs of *S. urginea Burkii*, which appeared to be less toxic than *Scilla maritima*. Chemical and microscopical examination were of little value in distinguishing between the three varieties of squills, and the actual administration to rats was the only ready means of identifying red squill in commercial preparations.

Mr. SMITH, in his reply, remarked that in view of his own experimental results, he could not understand the difficulty experienced in extracting the toxic principle with alcohol. Apparently the alcohol caused exosmosis to take place from the cells, and the glucoside was thus readily extracted.

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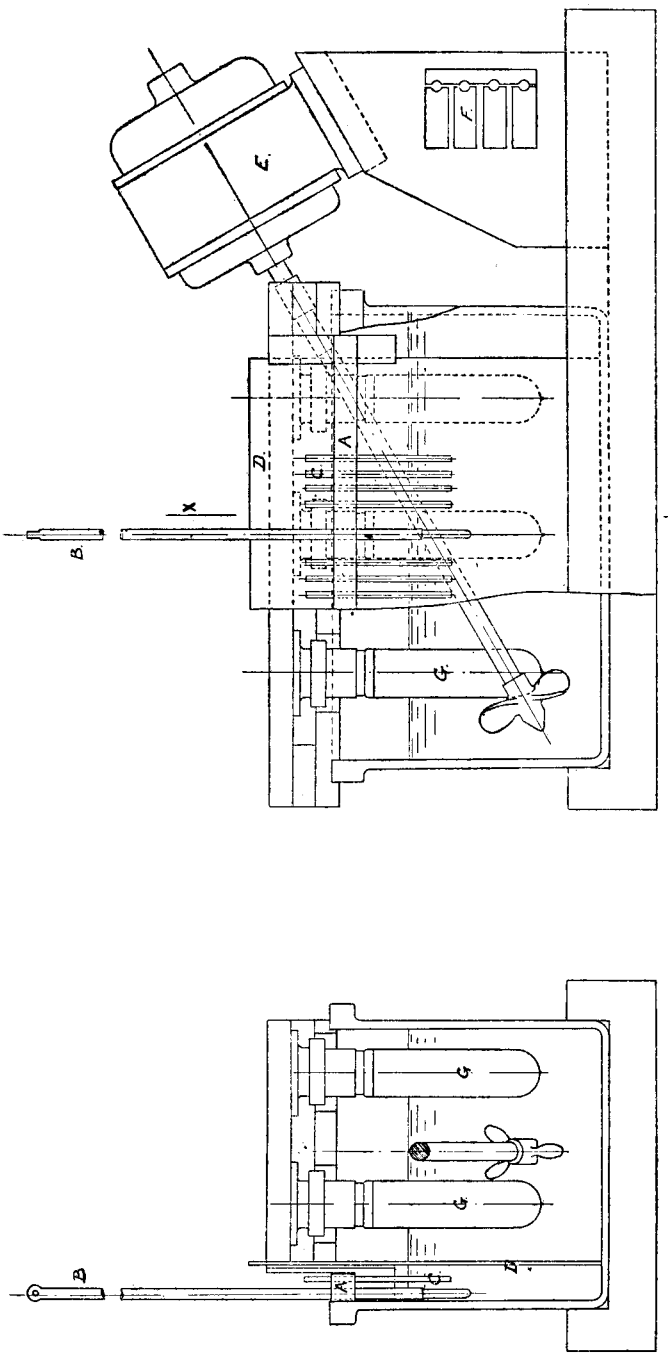
APPARATUS FOR THE ROUTINE DETERMINATION OF MELTING-POINTS OF FATS AND FATTY ACIDS.

BY S. H. BLICHFELDT AND T. THORNLEY.

(*Read at the Meeting, March* 2, 1921.)

A NEED has long been felt for a method of determining melting-points of fats which shall be rapid and accurate, giving strictly comparable and reproducible results, and capable of application by a relatively unskilled operator to a number of samples at one time.

It is difficult accurately to define the melting-point of such a complex mixture of glycerides as a fat. The melting-point varies enormously with the method of



SECTION ON XY

A. SUPPORT FOR THERMOMETER AND TUBES.
B. THERMOMETER.
C. TUBES.
D. OPAQUE GLASS SHEET.
E. MOTOR DRIVING PROPELLER.
F. PLUGS FOR CONNECTING UP LAMPS + MOTOR TO MAINS.
G. HEATING LAMPS.

preparation of the sample and with the method of carrying out the actual determination, and there is no doubt that many of the wide variations in the recorded melting-points are due to divergences in methods and determination.

By "melting-point" in this method is understood the temperature at which a column of fat of specified dimensions begins to move in an open tube of specified dimensions under a definite hydrostatic pressure.

METHOD.—A clean glass tube 6·5 cm. long, 1 mm. bore and 3 mm. diameter, is dipped into the melted mixed sample. The fat is allowed to rise just over 1 cm. in the tube, and the lower end of the tube is then brought into contact with filter paper or any absorbent material, and just so much melted fat is withdrawn that the column remaining is 1 cm. long. The column of completely liquid fatty material is solidified by placing the tube between two blocks of ice, where it remains during two hours.

The tube is then fixed vertically in a water bath capable of being slowly and regularly heated with constant stirring. The tube is immersed so far that the upper surface of the fat is 1 cm. below the level of the water. The water is heated so that the temperature rises 1° C. per minute, and the temperature at which the fat begins to slide up the tube is noted as the melting-point.

The apparatus shown is adapted for the simultaneous determination of melting-point of a number of samples. A glass tank carries a hard cover, on the under side of which are mounted sockets for six 16 c.p. carbon filament lamps. These are connected in three pairs with plug sockets, so that two, four, or six lamps may be connected with the supply. The fourth plug switches in the motor driving the stirring propeller.

The cover supports vertically a sheet of black or dark-blue glass, which divides the tank into two compartments. The large compartment contains the heating bulbs and propeller, and the smaller one the sample tubes and thermometer. The sheet does not quite reach the ends of the tank, so that, when the propeller is running, a rapid current of water flows past the tubes without too much commotion on the surface. Sufficient light is reflected round the ends of the sheet to provide ample illumination of the tubes. The bar carrying the tubes is supported by a pair of ebonite brackets, and the thermometer is slung by wire hooked over the top of the glass sheet.

By adherence to such a method, the wide divergences in melting-points of fats indicated by the published figures seem to be considerably reduced.

The melting-points of butter fat, oleo, and palm oil samples, determined after rapid and slow cooling are given in the following table:

	Quick Cooling. Two Hours in Ice.	Slow Cooling. Twenty-four Hours at Room Temperature.
	° C.	° C.
Butter	31·0	32·2
Oleo	29·3	32·1
Palm oil	35·0	37·4

THE DETECTION OF ADULTERATION IN BUTTER BY MEANS OF THE MELTING-POINT OF THE INSOLUBLE VOLATILE ACIDS.

By GEORGE VAN B. GILMOUR, B.Sc. (Lond.), A.R.C.Sc.I., A.I.C.

(*Read at the Meeting, March* 2, 1921.)

Use has already been made of the melting-points of the insoluble volatile acids from coconut and palm kernel fats for distinguishing between these fats in margarines and other mixtures (see Blichfeldt, *J. Soc. Chem. Ind.*, 1919, **38**, 150-152T). The object of this investigation was to determine the melting-point of the insoluble volatile acids from butter fat, to see how this varies with different varieties, and to find out if the determination of the melting-point would be useful in detecting the presence of coconut and palm kernel fats in butter.

The term "melting-point," wherever used in this paper, refers to the melting-point of the insoluble volatile acids.

The melting-point determinations were made upon the insoluble volatile acids obtained by using the Blichfeldt distillation apparatus, the procedure being as follows: After the distillation is completed, the apparatus is disconnected and the acids transferred while still warm to a clean test-tube. This is done by manipulating the apparatus so that the insoluble acids are first introduced into the upper bulb and then run out into the test-tube through the side delivery tube. The test-tube is filled almost to the top with warm liquid from the apparatus, and then gently rotated in the hand so that all the globules of fatty acids rise to the surface and coalesce to form a large globule. If the test-tube is not clean the globules have a tendency to stick to the walls of the tube.

The capillary tube method for determining melting-points as described by Blichfeldt and Thornley (Analyst, 1921, p. 180) was that used. The acids being at the top of the test-tube, a column can easily be introduced into the capillary tube by immersing one end of it in the acids.

Occasionally the quantity of insoluble volatile acids is so small that difficulty is experienced in getting a column to rise in the capillary tube without some water rising at the same time. This can be overcome by solidifying the acids and removing them from the test-tube to a watch-glass. After drying by touching them with a piece of filter paper, the acids are melted and a column introduced into the capillary tube.

In the first place it must be pointed out that it is just as important in determining the melting-point that the distillation should be carried out in exactly the same way as when estimating the soluble and insoluble volatile acids, because the melting-point depends on the quantity of acids that come over. This will be apparent when the following figures are considered where the melting-points of the insoluble volatile acids obtained from three successive distillations of the fatty acids from 5 grms. of fat are shown both in the case of coconut and palm kernel fats. The second and third distillations were carried out by adding 100 c.c. of water

to the distilling flask after the first or second distillations were finished, and distilling to the same mark as before.

	Coconut Fat.	Palm Kernel Fat.
	° C.	° C.
First distillation	10·2	23·4
Second ,, ...	19·6	28·3
Third ,, ...	26·0	32·0

The melting-points for a number of butters obtained from different sources are given below; in this table are shown the distillation figures of these butters determined by the author's method (ANALYST, 1920, **45**, 2-7), also the corresponding figures for the coconut and palm kernel fats used in the mixtures referred to later.

Sample.	Total Volatile Acids.	Soluble Volatile Acids.	Insoluble Volatile Acids.	Melting-Point of Insoluble Volatile Acids.
				° C.
Butter 1	31·0	22·6	8·4	15·8
2	30·4	22·3	8·1	16·0
3	30·3	22·9	7·4	16·8
4	29·2	22·1	7·1	17·9
5	28·7	22·2	6·5	19·5
6	29·8	23·4	6·4	20·0
7	28·8	22·5	6·3	20·3
8	26·3	20·9	5·4	25·6
9	26·5	21·2	5·3	21·7
10	25·4	20·3	5·1	22·1
11	27·7	21·8	5·9	23·0
12	29·4	22·4	7·0	22·3
Coconut fat	21·1	1·6	19·5	9·9
Palm kernel fat	13·7	1·1	12·6	21·2

The limits between which the melting-point can lie are fairly wide apart. In the above table the limits are 15·8° and 25·6° C. Early results seemed to indicate that the melting-point was inversely proportional to the quantity of the insoluble volatile acids present, but later determinations did not bear this out. It can, however, be said of the butters examined that those with high melting-points usually had low distillation figures, and *vice versa*.

From the behaviour of the melting-points of the insoluble volatile acids derived from mixtures of coconut and palm kernel fats (Blichfeldt, *loc. cit.*), where these melting-points are stated to lie between the melting-points of the acids from the pure fats and are approximately proportional to the amounts present, it was thought that similar results would be obtained by the addition of either of these fats to butter.

That this is not the case can be seen from the figures below, showing the melting-points of the acids from butters which contain coconut and palm kernel fats.

Butter Sample.	Butter + 10 per Cent. Coconut Fat.		Butter + 10 per Cent. Palm Kernel Fat.	
	Melting-point of Insoluble Volatile Acids.	Total Volatile Acids. (Calculated.)	Melting-point of Insoluble Volatile Acids.	Total Volatile Acids. (Calculated.)
	° C.		° C.	
1	10·5	30·0	13·0	29·3
2	12·1	29·5	13·7	28·7
3	11·5	29·4	13·1	28·6
4	12·5	28·4	16·1	27·6
5	10·9	27·9	14·2	27·2
6	12·0	28·9	15·1	28·2
7	12·1	28·0	16·8	27·3
8	15·2	25·8	18·1	25·0

Butter Sample 2, containing 5 per cent. coconut fat + 5 per cent. palm kernel fat, gave a melting-point of 11·3° C. Total volatile acids calculated = 29·1.

Butter Sample 4, containing 5 per cent. coconut fat + 5 per cent. palm kernel fat, gave a melting-point of 14·1° C. Total volatile acids calculated = 28·0.

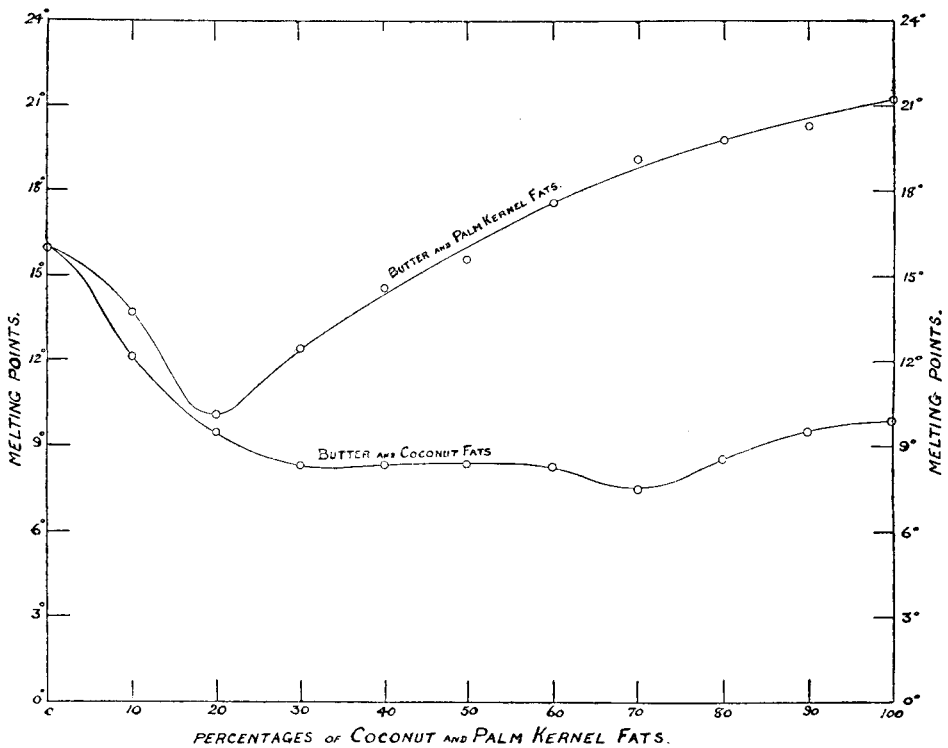
Butter Sample 7, to which was added 10 per cent. of margarine containing 80 per cent. of a mixture of coconut and palm kernel fats, gave a melting-point of 15·4° C. Total volatile acids calculated = 27·3.

The butters numbered 1 to 8 in the mixtures are the same as those bearing similar numbers in the previous table.

Per Cent. Butter (2).	Per Cent. Coconut Fat.	Melting-Point of Insoluble Volatile Acids.	Per Cent. Butter (2).	Per Cent. Palm Kernel Fat.	Melting Point of Insoluble Volatile Acids.
		° C.			C.
100·0	0·0	16·0	100·0	0·0	16·0
90·0	10·0	12·1	90·0	10·0	13·7
80·0	20·0	9·5	80·0	20·0	10·1
70·0	30·0	8·3	70·0	30·0	12·4
60·0	40·0	8·3	60·0	40·0	14·6
50·0	50·0	8·3	50·0	50·0	15·6
40·0	60·0	8·2	40·0	60·0	17·6
30·0	70·0	7·5	30·0	70·0	19·2
20·0	80·0	8·5	20·0	80·0	19·8
10·0	90·0	9·5	10·0	90·0	20·3
0·0	100·0	9·9	0·0	100·0	21·2

If the table of melting-points of the acids from pure butters be examined it will be seen that all the butters with figures for total volatile acids below 28 have

melting-points above 20·0° C., and no exception has so far been met with. Apparently, then, any butter with a total volatile figure below 28 and a melting-point below 20·0° C. can be assumed to be adulterated. In most of the above examples, where coconut or palm kernel fats, or both, were added to butter fat, the melting-points of the acids from the mixtures fell below 15·8° C., which is the minimum for pure butter. In the case of those examples where the melting-points were above 15·8° C., the total volatile figures were below 28, and, as none of the melting-points reached 20·0° C., it follows that adulteration has been detected in every case.



The effect on the melting-point when increasing quantities of coconut and palm kernel fats are added to butter fat was studied with the results given on p. 185.

A graphical representation of these results is shown in the diagram.

The addition of increasing amounts of coconut fat to the butter fat caused the melting-point to fall rapidly at first until there was about 25 per cent. present; then it remained fairly steady up to 60 per cent. At this point it began to fall again, reaching a minimum of 7·5° C. when there was 70 per cent. of coconut fat present. Further additions now raised the melting-point steadily until that of pure coconut fat was reached.

In the case of palm kernel fat the melting-point fell rapidly until about 20 per cent. was present, when the lowest melting-point—viz., 10·1° C.—was obtained. On

further addition, the melting-point steadily rose until that of pure palm kernel fat was attained.

The corresponding graph for mixtures of the insoluble volatile acids from coconut and palm kernel fats should be a straight line joining the melting-points of the acids from the pure fats. It can thus be assumed that the insoluble volatile acids from coconut and palm kernel fats are very similar in composition, but that those from butter fat differ considerably. This difference consists probably in the relative proportions of caprylic, capric, and lauric acids present.

The eutectic point on the graph for mixtures of palm kernel and butter fats is much more decided than that for coconut and butter fats.

The graphs also showed that the melting-points of the acids from a margarine containing a high percentage of coconut and palm kernel fats will be only slightly affected by the addition of 10 per cent. of butter, but that if the margarine contain a small percentage of these fats the addition of 10 per cent. of butter will have a more marked effect.

Conclusions.—The determination of the melting-point of the insoluble volatile acids is useful as a confirmatory test in the analysis of butter fat.

When the melting-point is taken into consideration with the figure representing the total volatile acids, it should give sufficient evidence of the purity of the sample. If the figures point to the sample being pure, the volatile acids need not be separated into soluble and insoluble fractions.

The melting-points for pure butters lie between 15·8° and 25·6° C. Butters with high total and insoluble volatile figures usually have low melting-points, and *vice versa*. Genuine butters give melting-points above 15·8° C., but if the total volatile figure falls below 28, then the melting-point should be above 20·0° C.

In many cases the melting-point will give an indication of adulteration when distillation methods fail to do so. In the case of a butter, for instance, with high distillation figures and a low melting-point, if adulterated with a fat which does not give rise to volatile acids the volatile acids will be reduced in proportion to the amount of adulteration, but not necessarily below the legal limit, whilst the melting-point will continue to represent the figures that the pure butter would give, and will probably be too low for the reduced total volatile figure.

The addition of either coconut or palm kernel fats to butter fat has the effect of lowering the melting-point of the insoluble volatile acids.

The melting-point will readily detect 10 per cent. of coconut or palm kernel fat. Smaller quantities can also be detected, but not with the same certainty.

Maypole Laboratory,
Southall, Middlesex.

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THE ESTIMATION OF STRYCHNINE IN SCALE PREPARATIONS CONTAINING QUININE AND OTHER CINCHONA ALKALOIDS.

By T. F. HARVEY, F.I.C., and S. BACK.

(*Read at the Meeting, April* 6, 1921.)

The estimation of strychnine in a scale preparation containing iron and quinine is a matter of some difficulty. The obvious method would be to extract the total alkaloids and then separate them by a reliable method.

The chief methods that have been proposed for the separation of these two alkaloids are:

1. Precipitation of the quinine as oxalate. The separation is not complete and the method does not appear to have been much used. Applied to the total alkaloids obtained from Easton's syrup this method gave a crude residue containing three and a half times as much quinine as strychnine.

2. A method published in the *J. Amer. Pharm. Assoc.*, 1919, **8**, 804–807, which is said to depend on the solubility of strychnine in water and its slight solubility in ether.

This method is not only cumbersome, involving the manipulation of considerable volumes of liquid and fourteen separations, but appears to be theoretically unsound, since strychnine is more soluble in ether than in water, and will partition itself between the two solvents according to well-established laws. However, we carried out an experiment using 475 mgrms. quinine and 25 mgrms. strychnine and extracted with nine consecutive quantities of ether of 8 c.c. each from a volume of 222 c.c. aqueous phase. The quinine fraction weighed 461 mgrms., from which we recovered 7 mgrms. of strychnine, whilst the strychnine fraction weighed 37·7 mgrms. but contained only 15·3 mgrms. strychnine.

We have found the solubility of powdered strychnine in ether of 0·720 sp. gr. at 15° C. to be 0·034 grm. per 100 grms. of solution, whilst Seidell's Solubilities gives 0·0432 grm. at 20° C., and 0·0513 grm. for wet ether. Taking our own figure as a basis, and the solubility of strychnine in water as 1 in 6,420, it can be calculated that the amount of strychnine remaining in the aqueous phase after the above treatment with ether should be 15·27 mgrms., which by coincidence is in exact agreement with the amount found.

3. Precipitation of quinine as tartrate with subsequent recovery of the strychnine and final washing with ether. (Harrison and Gair, *Yearbook Pharm.*, 1903, 564.) Harrison and Gair obtained very good results with mixtures of pure alkaloids, which were not seriously affected by the presence of other cinchona alkaloids occurring in the quinine sulphate of commerce. We find that this method gives quite good results on known mixtures of the pure (B.P.) alkaloids, as the figures in the table show. When, however, the method was applied to the dried alkaloidal residues obtained from iron, quinine, and strychnine citrate, results much in excess of the truth were obtained, and the method was obviously unsuitable for the purpose. The unwashed

strychnine (?) residues in these cases amounted sometimes to about 180 mgrms. instead of 50 mgrms., and it is impossible to complete the separation of the two alkaloids in a residue of this nature by means of ether. Washing with ether should only be resorted to for removing a comparatively small amount of impurity from a strychnine residue.

	Mgrms.	Mgrms.	Mgrms.
Quinine alkaloid taken	750	750	750
Strychnine taken	50	50	50
Unwashed strychnine obtained	59	60	56
Strychnine, after washing with 3 × 1 c.c. washed ether	53	51	51

4. A fourth method of separating these two alkaloids is that of Simmonds (ANALYST, 1914, **39**, 81). It depends on precipitation of strychnine ferrocyanide from 10 per cent. sulphuric acid solution followed by reprecipitation of the strychnine salt from 20 per cent. sulphuric acid.

We obtained the following results by Simmonds' method, working on pure alkaloids and using always 750 mgrms. dry quinine and 50 mgrms. strychnine:

Strychnine taken	50 mgrms.	50 mgrms.
Time of standing with ferrocyanide	21 and 23 hours	2½ and 2 hours
Strychnine residues obtained ...	56 mgrms.	54 mgrms.

These results, which are 12 and 8 per cent. too high, are probably due to the initial presence of a rather high concentration of quinine (750 mgrms. in 50 c.c.), but whatever the cause, the errors are large and even by washing carefully three times with 1 c.c. of washed ether as in Harrison and Gair's method they are only reduced to 6 per cent. Other experiments introducing slight variations have given similar results. When Simmonds' method is applied to the alkaloidal residue obtained from iron, quinine, and strychnine citrate, it is more unsatisfactory, and we found it necessary to seek some modification or combination of methods on the results of which one could rely with some confidence. It is obvious that the extracted and dried residue from scale preparations will differ in some degree from the pure alkaloids themselves, quinine being very prone to slight alteration during the processes of manufacture and analysis. It is therefore preferable to separate the strychnine from an undried alkaloid, and to make a separate estimation of the total alkaloid.

The method which has given the best results is a combination of the methods of Harrison and Gair and of Simmonds, but we found that the tartrate could be conveniently precipitated from the solution of the scales in water without previous isolation of the alkaloids. This direct precipitation of quinine has proved easy, and incidentally avoids shaking out with solvents at a stage at which emulsification is very prone to occur. To save time and avoid washing the bulky quinine tartrate, we adopted the practice of adjusting the total volume and taking an aliquot part of the filtrate, making the necessary percentage correction at the end.

The method is as follows, the time required for the actual manipulation being about two and a half hours. In the case of an iron quinine and strychnine scale preparation containing about 15 per cent. of quinine and 1 per cent. of strychnine, 10 grms. are dissolved in 70 c.c. of water in a beaker, 5 c.c. of $\frac{N}{1}$ sulphuric acid added, and the whole almost neutralised with ammonia, until the precipitated quinine only just redissolves. Thirty grms. of Rochelle salt are added, and the liquid is neutralised with dilute ammonia, the final reaction being left faintly acid to litmus paper. The mixture is stirred and heated in a water bath for fifteen minutes, cooled and transferred to a 100 c.c. measuring flask, the beaker being rinsed with water so as to make the volume up to 100 c.c. After standing for two hours the liquid is filtered, the first 10 c.c. being rejected. Fifty c.c. of the filtrate are extracted three times with chloroform, using 30 c.c., 10 c.c., and 10 c.c., and ammonia, and the mixed chloroform solutions are washed twice with 5 c.c. of water. The chloroform is extracted with 30 c.c. of 10 per cent. (weight/vol.) sulphuric acid, and twice more with 10 c.c. of the same acid, the acid liquids being collected in a small (60 c.c.) separator previously plugged with a small piece of cotton-wool. Five c.c. of freshly made 4 per cent. potassium ferrocyanide solution are added, the separator is practically filled with 10 per cent. acid (to exclude air), and after rotation the whole is allowed to stand in a dark place for two hours. It is advisable to be sure that precipitation has definitely occurred before placing aside. We prefer to use 10 per cent. acid in the precipitation. Simmonds makes use of 20 per cent. acid, but in that case the precipitation is very sluggish, and two hours' standing might be insufficient. At the end of this time the acid is forced out through the plug, the strychnine ferrocyanide washed twice with 3 c.c. of 5 per cent. sulphuric acid, and the strychnine recovered by shaking with 10 c.c. of 10 per cent. ammonia, and 15 c.c., 10 c.c., and 10 c.c. of chloroform.

Evaporation is carried out as usual, an addition of about three drops of amyl alcohol being made towards the end to prevent decrepitation of the strychnine crystals. When cold, the alkaloidal residue is washed three times with 1 c.c. of ether and dried at 100° C. It usually consists chiefly of characteristic crystals of strychnine, which are of a pale brown tint.

A correction of $-1{\cdot}7$ per cent. has been established for the volume of the quinine tartrate when following the above procedure. The following experiments seem to show the accuracy of the method for iron quinine and strychnine citrate.

A sample of iron and quinine citrate precipitated as described with tartrate left 160 mgrms. of alkaloid unprecipitated. This, on precipitation with ferrocyanide, yielded 2 mgrms. of residue which was entirely dissolved by the ether used for washing. One hundred mgrms. of strychnine were dissolved in $\frac{N}{1}$ acid and added to a solution of 10 grms. of the above iron and quinine citrate. The strychnine was then estimated as above. The weight of unwashed strychnine obtained was 50·5 mgrms., which, after washing with ether, weighed 49·0 mgrms. or 48·2 mgrms. (corrected). (Theory = 50 mgrms.)

Another experiment with a different sample of iron and quinine citrate gave 49·7 mgrms. (corrected).

Several commercial samples of iron quinine and strychnine citrate gave the

following results as compared with the expected 1 per cent.: 0·94, 0·94, 0·92, 0·94, 1·00 and 1·04 per cent.

The occurrence of other cinchona alkaloids as impurities has received some consideration, the results of which are shown in the table below.

Quinidine is the least likely to occur, but if present would, together with cinchonine, escape precipitation as tartrate. Quantities of 150 mgrms. of each of these alkaloids were dissolved separately in 10 per cent. and in 20 per cent. sulphuric acid and treated with potassium ferrocyanide solution. In no case did any precipitation occur during two hours' standing, and it is therefore not likely that small quantities of these alkaloids would introduce any inaccuracy. Cinchonidine, the most usual impurity in quinine salts, on the other hand, is very completely precipitated as tartrate. For instance, 300 mgrms. of cinchonidine were precipitated as tartrate under the above experimental conditions, and the amount of unprecipitated cinchonidine was only 4 mgrms., which, when subjected to the ferrocyanide precipitation, gave no trace of precipitate within two hours. The table shows that 150 mgrms. of cinchonidine dissolved and treated directly with ferrocyanide solution cause a considerable amount of crystalline precipitate to appear. On the other hand, we have found that 50 mgrms. of strychnine with 75 mgrms. of cinchonidine are easily and completely separated by Simmonds' method (two precipitations with ferrocyanide in 10 and 20 per cent. sulphuric acid). It is therefore highly improbable that any trace of cinchonidine sufficient to interfere with the method could get through from the tartrate precipitation.

There is left the possibility of the occurrence of quinicine as an alteration product of quinine. This alkaloid is hardly likely to occur in any quantity. It is, however, not precipitated by neutral tartrate, and is thrown down by ferrocyanide together with the strychnine. Its presence is made fairly obvious by the yellow colour of its acid solutions, and by the dark and abnormal appearance of the strychnine residue.

We have treated by the above method two alkaloid mixtures, with the results shown below:

Alkaloids taken.	Unwashed Strychnine Residue.	Ether-washed Residue.
	Mgrms.	Mgrms.
Strychnine, 50 mgrms. Quinicine, 37½ „ ...	73	71·5
Strychnine, 50 „ Quinicine, 37½ „ Quinine, 712½ „ ...	63	61

Thus, although dry quinicine is very soluble in ether, it is not removed from such residues by this means, and we have found by experiment that pure quinicine dissolves only slowly in ether. Attempts to soften the residues with alcohol and then treat with ether were not successful, far too much strychnine being removed. Experiments were then made with pure acetone, in which strychnine dissolves to the extent of 0·132 grm. per 100 c.c. (1 in 750), whilst quinicine dissolves both rapidly and completely in all proportions.

It will be seen from the accompanying figures that 10 mgrms. of quinicine can be separated from 50 mgrms. of strychnine by carefully washing the mixture twice with 0·5 c.c. of acetone, whilst in the presence of 20 mgrms. of quinicine the results obtained by washing are of very doubtful value.

Quantity Alkaloids taken.	Weight in Mgrms. after Washing with Pure Acetone.		
	2 × 0·5 c.c.	3 × 0·5 c.c.	5 × 0·5 c.c.
	Mgrms.	Mgrms.	Mgrms.
Strychnine, 50 mgrms. Quinicine, 10 ,,	49·0	—	—
Strychnine, 50 ,, Quinicine, 20 ,,	58·5	54·0 54·5	50·5

Solubility at 15° C. in grms. per 100 c.c. of Solution.

	Ether, Sp. Gr. 0·720.	Acetone.
Strychnine	0·244	0·132
Quinicine	Very soluble (slowly).	Soluble in all proportions.
Quinine	—	2·32

The authors are indebted to Messrs. Thomas Morson and Son, Ltd., in whose laboratory this investigation was made.

BEHAVIOUR OF CINCHONA ALKALOIDS WITH FERROCYANIDE IN ACID SOLUTION.

Alkaloid.	Mgrms. taken.	Strength Acid (Per Cent. by Volume).	Immediate Result.	Result after Two Hours in Dark.
Quinidine	150	10	Nil.	Nil.
		20	,,	,,
Cinchonine	150	10	,,	,,
		20	,,	,,
Cinchonidine	150	10	,,	Considerable crystalline ppt.
		20	,,	Smaller ppt. than in 10 per cent. acid.
Cinchonidine	100	10	,,	Few very small crystals (after 12 hours).
Quinicine	20	10	Nil (ppt. in 5 mins.)	Heavy ppt.
		20	Distinctly opalescent.	,, ,,
	40	10	Opalescence (ppt. in 5 mins.).	,, ,,
		20	Distinct ppt.	,, ,,
	150	10	Immediate ppt.	Considerable ppt.
		20	,, ,,	(About 50 per cent. ppted).

(The substance was dissolved in 50 c.c. of the sulphuric acid and 5 c.c. of recently made 4 per cent. potassium ferrocyanide solution were added with rotation.)

REFERENCES.

Harrison and Gair, *Year-Book of Pharmacy*, 1903, 564. Simmonds, ANALYST, 1914, 81. Allen's "Commercial Organic Analysis," VI., 461, 462, 543 ; IX., 518.

Quinicine: Pasteur, *Jahresberichte*, 1853, 473. D. Howard, *J. Chem. Soc.*, 1871, **24**, 61 ; 1872, **25**, 101. Howard and Chick, *J. Soc. Chem. Ind.*, 1909, 54. Henry, "Plant Alkaloids," 147.

A COLOUR REACTION FOR ACONITE.

BY S. MALLANNEH, M.D., D.P.H.

(*Read at the Meeting, April* 6, 1921.)

THE colour reactions at present known are not reactions of aconitine but of benzoic acid, which is one of the products of decomposition of aconitine. Hence the colour reactions are not specific.

Most of the colour reactions for alkaloids are such that they are useful only when applied to pure samples of alkaloids, but they are of no use when applied to crude substances containing alkaloids.

In medico-legal cases, especially in India, the poisoning is generally caused by the administration of crude substances in the form of powdered root, bark, or seeds, and not by the use of active principles. If the quantity of the vegetable poison present in the stomach be small, as is generally the case with aconite, it is next to impossible in most cases to get a sufficient amount of alkaloid extracted in order to prove the presence of poison by means of experiments on animals.

In India the vegetable poison also undergoes decomposition so quickly that it is almost impossible to detect its presence in a dead body, though distinct clinical symptoms of poisoning might have been present before death. But the cause of the failure to detect the poison in such cases is that, up to date, there is no reliable chemical test known for aconite.

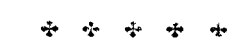
As the result of my experiments, I have discovered a test which I think is very useful for medico-legal purposes. If a minute particle of potassium ferricyanide be placed close to a minute portion of aconitine or a small portion of powdered root of aconite, and then a drop of formic acid added, a green coloration immediately appears. This is a very delicate reaction, as $\frac{1}{8000}$ grain of aconitine gives a positive reaction. Heat should not be applied for this test.

Morphine, atropine, digitalin, strychnine, eserine, and hyoscyamine do not react to this test, which therefore seems to be specific for aconite. It is not only applicable to the pure alkaloid, but also applicable to powdered root of aconite. Hence, if confirmed, it will be of great toxicological importance. Recently, in a case of human poisoning at Banswada Nizamaabad, I was able to test this reaction. A few black fragments found adherent to the stomach wall of the deceased gave positive reaction

to this test and the case was confirmed subsequently by experiments on animals. The police were able to procure from the house of the culprit a brown powder, which on examination was found to be powdered root of aconite.

DISCUSSION.

Mr. H. FINNEMORE said that, apart from the limited value of all colour reactions, this test was not specific for aconitine, since it appeared to be given by the Indian variety of aconite, which contained pseudaconitine but not aconitine.



A METHOD FOR THE DETERMINATION OF THE ACIDITY OF COLOURED SOLUTIONS.

BY J. L. LIZIUS, B.Sc., A.I.C.

(*Read at the Meeting, April* 6, 1921.)

THE filter paper of a small Buchner funnel (about 2 inches in diameter) is moistened with several drops of phenolphthalein solution. The pump is turned on, and when the excess of phenolphthalein has been removed the pump is turned off again. To 10 c.c. of the coloured solution $\frac{N}{10}$ alkali is added until it is just alkaline. The test for alkalinity is made by withdrawing a little of the solution by means of a fine capillary tube (a melting-point tube), and touching the filter-paper. The liquid remaining in the capillary tube is blown back into the solution. When the capillary tube test gives a pink colour the whole solution is run through the Buchner funnel. The filter-paper is left a pink colour. The contents of the Buchner flask are washed out into a beaker and 0·05 c.c. of $\frac{N}{10}$ acid is added. One or two drops of phenolphthalein solution are run on to the filter-paper, and the coloured solution is again poured through. If the filter-paper is not pink the volume of $\frac{N}{10}$ alkali added is the titration value. If the filter-paper is still pink the addition of $\frac{N}{10}$ acid is continued, 0·05 c.c. at a time, until the filter paper is no longer pink. The titration value is the volume of $\frac{N}{10}$ alkali—(volume $\frac{N}{10}$ acid $-0{\cdot}05$ c.c.). If the colour of the solution is adsorbed by the filter and tends to mask the pink colour, the difficulty can be overcome by washing through the paper a drop or two of phenolphthalein solution. Coloured alkaline solutions can be titrated by $\frac{N}{10}$ acid in a similar manner.

The pink colour left on the filter paper rapidly fades, owing to the action of the carbon dioxide in the air.

This method was tested by four different observers on a sample of stout. The results obtained by the above method agreed to within 0·1 c.c. $\frac{N}{10}$ alkali, whereas by the ordinary method of titration the results varied over 0·6 c.c.

In the case of blue-black inks, the determination is extremely difficult for the following reasons:

1. The ink itself acts as an indicator, becoming violet before the neutral point is

reached. The combination of the pink colour of phenolphthalein in alkaline solution with the colour of the ink also produces a violet colour.

2. The colour of the ink is very strongly adsorbed by the filter-paper, and this colour is not readily washed out by phenolphthalein. The method of decolourising with hydrogen peroxide (ANALYST, 1921, 131) is much simpler and more satisfactory for inks.

I have no doubt that the method will prove suitable for very dark vinegars and for molasses, as it has been shown suitable for stout. I have correctly determined the acidity of a sample of vinegar, and of a solution of hydrochloric acid to which caramel had been added, in this way.

The note is put forward as a more satisfactory method for the acidity of the coloured solutions the analytical chemist has to determine, but does not claim to be a general method of determining the acidity of every conceivable coloured solution.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Wet and Dry Feeding of Concentrates to Dairy Cows. R. A. Berry. (*J. Agric. Sci.*, 1921, **11**, 78-98.)—The effect of wet and dry feeding has been investigated on a herd of ten Ayrshire cows with special reference to the effect on health, condition, milk yield, and fat content. Water consumption and milk yield and temperature effects have also been studied. Feeding with wet meal for a period of five weeks raised the milk yield by about ¾ lb. per cow per day, and the fall in yield due to advanced lactation was reduced. On a ten weeks' wet feeding the yield is increased, but the advantage diminishes as lactation advances. Cows fed on wet food take in altogether more water *per diem*, the exact ratio of water consumed varying with the fodder, and increasing as the milk yield increases, the latter representing about 26 per cent. of the total intake. On a heavy-root feeding the milk yield decreases, but the fat content rises materially, the non-fatty solids remaining constant. The health of the cows is adversely affected. The mean temperature in the byre is 15° to 20° F. above the outside temperature; lowering the temperature decreases the milk yield and *vice versa*, and it has been shown (*cf.* Berry, *Bull.* 76, *W. Scot. Agric. Coll.*, 1916, 49) that the butyric acid content is reduced by low temperatures. H. E. C.

Coffee Substitutes from Narcissus Bulbs and Seaweed. C. Griebel and W. Rothe. (*Zeitsch. Unters. Nahr. Genussm.*, 1921, **41**, 69-73.)—Roasted narcissus bulbs have been used in Germany as a coffee substitute; the raw bulbs contain

an alkaloid, which acts as an emetic, but in a case of sickness supposed to be caused by the consumption of this coffee substitute the authors were unable to detect any trace of the alkaloid, possibly because it had been decomposed during the roasting process. The ground, roasted bulb has a characteristic appearance under the microscope, the long clusters of calcium oxalate crystals being particularly conspicuous. Another coffee substitute is prepared from seaweed (*Zostera marina*); this may also be identified by microscopical examination. W. P. S.

Poisonous Haricot Beans. Th. v. Fellenberg. (*Chem. Zeit.*, 1921, **48**, 201.)—The striated markings, or the network pattern, in the European variety of the bean *Phascolus lunatus* can be easily distinguished if the beans are treated with basic dyes, such as fuchsin and methyl violet. The poisonous properties of the beans may be destroyed by boiling them, but such a course does not always secure complete immunity. W. J. W.

Adulteration of Ground Cinnamon with Ochre. R. Windisch. (*Zeitsch. Unters. Nahr. Genussm.*, 1921, **41**, 78-81.)—Of nine samples of ground cinnamon examined, four yielded from 6 to 10 per cent. of ash, having a red-brown colour, and containing up to 4 per cent. of sand. Genuine cinnamon gave less than 3 per cent. of ash, containing a very small quantity (0·02 per cent.) of sand. The sand was estimated by extracting the ash with boiling 10 per cent. hydrochloric acid. A specimen of ochre tested by the author yielded 10 per cent. of soluble matters to 10 per cent. hydrochloric acid, and mixtures of this ochre with genuine ground cinnamon had properties similar to the samples in question. W. P. S.

Indian Kapok Seed. (*Bull. Imperial Inst.*, 1920, **18**, 335-337.)—The seeds of Indian kapok (*Bombax malabaricum*, D.C.), a large deciduous tree occurring throughout India and Ceylon, contained 8·9 per cent. of moisture and 22·3 per cent. of oil. The oil was bright yellow in colour, deposited some stearine on standing, and had the following characters: Sp. gr. at 15°/15°, 0·9208; $[n]_D^{20}$, 1·461; solidif. pt. of fatty acids, 38·0° C.; acid value, 9·3; saponification value, 193·3; iodine value, 78·0; soluble volatile acids, nil; and insoluble volatile acids, equivalent to 0·5 c.c. $\frac{N}{10}$ alkali per 5 grms. of oil. The oil therefore differs somewhat from that of Java kapok (*Eriodendron anfractuosum*), which has an iodine value 95 to 110. The composition of the residual meal was: Moisture, 11·4; crude proteins, 36·5; fat, 0·8; carbohydrates, 24·7; fibre, 19·9; and ash, 6·7 per cent. The cake obtainable from the seeds of Indian kapok and pressed to an oil content of about 7 per cent. would therefore contain about 34 per cent. of proteins in comparison with 25 to 26 per cent. in commercial undecorticated cotton-seed or Java kapok-seed cake. R. G. P.

Estimation of Coconut and Palm Kernel Oils in Fat Mixtures in which both Oils may be present. W. N. Stokoe. (*J. Soc. Chem. Ind.*, 1921, **40**, 57-58 T.)—The methods given by Burnett and Revis (ANALYST, 1913, **38**, 255) and by Blichfeldt (ANALYST, 1919, **44**, 290) having proved somewhat unsatisfactory, the author has devised the following method for estimating coconut and palm kernel oils

in mixtures. In the standard Reichert-Polenske process, after 110 c.c. have been distilled, the liquid is cooled and the insoluble acids collected on a filter; the condenser tube is rinsed down with 20 c.c. of water at 30° to 40° C., this being poured over the filter. The filter paper is washed with water at the same temperature, and as soon as the last drop of water has drained through, several capillary tubes of 1 mm. bore are filled to a depth of half an inch with the now melted acids. Two of the tubes are immediately attached, by means of a small rubber band, to either side of a thermometer graduated to read to 0·1° C., and the thermometer then fitted through the cork of a test-tube. The tube is supported in a beaker containing ether, the surface of which should be above the level of the acids in the capillary tubes. Whether the bulk of the acids is from palm kernel or from coconut oil will have been ascertained previously from the appearance of the acids in the original Reichert-Polenske distillate; if the acids are solid at the ordinary temperature it is necessary to warm the ether to about 30° C. A gentle stream of air is forced, by means of a foot-bellows, through the ether, so that the temperature of the latter is gradually lowered, the rate of cooling being adjusted to make the temperature of the ether 2° C. below that indicated by the thermometer within the test-tube. As the cooling progresses, the fatty acids become slightly cloudy, a distinct "seeding" or crystallisation afterwards occurring; at the first appearance of crystals the temperature is noted. This "seeding point" is quite definite and sharp, the acids in the capillary tubes appearing liquid, with a number of tiny white crystals along the sides of the tube; regular cooling is, however, essential.

The maximum, minimum, and average values obtained for the seeding point of four samples of coconut oil acids were 11·4°, 9·9°, and 10·75° C., whilst for four samples of palm kernel acids the corresponding values were 23·2°, 22·05°, and 22·75° C. For mixtures of acids of the two kinds, the seeding points, when plotted against the compositions, lie on a curve which is slightly concave towards the axis of temperature. The presence of other fats raises the seeding point somewhat, but the curves are approximately parallel to the original one. Thus, in the analysis of an unknown fat mixture, it is necessary first to ascertain the approximate proportion of the fats of the coconut group present by determining the saponification value and the Reichert-Polenske value, the relative proportions of coconut and palm kernel oils being read off from the corresponding setting point curve. The contents of the capillary tubes may be subsequently added to the alcoholic solution of the remainder of the insoluble acids, and the whole titrated as usual to obtain the Polenske value. T. H. P.

Composition of the Head Oil of the Sperm Whale. M. Tsujimoto. (*J. Chem. Ind., Japan*, 1920, **24**, No. 275 Reprint.)—A specimen of crude oil obtained from the head of a male sperm whale (*Physeter macrocephalus, L.*) was cooled at 6° C. and pressed through a bag to remove spermaceti. The expressed oil had a light yellow colour, an unpleasant odour, and the following physical and chemical characters: Sp. gr. at 15°/4° C., 0·8848; $[n]^{20}_{D}$, 1·4633; acid value, 0·99; saponification value, 147·1; iodine value (Wijs), 71·4; Reichert-Meissl value, 0·57; unsaponifiable matter, 36 per cent.; fatty acids, 65 per cent.; glycerol, 3·52 per cent.; cholesterol, 0·18 per cent. The unsaponifiable matter (wax alcohols)

consisted chiefly of cetyl alcohol and oleicalcohol together with a small amount of pentadecyl alcohol. The fatty acids consisted of about 19 per cent. of solid acids (including palmitic and myristic acids) and about 81 per cent. of liquid acids (mainly physetoleic and oleic acids). On hydrogenation, the physetoleic acid yielded an iso-palmitic acid, m.-pt. 55° to 56° C. W. P. S.

Fatty Oils from Reptilia. M. Tsujimoto and S. Kobayashi. (*J. Chem. Ind., Japan*, 1920, **23**, No. 273 Reprint.)—Oils obtained from a Japanese viper (*Agkistrodon blomhoffii*), python (*Python reticulatus*), toad (*Bufo japonicus*), and giant lizard (*Varanus*), had the following characters:

	Viper Oil.	Python Oil.	Lizard Oil.	Toad Oil.
Specific gravity at 15°/4° C. ...	0·9192	0·9165	0·9132 (40° C.)	0·9348
Refractive index at 20° C.	1·4723	1·4685	1·4747 (40° C.)	1·4742
Acid value	0·4	0·6	21·0	8·5
Saponification value	187·7	194·1	188·8	181·3
Iodine value (Wijs)	110·8	80·3	101·0	104·4
Unsaponifiable matter, per cent. ...	1·98	—	—	7·94
Ether-insoluble bromides, per cent.	7·8	2·1	9·7	1·0

W. P. S.

Test for Annatto in Fats and Oils. W. Brinsmaid. (*J. Ind. Eng. Chem.* 1921, **13**, 216-217.)—The usual test for annatto, in which the fat is treated with sodium hydroxide solution and filtered, the dried filter-paper being subsequently tested with a drop of stannous chloride solution, is unsatisfactory, since the paper is frequently so greasy that the reagent does not come into contact with the annatto. The following modification of the method overcomes this difficulty. Fifteen c.c. of the clear melted fat are mixed thoroughly with 15 c.c. of chloroform and 15 c.c. of 5 per cent. sodium hydroxide solution, and the mixture is heated at 55° C. until the emulsion is broken up; the soap froth is then transferred to a beaker, 10 c.c. of water and 2 c.c. of sodium hydroxide solution and a suitable quantity of filter-paper pulp are added, and the mixture is heated on a steam-bath for thirty minutes. The filter-paper pulp is then collected by filtering the mixture through a Gooch crucible containing a disc of filter-paper, slight suction being applied. If annatto be present, the pulp will exhibit an orange colour, which changes to pink on the addition of a few drops of stannous chloride solution. W. P. S.

Estimation of Maltose or Lactose in Presence of other Reducing Sugars: Use of Barfoed's Solution. Legrand. (*Comptes rend.*, 1921, **172**, 602-604.)—Since Barfoed's solution is reduced by monoses but not by bioses, the proportion of maltose or lactose in a saccharine solution may be estimated by difference from the separate reductions of Fehling's and Barfoed's solutions. Fifteen c.c. of the latter

solution and 5 c.c. of sugar solution containing at most 0·1 grm. of sugar are boiled for three minutes in a conical flask; the slight loss of acetic acid occurring under these conditions does not involve formation of basic copper salt. The cuprous oxide separating is collected on an ordinary filter and estimated, according to Bertrand's method, by means of ferric sulphate and permanganate. This method is the only one which is applicable to the estimation of the amount of sucrose converted into invert sugar in condensed milk. T. H. P.

Cattle Food prepared from Hydrolysed Sawdust. E. C. Sherrard and G. W. Blanco. (*J. Ind. Eng. Chem.*, 1921, **13**, 61-65.)—Comparison of the analysis of the original wood with that of the completed stock food obtained by hydrolysis shows a reduction in the pentosan yield from 9·40-9·86 per cent. to 4·38-4·66 per cent., the loss being probably due to partial conversion of the pentoses into volatile acids and furfural. Methyl pentosans in the wood are unaffected by the sulphuric acid treatment; the amounts in the original wood were 2·37 and 2·83, and in the hydrolysed product, 2·15 and 2·50 per cent. The yield of cellulose from sawdust was 55·79 per cent., and from the cattle food 37·08 per cent., equivalent to 34·11 per cent. if calculated on the original woods; and the yield of reducing sugars was 71·5 per cent. of the theoretical figure. Treatment of cellulose from sawdust with 17·5 per cent. sodium hydroxide left over 50 per cent. unattacked, the amounts of α-cellulose in two samples being 57·30 and 55·85 per cent. Cellulose from hydrolysed wood was converted by alkali treatment into a viscous, semi-transparent mass, having the appearance of collodion. W. J. W.

Estimation of the Alkalinity and Phosphate Content of the Ash of Foods. J. Tillmans and A. Bohrmann. (*Zeitsch. Unters. Nahr. Genussm.*, 1921, **41**, 1-16.)—Alkalinity due to oxides and carbonates is estimated by treating a portion of the ash with an excess of standard acid, boiling the mixture to expel carbon dioxide, adding 30 c.c. of 40 per cent. calcium chloride solution, and titrating the excess of acid with standard alkali solution, using phenolphthalein as indicator. The addition of the calcium chloride prevents the interference of the phosphates present. To estimate phosphates, another portion of the ash (at least 0·2 grm.) is boiled for one hour with 100 c.c. of $\frac{N}{10}$ hydrochloric acid, cooled, and titrated with $\frac{N}{10}$ sodium hydroxide solution, using methyl-orange as indicator; let the quantity of $\frac{N}{10}$ acid used $= b$. Twenty c.c. of saturated sodium oxalate solution are then added and the solution is titrated until a faint pink coloration with phenolphthalein is obtained; let the quantity of standard solution used for this titration $= c$. If the ash be alkaline (equal to a c.c. of $\frac{N}{10}$ acid solution), then orthophosphates only can be present, and the formula $\frac{3}{2}(b-a) \times 3{\cdot}167$ gives mgrms. of PO_4 present as orthophosphate. When the ash is not alkaline and the value c is positive, the orthophosphate $-PO_4 = 3c \times 3{\cdot}167$ and the pyrophosphate $-PO_4 = 2(b-2c) \times 4{\cdot}75$ mgrms. If c has a negative value, then pyro- and metaphosphates are present; in this case the pyrophosphate $-PO_4 = 2b \times 4{\cdot}75$ and the metaphosphate $-PO_4 = -c \times 9{\cdot}5$ mgrms. Milk ash contains a small quantity of alkali as oxides and carbonates, and 30 to 40 per cent. of orthophosphates; pyro- and metaphosphates are not present. Flour ash contains no true alkali, but a

mixture of the three phosphates. Beef and horse-flesh ashes contain no oxide or carbonate, but about 7 per cent. of orthophosphate and 50 per cent. of pyrophosphate. Fruit juice ashes consist chiefly of carbonates. Cocoa ash has a small alkalinity due to oxides and carbonates; it contains 33 per cent. of orthophosphates.

W. P. S.

The United States Pharmacopœia Method for the Estimation of Phosphoric Acid and Soluble Phosphates. A. E. Stearn, H. V. Farr and N. P. Knowlton. (*J. Ind. Eng. Chem.*, 1921, **13**, 220–225.)—The fairly accurate results yielded by the method prescribed in the U.S. Pharmacopœia are obtained only at the specific concentration given, namely, 0·0062 gr. per c.c.; at other concentrations the results obtained are untrustworthy. This is probably due to the formation of silver hydrogen phosphates which are slightly soluble, the amount increasing rapidly as the phosphate concentration is increased and the excess of silver nitrate simultaneously decreased. If the method be modified by converting the acid into the trisodium salt, the results obtained are trustworthy and are independent of the concentration. Ten c.c. of a 1 per cent. solution of the acid are treated with 5 grms. of sodium nitrate, cooled to 0° C., and titrated with $\frac{N}{10}$ sodium hydroxide solution, using phenolphthalein as indicator; the number of c.c. of alkali solution required is divided by two, and this quantity is added in excess. The mixture is then treated with 50 c.c. of $\frac{N}{10}$ silver nitrate solution, diluted to 100 c.c., mixed, filtered, and 50 c.c. of the filtrate are acidified with nitric acid and the excess of silver titrated with thiocyanate solution. The modified method may be applied to sodium phosphates; in this case the salts are first rendered anhydrous and the amount of alkali required is found by calculation. W. P. S.

Estimation of Santonin in Wormseeds. T. Kariyone and Y. Kimura. (*Pharm. Soc. Japan*, 1920, 927-940; *J. Chem. Soc.*, 1921, **120**, ii., 223.)—The method is essentially a determination of the saponification value, as a measure of lactone formation. The residue left after a Soxhlet extraction of 10 grms. of powdered *Artemisia Cina*, Berg., with ether, is boiled with 100 c.c. of 5 per cent. barium hydroxide solution, acidified with dry carbon dioxide and filtered. Eighty c.c. of the filtrate are extracted with 20 c.c. of chloroform and 10 c.c. of 15 per cent. hydrochloric acid, and then with three portions of 10 c.c. of chloroform. The extract is evaporated, the residue dissolved in 30 c.c. of alcohol, and the solution neutralised with $\frac{N}{10}$ potassium hydroxide solution, 20 c.c. of which are then added in excess. The mixture is heated under a reflux condenser for half an hour and titrated with $\frac{N}{10}$ hydrochloric acid, using phenolphthalein. When S′ and S are the amounts of $\frac{N}{10}$ acid required by the 20 c.c. of potassium hydroxide and the sample respectively, the santonin content per cent. is expressed by the formula $2{\cdot}462\ (S' - S)/8$.

H. E. C.

The Alkaloid and Oil Content of Meadow Saffron Seeds. C. Grimme. (*Chem. Abstracts*, 1921, **15**, 574; from *Pharm. Zentralhalle*, 1920, **61**, 521-4.)—The content of colchicine varied from 0·11 to 0·52 per cent., the actual amount present

being from 1 to 25 per cent. greater than indicated by the physiological activity. The proportion of alkaloid present was higher in the smaller seeds. The seeds yielded 6·6 to 8·4 per cent. of an oil having the following characteristics: Sp. gr., 0·9176 at 15° C.; solidifying-point, −9° C.; $[n]_D^{40}$, 1·4642; acid value, 20·3; saponification value, 184·3; iodine value, 128·5; and unsaponifiable matter, 0·71 per cent. The fatty acids had: M.-pt., 24° C.; solidifying-point, 22·5° C.; $[n]_D^{40}$, 1·4646; neutralisation value, 187·6; and iodine value, 131·0.

T. J. W.

Clove Oil from Clove Stems. S. T. Gadre. (*J. Indian Ind. and Labour*, 1921, **1**, 41-47.)—The oil was prepared by soaking the stems connecting the flowers to the branches in water overnight, and distilling with steam at a pressure of from 10 to 15 lbs. for five or six hours in a tin-lined copper still. A yield of 4·50 per cent. of a heavy, pale-yellow oil was obtained, equal in aroma and quality to the best English distilled clove oil. Examination of the oil gave the following results: Sp. gr., 1·0522 at 34° C.; $[n]_D$, 1·5345; total eugenol content (Umney's method), 93·09 per cent. (Thom's gravimetric method), 83·53 per cent.; free eugenol content by Karley and Bolsing's volumetric method, 69·86 per cent. The benzoyl-eugenol obtained in Thom's methods on recrystallisation from alcohol gave a m.-pt. of 70° C. The oil was soluble in half its volume of 80 per cent. alcohol, but with more than two volumes an opalescence was produced. T. J. W.

Essential Oil of Spearmint from South Africa. (*Bull. Imperial Inst.*, 1920, **18**, 350-351.)—Dried leaves of spearmint (*Mentha longifolia*) grown in the Cape Province yielded 2·4 per cent. of essential oil; the stems were practically devoid of oil. The essential oil showed the following characters: Sp. gr. at 15°/15°, 0·947; $[\alpha]_D = -47·6°$; $[n]_D$*, 1·4925; ketones (largely or entirely carvone), 70 per cent. The oil was similar in character to, but richer in ketones than, English and American spearmint oils (derived from *M. spicata*, Huds., or *M. viridis*, L.), which contain 30 to 48 and 35 to 66 per cent. respectively of ketones, and resembled Austrian spearmint oil, which contains up to 72 per cent. of ketones. R. G. P.

Reaction of Saccharin. (A Correction.) L. Thevenon. (*J. Pharm. Chim.*, 1921, **23**, 215.)—In dilute acid solution, β-naphthol reacts with nitrous acid and yields a red colouring matter. Consequently, the test for saccharin described recently (ANALYST, 1921, **54**) is not characteristic of this substance.

W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Vitamins and the Food Supply. A. Harden. (*J. Soc. Chem. Ind.*, 1921, **40**, 79-82 R.)—The vitamin requirements of the animal organism, the variations in the vitamin content of foodstuffs, and the influence of processes of manufacture or preparation, are considered. The three vitamins, fat-soluble *A*, water-soluble *B* or

* No temperature given.

anti-neuritic, and water-soluble *C* or anti-scorbutic, are termed, in accordance with Drummond's suggestion, vitamins *A*, *B*, and *C* respectively, and occur chiefly in (*A*) animal fats and products containing them, fish liver oils, green vegetables, and egg-yolk; (*B*) seeds (especially in the germ, pericarp, and aleurone layer), yeast, and egg-yolk; and (*C*) green vegetables (especially *Cruciferæ*), orange and lemon juice, tomatoes, germinated seeds, swedes, and turnips; they cannot be synthesised by the mammalian organism, and are required in relatively large proportions by the young growing organism. The amount of vitamin contained in natural foodstuffs appears to vary under different conditions of season, age, etc., and may also be modified by the treatment to which the material is subjected prior to consumption. Thus, the unbalanced use of milled rice is the main cause of beri-beri in the tropics. The effect produced by drying varies very greatly with the character of the foodstuff. Cabbage, the richest known source of vitamin *C*, loses 70 to 80 per cent. of its anti-scorbutic potency when dried, even at a low temperature, whilst orange and lemon juices may be evaporated *in vacuo* with little or no loss. If undue exposure to a high temperature be avoided, dried milk may be prepared but little inferior in anti-scorbutic properties to fresh milk, although many samples of dried milk actually on the market contain far less of the antiscorbutic principle than fresh milk. Vitamins *A* and *B* are less affected by desiccation than vitamin *C*, although with these also the effect varies with the nature of the foodstuff. Comparatively little is known of the effect of preservation on the vitamin content of food materials. Air-dried cabbage retains its slight anti-scorbutic potency unimpaired for long periods when preserved over phosphorus pentoxide, even at 37° C., but loses it rapidly when kept over calcium chloride. It seems probable that a perfectly dry powder will long withstand preservation at ordinary temperatures, whereas in the presence of even very small amounts of moisture deterioration may occur. The deterioration of lemon juice in this respect is prevented by the natural antiseptic furnished by the oil of the rind. Vitamin *A* is moderately stable towards rise of temperature provided that air is excluded, whereas in the presence of air it becomes rapidly inactivated, even at comparatively low temperatures; it remains almost unaltered when cabbage is cooked or when milk is autoclaved at 120° C. for an hour. Vitamin *B* is but little affected by heating for one to two hours at 100° C., and has even been found to survive boiling with sulphuric acid. As regards vitamin *C*, matters are less definite, the effect of heat in this case appearing to depend largely on the nature of the vitamin-containing material.

In the course of refining fish oils seem to lose much of their vitamin *A*, which is absolutely destroyed by hydrogenation—either by the actual reduction, or by the catalyst or the conditions employed. T. H. P.

Estimation of a Substance from Yeast and Rice Polishings which Accelerates Fermentation. S. Frankel and E. Schwarz. (*Biochem. Zeitsch.*, 1920, **112**, 203-235; *J. Chem. Soc.*, 1921, **120**, ii., 228.)—The fermentation-accelerating factor from yeast and rice polishings can be estimated as follows: The fats are removed from an 80 per cent. alcohol extract of yeast, which is then treated with basic lead acetate. Excess of lead is removed from the

filtrate by hydrogen sulphide, and mercuric chloride added. After decomposition of the precipitate with hydrogen sulphide, it is freed from hydrochloric acid and concentrated in a vacuum. An inactive picrolonate is precipitated by adding picrolonic acid, and the active substance precipitated with phosphotungstic acid. This precipitate is decomposed with baryta and sulphuric acid and concentrated in a vacuum, yielding an active base twenty-two times as strong as the original alcoholic extract. H. E. C.

Amylase of *Rhizopus Tritici*. L. L. Harter. (*J. Agric. Research*, 1921, **20**, 761-786.)—The mould was cultivated on a modified Czapek's solution with varying amounts of starch paste, glucose, or both, at 25° to 35° C., for seven to ten days. The felt of mycelium was washed with water, acetone, and ether, dried by exposure to air, and kept at 9° C. until required. The results of a large number of experiments are tabulated, the following results being deduced: an energetic amylase is produced during the growth of the mould, a proportion of which diffuses out into the culture solution. The enzyme exerts a vigorous action upon starch paste, but raw potato starch is attacked less readily, the optimum temperature being 45° C., whilst at 60° C. the amylase is completely destroyed in 100 hours. The addition of glucose causes retardation in the hydrolysis of starch by the amylase, but has no other influence upon the results. Since the determination of the reducing sugars does not account for the whole of the starch when an end point is shown by iodine, it is probable that dextrins are formed during the hydrolysis. Mycelium grown at 9° C. hydrolyses four times as much starch as that grown at 40° C., whilst cultivation at 29° C. yields an intermediate figure, although the fungus grows best at the latter temperature. The activity of the enzyme depends upon the influence which the carbohydrate added to the culture medium has upon the growth of the fungus, the most efficient source of carbohydrate being sweet potato bouillon. T. J. W.

Composition of Tubers, Skins, and Sprouts of Three Varieties of Potatoes. F. C. Cook. (*J. Agric. Research*, 1921, **20**, 623-635.)—Specimens of Rural New Yorker, Green Mountain, and Irish Cobbler varieties of potatoes were stored in a dark chamber at 70° C. for four to eight months, and the sprouts, skins, and tubers separated and analysed. Extensive details are given of the results obtained, for which the original paper should be consulted. The composition is practically identical for the three varieties, and spraying with Bordeaux mixture caused no variation in composition. The sprouts contained more protein and less basic nitrogen than the skins and tubers, and contained a larger proportion of water, ash, phosphoric acid, and nitrogen than originally present in the tuber. Copper was found in both sprayed and unsprayed samples, varying from fourteen to forty-one parts per million. T. J. W.

Digestion of Vegetable Fibres. W. Thomann. (*Chem. Zeit.*, 1921, **45**, 200.)—Digestion with ammoniacal copper oxide, as suggested by Mach and Lederle, gives results in close agreement with those obtained by the agency of ruminating animals. In the case of rabbits digestion is less complete, and values obtained by

Mach's method must be multiplied by 0·49 and 0·72 for straw fodder and grain food respectively. W. J. W.

Hemicellulose of Apple Wood. W. E. Tottingham, R. H. Roberts, and S. Lepkousky. (*J. Biol. Chem.*, 1921, **45**, 407-414.)—The base wood of young fruiting apple shoots was analysed by drying, extracting with ether and 90 per cent. alcohol, digesting with saliva, and boiling the residue with 2·5 per cent. (volume) sulphuric acid for two hours under a reflux condenser. The amount of material (hemicellulose) hydrolysed by this treatment amounted to 28·7 per cent., calculated on the original dry wood. The acid extract, after neutralising, decolorising, and purifying, yielded a very viscous pale brown solid, which on examination by fractional precipitation of osazones and quantitative tests proved to be a mixture of dextrose, xylose, and a small proportion of galactose, together with other unidentified substances. It is suggested that the hemicellulose forms a carbohydrate reserve in the metabolism of the apple-tree. T. J. W.

Detection of Albumoses. C. Achard and E. Feuillee. (*J. Pharm. Chim.*, 1921, **23**, 146-147.)—Albumin is first removed from the liquid (*e.g.*, blood-serum) as follows: 2·5 c.c. of the liquid are diluted with 10 c.c. of water in a 100 c.c. conical flask, 1 drop of acetic acid and 1·25 grm. of sodium chloride are added, and the flask heated over a wire gauze, with constant rotation and removal from the flame whenever the foam rises in the flask. After two to four minutes' boiling there is incipient coagulation, and at this point the liquid is allowed to cool for about three minutes, again heated, and immediately it boils is filtered into a tube 2 cm. in diameter, and made up to 10 c.c. with boiling water and filtered. The filtrate is treated with 10 c.c. of Tanret's reagent and again heated, care being taken that on removing the tube from the flame directly ebullition occurs the liquid is clear. After cooling the tube in a stream of water, the turbidity due to albumoses appears, and is compared, after standing for one hour, with standard tubes of Witte's peptone. R. G. P.

Estimation of Amino-Acids in Urine. W. Mestrézal. (*J. Pharm. Chim.*, 1921, **23**, 137-141.)—The following modification of Sörenson's method obviates the use of litmus as an indicator. Phosphates and carbonates are first removed as follows: To 50 c.c. of urine in a 100 c.c. graduated flask 2 grms. of powdered barium chloride and two drops of phenolphthalein solution are added, and when the barium chloride is dissolved, saturated barium hydroxide solution is added (5 c.c. in excess of that required to colour the phenolphthalein), and the liquid is allowed to stand in the corked flask for fifteen minutes and then made up to 100 c.c. After filtration, 20 c.c. of liquid (=10 c.c. of urine) are treated with ten drops of phenolphthalein solution (1 per cent.) and decolourised by adding dilute hydrochloric acid drop by drop; $\frac{N}{10}$ sodium hydroxide solution is then run in till a pale rose tint appears. Ten c.c. of neutral formaldehyde solution (35 to 40 per cent.) are next added, and the liquid is finally titrated with $\frac{N}{10}$ sodium hydroxide solution, the titration being continued until the decided red tint of the blank is reached. The blank (or standard

tint) is obtained by taking 10 c.c. of neutral formaldehyde solution, 20 c.c. to 30 c.c. of boiled distilled water, 1 c.c. of phenolphthalein, and adding $\frac{N}{10}$ sodium hydroxide (0·3 c.c. to 0·4 c.c.) until a decided red tint is obtained. It is necessary to continue the titration to this point, as the amino-acids react with formalin to produce derivatives (of the type $R-CH(N=CH_2)COOH$) which function as weak acids. Allowance is made for the retarding effect of ammonium salts by increasing the number of c.c. taken for titration by $\frac{1}{100}$. The amino-nitrogen is obtained by deducting the ammoniacal nitrogen determined separately by Schloesing's method.

R. G. P.

WATER ANALYSIS.

Analysis of Mineral Sulphide Waters. J. G. Fairchild. (*J. Washington Acad. Sci.*, 1920, **10**, 559-565; *J. Chem. Soc.*, 1921, **120**, ii., 126.)—In waters containtaining alkali and alkaline earth hydrosulphides and hydrogen carbonates the alkalinity increases with the escape of hydrogen sulphide or precipitation of sulphur. Calcium and magnesium chlorides are pronouncedly acidic towards the alkali sulphides, but have less effect on the hydrogen carbonates. Barium chloride aids in the decomposition of hydrogen carbonate ions in carbonate waters, but has no such effect where much alkali sulphide is present. It is proposed to estimate carbon dioxide present as hydrogen carbonate, and volatile hydrogen sulphide, by boiling the water rapidly for five minutes in a current of hydrogen, and absorbing the gases in an ammoniacal solution of cadmium and barium chlorides; on acidification of this solution with acetic acid the carbon dioxide is liberated and collected, and the cadmium sulphide remains. R. G. P.

The Effect of Denitrifying Bacteria in Water. K. Scheringa. (*Pharm. Weekblad*, 1921, **58**, 263-269.)—A culture of *B. pyocyaneus*, added to waters from various supplies, in no case exerted a denitrifying action; such action may in fact even be impeded by these bacteria, and nitrites are usually formed. With only a small content of organic matter, little change occurs; in presence of an appreciable amount, however, loss of nitrogen compounds results when samples of water are kept at moderately high temperatures in absence of air. W. J. W.

AGRICULTURAL ANALYSIS.

Colloid Chemistry of Indicators. G. Wiegner. (*Chem. Zeit.*, 1921, **45**, 200.)—The use of azolitmin as an indicator in soil analysis is based on the blue coloration which it gives with calcium ions; addition of potassium chloride increases the sensitiveness of the indicator owing to formation of calcium chloride. The retarding effect of humus is overcome in presence of excess of calcium ions.

W. J. W.

ORGANIC ANALYSIS.

Volumetric Method for the Estimation of Total Sulphurous Acid in Organic Substances. V. Froboese. (*Arbeit. Reichsgesundheitsamte*, 1920, **52**, 657-669.)—A quantity of the substance, containing not more than 90 mgrms. of sulphur

dioxide, is placed in a flask, together with 400 c.c. of water and 50 c.c. of 25 per cent. phosphoric acid; a current of carbon dioxide is passed into the flask, which is attached to a condenser, and the liquid is distilled, the distillate being collected in a receiver containing 40 c.c. of $\frac{N}{10}$ sodium hydrogen carbonate solution. When 200 c.c. of distillate have been collected, an excess of hydrogen peroxide is added, the mixture boiled until oxygen and carbon dioxide are no longer evolved, and the excess of alkali (carbonate) is titrated with $\frac{N}{10}$ hydrochloric acid, using methyl-orange as indicator. Each c.c. of $\frac{N}{10}$ sodium bicarbonate is equivalent to 3·2 mgrms. of sulphur dioxide. If the substance under examination contains volatile acids other than sulphurous acid, the distillation flask should be connected with the condenser by means of a long vertical tube; this arrangement is usually effective in preventing the distillation of the acids. The use of a current of carbon dioxide in the distillation is simply to aid the distillation of the sulphurous acid; it does not prevent the oxidation of the latter if any dissolved oxygen is present in the water.

W. P. S.

Estimation of Small Quantities of Iron in Organic Liquids, especially in Wines. Malvezin and C. Rivilland. (*Ann. Chim. anal.*, 1921, **3**, 90-92.)—The method depends on the titration of ferric salts with thiosulphate solution, using sodium salicylate as indicator. The ash obtained from 20 c.c. of wine is dissolved in 10 c.c. of 5 per cent. hydrochloric acid, the solution filtered, and the filtrate treated with 0·5 c.c. of hydrogen peroxide. After fifteen minutes the solution is boiled to decompose the excess of peroxide, 5 c.c. of 1 per cent. copper sulphate solution and 1 c.c. of 2 per cent. sodium salicylate solution are added, and the mixture is titrated with standardised (about 0·5 per cent.) sodium thiosulphate solution until the violet coloration is discharged. The copper sulphate acts as a catalyst and aids the reduction of the ferric salt.

W. P. S.

A Method of Purification of Methyl Alcohol. A. Lanzenberg and J. Duclaux. (*Bull. Soc. Chim.*, 1921, **29**, 135-136.)—A method of purification of methyl alcohol, especially from ethers and ketones of near boiling-points, is based upon the formation by methyl alcohol of a eutectic mixture of b.-pt. 50° C., whereas acetone, for example, forms an anti-eutectic mixture boiling 12° C. higher and easy to separate. One part of the crude alcohol is mixed with 7·5 parts (by weight) of chloroform and distilled in a rectifier, the fraction boiling between 52·5° and 53·5° C. is collected, acetone-chloroform mixtures and other impurities coming over above 64° C. This distillate is washed three times with an equal volume of water and the aqueous extracts are distilled. A second distillation will yield a practically anhydrous methyl alcohol. This product may contain about 0·25 grm. acetone per 100 c.c. as sole impurity.

H. E. C.

American Turpentine. (*U.S. Dept. Agric. Bureau Chem. Bull.* 898, 43-49.)—The following specifications are recommended by the U.S. Interdepartmental Committee on Paint Specification Standardisation (October, 1919) for turpentine and wood-turpentine — *i.e.*, turpentine distilled from resinous wood. The turpentine

shall be clear and free from suspended matter and water, its colour shall be "standard" or better, and it shall possess the characteristic odour of the variety of turpentine specified, or conform to that of an agreed sample; sp. gr. at 15·5°/15·5° C., 0·862 to 0·875; $[n]^{20}_{D}$, 1·468 to 1·478; initial boiling-point at 760 mm., 150° to 160° C.; fraction below 170° C. at 760 mm., at least 90 per cent. by volume; polymerisation test for turpentine (derived from oleo-resin), maximum residue 2·0 per cent. by volume with $[n]_{D}$ at 20° C. above 1·500; polymerisation test for wood-turpentine, maximum residue 2·5 per cent. with $[n]^{20}_{D}$ above 1·495.

Full details are given of methods of sampling and analysis. The determination of initial boiling-point and per cent. of distillate below 170° C. is carried out under standard conditions in an apparatus adopted by the American Society for Testing Materials for paint thinners, of which a dimensioned drawing is given. The polymerisation test is carried out by treating the turpentine (5 c.c.) with 38 *N*-sulphuric acid (20 c.c.) in a Babcock bottle; the turpentine is mixed with the cold acid, keeping the temperature below 60° C., until the mixture no longer warms up on shaking; the flask is then heated for not less than ten minutes to 60° to 65° C. on the water-bath, and shaken six times for half-a-minute each time (the stopper should not be used, as the flask may burst). After cooling to room temperature, sulphuric acid is added until the unpolymerised residue reaches the graduated neck and the volume of residue is read after separation by standing for twelve hours or by centrifuging. The residue should be viscous and straw-coloured, or darker. A comparative blank test should be made with turpentine of known purity. Colour is determined by matching in a tube graduated in millimetres. The following grades are recognised by the depth of column required to match a No. 1 yellow Lovibond tintometer glass: water white, 150 mm.; standard, 50 mm.; one shade off, 25 mm.; two shades off, 15 mm. R. G. P.

Syntheses of Cyanic Acid by Oxidation of Organic Substances. New Methods of Analysis of this Substance. R. Fosse. (*Bull. Soc. Chim.*, 1921, **29,** 158-203.)—Urea is an excretion product of vegetable as well as animal life, and results from the oxidation of both proteins, carbohydrates, and fats. Cyanic acid is the intermediate product. Urea is precipitated quantitatively in the form of crystals of urea dixanthylate by xanthydrol in methyl-alcohol solution in presence of acetic acid; cyanic acid may be similarly estimated by its hydrolysis by acid or alkalies to urea and precipitation with xanthydrol, which gives accurate results even with minute quantities. In the case of mixtures of cyanic acid and urea, two determinations of urea by xanthydrol are made, one direct and one after heating an hour with ammonium chloride; the difference is a measure of the cyanic acid. This acid may also be estimated by means of its silver salt; ammonium chloride and silver cyanate with water are heated for fifteen minutes on the water-bath, a little acetic acid and xanthydrol added, and the crystals separated by centrifuging; 0·5 mgrm. of cyanic acid may be so treated. Nitric acid interferes with the reaction. Cyanic acid may be identified microchemically by the characteristic filamentous crystals of the silver salt, which gives a blue colour with cobalt acetate. On shaking with water and amyl alcohol this is not transferred to the latter as in the case of a

sulphocyanate. Silver cyanate may be analysed quantitatively by heating it with ammonium chloride and weighing the resulting silver chloride, and estimating the urea as dixanthylate. H. E. C.

Characters of Resins used for making Varnishes. P. Nicolardot and C. Coffignier. (*Chim. et Ind.*, 1921, **5**, 150-156.)—To determine the relative hardness of different resins the authors employ an apparatus which consists of a vertical rod fitted with a steel ball at its lower end; the block of resin is supported below this rod, so that the ball rests on the surface, and weights are applied to the upper end of the rod. Usually the ball has a diameter of 2 mm., and the weight used is 5 kilos. After the ball has been pressed on the resin for varying periods—say, five seconds, ten seconds, and ten minutes—the diameter of the impression produced is measured by means of a microscope. During the test the resin is maintained at a definite temperature (0° C. or 25° C.). The hardness and other characters of resins are given as follows, the values for the hardness being the diameter of the impressions in millimetres, magnified by 44·5:

	Specific Gravity.	Melting-Point.	Hardness—		
			5 Seconds.	10 Seconds.	10 Minutes.
		C.			
Hard resins:					
Zanzibar	1·058	>300	22	—	23
Madagascar	1·056	>300	23	—	23
Demerara	1·047	180	23·5	—	23
Semi-hard resins:					
Congo	1·061	195	23	23·5	—
Benguela	1·058	165	24	24·5	—
Cameroon	1·052	150	23	24·5	—
Angola, red	1·066	>300	22	25	—
Kissel	1·066	110	22	25	—
Brazil	1·053	100	23	25	—
Angola, white	1·055	95	25	26	—
Sierra Leone	1·072	130	25	28	—
Soft resins:					
Aucoumea Klaineana ...	0·996	77	77	40·5	50
Hopea odorata	0·990	110	53	47	—
Hopea dealbata	1·061	142	82	72	—
Various:					
Amber, yellow	1·052	>300	22	25	—
Amber, cloudy	1·052	>300	29	29	—
Kauri, light	1·036	165	28	41	—
Kauri, bush	1·030	150	29	44	—
Kauri, crop	1·038	125	36	80	—
Manilla, hard	1·065	190	26	38	—
Manilla, soft	1·060	120	28	49	—
Pontianac	1·037	135	69	95	—

W. P. S.

Assay of Coal for Carbonisation Purposes. T. Gray and J. G. King. (*Fuel Research Board, Technical Paper* No. 1, 1921.)—The authors have devised a method and apparatus for the measurement and analysis of the primary products from coal carbonisation, by which secondary changes are avoided. The apparatus comprises an electric furnace; a hard glass or silica tube sealed at one end and closed by a rubber stopper at the other, and having a short lateral branch; a U-shaped condensing tube with bottom stop-cock; an absorption tube containing glass beads soaked in sulphuric acid and also provided with a stop-cock; and a gas-holder filled with glycerin and water. This gas-holder is connected at the bottom by means of a rubber tube with a glass reservoir suspended over two pulleys, having a counterpoise floating in a vessel, into which further liquid can pass from the reservoir by means of an overflow tube; by this arrangement any desired pressure may be maintained in the gas-holder. The sample of coal is ground to pass a 60-mesh sieve and dried at 105° to 110° C.; 20 grms. are then introduced into the hard-glass tube, previously weighed, and the weighed U-tube is attached, the whole being then connected with the remainder of the apparatus. After the furnace has been heated to 300° C., the sample tube is introduced and the temperature is raised to 550° to 600° C., in one hour, observations being made of the temperatures at which water and oil appear; heating is continued for one hour. The oil and water in the U-tube, after weighing, are washed into a graduated measure with chloroform or petroleum ether, by which means the aqueous portion is determined. The yield of coke is obtained by weighing the cooled sample-tube, and ammonia and bases are estimated in the contents of the absorption flask and in the aqueous washings from the U-tube. To ascertain the gas volume, water is removed from the vessel in which the counterpoise floats and is allowed to run into the reservoir, which is then raised till the top of the inlet is level with the liquid in the gas-holder, and the liquid in the manometer tube is level. From the weight and specific gravity of the liquid in the counterpoise vessel, the gas volume at observed temperature and atmospheric pressure is calculated. In a second test the gas from the first test, which is to some extent contaminated by air, may be used to displace air from the apparatus.

W. J. W.

Hydrolytic Alkalinity of Pure and Commercial Soaps. F. C. Beedle and T. R. Bolam. (*J. Soc. Chem. Ind.*, 1921, **40**, 27-29T.)—In continuation of previous work the authors have determined the hydrion concentration at 90° C. of aqueous solutions of different strengths of the sodium salts of oleic, palmitic and abietic acids, and of soaps of various types by the method of Francis and Geake (ANALYST, 1913, **38**, 522; 1916, **41**, 22; 1918, **43**, 358), depending on the decomposition of nitrosotriacetoneamine. Numerous results are given, and conclusions are drawn as to the hydrolytic alkalinity of different soaps and the irritant effect on the skin attributed to certain soaps—*e g.*, coconut oil soap. R. G. P.

Estimation of Cellulose in Bast Fibres. Y. Uyeda. (*J. Ind. Eng. Chem.*, 1921, **13**, 141-143.)—In estimations of cellulose, Dore (*J. Ind. Eng. Chem.*, 1920, **12**, 264) found that Renker's method of chlorination without hydrolysis (*Bestimmungs-*

methoden der Cellulose, 1910) gives better results than either of the methods proposed by Cross and Bevan, or Johnsen and Hovey (*Paper*, 1918, **21**, 36). An investigation of these methods has been applied to the estimation of cellulose in Korean hemp fibre, which was cut into pieces, 1 cm. long, for storage during the experiment. Preliminary treatment of the fibre consisted in drying 1 grm. samples for sixteen hours in an electric oven, and extracting them for six hours with benzene, and then for the same period with 95 per cent. alcohol. Here again, Renker's method was the most satisfactory, but the cellulose obtained by Cross and Bevan's method showed the highest degree of purity, as indicated by a determination of the α-cellulose by their mercerisation test; this test also shows the chemical behaviour of the fibre towards alkaline reagents. Pectin substances retained in the cellulose obtained by Renker's and Johnsen's methods are partly removed by the alkali in mercerisation. Pentosan and methylpentosan in bast fibres may be estimated by Tollen's and Kroeber's and Ellet and Tollen's (*J. Landw.*, 1905, **53**, 20) methods respectively; but hexosans also yield oxymethylfurfural. Hemp fibres have in some respects a similar composition to that of wood cellulose, and their cellulose may have an oxycellulose structure, as its affinity towards basic dyestuffs is much greater than that of cotton. The cellulose, as well as the α-cellulose, from these fibres, on distillation with hydrochloric acid, gives a mixture of furfural, methylfurfural, and oxymethylfurfural; and a brown phloroglucide, consisting of the two last-named substances, was obtained from the α-cellulose. W. J. W.

Constitution of Cellobiose (Cellose). W. N. Haworth and E. L. Hirst. (*J. Chem. Soc.*, 1921, **119**, 193-201.)—The structure of cellobiose has been investigated, and experimental evidence is brought forward in support of the constitutional formula proposed previously (*J. Chem. Soc.*, 1919, **115**, 809). An improved method for the preparation of cellobiose consists in stirring for five minutes 20 grms. of dried filter-paper with 80 c.c. of commercial acetic anhydride (80 to 95 per cent.) containing 11 c.c. of sulphuric acid, and heating the paste on a bath at 120° C. until, at about 112° C., the mixture becomes a dark-red, mobile liquid and begins to boil; immediately the liquid appears to be changing to black it is poured into 1·5 litres of cold water. The pale yellow precipitate of cellobiose oct-acetate, which separates after about ten minutes (and which should be soluble in boiling, but not in cold, alcohol), is allowed to stand in contact with water for six hours, filtered, washed, and dried at 40° C., and then dissolved in boiling 90 per cent. alcohol, the solution filtered hot, and the octa-acetate crystallised (m.-pt., 224° to 227° C.; yield, 25 to 35 per cent. of cellulose taken). The acetyl groups are removed from the cellobiose oct-acetate by alcoholic potassium hydroxide, and the resultant potassium cellobiosate converted to cellobiose by treatment with perchloric acid. Cellobiose is hydrolysed by emulsin and by cellase, with production of dextrose, but is not hydrolysed by maltase. It is not known whether the effect of emulsin is due to the presence of traces of cellase. The optical properties of derivatives of lactose and of cellobiose are strikingly similar, and it is considered very probable that the linking of the two hexoses in each is structurally and stereochemically similar. Cellobiose may be regarded as glucose-β-glucoside. R. G. P.

Solubility of Crystalline Substances in Rubber. G. Bruni. (*Giorn. Chim. Ind. Appl.*, 1921, **3**, 51-53.)—Thermal analysis of the binary systems formed by rubber with azobenzene, naphthalene, and *p*-toluidine shows that the rubber acts toward these crystalline compounds as an ordinary solvent, yielding true, saturated solutions, supercooling and supersaturation of these readily occurring. That complexes between the rubber and the crystalline components of the systems are formed is indicated by the pronounced concavity of the composition-temperature curves towards the axis of concentration. The molecular condition of the rubber is not altered by prolonged heating of the systems. T. H. P.

INORGANIC ANALYSIS.

Action of Iodine on Different Metals in the Cold: Process for the Detection of the Presence of Chlorine in the Atmosphere. C. Matignon. (*Comptes rend.*, 1921, **172**, 532-534.)—If a small crystal of iodine be placed on a leaf of beaten silver, the latter undergoes conversion into friable silver iodide, the crystal becoming surrounded by a yellow circle, the radius of which increases with gradually diminishing rapidity. Calculation shows that a sheet of silver 3μ in thickness should be completely converted into iodide beneath the iodine in about a second. Other metals give somewhat similar results. This phenomenon may be employed to indicate the presence of chlorine in the air, the procedure being as follows: On a strip of silver leaf 1 cm. wide is placed a thin layer of moistened potassium iodide reaching from side to side of the strip, which is used to close an electric circuit. When chlorine comes into contact with the iodide, the iodine liberated converts the metal into non-conducting silver iodide and thus interrupts the current; if an ampèremeter be inserted in the circuit, the movement of the needle may serve to actuate an alarm. If the width of the rectangle of potassium iodide be 3 mm. and the silver leaf be 3μ in thickness and 1 cm. in width, 0·1 c.c. of chlorine will suffice to liberate enough iodine to convert the corresponding surface of the metal into iodide. Lead iodide or mercuric iodide may be used in place of potassium iodide, and bromine vapour acts appreciably more slowly than chlorine.

T. H. P.

Electrometric Titration of Hydriodic Acid and its Use as a Standard in Oxidimetry. W. S. Hendrixson. (*J. Amer. Chem. Soc.*, 1921, **43**, 14-23.)—For estimating iodine ion in presence of other halides, a method of electrometric titration is used, in presence of an oxidising agent which can be standardised accurately and is sufficiently stable and yet sensitive enough to react completely at room temperature with hydriodic acid when the two are present in equivalent amounts. These requirements appear to be met by potassium permanganate. A potentiometer is employed, consisting of a rheostat having a coil 425 mm. long, made of about 650 turns of oxidised resistance wire closely contact-wound, and with a resistance of 169·45 ohms. A section of square brass beam, accurately graduated in mm., carries the sliding contact, and the contact points are narrowed so as to touch only two wires at once. Since the voltage of the titration cell never exceeds about 0·8 in

these measurements, the calculated resistance, 229 ohms, was added by means of a resistance box to the end of the instrument not traversed by the sliding contact. This makes the scale virtually 1 m. in length, and the value of 1 mm. equals approximately 2 millivolts when one storage cell is used as a balance. Calibration of the potentiometer without the extension to every 5 cm., together with the results of measurements of known voltages, showed that the error was nowhere greater than 0·4 per cent., while in the region of measurement it would be smaller, and with its virtual 1,500 turns of wire still smaller. A damped movable coil galvanometer, with rated sensitiveness 106 megohms, was used and was not found too sensitive; with this were used the Leeds and Northrup electric lamp and etched scale. The titration vessel consisted of a three-necked Woulff's bottle with a hole bored for the tip of the burette; one neck carried the drawn-out tube for the washed carbon dioxide or compressed air for vigorously stirring the solution, and the others the usual calomel electrode with a normal potassium chloride solution and the small, bright, platinum electrode. By means of this apparatus it is possible to titrate dilute sulphuric acid solution of iodine accurately with standard permanganate solution; chloride or bromide retards and diminishes in amount the sudden rise in voltage to an extent proportional to its concentration, but the presence of chloride in amount at least equivalent to the iodide, or one-fourth of this quantity of bromide, is permissible. Dichromate and iodate in 0·02 *N* and 0·05 *N* solutions may be titrated accurately by adding the solution to an excess of iodide in sulphuric acid and titrating the excess with permanganate, dichromate showing its theoretical oxidising capacity; silver has been determined in the same way. The high results obtained by Crotogino (*Zeitsch. anorg. Chem.*, 1899, **24**, 225) in titrating iodide with permanganate are not explainable by formation of iodic acid before the end-point was reached.

T. H. P.

Use of Spot Reactions in Qualitative Analysis. F. Feigl and R. Stern. (*Zeitsch. anal. Chem.*, 1921, **60**, 1-43.)—Tests made by adding a drop of the solution under examination to a drop of reagent placed previously on a plate, or, preferably, on a piece of filter-paper, are of wide application, and are often more sensitive than similar tests carried out in test-tubes. The authors give numerous examples of the usefulness of the method in the detection of individual substances and certain constituents in mixtures. Reactions of aluminium, uranium, and chromium are described, depending on the fact that salts of these metals yield coloured lakes with alizarin; manganese hydroxide yields a blue coloration with benzidine acetate solution.

W. P. S.

Separation of the Phosphorus from the other Components of Steel. R. Ariano. (*Gazzetta*, 1921, **51**, I., 1-31.)—The methods employed to separate phosphorus from the other constituents of steel are examined, the reactions involved being considered with the help of chemical statics and dynamics, and the conditions necessary to render this separation complete indicated. Following a discussion of the various methods employed for the elimination of the silicon and of the copper, arsenic, etc., the acetate method and the different modifications of the phospho-

molybdic method for precipitating the phosphorus are examined. In separating the silica, 16 c.c. of nitric acid of density 1·2 are used for the treatment of each grm. of steel, and, after each evaporation to dryness, the residue is taken up in hydrochloric acid of medium concentration. Ammonium phosphomolybdate does not appear to be a definite compound, and the ratio of the amount of P_2O_5 calculated from the weight of precipitate found on the basis of Carnot's formula, $P_2O_5, 24MoO_3, 3(NH_4)_2O, 3H_2O$, to the amount actually present, depends on the concentration of the molybdic anhydride, the quantity of phosphorus present, the temperature at which the precipitation takes place, the duration of the precipitation, stirring during precipitation, the nature of the metals present, the volume of the liquid, the nature and amount of the acids present, the concentration of the ferric ions, and the proportion of ammonium nitrate in the solution (see Jörgensen, *Zeitsch. anal. Chem.*, 1907, 370). The conditions favouring the precipitation are as follows: (1) High concentration of the molybdic anhydride; this, however, renders the precipitate less pure, so that the concentration must be regulated according to the subsequent treatment of the precipitate. (2) High temperature, even 100° C. being advisable provided that the arsenic has been previously removed; precipitation for twenty-four hours at the ordinary temperature does not yield very accurate results. (3) Stirring of the liquid during precipitation. (4) The solutions should be as dilute as possible, especially as regards iron. (5) The solution should be either neutral or very strongly acid, medium acidity rendering the precipitation incomplete. (6) The precipitate should be washed, not with nitric acid, but with water. (7) The same conditions should always be employed. (8) Addition of ammonium chloride, sulphate, etc., facilitates the precipitation and renders it more complete. T. H. P.

Volumetric Estimation of Arsenious Compounds by Means of Potassium Dichromate. R. Meurice. (*Ann. Chim. anal.*, 1921, **3**, 85-86.)—The arsenious compound in hydrochloric acid solution is treated with potassium bromide, and the mixture then titrated with standardised potassium dichromate solution. During the titration a current of air is aspirated through the mixture, and then conducted into a test-tube containing potassium iodide solution and starch; as soon as all the arsenious acid has been oxidised, the next drop of dichromate solution added liberates bromine, which is carried over with the air into the iodide-starch mixture, in which it produces a blue coloration. W. P. S.

Separation of Tin and Antimony in Hydrochloric Acid Solution by Means of Hydrogen Sulphide. G. Luff. (*Chem. Zeit.*, 1921, **45**, 249-251, 254-255, 274.)—With increasing amounts of concentrated hydrochloric acid, the temperature at which precipitation of antimony and tin salts by the treatment of their boiling solutions with hydrogen sulphide takes place, is lowered. Thus, with 8, 14, and 30 c.c. respectively added to 100 c.c. of solution, the temperatures are 102°, 102°, and 95° C. for antimony trisulphide; 102°, 100°, and 80° C. for the pentasulphide; and 90° to 95°, 75° to 80°, and 25° C. for stannic sulphide. The depression of the temperature is still more noticeable with ammonium chloride: with 36 grms. per 100 c.c., the trisulphide and pentasulphide are precipitated at 78°

and 60° C. respectively, whilst stannic sulphide does not separate, except after standing, even at normal temperature. The effect of varying concentrations of hydrochloric acid, with a fixed amount (16·5 per cent.) of ammonium chloride, on the separation of antimony and tin sulphides, has been investigated. Fourteen c.c. of hydrochloric acid (sp. gr. 1·193) per 100 c.c. of solution effects the clearest separation; the lowest amount to ensure separation is 8 c.c. per 100 c.c., and the maximum 35 c.c. per 65 c.c. of solution. Vortmann-Metzel's method is satisfactory for separation of antimonious sulphides, as well as modifications of it with 8 and 14 c.c. of hydrochloric acid respectively. Sufficiently accurate results are also obtained by the Panajotow-Prim method; if the ammonium chloride content exceeds 16·5 grms., however, the solution must be cooled in ice, introduction of hydrogen sulphide arrested at 20° C., and the solution filtered at 5° C.

W. J. W.

Estimation of Tungsten. G. Fiorentino. (*Giorn. Chim. Ind. Appl.*, 1921, **3**, 56-58.)—The volumetric estimation of tungstic acid requires (1) a solution containing 32·6893 grms. of crystallised lead acetate and about 2 c.c. of acetic acid per 2 litres, and (2) a solution containing 7·485 grms. of crystallised ammonium molybdate per litre. If the tungstic acid is in the form of dissolved alkali salt, the solution, which should not contain excess of alkaline salts, is treated with 8 c.c. of concentrated ammonia solution; hydrated tungstic acid, however, is heated gently with 25 c.c. of water and 8 to 10 c.c. of concentrated ammonia until solution is complete. The solution in either case is diluted to about 200 c.c., acidified with acetic acid, then made alkaline to the extent of 1 or 2 drops of ammonia solution, and boiled until a litmus paper in the liquid turns wine-red. A measured volume, in excess, of the standard lead acetate solution is added to the boiling liquid, with continual stirring, boiling being continued until the precipitate becomes pulverulent or crystalline. Solution (2) is then added from a burette to the boiling liquid until a drop of the latter gives a yellow coloration with a drop of a fresh solution of 0·1 grm. of tannin in 10 c.c. of water. The volume (usually 0·3 c.c.) of the molybdate solution required to produce a distinct yellow coloration with the tannin is determined by a blank test on 200 c.c. of water and applied as a correction. The amount of WO_3 present is calculated by multiplying the amount of lead precipitated by the tungstic acid by 1·202. The method is based on the reactions: $H_2WO_4 + Pb(C_2H_3O_2)_2, 3H_2O = PbWO_4 + 2C_2H_4O_2 + 3H_2O$, and $Mo_7O_{24}(NH_4)_6, 4H_2O + 7Pb(C_2H_3O_2)_2, 3H_2O = 7PbMoO_4 + 6NH_4{\cdot}C_2H_3O_2 + 8C_2H_4O_2 + 3H_2O$.

In the case of an ore or concentrate, 1·5 grm. of the finely powdered material is heated to 60° C., and occasionally stirred for an hour with 100 c.c. of concentrated hydrochloric acid in a covered beaker. The beaker is then heated more strongly on a sand-bath or asbestos card, the liquid being stirred continuously for thirty minutes, and then occasionally until reduced to about one-half of its original volume, when it is treated with 20 c.c. of nitric acid and evaporated to 25 c.c.; 5 c.c. of nitric acid are then added, and the solution evaporated to 15 to 20 c.c., mixed with about 180 c.c. of boiling water, left for some hours, and then filtered, the precipitate being washed by decantation with 1 per cent. nitric acid. Unless a very exact result is required,

the filter and its contents are returned to the beaker and heated gently with 8 to 10 c.c. of concentrated ammonia solution and 25 c.c. of water, which should dissolve all the tungstic acid and leave no undecomposed particles of ore. Either all or an aliquot part of this solution, containing about 0·25 grm. of WO_3, is treated according to the volumetric method described above. When great accuracy is necessary, the filter is washed with the mixture of 8 to 10 c.c. of ammonia and 25 c.c. of water into the beaker containing the bulk of the precipitate, the liquid being heated gently until all the tungsten trioxide is dissolved, filtered through the same filter, and the latter washed with very dilute ammonia solution. The liquid is then dealt with as above, and the residue on the filter dried and calcined at a moderate temperature, any tungsten trioxide it contains being estimated by the cinchonine method (see below). The acid liquor from the original attack of the ore and from the washing should also be concentrated and precipitated with cinchonine, since part of the WO_3 may be hydrolysed and dissolved if too little acid remains when the boiling water is added.

In the cinchonine gravimetric method, 0·5 to 1 grm. of the tungsten ore is heated to dull redness with a little wood charcoal, and about 1 grm. of sodium hydroxide, previously fused in an iron crucible. The fused mass is cooled and dissolved by boiling with water, finally with addition of 4 to 5 grms. of solid ammonium carbonate; the liquid is then filtered, the filter washed with very dilute sodium hydroxide solution, and the filtrate heated, treated gradually with 30 c.c. of concentrated hydrochloric acid, boiled for a few minutes, mixed with 8 to 10 c.c. of a solution of 30 grms. of cinchonine in 50 c.c. of concentrated hydrochloric acid and 250 c.c. of water, and allowed to stand for some hours. It is then passed through a filter containing a little pulped filter-paper, the precipitate being washed three or four times with dilute cinchonine solution (30 c.c. of the concentrated solution, 30 c.c. of concentrated hydrochloric acid, and 1,000 c.c. of water), and once only with cold water; the filtrate contains the tin, which may be estimated by the Pearce-Low method. The filter is introduced into the original precipitating beaker and heated gently, and stirred to break lumps, with 8 to 10 c.c. of ammonia solution and 25 c.c. of water, the solution being filtered and the filter washed with hot water containing a little ammonia. The filtrate is heated to expel the excess of ammonia, treated at once with 2 to 3 drops of hydrochloric acid and 8 to 10 c.c. of the concentrated cinchonine solution, and stirred well for some minutes. After one to two hours, the liquid is filtered through an ashless filter containing a little pulped filter-paper, and the precipitate washed as before with dilute cinchonine solution and once with water, dried, ignited in a platinum crucible and weighed as WO_3. A small proportion of silica sometimes occurs in the precipitate, and this may be expelled by treating the precipitate with 1 to 2 drops of dilute sulphuric acid and a little hydrofluoric acid, the latter being then driven off, 1 to 2 drops of nitric acid added, and this and the sulphuric acid then expelled by heating. Cinchonine may be recovered from liquors containing it by rendering them alkaline with ammonia. T. H. P.

New Method for Separating Ferric, Chromium, and Aluminium Hydroxides. M. and M. Lemarchands. (*Ann. Chim. anal.*, 1921, **3**, 86-87.)—The three

hydroxides are precipitated together in the usual way, and washed until free from ammonia; the precipitate is then boiled with 10 per cent. sodium hydroxide solution, and sodium perborate is added. Ferric hydroxide remains insoluble and is separated by filtration, whilst the chromium and aluminium hydroxides pass into solution as sodium chromate and sodium aluminate respectively. The filtrate containing these two compounds is divided into two parts: in one, the aluminium is precipitated as hydroxide by boiling with the addition of an excess of ammonium chloride, and in the other portion the chromium is precipitated as lead chromate. W. P. S.

Iodimetric Method for the Estimation of Chromium in Chromite. E. Little and J. Costa. (*J. Ind. Eng. Chem.*, 1921, **13**, 228-230.)—The substance is fused with sodium peroxide, and the resulting chromate estimated iodimetrically in hydrochloric acid solution, ammonium fluoride being added to prevent interference by ferric chloride. The details of the method are as follows: 0·4 grm. of the sample is mixed in an iron crucible with 3 grms. of sodium peroxide, the mixture is covered with a further 2 grms. of peroxide, then heated at a low red heat for five minutes, and fused for fifteen minutes at a higher temperature. After cooling, the fused mass is dissolved in 150 c.c. of water, the solution treated with a further 0·5 grm. of peroxide, and boiled to decompose the excess of the latter. The solution is then cooled, hydrochloric acid is added until the ferric hydroxide has dissolved, and 5 c.c. of concentrated acid are introduced for every 100 c.c. of solution. Ammonium fluoride is now added until the solution no longer gives a reaction for ferric iron with potassium ferrocyanide, an excess of 1 grm. of ammonium fluoride is introduced and, after the addition of 3 grms. of potassium iodide, the liberated iodine is titrated with standardised thiosulphate solution. W. P. S.

Entanglement of Lime and Magnesia by Chromic Oxide Precipitates. E. Toporescu. (*Comptes rend.*, 1921, **172**, 600-602.)—The proportion of lime contained in the chromic oxide precipitated by ammonia solution from a boiling solution containing calcium and chromic chlorides tends towards a limit corresponding with formation of calcium chromite, $Cr_2O_3,3CaO$; the lime may be removed by washing the precipitate on the filter with boiling 5 per cent. ammonium nitrate solution. Similarly, the proportion of magnesia carried down with chromic oxide from a solution containing magnesium and chromic sulphates and ammonium chloride is variable and, if the liquid be saturated with the ammonium salt, reaches a limit corresponding with the formula $Cr_2O_3,3MgO$; the magnesia is removable by washing the precipitate several times by decantation with boiling 5 per cent. ammonium nitrate solution. T. H. P.

The Quantitative Estimation of Calcium and Magnesium in Various Salt Mixtures. E. Canals. (*Bull. Soc. Chim.*, 1921, **29**, 152-158.)—The precipitation of calcium and magnesium as oxalates in ammoniacal and acetic acid solutions has been studied. The essential condition for precipitation of lime in the cold in presence of magnesium is that a very dilute solution (not more than 1 part in 400) be taken. Magnesium is not precipitated with calcium oxalate when the dilution of

magnesium is greater than 1 : 100, and magnesium oxalate can be removed from the precipitate by repeated washing with boiling water, in some cases as much as 500 c.c. being used. In acetic acid solution the lime can be accurately estimated if the concentration is about 1 : 700, and the precipitate be well washed with boiling water.

H. E. C.

Removal of Nitrates by Means of Alcohol. R. Schneidewind. (*Chem. and Met. Eng.*, 1921, **24**, 22; *J. Soc. Chem. Ind.*, 1921, **40**, 131A.)—Nitric acid and nitrates are removed by heating with alcohol and sulphuric acid. For example, 20 c.c. of nitric acid are diluted with 150 c.c. of water and treated with 15 c.c. of sulphuric acid, and when the liquid is nearly boiling 5 c.c. of ethyl alcohol are carefully run in from time to time until nitrous fumes cease to be evolved on adding more alcohol. The liquid is boiled until free from alcohol, and is then sufficiently free from nitrates not to react with ferrous sulphate or oxidise hydrogen sulphide.

R. G. P.

Estimation of Nitrous Acid in Mixed and Waste Acids. H. Toussaint. (*Z. angew. Chem.*, 1921, **34**, 102.)—A Woulff bottle with three necks is used for the estimation, through the outer ones of which pass a funnel, and an inlet tube for carbon dioxide. Seven hundred c.c. of air-free, distilled water is first placed in the bottle, and the air in this is displaced by carbon dioxide. The acid is then introduced through the centre neck from a burette, after which a glass tube, 10 cm. long, is fixed in the neck by means of a rubber sleeve, so that its lower end is level with the top of the Woulff bottle. Potassium iodide solution is run in through the funnel, and the mixture is then titrated with $\frac{N}{2}$ or $\frac{N}{10}$ thiosulphate, introduced through the centre neck, a stream of carbon dioxide being passed through during the operation, for which purpose the inlet tube should have been previously pushed down to the bottom of the bottle. Starch solution may be used as indicator if desired. Large amounts of ferric oxide in the acid affect the results; the iron must therefore first be removed by sodium hydroxide, free from nitrate, and the filtrate subsequently acidified with pure sulphuric acid.

W. J. W.

PHYSICAL METHODS, APPARATUS, ETC.

Electric Oven for Rapid Moisture Estimations. G. L. Spencer. (*J. Ind. Eng. Chem.*, 1921, **13**, 70-72.)—In an electric oven the material is enclosed in a crucible, preferably of metal, having a bottom of Monel metal filter cloth. The crucible is in close contact with its seat and above an annular channel connecting several crucible openings, and is in communication with a vacuum pump or ejector, by means of which electrically heated air is drawn through the oven. The heating element for the oven is placed inside to prevent loss by radiation; the temperature is regulated by means of a rheostat, and a time-switch rings a bell at the end of the drying period. The apparatus is suitable for drying any substance through which air can pass freely, and is especially adaptable to the drying of raw sugar and cane bagasse; with these latter materials tests were completed in ten and thirty minutes respectively.

W. J. W.

Laboratory Apparatus. G. Ajon. (*Giorn. Chim. Ind. Appl.*, 1921, **3**, 62-63.)—*Extractor with Continuous Flow of Liquid.*—This consists of a modification of the Soxhlet extractor, in which the syphon tube, just after it begins its downward course, is inserted into a wider tube connected in the ordinary way with the stem of the apparatus. *Multiple Hot Filter.*—This consists of an elongated, constant-level, copper water-bath, which has a number of small holes in the base and corresponding larger holes in the lid to take funnels, and is connected by means of two tubes with the upper and lower parts of a small boiler, so that continuous circulation of water takes place. It is intended especially for the filtration of calcium citrate in analyses by Warington's method. T. H. P.

Cold Test Apparatus for Oils. G. H. P. Lichthardt. (*J. Ind. Eng. Chem.*, 1921, **13**, 145-146.)—The apparatus consists of an inclined, galvanised iron refrigerator tank, 6 × 6 × 6 inches, in which are placed glass tubes of 0·3 inch internal diameter, which project appreciably through one side of the tank, and are bent upwards just beyond the other side and connected with an air supply. The samples of oil are put in the tubes so as to occupy lengths of about 6 inches, and the tank is then filled with a freezing mixture of acetone and carbon dioxide snow. The temperature at which, under an air-pressure of 16 inches of water, the oil becomes solid and fails to move is noted, after which the temperature is allowed to rise slowly, readings being taken at short, fixed intervals. The temperature at which the oil appears in the projecting portions of the tubes is then taken as the " cold test." W. J. W.

Melting-Point Apparatus. F. Friedrichs. (*Zeitsch. angew. Chem.*, 1921, **34**, 61.)—An apparatus for melting-point determinations consists of an outer glass vessel containing the heating liquid, and an inner one surrounding the thermometer, which serves to eliminate the error due to lag. Both vessels are oval in shape, but have flattened front and back surfaces, which enable microscopical readings to be made without interference due to curvature of the glass. Tubular extensions, fused obliquely into the base of the outer vessel, admit of the melting-point tubes being placed in position through openings in the inner vessel without removal of the thermometer. W. J. W.

Determination of Boiling-Point of Minute Quantities of Substances. V. Arreguine. (*Ann. Chim. analyt.*, 1921, **3**, 40-49.)—The method, which is similar in principle to that of Schleiermacher, is based on the vapour tension of the substance, and can be employed in cases where only a few mgrms. of material are available. The apparatus consists of a test-tube about 80 to 85 mm. in length and 9 mm. in diameter, which is half-filled with mercury, and a piece of open tubing (about 5 mm. in diameter) is selected of such bore that when immersed in the mercury the meniscus in the open tube is level with that in the test-tube, thus eliminating error due to capillarity. One end of the open tube is then sealed and blown into a small bulb, so that it fits inside the test-tube, leaving an annular space of 0·5 to 1 mm., and the tube is cut to a length of about 52 mm. For the determination of boiling-points, the inner tube is filled with mercury to within about 1 mm. of the top ; a drop of

the liquid is then added, precautions being taken (*e.g.*, gentle heating) to eliminate air from the tube and its contents. In the case of solids, a small quantity of the substance is melted in the bulb and solidified by cooling before filling the tube with mercury. The filled tube is inverted and placed inside the test-tube, the open end being submerged below a short column of mercury in the test-tube. The inner tube is then fixed in position by filling the space in the test-tube above it with glass beads, and covering with a metal cap clipped over the rim of the test-tube. The test-tube, with its inner tube, is then fixed near a thermometer in a bath, which is heated as evenly as possible; on raising the temperature, the point at which the meniscus of the mercury in the inner tube containing the substance is level with that in the test-tube is taken as the boiling-point. The chief difficulty of operation is that due to local inequalities in temperature of the heating bath. The method only yields approximate results, and is unsuitable for substances boiling above 210° to 220° C., owing to errors produced by the vapour pressure of the mercury; fusible alloys were found to be unsuitable as substitutes for mercury. Formulæ are quoted for the correction of errors due to the vapour tension of the mercury and to excess of liquid above the mercury in the inner tube. The following figures were obtained without corrections: Water, 99·6°; carbon disulphide, 46°; ethyl alcohol, 78·1°; ether, 34·96°; aniline, 180·3°, 183·5°; acetone, 55·8°; and monobrombenzene, 153° C. The calculated corrections for a column of 1 mm. of liquid above the mercury in tube were generally very small (0·001° to 0·005° C.) and therefore negligible. The following corrections were calculated for the vapour tension of mercury: With water, 0·015°; with carbon disulphide, 0·001°; with alcohol, 0·005°; with monobrom benzene, 0·17°; with aniline, 0·64° C. R. G. P.

Determination of the Flash Points of Mixtures of Air and Benzine. W. Reinders. (*Chem. Weekblad*, 1921, **18**, 157-159.)—An apparatus for determining the maximum and minimum flash points of air saturated with benzine vapour consists of a glass tube of 35 cm. length and 2 cm. diameter, having two platinum wires fused into the top part, and terminating at its upper end in a narrow tube with funnel and tap. It is surrounded for the greater part of its length by a glass water-jacket, provided with stirrer and thermometer, and is connected by rubber tubing with a levelling tube. The tube is first filled with water, after which, by manipulation of the levelling tube, a layer of about 10 cm. of benzine and a few cubic centimetres of air are introduced. The water in the jacket is brought to any desired temperature, and the apparatus is allowed to stand for five minutes to ensure saturation of the air with benzine vapour. An electric spark is then passed through the mixture. For determinations at temperatures below O° C., the jacket is filled with a freezing mixture, and the benzine layer must fill the whole portion of the tube which is enclosed in the jacket, or alternatively, the water below the benzine layer must be superseded by a saturated salt solution. Results obtained with various benzine fractions are tabulated. The boiling-points of the fractions themselves may vary between wide limits, and consequently afford but little information as to the volatility of the benzine; a specific gravity determination must therefore be made. W. J. W.

Experimental Data and Balance for Fixing the Exposure to X-Rays in Radiography and Radiotherapy. F. Miramond de Laroquette and S. Millot. (*Comptes rend.*, 1921, **172**, 525-527.)—The results obtained by the authors do not confirm the proportionality of the chemical or biological effect of X-rays to the square of the voltage, or to the square of the spark-length. Tests made with various forms of tubes and apparatus show, indeed, that for purposes of calculation it is preferable to retain the degree element *B*, and to measure this directly with a radiochromometer, and not by the spark-length, the variations in the two cases being sometimes discordant. When the radiation increases by 1 *B* unit, the effect produced superficially and in depth increases on the average by about 33 per cent. between 4 *B* and 7 *B*. The absorption of X-rays by different soft tissues, such as skin, fat, and muscle, is virtually the same, aluminium exhibiting a tenfold greater coefficient of absorption; theoretically, the aluminium used for filtration in radiotherapy should not be more than 3 mm. in thickness. For the various factors involved in the calculations, graphic scales have been constructed, these being arranged on a plate oscillating about a horizontal axis. Equal weights are placed on the scales at the graduations corresponding with the data fitting the particular case, a similar weight being then moved along the scale of the unknown factor until equilibrium is established. The different formulæ, being calculable by means of logarithms, may thus be transformed into equilibrium equations. By this means it is possible to determine instantly the necessary exposure for any radiographic or radiotherapeutic case, to make any combination by varying the current and voltage, the distance of the tube and the thickness of the filter, and by interposing a screen, and also to ascertain in Holzknecht units (*H*) the quantity of the incident radiation and also that which is absorbed and thus acts at any particular depth in the tissues. T. H. P.

REVIEWS.

L'Équilibre des Substances Hétérogènes. Abridged account by Willard Gibbs, translated by G. Matisse, with complete explanatory notes. Pp. 102. Paris: Gauthier, Villars et Cie. 1919. Price 3 fr. 50, plus 50 per cent.

The well-known treatise of Gibbs on heterogeneous equilibrium has already been translated into French, but it is difficult for the non-mathematical reader to understand the abstract manner in which the subject is expounded. In 1878, in the *Scientific American*, Gibbs published an abbreviated account of the essentials of his theory of heterogeneous equilibrium and set out the principal formulæ. The present volume is an exact translation of Gibbs's synopsis, together with notes by the translator explaining the derivation and application of the formulæ given in the text.

The book is addressed to those who have not the mathematical equipment necessary to understand the larger treatise and to those who, having mastered the large work, wish to summarise and retain the principal propositions. The notes are very full and clear, and should enable the reader, who is possessed of the necessary

quantum of mathematics which is essential for the understanding of modern physical chemistry, readily to grasp the fundamental theory of heterogeneous equilibrium.

H. E. Cox.

PUBLIC HEALTH CHEMICAL ANALYSIS. By ROBERT C. FREDERICK and AQUILA FORSTER. Pp. 284 and Appendix. London: Constable and Co. Price 21s.

The subject-matter of this excellently printed volume includes the analysis of air, water, sewage, and trade effluents, most of the ordinary foods, condiments, and drinks, disinfectants, soaps, and rag flock, and as the whole ground is covered within the compass of 286 pages, it will be obvious that the authors have had to exercise both discrimination and restraint. Only in relatively few cases could alternative methods be included, and much discussion of theoretical principles could not be expected.

In spite of these limitations the authors have acquitted themselves well, and have produced a volume which is essentially practical and extremely clearly written.

Judging from its title the book is intended mainly for those preparing for the Diploma of Public Health, and if this be so it will account for the relatively large amount of space devoted to the analyses of air and water, which between them occupy nearly one-third of the total number of pages. It may, perhaps, also account for the insertion of an introductory chapter, describing the fundamental operations of analysis, weighing and measuring, the care of platinum dishes, the use and preparation of normal solutions, etc. Except for the revolutionary recommendation that "small quantities of infusible precipitates, such as barium sulphate, should be weighed direct on the pan of the balance," no exception can be taken to the instructions given, but surely they should be quite unnecessary for those competent to undertake the wide range of analytical operations described.

In dealing with the examination of air, the writers seem to have been carried away by their partiality for the Haldane apparatus, to which some twenty pages are allotted, and undue space is given to the relatively simple determination of carbonic acid. Haldane's hæmoglobin method for the determination of carbon monoxide is a dangerous one to put into the hands of any but experts. It sounds delightfully simple, but only the happy possessors of a retina extremely sensitive to the red end of the spectrum can hope to apply it with any chance of success. The authors warn the reader that experience is required, but unless the eyes of the operator possess the requisite red sensitiveness no amount of experience will suffice.

The chapter on water is commendably full, and a series of fourteen typical analyses of waters from various sources, with appropriate criticisms of the results, will be of considerable help to the beginner.

An alternative to the phenol-sulphonic acid method for the determination of nitrates might with advantage have been given, and the recommendation to evaporate 5,000 c.c. of the water to dryness for the estimation of the silica and the ordinary bases is obviously a counsel of perfection which it would be very inconvenient to follow.

The section on milk and milk products is well up to date, but with regard to matters of detail the writers here again show a tendency towards the academic, and the

procedure recommended is often unnecessarily laborious and expensive. The determination of total solids in milk by weighing 10 c.c. into large platinum dishes, each containing $3\frac{1}{2}$ inches of platinum wire (to break up the skin that forms on heating) would hardly commend itself to the busy practitioner in these hard times. The omission of such an accurate, simple, and widely applicable method as that of Gottlieb for the determination of fat is to be regretted even though the Adams, Werner-Schmidt maceration and two centrifugal processes are described.

Another rather more serious omission is the failure to mention the use of powdered pumice in the Reichert-Polenske process, as the employment of broken porcelain, which the authors recommend, might seriously vitiate the results.

To the other articles of food, etc., less space is allotted, but there is a chapter on the general methods of examination of sugars, which on the whole is very thorough and systematic, and the treatment of the problem of the analysis of mixtures of various sugars is much in advance of what is found in much more pretentious works. There are, however, one or two statements of a misleading or otherwise objectionable character to which attention should be drawn. The Clerget formula (for an angular degree instrument) is given as $S = \frac{100\ D}{95 - \frac{t}{3}}$ where D is stated to be the "angular deviation" of 1 dcm. of the solution after inversion. The expression of the algebraical sum of the readings before and after inversion by the single symbol D, coupled with the ambiguous term "angular deviation," might easily mislead the unwary, although the matter is more clearly stated on a later page. The verbal epicure would also be shocked by the use of the term "negative rotation" to signify lævo-rotation, and of the objectionable word "opticity" for rotatory power.

The statement that in the manufacture of beer the malt is digested with water for two or three hours at 30° C. is not in accordance with facts, as the temperature usually ranges between 61° and 68° C.

In the case of tea and pepper the student is told to determine the fibre by acid treatment alone; under Cocoa the fibre is not mentioned at all.

In dealing with the Marsh-Berzelius process no precautions are recommended to secure the sensitiveness of the zinc. It is to be regretted that no reference is made to the much simpler and now official Gutzeit process.

The illustrations are excellent and to the point, but the photomicrographs of starches, condiments, etc., suffer from an unnecessarily high magnification, and, as is generally the case, hardly justify their existence and are quite unnecessary if, as should be the case, authentic specimens of the substances are examined.

There are very few misprints, the arrangement and general style of the volume is excellent, and taken as a whole the work is a credit to authors and publisher alike.

C. H. Cribb.

Dairy Chemistry: A Practical Handbook for Dairy Chemists and Others having Control of Dairies. By Henry Droop Richmond, F.I.C. Third Edition, Revised and Reset. London: Charles Griffin and Co., Ltd. 1920. Price 25s.

The last edition of this standard work was published in 1915, and the appearance of a new edition in such a comparatively short period as five years indicates

the value of the book. The general plan of the book remains the same, but advantage has been taken of the present revision to collect scattered items into the part allocated to the particular subject and to rearrange other details in a more useful order.

The book is now divided into three parts dealing respectively with the Constituents of Milk, the Analysis of Milk and Milk Products, and Technical Applications. Each of these parts is full of information, the latest researches have been included, and new work appearing while the book was in the press is given in addenda.

An improvement, which adds greatly to convenience in using the book, is the elimination of the cumbersome folding tables, these being now incorporated in the text.

One or two of the illustrations are poor, particularly those showing the lactometer (p. 75) and butter and margarine under polarised light (p. 270), and those who are desirous of seeking further information on any point will possibly regret that all references to literature are omitted from the text.

Mr. Richmond is to be congratulated in that he has improved a book which had already a world-wide reputation as a standard. In addition to dairy chemists, food analysts in general will find the book to be a valuable addition to their libraries.

W. P. SKERTCHLY.

RAPID METHODS FOR THE CHEMICAL ANALYSIS OF SPECIAL STEELS, STEEL-MAKING ALLOYS, THEIR ORES, AND GRAPHITES. By C. M. JOHNSON. Third Edition. Pp. xi + 552. New York: John Wiley and Sons; London: Chapman and Hall. 1920. Price 36s. net.

The first edition, numbering 221 pages, was reviewed by L. Archbutt in the ANALYST of 1909, p. 252; in the present work the increase of the text matter over the second edition represents more than 100 pages. The processes described for testing special steels and steel-making alloys are the result of the practical experience of a steel works chemist; as such they should prove a valuable guide to other analysts working in this field, and a careful perusal of the book will disclose a number of helpful suggestions. The same cannot always be said of the processes recommended for analysing ores of the elements which enter into the composition of special steels. In this class of work many of the methods used by the author are laboursome, involved, and anything but "rapid." To take one example, that of wolframite: the decomposition by strong hydrochloric acid of the ore "ground to the finest flour" requires far less than six hours, whilst the use of a double filter and addition of "a three-quarter inch ball of filter pulp" to the tungstic acid "to hasten filtration and secure a perfect washing" are not only unnecessary, but even detrimental in this case. The excess of filter pulp containing the tungstic acid from 1 grm. of ore must be given "not less than sixty washings" to remove the potassium salts derived from the chlorate, which is used as an oxidiser. Nitric acid would answer just as well without introducing any fixed constituents.

The author next commits what the reviewer regards as an error of judgment in igniting the impure precipitate and weighing it "as WO_3 plus some Fe_2O_3, Al_2O_3, SnO_2, Mn_3O_4, CaO, Ta_2O_5, Nb_2O_5, MoO_3, CuO, and all of the SiO_2," and then

evaporating with hydrofluoric acid to eliminate silica even if it reaches "from 30 to 60 per cent., as it often does in unconcentrated ores and slimes"; incidentally, the *aqua regia* method does not give good results with low-grade ores. Considering that the bulk of such silica is usually present in the form of quartz, which is not easily attacked and volatilised, it would be far wiser to eliminate it before ignition—*e.g.*, by dissolving the tungstic acid in ammonia. The tungstic acid is then weighed, fused, and submitted to a series of manipulations the object of which is to extract and weigh the impurities, tungstic acid being found by difference. Removal of the impurities followed by direct weighing of the purified tungstic acid appeals to the writer as being more rapid and reliable. For the quick and accurate estimation of tin in tungsten ores, the author would be well advised to refer to and adopt Powell's excellent method (*J. Soc. Chem. Ind.*, 1918, **37**, 285T). Arsenic is determined in tungsten ores by weighing as pentasulphide; a volumetric method would be preferable for speed and accuracy.

On p. 197 iron is titrated in presence of titanium after reduction with zinc, though on p. 52 this is rightly stated to be inadmissible. In the analysis of crude zirconia the precipitate obtained by sodium phosphate and washed "at least sixty times with water" is described as "pure phosphate of zirconium." This can hardly be the case unless zirconium is precipitated by ammonium phosphate from a solution free from alkalies, and the precipitate washed with ammonium nitrate solution; on ignition it yields pyrophosphate, ZrO_2 factor 0·4632 (Lundell and Knowles, *J. Amer. Chem. Soc.*, 1919, **41**, 1801). A general criticism must be directed against the author's habit of washing a precipitate forty, sixty, or even eighty times.

The book is characterised by lack of systematic arrangement and a wealth of minute instructions: the more or less detailed description of the precipitation of molybdenum sulphide occurs more than ten times. In every case the sulphide is roasted to trioxide, which is weighed, whilst the more reliable estimation as lead molybdate is described once as a check method.

Misprints and errors in the text are not infrequent; the author's style is often careless and distinctly Transatlantic—*e.g.*, "the crucible is attacked some by the niter" (p. 327). The following quaint direction is worth remembering in case of a sudden shortage of litmus paper: "Wash . . . until the outside of the filter has no sour taste, especially along the double thickness" (p. 319).

The index is rather poor, the references under each letter not being arranged alphabetically.

In short, the book contains much that is of value, but in a crude state. It would be greatly improved by extensive excision and careful revision.

W. R. SCHOELLER.

RECOVERY OF NITRATE FROM CHILEAN CALICHE. By A. W. ALLEN. Pp. xvi + 50. London: Charles Griffin and Co., Ltd. Price 6s. net.

In a preface the author points out that, although the Shanks system of caliche treatment has effected notable economies in the Chilean nitrate industry, its inefficiency is manifesting itself more and more as the richer deposits become exhausted, since it cannot be successfully applied for the recovery of nitrate from low grade caliche

and waste products (ripio). Estimates of the latter in numerous dumps vary between 100,000,000 and 150,000,000 tons, whilst the quantity of the former probably exceeds a billion tons. The fact is emphasised that only by successful and economical treatment of these at present valueless products can the nitrate industry be placed on a secure footing and enabled to compete with the steadily progressing synthetic processes for production of nitrogen compounds; nevertheless, the present position of the latter industry tends to negative the author's implied denial on p. 19 of its commercial feasibility. In the first part of this volume the Shanks process is described; its defects are said to be: high loss of nitrate in ripio and borra, high cost of operation and heavy overhead charges, excessive labour requirements, and inaccurate accounting of yields. The latter part of the book deals with the new Allen extraction process, which is briefly described. Its advantages are: even percolation on account of homogeneity of the charge, improved and shorter treatment, better displacement and more rapid solution, possibility of recovery of nitrate from waste products, and an increased yield up to 90 to 95 per cent. Alternative methods for evaporation and crystallisation are also discussed. The information is clearly conveyed and a glossary of Spanish terms forms a useful guide, but the index might, with advantage, be reconstructed. The book should serve to call fresh attention to the imperative necessity for improved methods in treating caliche, whilst, at the same time, the lines along which development should proceed are indicated.

W. J. WRIGHT.

A DICTIONARY OF APPLIED CHEMISTRY. Volume I.: A to Calcium, by SIR EDWARD THORPE, C.B., F.R.S., assisted by eminent contributors. New and revised edition, 1921. London: Longmans, Green and Co. Price 60s. net.

It is clearly impossible for a reviewer to comment adequately on all the sections of such a comprehensive and authoritative treatise as Thorpe's "Dictionary of Applied Chemistry." The names of the various writers and the established reputation of the book are sufficient guarantee of its general excellence.

The new edition is a distinct improvement on the last (1912) edition, and contains so much new matter that it is proposed to issue six or seven volumes instead of five; the scale of the revision and enlargement may be judged by the expansion of A to Calcium from 614 to 752 pages. Several articles have disappeared or been transferred to other headings in later volumes and a number of new ones included; the whole is brought up to date so far as is possible in so large a work. References to literature are up to 1919, and atomic weights are those of 1920. Paper and binding are of similar good quality to the 1912 edition.

The article on Acetic Acid and Vinegar manufacture has been rewritten by C. A. Mitchell, and indicates a notable advance in this industry. A few obsolete passages, however, have escaped excision; thus on page 21 it is remarked that perry and crab-apple vinegar are used in Wales and Monmouthshire; no evidence of this has been observed by the writers in recent years.

Under Acetone details of the new catalytic processes are not given for reasons, stated in the Preface, of inexpediency.

Some of the articles are not so up to date as others. The section on Acidimetry appears to the reviewers to be lacking in this respect; references are only to 1915 and the portion dealing with indicators does not give their [H·] ranges and exponents. A table of the [H.] values at which the colour changes would be most useful, much more so than the rather old-fashioned table on page 59. The bibliography under Indicators should certainly include Prideaux's excellent book on the theory and use of indicators; similarly the general references (p. 64) should be more recent and include the 1911 edition of "Sutton."

Under Alkaloids a new article appears by Barger incorporating part of the article on Vegeto-Alkaloids of the 1912 edition with a general account of this group of substances. The article on Aluminium has been extended, but it is singular that no mention is made of the phosphate method of estimation, although the hydroxide precipitation is described. On page 171 there occurs a reproduction of the 1912 edition of the misspelling of Le Chatelier's name.

The section on Analysis, like that on Acidimetry, is not sufficiently revised; references are to old editions of well-known textbooks. Mellor's textbook of quantitative analysis is not included in the bibliography. Under Assaying three figures of commonplace implements have been deleted and more valuable information inserted, but the section on the assaying of coal is so sketchy as to be of little value; the determination of calorific power by mixture with potassium chlorate and nitrate in Thompson's calorimeter is obsolete; this subject might well be left for adequate treatment in a later volume under Fuel.

The subject of radioactive constituents of the atmosphere has progressed considerably of recent years, so that the article containing only references to 1909 is not quite worthy of a 1921 edition.

There is a valuable and excellent fourteen-page addendum to the article on Balance, dealing with highly refined weighing and discussing all factors affecting this rather difficult matter; various types of micro-balance are clearly explained.

It may surprise many readers to find an account of the solution usually known as Fehling's described and discussed in detail under Barreswil's name. However accurate it may be to ascribe this solution to Barreswil, it is so long associated with Fehling's name that such change is rather startling.

The section on Boiler Incrustations, by Brame, is new and that on Brewing, by Stern, is much condensed, so that we now have a short and concise article which is highly desirable for a subject which for adequate treatment requires several volumes of its own. The numerous references to literature will enable a reader readily to obtain supplementary information on any desired point.

The work as a whole is beyond criticism, and the new edition deserves a place on the bookshelf of every technical chemist; there are remarkably few typographical errors, and great care has evidently been taken by the Editor. Of the many references turned up by the reviewers, taken at random, not one was found incorrect.

G. R. Thompson.
H. E. Cox.

TECHNICAL CHEMISTS' POCKET BOOK. By ROBERT ENSOLL. London: E. and F. N. Spon, Ltd. Price 8s. 6d. net.

The one hundred and ninety pages of this book contain an immense amount of material useful to technical chemists, metallurgists, and chemical engineers, the aim of the author being to produce a complementary volume to the well-known "Bayley's Chemists Pocket-Book." The greater part of the information is given in tabular form and the scope of the work may be judged from the following selection of subjects taken from the contents: Weights, areas, etc., calibration of storage tanks, combustion data, furnace construction, fuel consumption, boilers, properties of metals, strength of materials, alloys, thermal and electrical conductivities, specific gravities of solutions, metals, solids, etc., compression of gases, fuse wires, injurious effects of various gases, and typical analyses.

The tables giving the strength of alcoholic solutions from the specific gravities do not show a greater alcoholic strength than 42·18 per cent. by weight, and would be of enhanced value if they were extended to include stronger alcoholic solutions.

References to literature are up to date and but few errors have been noticed in the text. The name "Inchley" at the bottom of page 63 should be "Hinchley"; on page 124 the expression "< 99" under the heading of tin should be "> 99."

The book will be found extremely useful for reference, whether it is carried in the pocket or used on the table or bench, but it is a pity that the paper is not of better quality, as this affects the clearness of the printing.

W. P. SKERTCHLY.

MINISTRY OF HEALTH.

REPORT OF THE DEPARTMENTAL COMMITTEE ON THE CONTROL OF CERTAIN THERAPEUTIC SUBSTANCES, 1921 (CMD. 1156).

THE substances included in the terms of reference were classified into three groups: (*A*) Bodies described in the U.S. Regulations of 1919 as "biologic products," —*i.e.*, vaccines, sera, toxins, antitoxins, and analogous products. (*B*) Synthetic remedies, such as salvarsan and its analogues. (*C*) Substances corresponding more nearly with the popular definition of ordinary "drugs"—*e.g.*, preparations of digitalis, strophanthus, squill, ergot, cannabis indica, pituitary gland, etc.

An account is given of the present position as regards standardisation and control of substances which cannot be adequately tested by direct chemical means, together with an outline of the systems of control adopted in U.S.A. and Germany.

Evidence was given before the Committee by scientific authorities and representatives of commercial and manufacturing interests, and the following is a summary of the principal recommendations based upon this evidence:

(*a*) That therapeutic substances which cannot be tested adequately by chemical means should be subject to supervision and control.

(*b*) That the Controlling Authority should be the Committee of the Privy Council which was appointed by an Order in Council dated 11th March, 1920, under powers conferred by the Ministry of Health Act, 1919.

(*c*) That the Controlling Authority should be assisted by an Advisory Committee which might consist of members nominated by the Minister of Health, the Secretary for Scotland, the Chief Secretary for Ireland, the Naval and Military Authorities, the General Medical Council, the Medical Research Council, and the Pharmaceutical Society.

(*d*) That there should be a Central Laboratory under Government control, wherein standards would be prepared and maintained, research carried out in connection therewith, and tests made to ascertain that the products issued by manufacturers conform with the standards laid down. The Medical Research Council, who already possess the requisite organisation, should be responsible for this Central Laboratory.

(*e*) That the method of control should include the licensing of manufacturers, inspection of their plant, premises, and processes, and the testing of the finished products.

(*f*) That, with certain exceptions, testing by the Central Laboratory should be confined to samples taken from makers' stocks or bought in the open market, leaving to the manufacturers the primary responsibility for securing that the products conform with the prescribed standards and tests. That power should, however, be taken to require a manufacturer to submit for central testing, for a stated period, samples of every batch of a substance made.

(*g*) That power should be taken to inspect all premises where the substances are made and the several processes of their manufacture, but that in the case of salvarsan and its analogues (Group *B*) and of the galenical and other preparations which we have classified as Group *C*, inspection should ordinarily be confined to the records and methods of biological testing, and, when necessary, to the filling and sealing of the containers.

(*h*) That products made abroad should be subject to restrictions similar to those applying to products made in the United Kingdom, and that licences should be granted to approved manufacturers based upon a preliminary inspection of plant, premises and processes, and testing of samples. In addition, steps should be taken to ensure that each consignment attains the standards laid down for similar products of home manufacture.