

THE ANALYST.

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

NOTES ON RUBBER ANALYSIS.

By A. R. PEARSON, B.Sc., F.I.C., LL.B.

THE following notes may be of use to chemists interested in the chemical testing of manufactured rubber goods :

1. *Determination of Total Sulphur.*—Numerous methods and modifications of methods have been used for determining total sulphur in rubber, and the very multiplicity of suggestions shows that the ideal is still to seek. An elaborate review of these methods is given by Tuttle and Isaacs in the *Journal of Industrial and Engineering Chemistry* (1915, VII., 658), and it is not proposed to criticise them in detail here.

The following modification of the nitric acid method has, after some years' experience, proved accurate and easy to carry out, and it requires no special apparatus, although it must be admitted that it leaves something to be desired as regards rapidity.

Twenty c.c. of fuming nitric acid (sp. gr. 1.5) are placed in a wide-necked flask of about 150 c.c. capacity, and exactly half a gram of the sample is dropped into the acid bit by bit, in pieces small enough to dissolve without fuming or inflaming. The flask is now closed with a small funnel and warmed by slow degrees, so as to avoid violent action and frothing. Finally, it is made quite hot on a water-bath or hot plate, and kept so for half-an-hour. At this stage the sulphur has probably all been oxidised to sulphuric acid, but it cannot be directly precipitated owing to the presence of complex nitro-compounds, which make filtration impossible. It remains, therefore, to get rid of the organic matter, and this can be conveniently done by means of potassium permanganate. The solid permanganate is added in small portions so as not to have the reaction too violent, and until some oxide of manganese remains unreduced after about an hour's heating. At the end of that time 20 c.c. of concentrated hydrochloric acid are added, and the liquid is again heated. When action has ceased, the solution is evaporated to dryness in a porcelain dish ; the residue is again treated with hydrochloric acid, and taken to dryness. The

mixed salts are now taken up in hot dilute hydrochloric acid, the solution is filtered and sulphates in it determined as barium sulphate.

The precipitated barium sulphate sometimes carries down a little manganese, but this can be removed by boiling the precipitate for a short time with dilute hydrochloric acid before washing.

All the reagents required for these operations are to be obtained practically free from sulphates; but it is advisable to carry out a blank experiment with every batch of estimations.

It is to be remarked that imperfect sampling is responsible for many errors and discrepancies in rubber analysis, and especially so in the determination of sulphur; for it is well known that sulphur has a tendency to accumulate near the surface of a vulcanised article. For example, in one case raspings from the surface of a sample whose average sulphur content was 2.5 per cent., yielded 5.4 per cent. of sulphur. Consequently the portion taken for a representative sample must consist of the whole of a section or sections right through the sheet or other article under examination. Rasping on a coarse carborundum wheel is a rapid and effective way of obtaining such a sample in a form suitable for analysis. Provided the sample is quite uniform and representative, duplicate determinations of sulphur by the above method never differ by more than 0.05 per cent.

2. *Determination of Carbonates in Rubber Mixings.*—Chalk and magnesium carbonate are frequently used as fillers in rubber manufacture, and it is therefore useful to know the percentage of carbonic acid in such a mixing.

An examination of the ash is useless for this purpose. The usual method is to dissolve away the rubber hydrocarbon in solvents such as nitrobenzene and examine the residue; but this is a tedious and not very reliable process. It is clearly preferable to decompose any such carbonates *in situ* by treating the rubber with some suitable acid. The aqueous mineral acids do not penetrate rubber, but acetic acid is sufficiently miscible with rubber to penetrate it completely either before or after vulcanisation, and so to decompose any carbonates which may be present. The carbon dioxide may then be purified, absorbed in soda lime, and weighed.

A convenient form of reaction-flask for this process is shown in the figure. Its capacity is about 100 c.c. A tap-funnel is ground into the neck, and a water-cooled reflux condenser is sealed to the side-tube. The absorption train which is connected at O consists of:

(1) A U-tube filled with solid neutral lead acetate, to absorb sulphuretted hydrogen.

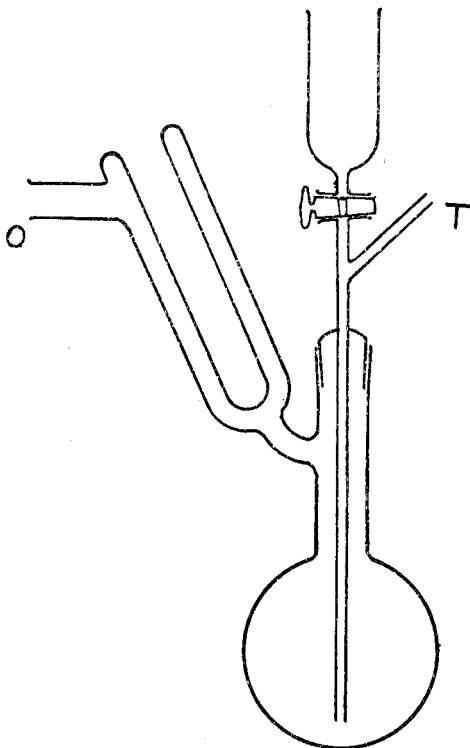
(2) A U-tube of which the first half contains sodium acetate which has been previously fused and powdered, and the second half contains calcium chloride. This arrests acid vapours and dries the gas. The purifying materials must of course be saturated with carbon dioxide before use.

(3) Two weighed tubes containing soda-lime and calcium chloride, to absorb the carbon dioxide.

One gram of the comminuted sample is introduced into the flask, and glacial acetic acid (25 c.c.) is run in by the tap-funnel. The mixture is heated so that it barely simmers, while a slow current of air, freed from carbon dioxide, is drawn

through the apparatus by way of the inlet tube T. The length of time required depends on the state of division of the sample. With raspings of the ordinary degree of fineness, and with a current of air passing at the rate of two bubbles per second, a treatment of one and a half hours is sufficient. The foot of the inlet tube should be protected by a small sheath-tube, otherwise it may become blocked by matter deposited from solution.

In calculating results it should be remembered that commercial magnesium car-



bonate is a basic carbonate of rather variable composition. The literature contains a number of suggested formulæ for it, and it generally has the approximate composition :

Magnesium oxide	42 per cent.
Carbon dioxide	34-35 "
Water of hydration	22-25 "

Since it is seldom possible to examine a sample of the carbonate used in a given mixing, some average factor must be employed to calculate, from the percentage of carbon dioxide found, the corresponding percentages of basic carbonate and of magnesium oxide: these two empirical factors are suggested :

$$\begin{aligned} [\text{CO}_2] \times 2.8 &= \text{Basic carbonate.} \\ [\text{CO}_2] \times 1.22 &= \text{Magnesia present in the form of carbonate.} \end{aligned}$$

3. *Determination of Sulphides in Rubber Mixings.*—The sulphides chiefly used as fillers are those of zinc and antimony. Lead sulphide also is sometimes found, for, although it is not often added as such, it is always formed to a greater or less extent during the vulcanisation of mixings containing litharge.

The coefficient of vulcanisation is a most important figure in rubber analysis, and as the sulphur of vulcanisation is usually determined by difference, it is necessary to know how much of the total sulphur is present in the form of sulphides.

As in the case of carbonates, a reagent is required which will penetrate the rubber and decompose these sulphides. Stevens (*ANALYST*, 1915, 40, 275) uses hydrochloric acid in a medium of benzene or ether, and determines iodimetrically the sulphuretted hydrogen which is evolved. Another method which has been found useful, where a considerable number of analyses is to be done, consists in decomposing the sulphides with a mixture of acetic and hydrochloric acids and absorbing the sulphuretted hydrogen by solid copper sulphate, so that it can be weighed directly.

The reaction is carried out in a flask similar to that described above. After the sample has been introduced, nitrogen from a cylinder fitted with reducing valve is passed through the flask until all air is displaced. Then the absorption train is connected, and a mixture of acetic acid (15 c.c.), concentrated hydrochloric acid (5 c.c.), and water (5 c.c.), is run in by the tap-funnel. The absorption train consists of:

1. A tube packed with sodium acetate and calcium chloride as described above. This should be saturated with sulphuretted hydrogen, and then filled with nitrogen before use. The same tube should not be used for determination of carbonates and of sulphides.

2. A weighed U-tube for absorbing the sulphuretted hydrogen. This is filled, as to its first two-thirds, with copper sulphate in fine crystals which has been dried at 105° C. ($\text{CuSO}_4, \text{H}_2\text{O}$), and then, as to its last third, with calcium chloride. The air in this tube must be displaced by nitrogen before weighing.

The operation is carried out in the same way as the determination of carbonates above. The following points are worthy of remark:

1. Air must be excluded as far as possible, because the copper sulphide absorbs oxygen slowly. It is inevitable that a little air should remain in the connections of the apparatus, and even this small amount causes results to become slightly higher when the sulphide accumulates from repeated experiments. This is shown in the following series of test experiments, in which a mixture of zinc sulphide and zinc oxide was used:

Experiment.	Weight of Mixture.	Weight of H_2S found.	Percentage of H_2S .	Percentage of ZnS .
1	0.4866 gm.	0.0784 gm.	16.11	46.07
2	0.2392 "	0.0386 "	16.14	46.13
3	0.4022 "	0.0654 "	16.26	46.49
	Actual percentages:	16.09	46.00

It is advisable, therefore, to carry out a blank experiment from time to time, and to make a corresponding deduction if any increase in weight is observed.

2. A nitrogen atmosphere is preferable to one of carbon dioxide, because the sodium acetate and calcium chloride always contain traces of basic compounds, and variable equilibria might be set up, such as : $\text{Na}_2\text{CO}_3 + \text{H}_2\text{S} \rightleftharpoons \text{Na}_2\text{S} + \text{H}_2\text{O} + \text{CO}_2$.

3. None of the combined sulphur is expelled by this treatment, for soft vulcanised rubber containing practically no mineral matter yields no sulphuretted hydrogen.

4. Commercial zinc sulphide may contain notable quantities of zinc oxide, free sulphur, and other substances. Commercial antimony sulphide is also of variable composition : it may contain trisulphide, pentasulphide, and basic compounds of antimony, together with free sulphur and compounds of lime and alkalis. Of course, only that sulphur corresponding to the trisulphide is estimated by the above method ; but it is precisely this sulphur which is not available for the reaction of vulcanisation.

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AN IMPROVEMENT IN THE HALDANE GENERAL AIR ANALYSIS APPARATUS.

BY ROBERT C. FREDERICK.

FOR general air analysis the writer considers the Haldane apparatus to have no superior, and in an extensive experience of its use a number of minor improvements in arrangement and manipulation have suggested themselves. Some of these have been referred to elsewhere,* and this note concerns a small modification which, gives distinct practical advantages.

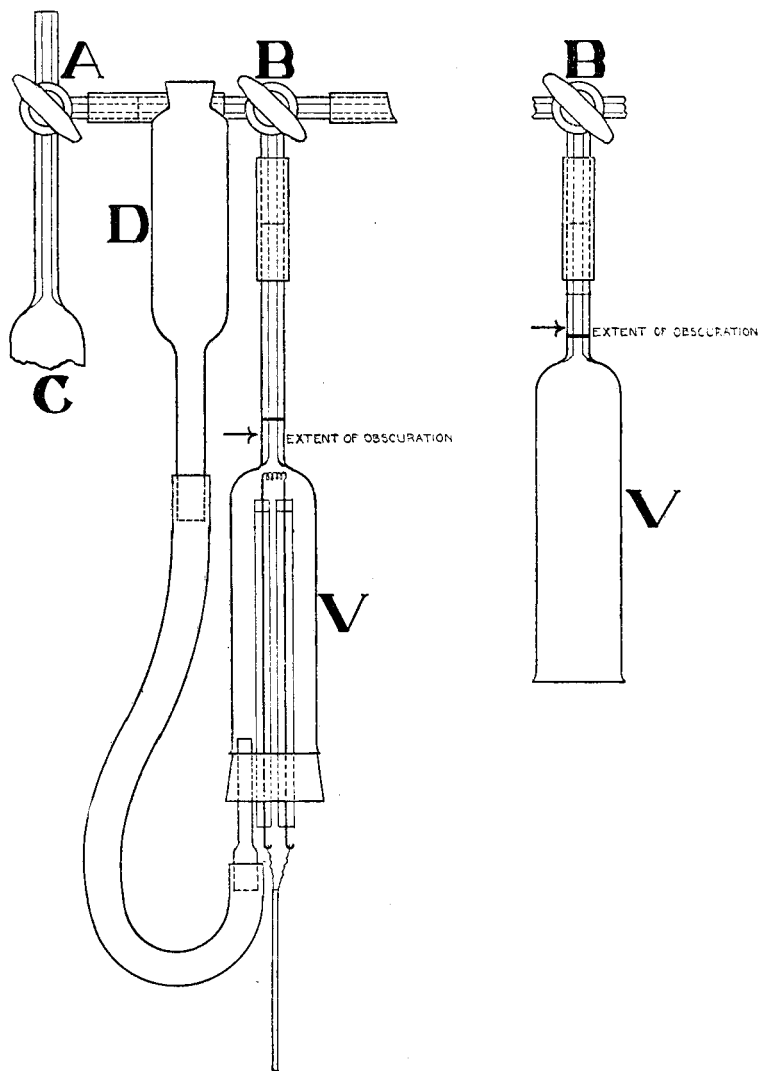
In brief, this improvement consists of using a combustion chamber V in which the wide part is $\frac{3}{8}$ inch shorter in length, the stem $1\frac{1}{8}$ inches longer, and the mark on the stem is $\frac{1}{2}$ inch from the shoulder ($\frac{3}{8}$ inch higher) ; the fitting of this does not necessitate any rearrangement of the other parts.

The object is twofold. In practice it is found that the lower part of the stem of the combustion chamber rapidly becomes obscured by the combustions, and in the original this obscuration extends over the graduation, so that after very little use the part has to be dismantled and cleaned ; with V modified, as shown, the obscuration remains well below the graduation, and this frequent cleaning is not required.

Again, in using this apparatus for examination of samples contained in bottles, these are opened under mercury, and the air is transferred by means of a bent tube with a three-way stopcock. In this manipulation a globule of mercury is almost invariably retained in the stopcock A of the apparatus and must be removed. The writer does this by turning A open from C (air burette) to B only, and B open from V towards A only, and then, by gently pressing the two limbs of the rubber tube connecting D and V against each, displacing the globule. In doing this with the

* " Public Health Chemical Analysis " (Constable and Co.).

original apparatus it frequently happens that the mercury is pushed up in the stem of V until it reaches the stopcock B, without freeing the globule from the narrow



Scale, one half.

tube below A ; the increased length of the stem of V enables the displacement to be made without difficulty.

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NOTES.

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

SOUR MILK SAMPLES AND GERBER'S FAT METHOD.

I HAVE recently had occasion to re-test, after a lapse of several weeks, a number of milk samples for fat by Gerber's method. These had been "preserved" by the addition of about 0.05 per cent. of formalin, which of course is not effectual for more than a few days, and the samples were very sour and decomposed. About 2 per cent. of strong ammonia was added to make pipetting possible, and, after correction for this, the results were up to 0.2 per cent. higher than those of the original tests. As would be expected, the ammonia is not responsible for this, as eight tests on five different milks averaged—

With ammonia	3.67
With ammonia, corrected	3.74
Without ammonia	3.76

The possibility of the formation of amyl esters suggested itself, and a sample of milk was divided into several parts, to which acetic, lactic, and butyric acids in known amounts were added. To make all comparable, 0.05 per cent. of formalin was also added, and 2 per cent. strong ammonia to neutralise the acidity. The following table shows the apparent increase in fat over the control, also the apparent increase in the case of several sour samples tested at the same time.

	Total Acid (as Lactic Acid).	Approximate Increase in Fat per Cent.
Milk + 0.5 per cent. acetic acid	0.57	0.10
" + 0.5 " butyric " 	0.42	0.32
" + 1.0 " " " 	0.63	0.57
" + 0.5 " lactic " 	0.70	0.05
" + 1.0 " " " 	1.20	Nil
" + { 0.17 " acetic " }	0.57	0.12
{ 0.17 " butyric " }		
{ 0.17 " lactic " }		
" + { 0.33 " butyric " }	0.80	0.19
{ 0.33 " lactic " }		
Milk 3 days old, no smell of butyric acid	0.72	-0.06
" 1 week old, " " " 	1.02	0.18
" 3 weeks old, smell of butyric acid	1.12	0.12
" 5 " " " " 	2.10	0.19

The results in the case of the artificially soured samples were corrected for the volume of acid added, and those of the naturally sour samples for the ammonia. The fat separated from the most acid samples had a distinct odour of amyl esters.

It is evident that the use of Gerber's method for sour milk is apt to give very

misleading results, owing to the formation of amyl esters of the lower aliphatic acids. Lactic acid appears to have no effect, acetic acid little, but butyric acid a large effect. I was unable to try the effect of propionic acid, but one would expect it to be intermediate between that of acetic and butyric acids, in which case it would be an important source of error.

F. E. DAY.

LANSDOWNE, LIMERICK.

THE IDENTIFICATION OF ACIDS BY PHENACYL BROMIDE.

A useful method for the identification of organic acids by means of the m.-pt. of their phenacyl esters has been described by Rather and Reid (*J. Amer. Chem. Soc.*, 1919, **41**, 75) and abstracted in the *ANALYST* (1919, **44**, 103).

The method depends upon the use of ω -bromoacetophenone for the esterification of the acids; and the writer thinks it worth while to call attention to the fact—learned by unpleasant experience—that special care should be taken in preparing and using this substance and its solution. Even small quantities produce very painful blisters on the skin if allowed to come in contact therewith. The substance also has a very irritating effect upon the eyes.

H. E. COX.

NEWPORT, MON.

MASKING THE PRESENCE OF FREE SALICYLIC ACID IN ACETYL SALICYLIC ACID.

The writer, (*Pharm. J.*, 1920, **51**, 90), in a paper describing a new method for the estimation of free acetic acid in acetyl salicylic acid, also drew attention to the masking of the free salicylic acid present in aspirin tablets when tested for with ferric chloride test solution. The substance causing this was tartaric acid, some colourless non-ionised salt of iron being formed, there being no free ferric ions available for the formation of the violet-coloured ferric salicylate. Certain other strong acids will produce the same phenomenon, particularly citric acid (1.0 per cent. of the latter being sufficient to mask the presence of about 0.2 per cent. of free salicylic acid).

Tartaric acid, citric acid, etc., when present, can readily be detected after removal of the salicylic acid, acetyl salicylic acid, etc., from a cold aqueous extract of the tablet, and, conversely, the free salicylic acid can be estimated colorimetrically, after separation by means of some suitable solvent, such as ether, chloroform, etc., which does not extract the masking substance; when either tartaric or citric acid is present, a mixture of equal parts of ether and petroleum ether is a better solvent than ether alone, since both acids are slightly soluble in ether. All samples of aspirin tablets, therefore, which, after being powdered, give no violet coloration with ferric chloride test solution, should be viewed with suspicion.

A. NUTTER SMITH.

MESSRS. BOOTS' PURE DRUG CO., LTD.
NOTTINGHAM.

THE TREATMENT OF RED LITMUS PAPER.

Owing to the sizing of the original paper with rosin (colophony), it is frequently found that red bibulous litmus papers are not readily wetted by aqueous solutions, and that the colour indication is indefinite and slow in appearance.

This trouble may be obviated by the following procedure: A number of litmus books are rolled into a double layer of filter paper and inserted, backs downwards, in a Soxhlet extractor. They are then covered with 85 per cent. (or stronger) alcohol,

containing 1 per cent. of concentrated hydrochloric acid, and allowed to soak overnight, when a further quantity of acid alcohol is added, and extraction carried through in the usual manner, until no more red colouring matter is removed. The books are then squeezed dry in a cloth, and finally dried in an oven at 100° C. The alcohol removes, in addition to the rosin, some of the colour from the litmus and some from the red dye of the bookbinding.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Detection and Identification of Dextrose in Vegetables by a Biochemical Method. E. Bourquelot and Bridel. (*J. Pharm. Chim.*, 1920, **22**, 209-215.)—The method depends on the formation of methyl glucoside when dextrose in methyl alcohol solution is treated with emulsin. For instance, a solution containing 2.0565 grms. of dextrose per 100 c.c. of 70 per cent. methyl alcohol (by weight) showed a rotation of +2° 20' in a 200 mm. tube; this solution was then treated with 0.5 gm. of emulsin, and placed aside at the ordinary temperature. The rotation decreased gradually (during some days) to a final value of -50', and 82.5 per cent. of the dextrose had combined with the alcohol. Experiments with mannose, lævulose, and arabinose showed that these sugars do not combine with methyl alcohol. The methyl glucoside may be separated by evaporating the alcoholic solution under reduced pressure, and extracting the residue with boiling ethyl acetate; crystals of the glucoside form as the solution cools. The method may be applied to the detection of dextrose in the reducing sugars isolated from plants and vegetables; the author finds that the reducing sugars from juniper berries contain 15 per cent. of their weight of dextrose.

W. P. S.

Estimation of Methyl Alcohol in Cacao Nibs and Husks. Von Koers. (*Pharm. Weekbl.*, 1920, **57**, 924; through *Chem. Zeit. Rep.*, 1920, **44**, 234.)—Von Fellenberg proposed to estimate the amount of adulteration in cacao by determining the methyl alcohol content after liberation by pectin. The author finds the same values as Von Fellenberg for nibs, but lower ones for husks and for certain cocoas on the market; hence the estimation of husk by this method appears impracticable.

O. E. M.

Hehner Value of Fats and Oils. Goldschmidt. (*Zeitsch. Öl-Fett-Ind.*, 1920, **40**, 406; through *Chem. Zeit. Rep.*, 1920, **44**, 244.)—The following procedure is recommended for the determination of this value. Five grms. of the sample are saponified, unsaponifiable matters are extracted from the soap solution in the usual way, and the solution is then acidified and the fatty acids extracted with ether. The ethereal solution is dried for two hours over 10 grms. of anhydrous sodium sulphate, filtered

into a weighed flask, and the sodium sulphate and filter are washed with ether (also dried over sodium sulphate). The greater part of the ether is removed by distillation, and the residue of fatty acids is dried at 50° to 60° C. in a current of air.

W. P. S.

Estimation of Oxy-Fatty Acids in Extracted Olive Oils. Stadlinger. (*Zeitsch. Öl-Fett-Ind.*, 1920, 40, 437; through *Chem. Zeit. Rep.*, 1920, 44, 244.)—A grade of oil obtained by extracting olive residues with carbon bisulphide is particularly rich in oxy-fatty acids, the average quantity of the latter being 6 per cent. To estimate the amount of oxy-fatty acids, the total fatty acids obtained from the oil are melted, treated with petroleum spirit (b.-pt. below 50° C.); the insoluble oxy-fatty acids are collected on a filter, washed with petroleum spirit, dissolved in warm alcohol (or a mixture of alcohol and chloroform), the solution evaporated, and the residue of oxy-fatty acids weighed.

W. P. S.

Use of the Refractometer in Ascertaining the Purity of Certain Refined Edible Oils. F. H. Trim. (*J. Soc. Chem. Ind.*, 1920, 39, 307-308 r.)—The melting-point, determined under standard conditions, will afford an indication of the amounts of constituents in dual mixtures, such as palm-kernel oil or coconut oil with each other or with arachis oil. In the case of mixtures of all three fats another constant is also required, and for this purpose the refractive index is the most useful. Standard samples should be prepared from the original material by extraction or expression, and the crude oils so obtained treated by laboratory methods similar to those used in the works. The following results were thus obtained with refined edible oils :

Oil.	Standard [n] _D 40° C.	Variation.	Melting-Point ° C.	Free Fatty Acids (as Lauric), per Cent.
Arachis ...	1.46235	± 0.00020	- 8.0	0.05
Palm-kernel ...	1.45097	± 0.00020	28.35	0.04
Coconut ...	1.44897	± 0.00010	24.85	0.03

Coconut oil showed the least variation in refractive index. The following results were given by refined oils from four different copras : Ceylon, 1.44887 ; Straits, 1.44902 ; Singapore, 1.44887 ; and Malabar, 1.44897. The melting-points and refractive indices of dual mixtures of pure arachis, palm-kernel, and coconut oil were plotted in graphs against the percentage compositions. The results showed that the refractive index was directly proportional to the percentage composition, and that the addition of 10 per cent. of arachis oil lowered the melting-point by only 0.4° C. If the refractive indices are plotted as ordinates and the melting-points as abscissæ a closed graph is obtained. This may be divided into sections, and any particular section used for the estimation of the constituents of either dual or triple mixtures. Allowances must be made, however, for natural variations in the natural products having an influence on the constants of the refined material.

C. A. M.

New Method of Estimating Quinine. C. Bamberger. (*Pharm. Zentralb.*, 1920, 61, 257-259; through *Chem. Zeit.*, 1920, 44, *Rep.*, 223.)—Two and a half grms. of cinchona bark are heated for ten minutes over the water-bath with 2 c.c. of hydrochloric acid and 20 c.c. of water, and the mixture then cooled, shaken with 25 grms. of chloroform and 50 grms. of ether, and treated with 5 grms. of sodium hydroxide solution. The contents of the flask are vigorously shaken (about 300 times) within two to three minutes, and treated with sufficient plaster of Paris (about 40 to 50 grms.) to clarify the liquid, and 60 grms. of the clear chloroform-ether layer transferred to a separating funnel. It is shaken twice (for two minutes each time) with 5 c.c. of $\frac{N}{10}$ hydrochloric acid, and twice with 10 c.c. of water, and the extracts united and titrated with $\frac{N}{10}$ potassium hydroxide solution, methyl red being used as indicator. The results agree closely with those obtained by the Swiss Pharmacopœia method, and the methods of Frerichs and Mannheim. C. A. M.

Laboratory Control of Wheat Flour Milling. B. R. Jacobs and O. S. Rask. (*J. Ind. and Eng. Chem.*, 1920, 12, 899-903.)—The authors discuss fully the composition of the wheat kernel and its component parts, and describe methods employed in mill control and the examination of commercial mill products. Methods for determining the "flour content" of wheat and flour mill products are fully entered into, while calculations are embodied which may be employed to check miller's yield reports. It is shown that the amount of fat and pentosans in any grade of flour is a measure of the amount of offal (germ and bran) contained in that flour. The amount of starch contained in wheat or any mill product is a measure of the amount of inner endosperm, and therefore of the amount of flour contained in that product while the determinations of pentosans and starch may be used as a control of the miller's yield reports, various formulæ being given which are suitable for such a calculation. H. F. E. H.

Analysis of Wines. W. Fresenius and L. Grünhut. (*Zeitsch. anal. Chem.*, 1920, 59, 209-232.)—Directions are given for the estimation of ash, total alkalinity of the ash, alkalinity of the soluble ash, and phosphoric acid (see also ANALYST 1920, 304). W. P. S.

Detection and Estimation of Yellow AB and Yellow OB in Mixtures. W. E. Mathewson. (*J. Ind. and Eng. Chem.* 1920, 12, 883.)—These dyes, benzeneazo- β -naphthylamine (AB) and *o*-tolueneazo- β -naphthylamine (OB) are the only oil-soluble coal-tar colours allowed in food products by the U.S. Department of Agriculture. The usual liquid mixtures sold for colouring purposes consist of 2 to 3 per cent. solutions of the dyes in oil, and are tested by diluting with from 500 to 1,000 times their volume of petroleum spirit, extracting 30 c.c. with 1 or 2 c.c. of 7*N* sulphuric acid, followed by two extractions with 18*N* acid, and finally, if the petroleum spirit still contains colour, with 24*N* acid. The 18*N* extract contains practically all the yellow AB and the yellow OB. Carrotin and annatto remain in the petroleum spirit after shaking with 24*N* acid, although bixin is removed by acid of this strength. The 18*N* acid extracts after dilution are submitted to an elaborate

series of extractions and distillation, and the dyes are finally obtained in 100 c.c. of 93 per cent. alcohol. The light absorption constant at wave length 0.436μ is then determined by means of a spectrophotometer, using a quartz mercury lamp as the source of illumination. By careful measurement it is possible to estimate the proportion of each dye present. Numerous details of experimental work performed in the working out of the above method are described, and a few colour tests are given, which, however, are of little value when mixtures of the two dyes are present, owing to similar results being given by each.

T. J. W.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Determination of Cellulose in Woods. S. A. Mahood. (*J. Ind. and Eng. Chem.*, 1920, **12**, 873-875.)—A uniform size of particle is essential, if comparable results are to be obtained in the determination of cellulose in woods. Material which passes an 80-mesh standard (American Society of Civil Engineers) sieve, but is retained on a 100-mesh sieve, has been found to be most satisfactory from the standpoint of both yield and manipulation. Material obtained by a single mechanical process of disintegration may give a sample on sifting which is not representative; to avoid this, a combination of two processes, sawing and grinding, has been used. The procedure recommended by Sieber and Walter—viz., allowing the material to remain in the crucible throughout the treatment with chlorine and sodium sulphite for chlorination—gives a lower yield of cellulose than the original Cross and Bevan method, probably because of the higher temperature at which chlorination takes place. The modification of the Cross and Bevan method proposed by Johnsen and Hovey, who subject the wood samples to a preliminary hydrolysis with acetic acid in glycerol at $135-140^{\circ}\text{C}$., appears to be of doubtful value, since the cellulose as well as the hemi-celluloses and furfural-yielding constituents are attacked. Dore, in a recent article (*J. Ind. and Chem. Eng.*, 1920, **12**, 264), shows that the more resistant portion of the cellulose, the α -cellulose, is attacked by the acetic acid-glycerol mixture. Experiments are described giving the results obtained for cellulose with varying the size of the material between 40 and 120 mesh, and also comparative determinations when using the methods of Johnsen and Hovey (Sieber and Walter's apparatus), and of Cross and Bevan as modified by Benker both in Schorger's and in Sieber and Walter's apparatus.

H. F. E. H.

Determination of Phytin in Plant Extracts. A. Rippel. (*Biochem. Zeitsch.*, 1920, **103**, 163-172; through *J. Soc. Chem. Ind.*, 1920, **39**, 582A.)—Phytin can be precipitated more or less quantitatively in the presence of inorganic phosphates by copper acetate in acetic acid solution. Satisfactory results can only be obtained with definite concentrations of copper acetate and acetic acid. An extract from barley yielded 63.3 per cent. of the phytin P_2O_5 by precipitation with copper acetate in 1 per cent. acetic acid. The same method accounted for 92.8 per cent. of the phytin in standard solutions of that substance. The phytin in an acetic acid extract from peas could not be precipitated by copper acetate at all.

ORGANIC ANALYSIS.

Rapid Method of Estimating Aromatic Hydrocarbons in Petroleum Spirits. W. Hess. (*Zeitsch. angew. Chem.*, 1920, **33**, 147, 176; through *Chem. Zeit.*, 1920, **44**, *Rep.*, 259.)—A method of estimating aromatic hydrocarbons in petroleum spirit is based on their conversion into mono-nitro-compounds, and their separation from the unchanged non-nitrated hydrocarbons by means of concentrated sulphuric acid, in which the nitrated hydrocarbons are soluble. An apparatus of special form is used for the estimation, in which it is essential that the relative proportions of the reacting substances and the concentration of the acid should be kept as constant as possible. The nitric acid used is freed from nitrous acid by means of a current of air at 40° to 45° C. If necessary, a temperature correction must be applied when reading the volume of non-nitrated hydrocarbons.

C. A. M.

Estimation of Benzene in Turpentine. A. Allina and H. Salvatena. (*Chem.-Zeit.*, 1920, **44**, 673-674 and 697-698.)—The methods of Herzfeld and Marcusson depending on the destruction of the terpenes are subject to large errors. The authors have investigated four methods :

(1) Temperature rise with sulphuric acid. The sample, 100 c.c., is stirred with a thermometer in a smaller beaker, enclosed with glass wool packing in a larger one, with 10 c.c. of 2:1 sulphuric acid. The rises of temperature, of which a table is given, varied from 2° C. for substitute to 137° C. for fresh Neustadt turpentine.

(2) Specific dilatation. Although the terpenes have a lower coefficient than benzene the method has no quantitative value.

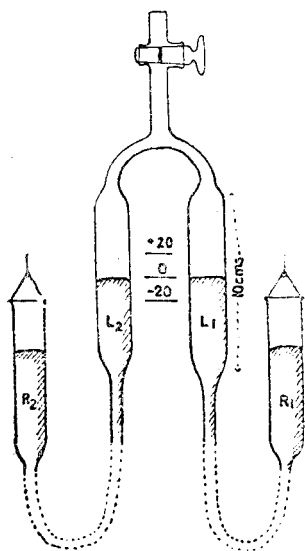
(3) Raising the sp. gr. by bromination. Irregularities in bromination, which was carried out by shaking with potassium bromide and bromate, with addition of hydrochloric acid, confine the method to distinguishing between genuine turpentine and substitute, giving an approximate value for the benzene.

(4) Bromination followed by steam distillation. The boiling-point of the turpenes is raised, whatever the nature of the bromination (substitution or addition), so that irregularities in this respect do not invalidate the method.

The bromination is carried out in a Claisen flask with 20 grms. of potassium bromate in 10 c.c. of water; 20 c.c. of the sample, and 50 c.c. hydrobromic acid (50 per cent.), 1 c.c. at a time, are added with shaking, and with cooling at first. Later, a reflux condenser being attached, the temperature is allowed to reach 40-50° C., at which it stands for an hour. The liquid is then decolorised with sodium carbonate and arsenic trioxide in powder. The side limb of the flask is filled with beads and glass wool for the steam distillation. A U-tube with one graduated limb (20 c.c. by tenths) surmounted by a cock and funnel, and having a cock below the graduations, a run-off cock at the lowest point, and another in the plain limb level with the highest graduation, is used as a receiver. This is filled with water, and the quantity of benzene per 5 c.c. of water coming over is noted. When distillate heavier than water, which may carry down benzene, appears, the water is run off, and after shaking with fuming sulphuric acid, the quantity of benzene is noted. Up

to 50 per cent. of benzine, 40 c.c. of condensed water suffices, but more is necessary for higher contents. The use of a water-cooled Hempel column for the distillation is recommended. A table is appended showing results, ranging from 11.1 to 89.4 per cent. respectively for 10 and 90 per cent. benzine taken in the experimental sample, with 40 c.c. condensed water.

O. E. M.



Consistency of Greases. F. S. Clulow and C. W. Taylor. (*J. Soc. Chem. Ind.*, 1920, 39, 291-295r.)—The viscosity or consistency of a thick grease at a definite temperature is reduced when the grease is used under working conditions, although the temperature remains constant. The apparatus shown in the illustration was employed to measure any change of viscosity due to mechanical treatment. The two mercury reservoirs, R_1 and R_2 , are connected by means of rubber pressure tubing to the lower ends of the tubes L_1 and L_2 , and the reservoirs are connected by a gut string passing over small pulleys, so that when one moves upwards the other moves downwards by an equal amount. The apparatus is surrounded by a water jacket. To use the apparatus, the temperature of the water jacket is raised a few degrees above the melting-point of the grease, both reservoirs are raised, with the tap of the apparatus open, so as to fill the tubes L_1 and L_2 with mercury, and the melted grease is poured in through

the tap, the reservoirs being lowered gradually until the mercury in both bulbs is at the zero mark. The tap is closed and the grease brought to the temperature at which the determination is to be made. The right-hand reservoir is then brought to the zero mark, and the gut string so adjusted that the mercury level in the left-hand reservoir is 140 mm. below this level. As the mercury level in the right limb L_1 moves upwards, the right reservoir is raised slowly, keeping the mercury surfaces level; the left reservoir descends at the same rate. The time noted is that required for the mercury surface to move 20 mm. above and below zero in either limb. By reversing the process the grease is forced in the opposite direction through the apparatus. The rate of flow of the grease increases each time it is forced through the tube, until a constant time is at last obtained which cannot be decreased by further working. For instance, at 32° C., a specimen of grease had an initial time of flow of 720 seconds, but after some fifty passages through the tube the time of flow was reduced to 15 seconds (constant). When re-melted and cooled to 32° C., the grease regained its high initial viscosity, which gradually dropped to the minimum value after working. This decrease of viscosity, brought about by mechanical treatment, is due to the destruction of the original crystalline structure of the grease. The apparatus may be standardised against castor oil, and it is suggested that such a head of mercury should be used as will produce a time value of 13.5 seconds for castor oil at 0° C.

W. P. S.

Reactions of Dichlorodiethyl Sulphide ("Mustard Gas"). F. Martin. (*J. Pharm. Chim.*, 1920, **22**, 161-165.)—Dichlorodiethyl sulphide is oxidised by nitric acid and other oxidising substances, and the solution obtained yields the usual reactions for sulphuric acid. If a few drops of dichlorodiethyl sulphide are heated with potassium hydroxide solution, the vapours produced give a white precipitate with Dénigès' reagent (an acid solution of mercuric sulphate). When the vapours are absorbed in water an odour resembling that of mercaptan is observed on the addition of a drop of iodine solution. The substance is hydrolysed by water, especially by acidified water, and the solution absorbs bromine. The presence of a few cgrms. of the substance in 1 litre of water may be detected by means of a solution of 8 grms. of potassium iodide, 0.8 gm. of copper sulphate, and 1 c.c. of gum arabic solution in 33 c.c. of water; this reagent is set aside for two hours, and then filtered before use. On mixing the aqueous solution of dichlorodiethyl sulphide with one-fourth of its volume of the reagent, a white turbidity develops, due to the formation of diiododiethyl sulphide. If the gum arabic is omitted from the reagent the reaction is slower, and the diiododiethyl sulphide separates in the form of crystals. W. P. S.

Determination of the Jellying Power of Gelatines and Glues by the Polarimeter. C. R. Smith. (*J. Ind. and Eng. Chem.*, 1920, **12**, 879.)—The optical rotation of solutions of all gelatines and glues at 35° C. is practically constant, ranging from -6.70 to -7.20 degrees Ventzke per gm. of solid material, but on cooling to 15° C. the rotation increases proportionately to the viscosity of the solution. Increase in concentration gives higher values, but by keeping the solutions for several hours at 15° C. a gradual decrease is observed, so that solutions of all strengths under the above working conditions yield a constant rotational value for any particular sample. Determinations of the polarimetric values are made by soaking 3 grms. of the powdered air-dried gelatine in cold water for thirty minutes, immersing in a boiling water-bath until solution is complete, cooling to 35° C. and diluting to 100 c.c. If clarification is necessary 5 grms. of light magnesium carbonate are added to the solution, which is kept at 30-40° C. for an hour, and filtered bright. The first rotation is measured in a 200 mm. tube at 35° C., the solution then being kept at 15° C. overnight, and the rotation again observed next day. The results are expressed by the formula $\frac{\text{Rotation at } 15^{\circ}}{\text{Rotation at } 35^{\circ}}$, and are inversely proportional to the percentage of gelatine required to produce a standard jelly at 15° C. This standard is defined as a solution of such strength that a bubble of air 4 to 5 mm. diameter admitted to the polarimeter tube moves vertically with a motion of 4 cm. per second. Two tables are provided, giving a large number of results obtained, and a third one showing the comparison between the polarimetric values and jelly strengths obtained in the following manner: A 60° funnel is partly filled with mercury, 50 c.c. of the gelatine solution are poured upon the surface, and allowed to set at 10° C. The mercury is then run out, and a partial vacuum of 600 mm. of water produced below the jelly, when the depression of the upper surface is measured by a micrometer gauge. The results obtained by this method bear a moderately definite ratio to those given by the polarimetric method. T. J. W.

Application of Devarda's Method to the Estimation of Nitrogen in Nitroglycerine. Koehler, Marqueyrol, and Jovinet. (*Ann. Chim. anal.*, 1920, 2, 271-272).—A quantity of about 0.5 gm. of nitroglycerine is mixed with 40 c.c. of hydrogen peroxide (12 vols.) and 5 c.c. of potassium hydroxide solution in a conical flask, and the mixture is heated at 40° C. for not less than forty minutes. The mixture is agitated thoroughly during this period by means of a mechanical stirrer (1,500 revs. per minute). The inside of the flask is then rinsed down with a few c.c. of alcohol, and the heating continued for a very short time at 70° C. The subsequent reduction of the nitrate with Devarda's alloy and the distillation of the resulting ammonia are carried out in the usual way. W. P. S.

Relation between the Quantities of Tarry and Carbonised Substances in Heavy Mineral Oils. W. Steinkopf and H. Winternitz. (*J. prakt. Chem.*, 1920, 101, 82-92).—A large number of experiments were made with the object of determining to what extent the formation of carbonised matter (coke) in heavy mineral oils is influenced by the amount of hard and soft tarry substances present, and also to determine the difference of coke formation at ordinary pressure and *in vacuo*. The hard tarry substance was estimated by Holde's method and the soft tarry substances by Holde and Meyerheim's method (*ANALYST*, 1911, 36, 298); the distillations for the estimation of carbonised matter were made in an atmosphere of nitrogen, and during the vacuum distillations the flask was shaken continuously. Generally, an oil containing a large quantity (2 to 6 per cent.) of hard tarry substances yields a considerable amount of carbonised matter (5 to 8 per cent.); but in the case of a few oils (Argentine), from 40 to 60 per cent. of the soft tarry matters are carbonised during distillation. Excepting very tarry oils, the results obtained by distillation at ordinary temperature and *in vacuo* differed to a slight extent only as regards the yields of carbonised matter. W. P. S.

Determination of the Specific Heat of Heavy Mineral Oils. H. S. Bailey and C. B. Edwards. (*J. Ind. and Eng. Chem.*, 1920, 12, 891-894).—The method depends upon the correct determination of the electrical energy required to heat a definite mass of oil through a given range of temperature. Determinations can be carried out rapidly at any temperature below the point where the vaporisation of the sample causes an appreciable error, the results being satisfactory for the calculation of heat transference data in oil circulating systems. The apparatus employed is described in detail, together with the methods of calculating results: 250 grms. of the oil are employed in the calorimeter, which is a 1-quart vacuum-jacketed glass vessel 25 ins. in diameter and 11½ ins. deep enclosed in a copper jacket, sold under the name of "Thermos Fruit Jar." Accuracy of any desired degree may be obtained by refinements in temperature and electrical energy measurements.

H. F. E. H.

Commercial Examination of Sulphonated Oils. G. F. Pickering. (*J. Soc. Chem. Ind.*, 1920, 39, 305-306T).—A few c.c. of the sample are boiled with three times the volume of hydrochloric acid until the fatty layer is clear, and the acid layer

is then drawn off and tested with barium chloride, a precipitate indicating the presence of sulphonated oil. If the oil layer is charred, the methods suitable for oils of high iodine value must be used. *Water*.—Distillation with xylene is the most accurate method, but in the case of oils of low iodine value it is sufficient to heat about 2 grms. in a $1\frac{1}{2}$ in. Petri dish in the water-oven. The amount of any ammonia present must be deducted from the loss of weight. *Total Fat in Oils of Low Iodine Value*.—About 5 grms. are boiled with dilute (1 : 1) hydrochloric acid until the fatty layer is clear, and, after cooling, extracted twice with ether. The united extracts are washed free from acid and distilled, and the residue of fat dried in the water-oven. *Oils of High Iodine Value*.—Five grms. are dissolved in 50 c.c. of pyridine, and the solution heated on the water-bath with 25 c.c. of strong hydrochloric acid for at least one and a half hours, with frequent shaking. It is then cooled, and extracted twice with ether, as described above. *Total Sulphuric Acid*.—About 2 grms. of the sample are distributed in small drops over a thin layer of powdered sodium carbonate in a platinum dish, and then covered with a layer of about $\frac{1}{4}$ in. of the carbonate, and then heated, first over a luminous Argand flame until the oil is carbonised, and then over a Teclu burner until the contents of the dish are white. The mass is then dissolved in hot dilute hydrochloric acid and the solution treated with barium chloride. *Free Sulphuric Acid*.—About 10 grms. of the sample are dissolved in 50 c.c. of ether, and the solution shaken with three successive portions of 50 c.c. of saturated brine of known sulphuric acid content. The united brine extracts are titrated with $\frac{N}{4}$ alkali, with methyl orange as indicator. *Alkali Salts*.—The only salts used for washing sulphonated oils are sodium sulphate and chloride, and, since the latter is converted into sulphate during the process, the salts left in the oil may be reported as Na_2SO_4 . The brine extracts from the titration are heated to boiling, the sulphate precipitated, and any free sulphuric acid present deducted. *Alkalis*.—Ten grms. of the sample are dissolved in water, and the solution titrated with $\frac{N}{2}$ acid, with methyl orange as indicator. In the absence of ammonia the result is expressed as sodium oxide. If ammonia be present, another 10 grms. of the oil are distilled with excess of sodium hydroxide, and the ammonia received in 25 c.c. of $\frac{N}{2}$ acid, and the difference between the amount of ammonia and total alkali is recorded as sodium oxide. *Unsulphonated Oil*.—A solution of about 5 grms. of the sample is neutralised with $\frac{N}{2}$ acid, and extracted with 100 c.c. of ether. The ethereal extract is repeatedly washed with water, and then shaken with 25 c.c. of water and 25 c.c. of approximately $\frac{N}{4}$ -potassium hydroxide solution, the soapy layer separated, and the ethereal layer washed with water until free from soap, the washings being united with the soap solution. The ether is then distilled and the residue of unsulphonated neutral oil dried and weighed. The water-soluble substance in the flask may be salted out and the solution shaken with a mixture of five parts (by vol.) of ether and one part of alcohol. The soap solution and washings are decomposed with acid and shaken with ether, the extract washed with water and distilled, and the residue dried and weighed. Good samples of sulphonated oil should not contain more than 15 per cent. of unsulphonated oil. A table is given showing the results thus obtained with seventeen samples of commercial sulphonated oils from castor, maize, seal and herring oils, wool grease oleine, etc.

Colour Test for Oxalic Acid. L. H. Chernoff. (*J. Amer. Chem. Soc.*, 1920, **42**, 1784.)—In 5 c.c. of the solution a few crystals of resorcinol are dissolved by gentle heating and the mixture cooled. Sulphuric acid is then slowly run in to form a layer beneath the solution without the generation of much heat, when, if oxalic acid is present, a blue ring will be produced at the junction of the liquids. Should no colour be produced the contents of the tube are mixed, cooled, and 5 c.c. more of sulphuric acid added as above. If still no colour is formed the solutions are again mixed and warmed over a flame. Any blue colour produced disappears on cooling the solution in ice-water, and reappears on warming. On boiling the mixture for a few minutes, the colour becomes dark green, changing to light yellowish-green on cooling, when, if sulphuric acid is again added, the blue colour reappears. This reaction is sensitive to 1 mgrm. of oxalic acid if dissolved in a few drops of water, and if dilute solutions are to be tested it is advisable to concentrate to about 10 per cent. The reaction is unaffected by the commonly occurring organic acids, and interfering substances may be removed by precipitating the oxalic acid as the calcium salt from ammoniacal solution, washing with water, and applying the test to the aqueous suspension of the salt.

T. J. W.

Paracymene as a Solvent. A. S. Wheeler. (*J. Amer. Chem. Soc.*, 1920, **42**, 1842.)—Paracymene is a by-product of the manufacture of spruce pulp by the sulphite process, and, owing to its recent fall in price, should be available as a useful solvent, being colourless and without objectionable odour. The boiling-point is 176–176.5° C. and the refractive index 1.4905 at 12.5° C. The author describes and criticises the methods of purification, and furnishes two tables showing the solubilities at different temperatures of various organic compounds and acids. Pure paracymene remains colourless on exposure to light, but, if impure, rapidly develops a yellow or red colour.

T. J. W.

Diffusing Power of Pigments. W. K. Lewis and F. P. Baker. (*J. Ind. and Eng. Chem.*, 1920, **12**, 890-891.)—A new testing method for pigments is described, which, while not measuring the covering power, is simple both in technique and apparatus, and gives results expressed in absolute figures capable of duplication by different observers in different laboratories. If a finely divided solid be suspended in a suitable fluid, light passing through the suspension is diffused, producing a "ground glass" effect. The diffusion increases greatly with the number of particles, and is probably nearly proportional to the total number of particles, and relatively independent of their size. This diffusion is the principle employed in every turbidimeter; that is, some object is observed through the suspension, and the thickness of suspension just required to blur the object is measured. In the present method the filament of an incandescent lamp is observed, and the thickness of pigment suspension needed completely to blur the filament is determined. The suspension does not shut off the light from the filament, but the light is diffused to such a degree that the outline of the filament is no longer perceptible. The depth of suspension just necessary to obscure the filament can be determined with remarkable precision. The pigment to be tested is suspended in a suitable

fluid, and the suspension is maintained by any satisfactory method of stirring, such as using a stirring rod or blowing air through the liquid. The apparatus consists of a vertical cylinder of metal or glass, to which is cemented a plate glass bottom, plate glass being used to make sure that the two faces are absolutely parallel. The sides of the cylinder are preferably blackened, but this is not essential. The suspension, the pigment concentration of which is known, is poured into the cylinder until the filament of an incandescent lamp held just below the bottom can no longer be distinguished, and the depth of suspension is accurately noted. It has been experimentally determined that the depth of suspension observed is inversely proportional to the concentration of the pigment. This means that the disappearance of the filament is determined by the amount of pigment per unit area of cross section of the cylinder, and is uninfluenced by the position of the pigment particles in the direction in which the light is travelling. It is this weight in grams required for each square centimeter of section which is measured. The authors report the reciprocal of this quantity, *i.e.*, the sq. cs. of surface which 1 grm. of pigment is capable of covering, and this is called the diffusing power of the pigment, and, though expressed in square centimeters per gram, it will be equally satisfactory if expressed in square feet per lb. The diffusing power of a pigment is a function of the completeness with which it is dispersed in the fluid employed, and in the investigation linseed oil has usually been employed. The diffusing power must not be confused with covering power, which is unquestionably influenced by a pigment's light-absorption capacity, a factor which is entirely eliminated in the determination of diffusing power. H. F. E. H.

Determination of Sulphur Compounds in Coal. A. R. Powell. (*J. Ind. and Eng. Chem.*, 1920, 12, 887-890.)—The author has employed the methods described by Powell and Parr (University of Illinois Engineering Experiment Station, 1919, Bull. III., p. 10) with certain modifications which are now described as suitable for rapid routine work. After the sulphate and pyrites determinations are completed, the organic sulphur may be reckoned from the total sulphur by difference. The extraction of sulphate may be completed in a few minutes by boiling with hydrochloric acid (sp. gr. 1.12) rather than by slow extraction with the very dilute acid. The boiling acid does not decompose pyrites to any appreciable extent. The extraction of pyrites by dilute nitric acid may be completed in an hour or two by the use of a shaking machine. Dilute nitric acid will extract small amounts of the unstable phenol-soluble form of organic sulphur from certain coals. To avoid this error in laboratory determinations it is recommended that pyritic sulphur be calculated from the pyritic iron value. The dilute nitric acid not only extracts pyrites quantitatively but also the same iron and sulphur which is taken into solution by dilute hydrochloric acid. For this reason, samples intended for dilute nitric acid extraction need not be extracted with hydrochloric acid first. Semi-bituminous coals and the harder varieties of bituminous coals must be digested for a long time with concentrated nitric acid before the organic matter will become completely soluble in ammonia. The Eschka method is preferable to the sodium peroxide method for the determination of sulphur in coal and coal residues, unless the sodium peroxide used has a low and constant sulphur content. The following table summarises

the results of analyses of some samples of coal, and shows that all the sulphur of these coals has been accounted for in the four forms of pyrites, sulphate, and the two forms of organic sulphur.

SUMMARY OF ANALYSES FOR SULPHUR COMPOUNDS IN COAL.

Pyritic sulphur	0.47	0.79	0.08	0.13	1.75	1.99
Sulphate sulphur	0.07	0.23	0.01	0.04	0.71	0.32
Humus organic sulphur	0.50	0.46	0.44	0.42	1.01	0.39
Phenol-soluble organic sulphur	0.12	0.20	0.02	0.09	0.77	0.32
Total	1.16	1.68	0.55	0.68	4.24	3.02
Total by direct analysis	1.21	1.72	0.56	0.71	4.25	3.06
Difference between totals	0.05	0.04	0.01	0.03	0.01	0.04

H. F. E. H.

Determination of Free Sulphur and the Coefficient of Vulcanisation in Vulcanised Rubber. W. J. Kelly. (*J. Ind. and Eng. Chem.*, 1920, 12, 875-878.)

—Two new methods for rubber analysis are presented; the first gives the correct value for free or elementary sulphur in rubber goods, and the second a lower and more accurate value for the coefficient of vulcanisation than is obtainable by any of the older methods. The work was all done on pure gum stock without any accelerator, and the methods described are being extended by the author to meet the conditions which obtain in compound stocks. The sulphur present in vulcanised rubber may be divided into four parts: Sulphur soluble in acetone: (1) Combined with resins and proteins, (2) true free sulphur. Sulphur insoluble in acetone: (1) Combined with rubber, (2) combined with resins and proteins. As regards the sulphur soluble in acetone it is unfortunate that all solvents for either will dissolve both to a greater or less extent. The entire extract very readily dissolves in 95 per cent. alcohol, and this alcohol will also dissolve about 0.04 gm. sulphur in 100 c.c., the amount of sulphur usually obtained in an ordinary analysis. If, however, the extract is treated with alcohol already saturated with sulphur, it will dissolve all organic material present, but none of the elementary sulphur, which remains in the flask after the solution is decanted, and can be weighed as such, or oxidised and weighed as barium sulphate. The saturated solution of sulphur in ethyl alcohol, referred to below as saturated alcohol, is therefore prepared by heating an excess of sulphur for about three hours at 70° C., and allowed to cool slowly. *Method.*—A 1-gm. sample of the ground rubber is extracted with acetone for sixteen hours in an Underwriters' apparatus. The acetone is distilled off, the residue dried at 60° to 65° C. for half an hour and treated with 50 c.c. of saturated 95 per cent. alcohol, and the flask and contents weighed. An accuracy of 0.5 gm. is sufficient for this weighing. It is then warmed to about 50° C. for a few minutes, to insure complete solution of all the organic material, and allowed to cool slowly to room temperature. Before it has reached this temperature the flask is again weighed, and any alcohol lost during the heating is replaced with pure 95 per cent. alcohol. The flask is then covered and allowed to stand for twenty-four hours, to permit

the complete crystallisation of any of the elementary sulphur of the extract which may have dissolved. The liquid is decanted and the residue washed two or three times by decantation with 5 c.c. of saturated alcohol, and dried. It may then be weighed directly, or oxidised and weighed as BaSO_4 . The figure thus obtained is the value of the true free or elemental sulphur which is available for further vulcanisation of the rubber. The remainder of the acetone-soluble sulphur is combined with resins, etc., and does not participate in further vulcanisation.

Coefficient of Vulcanisation.—A 1-grm. sample of ground rubber is extracted sixteen hours with acetone, and the residue dried. It is then boiled for eight hours with 75 c.c. of a 5 per cent. alcoholic potash solution and washed once or twice with hot alcohol. As the removal of the last traces of alkali is very difficult, the sample is extracted sixteen hours (over night) with alcohol in an Underwriters' apparatus. The solutions are then mixed, and the alcohol distilled off. The residue is oxidised first with 15 c.c. of a solution of bromine in potassium bromide (120 grms. KBr and 160 grms. Br_2 in 1 litre of water), in order to prevent the violent action of fuming nitric acid on alkali, and finished with nitric acid. The barium sulphate is precipitated in the usual manner. As there is always some silica derived from the glass, it is necessary to evaporate twice with hydrochloric acid, thoroughly dehydrate, and remove the silica before precipitating the sulphate. The figure obtained for the combined sulphur, after extraction with alcoholic potash, is much more nearly correct than any obtained by methods which do not take into account the acetone-insoluble compounds of sulphur with resins and proteins. It is still possible that there is some sulphur combined with proteins and resins, insoluble in acetone and not attacked by alcoholic potash. For the present, therefore, the figure obtained by the method presented here must be considered as a maximum value for the coefficient of vulcanisation.

H. F. E. H.

Detection of Mangrove [Tannin] in Mixed Tannin Extracts. E. Schell (*J. Soc. Leather Trades' Chem.*, 1918, 2, 284-286; through *J. Soc. Chem. Ind.*, 1920, 39, 496A.)—A review of the several methods suggested for the detection of mangrove in tanning extracts. The solubility of mangrove tanning in ethyl acetate is a very uncertain guide. The amount of chlorine or lime in the ash is also a doubtful indication of the presence of mangrove, since these constituents may have been introduced by using hard water. The Mulhouse dyeing test with mordanted cotton is unreliable. The precipitates obtained in the Schell test, in which a cobalt solution is added to a solution of the tanning extract, are not sufficiently well defined in colour to render it an efficient test. It can only be employed with normal extracts, and fails with "solubilised" extracts, and with those which have been subjected to a treatment with alkaline compounds and which give an alkaline reaction in solution.

INORGANIC ANALYSIS.

Estimation of Ammonia in Oil and Fat Preparations. N. Welwart. (*Chem. Zeit.*, 1920, 44, 719.)—Certain Turkey-red oils which contain ammonia, in addition to sodium compounds, are not completely decomposed when boiled with

dilute mineral acids, and in such cases estimation of the ammonia by the usual methods gives too low results. A more accurate method is to dilute a weighed quantity of the oil preparation with water, to add a fragment of pumice and an excess of calcium chloride solution, and to distil the ammonia into standard acid, after adding excess of sodium hydroxide solution.

C. A. M.

Estimation of Antimony in Lead-Antimony Alloys. L. Bertiaux. (*Ann. Chim. anal.*, 1920, 2, 273-278.)—The method depends on the titration of the antimony with potassium permanganate solution in the presence of hydrochloric acid. Poirrier's orange is added as indicator; as soon as all the antimony has been oxidised, the next drop of permanganate solution added reacts with the hydrochloric acid, liberating chlorine, which at once decolorises the indicator. The details of the method are as follows: Five grms. of the alloy (*e.g.*, antifriction metal) are dissolved by heating with a mixture of 40 c.c. of concentrated sulphuric acid and 10 grms. of potassium sulphate; after cooling, the mixture is diluted with 200 c.c. of water, 50 c.c. of hydrochloric acid, and 2 drops of a 0.1 per cent. Poirrier's orange solution are added, and the solution titrated with standardised permanganate solution. The latter must be added slowly, and the solution stirred thoroughly during the titration. Lead, tin, bismuth, copper, and arsenic do not interfere; iron, if present, is titrated together with the antimony, but it may be estimated colorimetrically in the titrated solution by means of thiocyanate and an allowance made for its quantity.

W. P. S.

Estimation of Calcium and Magnesium in the Presence of Iron and Aluminium Salts. E. Canals. (*Comptes rend.*, 1920, 171, 516-518.)—On treating a solution of ferric sulphate and magnesium sulphate with sodium phosphate solution containing two drops of sulphuric acid, then rendering the liquid alkaline with ammonia, and adding acetic acid, some of the magnesium, if it exceeds a certain concentration, will be retained by the insoluble ferric phosphate. Analogous results were obtained with magnesium in the presence of aluminium salts, and with calcium in the presence of iron salts. Accurate results were obtained with quantities of 0.02 to 0.23 gm. of magnesium and 0.01 to 0.125 gm. of calcium, in the presence of iron and aluminium, by adding ammonia to the solution of the mixed salts and sodium phosphate solution until the precipitation was complete, and then adding acetic acid drop by drop, with vigorous agitation, which was continued for some minutes, until the whole of the calcium and magnesium had dissolved.

C. A. M.

Indicators for Carbon Dioxide and Oxygen in Air and Flue Gas. L. H. Milligan, D. O. Crites, and W. S. Wilson. (U. S. Bureau of Mines, Tech. Paper No. 238, 1920, 24 pp.)—A portable apparatus described consists essentially of an absorption pipette connected with a graduated measuring tube. Sodium hydroxide solution (10 per cent.) is used as the absorbing liquid in the carbon dioxide apparatus and alkaline pyrogallol solution in the oxygen apparatus. The liquids in the apparatus are covered with a layer of petroleum oil to prevent absorption during the admission of the gas to be tested. Arrangements are provided for saturating the gases with moisture before they are admitted to the apparatus, for removing carbon dioxide in

the estimation of oxygen, and for cooling hot flue gases. The graduations of the scale are such that the percentage of carbon dioxide or oxygen, respectively, is indicated directly by the decrease in volume during the absorption. W. P. S.

Titration of Lead as Lead Chromate. J. M. Kolthoff. (*Pharm. Weekbl.*, 1920, 57, 934; through *Chem. Zeit. Rep.*, 1920, 44, 241.)—For small quantities of lead the neutral solution containing the metal is treated with a few drops of 5 per cent. aluminium chloride solution (to accelerate the precipitation of colloidal lead chromate), heated to 40° C., an excess of potassium chromate is added and the mixture boiled. The precipitate is collected on a filter, washed with hot water until free from potassium chromate, and then dissolved in dilute hydrochloric acid. The solution is diluted, potassium iodide is added, and the liberated iodine titrated with thiosulphate solution. Each c.c. of $\frac{N}{10}$ thiosulphate solution is equivalent to 6.9 mgrms. of lead. In the case of large amounts of lead, the solution may be treated with an excess of $\frac{N}{10}$ potassium dichromate solution; potassium iodide is then added, and the liberated iodine titrated. The error in this method amounts to about 0.15 per cent. W. P. S.

Estimation of Molybdenum. J. P. Bonardi and E. P. Barrett. (Technical Paper, No. 230, Bureau of Mines, Department of Interior, U.S.A., 1920, 35 pp.)—From a study of different methods for the estimation of molybdenum, the most satisfactory for the analysis of low-grade ores was found to be precipitation as lead molybdate, and titration with potassium permanganate after reduction of the molybdenum solution. In the volumetric method the ore is decomposed either by acid or by fusion with sodium peroxide into a soluble molybdate and insoluble compounds of the other constituents of the ore. The solution of the molybdate is filtered, acidified, and passed through a Jones "reductor" (Lord and Demorest, *Metallurgical Analysis*, 1916, 29-30), where the MoO_3 is reduced to Mo_2O_3 , which is then titrated with permanganate. In the acid method of decomposing the ore 0.5 to 5 grms. (containing not more than 0.08 gm. of molybdenum) are digested with *aqua regia*, and the liquid evaporated with sulphuric acid, and then boiled with ammonia. Copper and arsenic, if present, are removed by adding several grms. of zinc to the acid solution before passing it through the "reductor." In the decomposition by fusion with sodium peroxide a little molybdenum may be retained by the insoluble iron and copper hydroxides, and therefore in the case of high grade material the precipitate should be redissolved and again precipitated. $\frac{N}{20}$ permanganate solution is the most suitable strength for the titration, and the following amounts of ore should be taken: Five grms. of one containing less than about 3 per cent. of molybdenum (1 gm. for 15 per cent. and 0.5 gm. above 15 per cent.). If more than 16 c.c. of permanganate solution are required by a molybdenum solution no deduction is made for a blank estimation; but with a weaker molybdenum solution a small deduction is required. The volumetric method is accurate within 0.01 per cent. on the basis of a 1 gm. sample. For the gravimetric estimation the ore is decomposed with acids and treated with ammonium hydroxide. The hot solution of ammonium molybdate is treated with lead acetate in an excess of 2 to 3 c.c., the mixture heated for a few

minutes, and filtered. The precipitated lead molybdate is then ignited, cooled, and weighed. Any lead must first be removed as sulphate, and phosphorus by precipitation with magnesia mixture. Vanadium is also precipitated as lead vanadate, and a special method must be used for its separation from molybdenum. Sulphates do not interfere with the estimation. The gravimetric method is applicable to both high grade and low grade material, but is not practicable when fusion is necessary to decompose the ore.

C. A. M.

Electro-Analytical Separation of Nickel or Cobalt from Arsenic. N. H. Furman. (*J. Amer. Chem. Soc.*, 1920, **42**, 1789.)—When an ammoniacal solution of a nickel salt containing an alkali arsenate is electrolysed, the nickel is deposited free from arsenic; but with a similar solution containing cobalt in place of nickel, the deposit of cobalt is invariably contaminated with arsenic. Using a mixture of the two metals no arsenic is deposited with the alloy when the ratio of cobalt to nickel is less than 1:2.5, and the total weight of the mixed metals is 0.1 gm. The method used and tested by the author was as follows: Niccolite (native nickel arsenide) was dissolved in a mixture of concentrated nitric, hydrochloric, and sulphuric acids, the excess of acid evaporated and any iron present precipitated by addition of ammonium hydroxide, the ferric hydroxide being dissolved, and the precipitation repeated until free from nickel. To about 100 c.c. of the solution 2 grms. of ammonium sulphate and 15 c.c. of ammonium hydroxide (sp. gr. 0.90) were added, and a current of 0.5 amps. with a pressure of about 3.6 volts passed from ten to twelve hours. By the use of an electrode rotating from 600 to 800 revolutions per minute and a current density of 1.25 amps. deposition is complete in about one hour. After removal of the nickel the arsenic present in the solution was estimated as magnesium pyroarsenate, or as arsenic pentasulphide. The results obtained are in excellent agreement with those given by Moore's volumetric method for nickel and cobalt.

T. J. W.

Estimation of Potassium and Sodium in the Presence of Each Other. A. Quartaroli. (*Gazz. Chim. Ital.*, 1920, **50**, 64-65.)—The mixture of the chlorides of potassium and sodium is weighed, and the chlorides converted into nitrates, which are dried in a water-oven at 100° C. A small quantity of the mixture is then melted, and the solidification point of the fused mass determined. The relative proportions of potassium nitrate and sodium nitrate may be obtained directly from the result, in accordance with the following table constructed from determinations with the pure salts:

	Solidification Point. ° C.		Solidification Point. ° C.		Solidification Point. ° C.
KNO ₃ , pure...	336	65 per cent. KNO ₃	231	30 per cent. KNO ₃	254
95 per cent. KNO ₃	312	60 " "	225	25 " "	265
90 " "	297	55 " "	220	20 " "	273
85 " "	283	50 " "	225	15 " "	280
80 " "	270	45 " "	231	10 " "	287
75 " "	255	40 " "	237	5 " "	297
70 " "	243	35 " "	245	NaNO ₃ , pure	306

The method is much more accurate than the usual indirect methods, since an error of 10° C. in the determination is of less importance than an error of 1 mgrm. in the estimation of the chlorine in the mixed chlorides.

C. A. M.

Volumetric Estimation of Thiocyanates by Means of Potassium Permanganate. R. Meurice. (*Ann. Chim. anal.*, 1920, **2**, 272-273.)—Direct titration of thiocyanates in sulphuric acid solution with permanganate solution yields low results, but the oxidation is complete if an excess of permanganate (equal to at least one-half of the amount required for the oxidation) is added and allowed to react for a short time. The excess of permanganate may be titrated with standardised hydrogen peroxide solution.

W. P. S.

Influence of Vanadium on the Estimation of Chromium in Tungsten High-speed Steels. P. Slawik. (*Chem.-Zeit.*, 1920, **44**, 633.)—During the volumetric estimation of chromium in special steels a black to red-brown coloration is produced on the addition of ferrous sulphate, which changes to yellow on back-titration with permanganate, thus confusing the end-point. This was shown to be due to the formation of complex compounds of vanadic and tungstic acids. Parallel determinations in presence and in absence respectively of tungstic acid showed that its presence does not affect the result, except that the colour at end-point is yellowish brown instead of pink.

O. E. M.

Estimation of Zirconium. M. M. Smith and C. James. (*J. Amer. Chem. Soc.*, 1920, **42**, 1764.)—Zirconia ore is gradually heated with ten times its weight of potassium hydrogen fluoride until it just fuses to a clear liquid. After cooling, the melt is treated with 50 per cent. sulphuric acid, heated until dense fumes are evolved, and, when cold, dissolved in boiling water, cooled, and diluted to 250 c.c. Of this solution 100 c.c. are diluted, boiled, and precipitated with ammonium chloride and ammonium hydroxide and filtered, after which the hydroxides are washed, treated with 36 c.c. concentrated hydrochloric acid and an equal volume of water, boiled and diluted to 700 c.c. This volume is then precipitated by the addition of selenious acid solution containing approximately 12.5 per cent. of selenious acid, filtered, washed with 3 per cent. hydrochloric acid, dried, and ignited. The results obtained are in close agreement with those given by the "cupferron" method. Various modifications given in detail in the original paper are adopted in the presence of iron, titanium, and phosphoric acid, but aluminium and the rare earth metals exert no effect upon the results obtained.

T. J. W.

APPARATUS, ETC.

Chemical Analysis with Ultra-Filters. L. Moser and Kittl. (*Chem.-Zeit.*, 1920, **44**, 637-638.)—The ultra-filter of Zsigmondy (*Zeitschr. anal. Chem.*, 1919, **58**, 2413) is valuable in analytical work for the collection of colloidal precipitates or hydrosols, but not for general analytical work. The constant attention of the operator is necessary to avoid, on the one hand, contact of the precipitate with the

rubber joint ring, and, on the other, the running dry of the precipitate, which causes the formation of cracks through which the washing liquid passes uselessly. To remove the precipitate in the prescribed manner by means of a spatula and brush was not always possible; in the case of colloidal bismuth and silver the last traces had to be removed with moderately strong nitric acid. If the precipitate is allowed to dry it is very difficult to remove it from the filter.

O. E. M.

Apparatus for the Uniform Addition of Small Proportions of one Gas to another. R. Mezger. (*Chem.-Zeit.*, 1920, **44**, 658-659.)—Ammonia generated from slaked lime and ammonium chloride in a round flask heated by a mercury bath passes off to a mixing column filled with beads, at the bottom of which the other gas is admitted. On its way it passes a manometer and capillary. In communication with the flask is a U-tube containing mercury, which opens or closes the oblique mouth of a tube supplying gas to the burner heating the bath, so that as the pressure of the ammonia increases the flame is diminished, and *vice versa*. The capillary is chosen in accordance with a simplified form of Poiseuille's law, $V = KH$, in which V is the volume of gas passing in c.c. per second, H the difference of pressure, and K a constant for the capillary and the gas. This is determined for a given value of H , obtained by raising or lowering the oblique-ended tube, by absorbing the issuing gas in acid, titrating back, and calculating to c.c. per second. The constants of a range of tubes are determined. The method is applicable to other gases.

O. E. M.

Air-Oven Thermo-Regulator. H. M. Atkinson. (*J. Soc. Chem. Ind.*, 1920, **39**, 298t.)—A U-shaped tube has one limb connected with a bulb in the oven, whilst the other limb is closed with a stopper through which passes a short length of glass tube. The U-tube contains a quantity of mercury, and a float, having a capillary tube at the top, is placed in the limb carrying the stopper and glass tube, the capillary extending upwards into the latter. Gas is admitted through a side tube on this limb, and passes between the "shoulder" of the float and the lower end of the glass tube, and thence through the latter to the burner. As the pressure in the bulb in the oven increases, the mercury in the U-tube is gradually forced from one limb to the other, thus raising the float until the shoulder comes into contact with the end of the glass tube. The gas supply to the burner is thus shut off, but a small by-pass may be arranged by making a hole in the side of the float; the diameter of the capillary then controls the amount of gas passing through the by-pass. The lower end of the glass tube and the shoulder of the float may be ground to make a gas-tight joint, or a mercury seal can be provided at this point.

W. P. S.

Fischer's Viscosimeter. (*Chem. Zeit.*, 1920, **44**, 622.)—A new form of viscosimeter is described which is suitable for determining the viscosity of the most viscid liquids under constant conditions of temperature, etc. It consists essentially of an inner tube, at the base of which is a tap, and which is surrounded by a water-jacket similar to an upright Liebig's condenser. A thermometer is fixed in this water-jacket, and there is a second thermometer in a side tubulure which

communicates with the inner tube through the wall of the condenser. Water at the required temperature (usually 60° to 80° C.) is run through the condenser tube, and the efflux velocity of the liquid is measured by the time required for an aluminium or brass bulb to fall between two marks on the inner tube. The difference between the temperature of the heating water and of the liquid in the inner tube was found to be a constant of 4° C. For use with the aluminium bulb the apparatus is standardised upon chemically pure glycerol at 58° C., whilst, in the case of the brass bulb, the standardising solution is prepared by mixing 25 grms. of potato flour with 350 c.c. of water at 50° C., adding 50 c.c. of $\frac{N}{10}$ potassium hydroxide solution, and allowing the liquid to stand until cold. It is then heated to 70° C., introduced into the inner tube, and cooled to 58° C. by means of water in the jacket at 62° C.

C. A. M.

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REPORT.

Report on the Work of the Government Analytical Laboratory and Assay Office, Cairo, Egypt, during the Period 1913-1919. A. Lucas.— During the period covered by the Report, from 5,000 to 6,000 samples were examined annually. The analytical work comprised chiefly the examination of materials of all kinds from almost every Government Department, special war work for the Army, and investigations, etc., for the police and Ministry of Justice. There was also a considerable amount of advisory and consultative work, and during the last few years research work has taken a definite place in the programme of the Department; several papers by members of the staff of the Laboratory have been published in *THE ANALYST*. Every endeavour has been made to employ Egyptians as far as possible in the Laboratory, etc., and they now form more than 85 per cent. of the staff.

W. P. S.

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REVIEWS.

THE MANUFACTURE, REFINING AND ANALYSIS OF ANIMAL AND VEGETABLE OILS, FATS, AND WAXES, INCLUDING THE MANUFACTURE OF CANDLES, MARGARINE, AND BUTTER. By GEOFFREY MARTIN, D.Sc., Ph.D., F.I.C. London: Crosby, Lockwood and Son. Price 12s. 6d. net.

This volume, despite its somewhat ambitious title, consists of little more than 200 pages, and is stated in the Preface to be written for "practical men, industrial chemists, and manufacturers." The amount of condensation necessary to compress so extensive a subject within such limits is so extreme as seriously to prejudice its practical value. Thus, it is obviously next to hopeless to attempt, as the author does, to describe the manufacture and analysis of butter in five pages, of which two are taken up with tables of tests for colouring matters. Even more futile is it to essay the description of the chemistry and technology of margarine in seven pages, three of which are devoted to trade statistics.

It is a little difficult to see what good end can be served by this species of précis writing, except, indeed, as a popular exposition of the subject for the general reader, which is in this case specifically disclaimed.

One of the defects which arises from this attempt is the lack of coherence and proportion between the various sections of the subject. For example, the chapter on the hydrogenation of oils, occupying some thirty pages, is of quite disproportionate length when compared with the rest of the book. Incidentally the space in question is chiefly taken up with the text of patent specifications, without, in many cases, any accompanying explanatory matter, and without attempt at arrangement or classification. The insertion of chapters on the treatment of waste rags, and the manufacture of fish meal, is surely somewhat irrelevant, especially in view of the exclusion of so much matter of primary importance to the subject.

The portions of the book devoted to the chemistry of the oils and fats are brief and sketchy, and show the patchiness previously mentioned. Further, a good deal of the data appears to have been obtained from obsolete publications. Thus, in the two pages which are all that can apparently be spared to the chemical composition of the oils and fats, the substance *clupanodonic acid*, the characteristic fatty acid of the fish oil group, which was discovered in 1906 by Tsujimoto, is omitted from the table of fatty acids; and, although fish oils are mentioned in many places, there is apparently no reference to this distinguishing component either as fatty acid or as a glyceride.

Such an omission, although greatly to be deplored, might conceivably be due to inadvertence, but, unfortunately, there are not a few similar instances. Thus, the

table on page 4 contains "margaric acid," long ago shown to be a eutectic mixture of palmitic and stearic acids, and omits daturic acid, isolated by Meyer and Beer in 1912. Further, cerotic acid is given an odd number of carbon atoms (27 instead of 26), and several fatty acids are included which have never been obtained from natural oils and fats, while elæostearic acid is stated to have the formula $C_{19}H_{30}O_2$, whereas this was shown by Kametaka in 1903 to be erroneous.

In the section on analysis, confined to eleven pages, there are many similar instances of errors and omissions which are even less excusable. On page 151, the specific gravity of beeswax is given as lower than that of palm oil, while, further on, the "titer" of fats is defined as the *melting-point* of their fatty acids—a statement which is incidentally contradicted on the same page.

After all that has been written on the desirability of a uniform temperature for recording the refractive index of oils and fats, resulting in the virtually unanimous agreement of the figure of 40° C. as the most suitable, the statement is made on page 152 that "the refractive index of oils is usually determined at 60° C."

Similarly in the description of the Polenske value—erroneously spelt with an acute accent over the final "e"—one is still directed to "take a few fragments of pumice," notwithstanding that this very statement has been pilloried many times in this journal and elsewhere.

On page 162 are given percentage yields of insoluble bromides from oils without any mention of the conditions under which these were obtained, which, of course, vitally affect the figures. Similarly, the Maumené test is stated to be made with "concentrated sulphuric acid," with no further indication of strength.

In spite of much recent work on the "Valenta test," this is dismissed in two or three lines as a test for butter only, no mention being made of its general application. Further, the strength of the acetic acid on which the temperature figure entirely depends is not mentioned otherwise than as "glacial," which has been shown to be useless as a definition for this test.

In the section on butter, no mention is made of the Kirschner or even of the Polenske test in determining the purity of butter fat, but it is stated that the iodine value is "particularly useful in detecting adulteration with coconut fat."

There are several misprints and other signs of hurried proof correction, but these are of small moment compared with the foregoing inaccuracies.

Although, therefore, the book may present features of interest to the technical reader, it is scarcely possible to recommend it from the chemical and analytical standpoint.

P. J. FRYER.

DICTIONARY OF EXPLOSIVES. By ARTHUR MARSHALL. Pp. xiv+159. London: J. and A. Churchill, 1920. Price 15s. net.

The present volume is Mr. Marshall's third publication on the subject of explosives. It is a careful compilation on the composition, and certain of the properties, of about four hundred explosives. The subject-matter is restricted almost entirely to industrial explosives, which are more or less complex mixtures. The military explosives, which are either single chemical compounds or simple mixtures, are dealt with at length in Mr. Marshall's other works.

Explosives of foreign origin are included, but the explosives described are almost entirely those which are or have been in practical use. The many hundreds which have been proposed, but have failed to pass the test of actual performance, are not mentioned unless of special historical interest.

The dictionary is divided into three sections. The first contains a classification of explosives according to the purposes for which they are intended to be used; the second, which constitutes the principal part of the book, gives short descriptions of the explosives arranged in alphabetical order; and the third section is a list of the separate ingredients, with an indication of the explosives in which each occurs.

Many of the descriptions are necessarily somewhat vague, on account of the reticence of manufacturers to disclose the composition of their products. This is especially noticeable in regard to the American explosives, for many of which the only information available is a statement of the type to which they belong.

Within its somewhat restricted range, the volume contains a large amount of information, and should prove of considerable value as a work of reference. It is well printed, and no typographical errors have been observed. The style of binding is, however, not such as to produce a good impression, and the price seems very high for the size of the book.

W. L. TURNER.
