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THE ANALYST

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

An Ordinary Meeting of the Society was held on Wednesday, November 7, 1923, in the Chemical Society's Rooms, Burlington House. The President, Mr. P. A. Ellis Richards, F.I.C., was in the chair.

Certificates were read for the first time in favour of:—Messrs. Robert Charles Frederick and Hubert Thomas Stanley Britton, M.Sc. (Lond.), F.I.C.

Certificates were read for the second time in favour of:—Messrs. Laurence Eversley Campbell, M.Sc. (Lond.), F.I.C.; John Troubridge Hannen, B.A. (Cantab), A.R.C.Sc., A.I.C.; Cyril Langley Hinton, F.I.C.; Douglas William Kent-Jones, B.Sc. (Lond.), F.I.C.; Thomas William Alan Shaw, M.Sc. (Liv.); William Hall Simmons, A.I.C.; Kenneth Edward Nethercoate Williams; and Percy Noel Williams, M.Sc. (Liv.), A.I.C.

The following papers were read:—"A Quantitative Study of the Limitations of the Reaction between Ammonia and Sodium Hypobromite," by Dinshaw Rattonji Nanji and William Simpson Shaw; "The Gold-Beaters' Skin Test for Tannins," by Phyllis H. Price, B.Sc.; "The Estimation of Nitrogen in Coal," by W. Donovan, M.Sc.; and "The Estimation of Lead in Water and Urine," by J. C. Thresh, D.Sc., M.D., F.I.C.

Deaths.

We regret to have to record the deaths of two of our Members :—

Dr. J. E. Stead, F.R.S.
Mr. J. M. Wilkie.

The Sampling of Coal: The General Problem and Some Experiments.

By J. H. COSTE, F.I.C., E. R. ANDREWS, F.I.C., and W. E. F. POWNEY, F.I.C.

(*Read at the Meeting, October* 3, 1923.)

The analyst who receives for examination a sample from a client is concerned only with the composition and properties of what he receives. He knows nothing of the bulk supposed to be represented by the sample but what that sample can tell him. This attitude of detachment may, however, be disturbed by the fact that another party interested in the bulk in question has sent a second sample to another analyst, who finds the composition and properties of his sample to differ materially from the results returned by the first analyst. However, the matter is finally adjusted, there is a tendency on the part of clients to consider any allusion to variations of sample as a professional "face-saving" convention invoked to conceal errors of analysis.

Causes of Variation.—Two more or less fallacious assumptions appear to lead to this attitude of distrust. The first is the gross one that a bulk of, say, coal, is sufficiently homogeneous for a few pieces, or a few shovelsful of slack to represent with certainty the bulk within the very narrow limits of analytical error. The second is the apparently more reasonable one that an average sample is most truly representative of the bulk. The difference between the two assumptions is that the first is not true at all in the rigid sense, and in the case of such relatively homogeneous things as standard solutions is only statistically true, whilst as to the second, the average sample does represent one way of considering the bulk, but only one way.

Suppose that two bulks of coal are each carefully sampled in, say, 12 places, and that each of the two sets of twelve samples is analysed. Let the means of the two sets be identical within the limits of experimental error, whilst in one set the results are all very close and in the other they are widely discrepant. Can it be contended that the two coals are fully described relatively to one another by returning the average results only? In actually using the bulk for power-raising would the two coals be of equal value? They probably would, if the whole of each could be burnt at once, but the variable one would require much greater care in stoking than the uniform one.

The above is a plea for the recognition of the fact that variableness in material is a real property of the bulk, no more to be neglected than, say, variations of height in man. The bed of Procrustes may have fitted the average man conveniently, but his hospitality caused great inconvenience to the tall and the short whom he may have felt that he statistically balanced in a most equitable manner.

MEANING OF AVERAGE SAMPLES.—It is, however, usually the average that is sought, and, so long as it is remembered and, if need be, insisted on by the analyst that this does not necessarily represent any part of the bulk, but is merely a convenient means of assigning a value to the whole, no misunderstanding need result. It is, however, to be regretted that when a bulk exceeds, say, 100 tons, it is unusual to make some partial examination of sub-samples before mixing to make the final sample.

It must be remembered that the final sample is, at the best, the result of a series of irrevocable choices, none of which is free from error, and each of which introduces new error. Variableness in bulk must be recognised in sampling, unless errors, possibly large and of a kind only to be discovered in an unpleasant way, are to be allowed. At the best, the composition of the sample can only, except by accident, approximate to that of the bulk. The approximation may be relatively good with careful sampling, but different sets of samples will yield different values, which may vary considerably from one another and from the unascertained and unascertainable true value.

ERRORS IN SAMPLING.—All people who are accustomed to serious sampling realise that, if a bulk is too large for sampling by quartation or similar means, it is necessary to take sub-samples from various parts, in order to obtain something which may be representative.

In the special case of coal, the most troublesome cause of error in sampling is, not that a bulk may be composed of various kinds of coal, but that it may consist mainly of two widely diverse things—coal and not-coal (slaty material). This is much more dense than coal, and uniformity of distribution is unlikely. In the extreme case a sample might be all coal or all slate. This could only be possible if the sample were small relatively to the size of lumps of the bulk. If it were 10 or 20 times as big as the largest lump, such a case would be very improbable; in fact, the already low probability would become negligible.

The theory of errors, confirmed by all experience, suggests that, if a series of samples are taken, the variations from their average will usually be approximately equal in number above and below, and thus values should be most closely clustered around the average; that is, the normal distribution of deviations is for their frequency to be some sort of inverse function of their magnitude. However many or few samples may be taken, the ordinary arithmetical average of them all is the most probable approximation to the true mean value which they will yield. Bailey (*J. Ind. Eng. Chem.*, 1909, **1**, 1161) has shown that the divergences in ash content of a large number of samples from one bulk of coal did, in fact, conform to the usual binomial or exponential distribution of errors.

CALCULATION OF "PROBABLE" ERROR.—The equations connecting precision with the distribution of divergences around an average show that in a series of measurements 1 to n, yielding a numerical average, from which individual measurements differ by $e_1\, e_2\, e_3 - - e_n$, respectively, the divergence of an imaginary typical measurement is

$$\mathrm{E} = \pm \sqrt{\frac{\Sigma e^2}{n-1}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (1)$$

whilst this, multiplied by 0·6745, will show the divergence within which half the measurements will fall the "probable" error where the odds are even as to larger or smaller errors. The value of either of these expressions is proportional to the reciprocal of the precision. If the whole series of measurements is considered, the mean square error is reduced to

$$E_s = \pm \sqrt{\frac{\Sigma e^2}{n(n-1)}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (2)$$

Of these, (1) expresses the obvious fact that however many measurements are made (or sub-samples are analysed), the accuracy of each individual is unaltered, if the method remains the same, but the relation of each to the desired quantity is more clearly defined by the greater precision of the average as shown in (2). As to the advantage of taking many sub-samples, it is marked, but a sampler who had taken 10 sub-samples would be inclined to gasp when he found that to double their accuracy he must take 100.

It is clearly necessary to take a number of samples of a variable bulk, but as the accuracy of the result only increases as $\sqrt{n}$, a practical limit is soon reached. On the other hand, the reduction of variation in sub-samples is a more potent means of increasing the probability of the average. This is the point where the problems of sampling from a large unwieldy bulk, and from one which can be turned over, differ.

General Considerations in Sampling.—A large bulk of coal, upwards of, say, 100 tons, may be in a heap at a siding, in a bunker, the hold of a ship, a small fleet of barges, or a train of trucks. It clearly cannot be stirred up, coned, quartered or crushed. We feel that so long as coal is in such bulks its sampling is most difficult, and the samples themselves of doubtful value. Such aggregates should be sampled either in process of formation or dispersal. The general considerations that should govern details seem to be these:

(1) The sample from which a smaller one is to be prepared for the laboratory by crushing, quartation, etc., must not be so large that care taken in its selection may be nullified by the difficulty of these operations, nor must the cost of preparation be prohibitive.

(2) The method to be adopted in sampling must be determined not only by the form in which the bulk is presented, but by the size and variation of size of the individual lumps or particles.

If a bulk can be sampled while it is being handled, so much the better; grabsful, trolley loads, or a container on a conveyor approach closer to the ideal form of a long ribbon or a curve fluctuating about a mean ordinate than any large stationary mass can. Bulks in dumps present great difficulty, and we can only suggest that, as much as possible, they should be sampled during formation or dispersal.

The number of sub-samples must, in practice, be more or less an inverse function of their size. If only a given quantity of coal can be handled, it is better, in the case of a small coal, peas or nuts, to take many small sub-samples, whilst, if the lumps are large, the sub-samples must necessarily be proportionately large, so that many pieces are included in each sub-sample. The advantage of large sub-samples, if the weight of the final sample is limited, can only be that a large number of pieces are taken. It cannot be contended that the greater value of, say, a 4 pound sub-sample over one of one pound is more than that of four one-pound samples, except in so far as lumps are so large that a 1 lb. sample must be unrepresentative. If a large sub-sample can be taken and coarsely crushed it is possible to select a representative portion of this for inclusion in the final sample, but it involves a lot of work.

(3) One strong point we would make is that a set of samples increasing, within reasonable limits, in number with the size of the bulk should be taken and kept apart for individual partial analysis, say, ash determination, and that the analyst's report should show the composition of the mean sample with an indication, either maximum and minimum and mean square error, or "probable" error of the variableness of the samples. If the analysis is sufficiently important to undertake, it is worth showing the sort of deviation from the mean result which may be expected.

The actual details of sampling must vary in each case. We may refer, in addition to the paper by Bailey already mentioned, which is very full, and to a bulletin by F. S. Sinnatt, which includes a Board of Trade leaflet on Coal Sampling. The subject is now under consideration by a Committee, whose report is looked for with interest.

VARIATIONS IN THE ASH OF SAMPLES.—During and since the war low grade coals, *i.e.* those yielding high percentages of ash, have been utilised for steam-raising purposes, and, no doubt, in some instances this has been found to be an advantageous procedure when costs and results have been compared. After all, it is not necessary to burn an expensive high-class coal if it can be shown that a more moderately-priced fuel will meet the requirements of the case, and it is in dealing with coals which come within the latter category that the importance and difficulty of sampling become apparent.

With uniformly large, clean coals containing relatively small amounts of ash, we have not experienced any difficulty in obtaining duplicate samples which gave practically identical results on analysis. (Of course, this refers to the samples received in the laboratory, as representing the delivery.)

When we come to the class of coal which is used largely for mechanical stoking, we have a very different state of things; the coal is small and may contain a large amount of ash; it is often obviously admixed with slaty shale, and not infrequently is so small that it appears to consist largely of dust. In such cases the sampling of the "laboratory" sample has been found to be an extremely difficult and unsatisfactory matter.

The following experiment was undertaken to ascertain the variation in the composition of a "nutty slack." The method of taking the different samples was as follows:

COAL SAMPLING EXPERIMENT.

<table>
<tr><td colspan="11">4 cwt. of Nutty Slack; Mixed by coning 4 times, and then quartered down to 1 bucketful (25–30lbs.);—</td></tr>
<tr><td colspan="3" rowspan="3">SAMPLE A (25–30lb.).
Passed repeatedly through Sturtevant crusher until a 5lb. sample tin was filled at the sample spout.</td><td colspan="8">Remainder; Spread out in layer about 4″ deep. 10 × 5lb. lots taken from all parts for sieving test on 3,8″ mesh sieve;—</td></tr>
<tr><td rowspan="7">50lb. SAMPLE B.
Coarse = 21lb.
Treated as in Sample A to produce;—
5lb. Sample B1 coarse
5lb. Sample B2 coarse
4oz. Sample B3 coarse
Fine = 29lbs.
Treated as in Sample A to produce;—
5lb. Sample B1 fine
5lb. ,, B2 fine
4oz. ,, B3 fine</td><td colspan="7">Remainder; Shovelled into a conical heap from the middle of which a single shovelful was taken;—</td></tr>
<tr><td rowspan="6">SHOVEL SAMPLE C.
Treated as in Sample A to produce;
5lb. Sample C1.
5lb. Sample C2.
4oz. Sample C3.</td><td colspan="6">Remainder; Spread out on floor and larger pieces crushed (by hand stamping) to 3/4″–1″ size. The whole was then mixed by coning 3 times and quartered down to 1 bucketful (25–30lbs.);—</td></tr>
<tr><td rowspan="5">5lb. Sample A1.</td><td colspan="2">Remainder quartered down to about 5lb.</td><td rowspan="5">SAMPLE D.
Treated as in Sample A to produce;
5lb. Sample D1.
5lb. Sample D2.
4oz. Sample D3.</td><td colspan="5">Remainder; Passed twice through Sturtevant Crusher, then mixed by coning 3 times and quartered down to 10–12lb.;—</td></tr>
<tr><td rowspan="4">5lb. Sample A2.</td><td rowspan="4">Rem.; One 4oz. bottle filled indiscriminately.
4oz. Sample A3.</td><td rowspan="4">SAMPLE E.
2 × 5lb. tins and 1 × 4oz. bottle filled indiscriminately;
5lb. Sample E1.
5lb. Sample E2.
4oz. Sample E3.</td><td colspan="4">Remainder; Coned once, after which 2 × 5lb. tins and 1 × 4oz. bottle were filled indiscriminately.</td></tr>
<tr><td rowspan="3">SAMPLE F.
5lb. Sample F1.
5lb. Sample F2.
4oz. Sample F3.</td><td colspan="3">Remainder; Passed through Sturtevant Crusher, and during this operation 2 × 5lb. samples and 1 × 4oz. sample were collected in succession from the sample spout.</td></tr>
<tr><td rowspan="2">SAMPLE G.
5lb. Sample G1.
5lb. Sample G2.
4oz Sample G3.</td><td colspan="2">Remainder; Spread out. 3000 grms. taken, in small quantities, from all parts and sifted throught 30-mesh sieve.</td></tr>
<tr><td>SAMPLE H.
Coarse = 75·7 per cent.
Placed in tin marked H Coarse.
Fine = 24·3 per cent.
Placed in tin marked H Fine.</td><td>Remainder:
Not used.</td></tr>
</table>

The results of the examination of the samples are given below. The figures represent the percentage of ash calculated on the dry coal. The moisture figures are not given, but it may be recorded that they were all about 9 per cent.

	1	2	3
	Per Cent.	Per Cent.	Per Cent.
A	20·8	19·1	21·8
B	Coarse 18·2 Fine 30·2 } 25·2	Coarse 18·9 Fine 25·9 } 23·0	Coarse 17·4 Fine 27·7 } 23·4
C	23·0	23·3	26·7
D	23·9	24·3	25·1
E	23·1	23·0	21·3
F	21·4	20·9	24·8
G	20·2	21·0	26·0
H	Coarse 20·4 Fine 30·4 } calc. on original H sample		22·8

The results given for B1, B2 and B3, and for H, were obtained by combining those found for the coarse and fine samples after sieving. It is interesting to note how heterogeneous the bulk must have been. The following results show this:

	B1.	B2.	B3.	H.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Fine	30·2	25·9	27·7	30·4
Coarse	18·2	18·9	17·4	20·4

In the two experiments described below mechanical separation was adopted to demonstrate this.

EXPERIMENT WITH DENSE LIQUID.—Five grams of the H Fine sample were stirred well with 50 c.c. of carbon tetrachloride and allowed to settle overnight. The lighter portion was filtered off, and the coarse fraction again stirred with another 50 c.c. of the tetrachloride, and filtered as before. The combined lighter fractions of the coal were finally treated with the same liquid, and the very small denser portion was added to the bulk of the heavier fraction. The separated portions were dried and the amount of ash was estimated in each. The figures obtained were:

(*a*) Lighter portion dried 2·00 grs. ash = 10·74 per cent.
(*b*) Heavier ,, ,, 2·32 grs. ,, = 47·30 per cent.

EXPERIMENT WITH WASHING OVER WITH WATER.—To ascertain the proportion of "natural" coal ash as distinguished from the adventitious ash present in the "nutty slack" on which the above investigation was made, some of the crushed coal remaining from the previous samplings was treated as follows:

About 8 oz. of the 30-mesh and 60-mesh material were "panned" twice to remove the bulk of the ash-bearing fraction and the specifically lighter fraction was twice subjected to elutriation in a glass cylinder twelve inches deep, a slow current of water being employed. The final "light" product so obtained, weighing about 2 ozs., was air-dried in a warm atmosphere, ground to pass through a 60-mesh sieve, and finally dried at 105° C.

The percentage of ash was found to be 2·17 per cent.

In the following table results obtained from the examination of three different sub-samples are given. The samples were taken from a bulk sample of 60 to 70 lbs. in the following manner:

The whole of the bulk sample was passed three times through a Sturtevant crusher (from which the *maximum* size would be $\frac{3}{16}''-\frac{1}{4}''$), and the whole was then rapidly mixed and quartered down to obtain three 5 lb. samples for the estimation of moisture. The remainder was then passed through the crusher as many times as necessary for the filling of six 5 lb. sample tins from the sample spout of the machine. (The sample spout is supposed to deliver 10 per cent. of the bulk, but the actual proportion varies with the nature of the coal.)

The figures given are the percentages of ash calculated on the dry coal, and each set of three refers to the three sub-samples of one bulk sample.

11·2	15·1	14·4	17·1	16·0	15·4	14·8	12·0	12·5	12·9
11·7	14·2	12·1	15·4	14·1	12·0	12·0	10·7	12·7	12·2
9·8	15·0	15·0	13·5	13·7	13·2	10·1	9·7	14·5	13·4

These figures show a real variation in the composition of the bulk sample, and we feel that such variation is characteristic of this class of fuel, and must be accepted, and that the nearest approach to the true ash is obtained by averaging the results of as many sub-samples as possible.

DISCUSSION.

Mr. W. PARTRIDGE asked whether in the valuations of coal any account had been taken of the size of the particles, and remarked that an ash that would remain in the grate and keep hot was better than one that would fall through the firebars at once.

Mr. G. N. HUNTLY said that he agreed with the authors on the difficulties of sampling coal. Usually an analyst simply had the sample sent, and had no knowledge of the care taken in sampling. For boiler trials he always made a point of taking two independent samples from the bulk, which in the laboratory were not found to differ by more than 1 per cent. He had had the opportunity of discussing this matter with the late Prof. Constam, head of the Federal Fuel Laboratory at Zurich, who agreed that ± 1 per cent. was the usual accuracy of careful hand-sampling.

Mr. RUDD THOMPSON said that he was astonished at the somewhat extreme variations in ash, as shewn by the authors. He was in the habit of doing regular analyses of coal, perhaps, in an average year, having two or three million tons to deal with, representing shipments between the collieries, foreign Governments, and foreign railways, and it was extraordinary, considering the different sampling, how much the shipments agreed. A sample was taken at the colliery, at the port of shipment, actually during shipment in the ship's hold, and again while the coal was being discharged. The variations on a particular cargo were not more than 2 per cent. He deplored the method, mentioned by the authors, of placing samples in tins of about 5 lbs. and sending them to different chemists, as in such a large sample there was a very much greater source of error. He recommended ½ lb. samples. In his opinion, if the authors' method were adopted on a commercial scale in South Wales the methods of sampling there would have to be changed. For analysis he did not advise any elaborate formula, but preferred putting in a muffle and taking the ash as found.

Mr. COSTE, replying, said their experiments were made on a fine coal, from the Midlands, and not on a large coal of low ash content, which would be more uniform in composition. The variations in the ash were in the same order as those found by Bailey (*loc. cit.*). As regards the reference to engineers' gauges, he did not think that engineering measurements were usually of a high order of accuracy, but engineers had such a high idea of the accuracy of analysis that they were horrified if the chemist was "out" by a very small amount. Regarding Kent coal, he did not know how the problem mentioned could be dealt with. In their paper they had not touched on moisture nor on calorific value, but had used ash as a more direct measure of variation. Mr. Huntly and he had dealt with the question of moisture years ago in a paper (based, in part, on the report of an International Committee, written by the late G. T. Holloway and himself, in which Mr. Huntly and the speaker's co-authors had taken part) dealing with the variations in moisture, which, in that particular case, were very remarkable.

As regards colliery samples: some years ago they had examined a series of samples of coal received direct from collieries, and in every case they had found they agreed very fairly with what the colliery owners considered the general quality of the mine. It was probable that people who had not found great variations in the ash were dealing with a better class of coal than they had used for the experiments referred to in the paper; the coal in question was used for a very large power station.

In examining coal one naturally made the usual conventional partial analysis and determined the calorific value.

The Volumetric Estimation of Vanadium in Steel.

By A. T. ETHERIDGE, B.Sc., F.I.C., M.B.E.

(*Read at the Meeting, October* 3, 1923.)

In the well-known estimation of vanadium in steel by reduction with ferrous sulphate and titration with dichromate, with the use of ferricyanide solution as indicator on a spotting plate, the author discovered that with an increasing vanadium content the potassium ferricyanide is reduced by tetravalent vanadium, and therefore gives an erroneously low vanadium reading. This observation is confirmed by Cain (Reprint 161, *Bull. Bureau of Standards*, U.S.A.), and also by Ibbotson (*Analysis of Steel Works Materials*).

The author gave attention to the modified process of Hammer (*Chemical Engineering*, Vol. 17, No. 5, p. 207), where excess of ferrous ions in the presence of 5 c.c. of phosphoric acid is destroyed by adding a slight excess of ammonium persulphate, the tetravalent vanadium being then titrated with permanganate. It was found impossible to control the limits of excess of ammonium persulphate added. For example, in 500 c.c. volume, vanadyl salt was not appreciably oxidised by 7 c.c. of one per cent. solution of ammonium persulphate in three minutes, but was appreciably oxidised by 10 c.c. of the same solution; and, moreover, as ferrous ions are but slowly oxidised by ammonium persulphate as compared with oxidation by dichromate, it becomes impossible to ascertain the point at which titration with permanganate should commence. Tests made by Hammer's method have given erratic results.

Diphenyl-carbazide, first used as an indicator by Brandt (*Zeitsch. anal. Chem.*, **45**, **95**; idem, **1913**, **53**, p. 1; *J. Soc. Chem. Ind.*, **1914**, p. 44) has been employed as a colour test for traces of chromium by Cazaneuve (*Bull. Soc. Chim.*, 1900 (3), **23**, (18), 769; *J. Soc. Chem. Ind.*, 1900, p. 1007; idem, p. 1040) and Metzger and Zons (*J. Ind. Eng. Chem.*, 1912, **4**, 493; *J. Soc. Chem. Ind.*, **1912**, p. 772), and as an indicator for the titration of ferrous salt with dichromate by Barneby and Wilson (*J. Amer. Chem. Soc.*, **1913**, **35**, 156), who there describe its preparation. As a result of experiments with diphenyl-carbazide the following method applied to steel has been worked out:

Two grms. of steel are dissolved in 5 c.c. of dilute sulphuric acid (1:7 by vol) in a 500 c.c. flask. When dissolved, 2 to 3 c.c. of nitric acid (sp. gr. 1·42) are added and, if necessary, any residual carbide dissipated by digestion with a few more c.c. of nitric acid. The solution is evaporated until sulphur trioxide appears, but short of vigorous fuming. Nitric acid destroys the indicator and must be removed. The sulphates are dissolved by addition of 400 c.c. of water and boiled, with a porous plate added to prevent bumping, after which saturated solution of potassium permanganate is added, drop by drop, until the formation of a permanent precipitate, and boiling is continued for 15 minutes. A large excess of manganese dioxide must be avoided or chromium and vanadium may be associated with the precipitate. The solution is cooled, filtered on asbestos, and the precipitate washed with cold water, the filtrate being received in a large Erlenmeyer flask. Fifty c.c. of syrupy phosphoric acid are added and, after cooling, a slight excess of ferrous ammonium sulphate to reduce chromate and vanadate.

This excess is titrated with N/10 permanganate to permanent pink, which should exist for at least five minutes. This end-reaction may be controlled by comparison with a blank solution of the same steel treated in similar fashion, but short of the end-point, with permanganate.

Fifty c.c. of concentrated sulphuric acid are now added and the solution cooled, after which sufficient standard ferrous ammonium sulphate solution is added to reduce all vanadium and also to leave a small excess, the approximate amount of vanadium present being found by preliminary tests, either by this process, or by colour test with hydrogen peroxide. After thorough shaking, standard 0·1 *N* dichromate solution is added till within a few c.c. of the end, this end-point being approximately known from the preliminary test.

At this point half of the prepared colour indicator is added. This is made as follows: Diphenyl-carbazide (0·05 grm.) is dissolved in a few c.c. of glacial acetic acid and diluted to 500 c.c. Five c.c. of this solution are acidified with 3 drops of dilute sulphuric acid (1:3) and 1 drop of 0·1 *N* dichromate solution added. Approximately half of this solution is added to the liquid in the flask and dichromate solution continually added (the beaker being shaken well between each addition) until the purple colour has faded away. The remainder of indicator is now added, and the titration continued until the purple colour is discharged and the solution becomes apple green. This is the end-point, and marks the appearance of the first slight excess of dichromate.

The ferrous ammonium sulphate is standardised under the same conditions of acidity and volume as in the test. The difference in the two titrations represents vanadium in terms of 0·1 *N* dichromate solution. The process has been tested with 2 grms. of electrolytic iron to which known amounts of vanadium (as ammonium vanadate) and chromium (as dichromate) had been added.

Up to 2 per cent. of vanadium results correct to within 0·02 per cent. are obtained, but beyond 2 per cent. of chromium the green chromic sulphate obscures the end-point, and for 4 per cent. chromium steel it is recommended that work be carried out on a 1 grm. sample.

The presence of cobalt interferes with the permanganate end-point. Owing to the slight colour remaining after the addition of phosphoric acid to ferric sulphate, it has been found that 2 grms. of sample is the maximum weight to be taken.

Tungsten steels are treated as usual for the removal of tungsten as WO_3 in hydrochloric acid solution. After filtration, the liquid is evaporated with sulphuric acid to eliminate hydrogen chloride, and is then treated as described.

HIGH SILICON STEELS.—The silica separated in the dilute sulphuric solution is filtered off before proceeding further.

FERRO-VANADIUM.—A smaller weight is taken than for steels, *e.g.* 0·5 grm., or less. These alloys usually dissolve in dilute sulphuric acid. If not, they must be fused first by the usual methods and be taken up afterwards in dilute sulphuric acid. The green colour of the vanadyl salt changes to light yellow on oxidation with permanganate, so that there is no difficulty in obtaining the first end-point. The second end-point is somewhat obscured if too much vanadium is present. This can be overcome by limiting the amount of alloy taken for analysis.

NOTE.—Experiments have established two necessary conditions for the effective use of this indicator, *viz.*:

(1) Preliminary action of ferrous iron. For the amount of indicator used a very small quantity of ferrous iron is sufficient. Experiment showed a minimum of about 0·5 c.c. $N/100$ (or one drop of $N/10$) ferrous ammonium sulphate solution.

(2) Considerable excess of sulphuric acid, *i.e.* about 10 per cent. by volume.

Both these conditions are satisfied in the directions given. No. (1) condition is allowed for by having a slight excess of standard ferrous sulphate left in the solution when the indicator is added. No. (2) condition is allowed for by adding sulphuric acid after the chromium titration is finished.

Finally, the amount of indicator used is a fifth of that used by Burneby and Wilson, so that the "blank" is reduced to a negligible quantity.

RESEARCH DEPARTMENT,
WOOLWICH ARSENAL.

A New Test for Distinguishing Castor Oil from Other Oils and Determining its Purity.

BY H. B. STOCKS, F.I.C.

(*Read at the Meeting, October* 3, 1923.)

CASTOR oil is distinguished from most other oils by its high specific gravity and high viscosity, its solubility in alcohol, its insolubility in petroleum spirit, and also by its acetyl value.

These characteristics of castor oil are made use of for determining its purity, and, although quite sufficient for the ordinary purposes of analysis, it is doubtful if any one of these is more distinctive than the test about to be described.

In an investigation of various soaps the author found a very marked difference between castor oil soap and the soaps made from other oils. Castor oil soap is not as well known as the soaps from other oils, but it has characteristic properties which render it useful for certain purposes. The soda soap is hard and waxy in appearance, but dissolves with very great ease in warm water, forming a very fluid solution which shows no tendency to gelatinise on cooling.

One well-known property of soap solutions is their alkalinity. Generally speaking, all soaps, even neutral soaps, when dissolved in water, respond to the test for free alkali—that is, they give a pink colour with phenolphthalein. Commercial soaps may contain free caustic alkali, which is estimated by titration in an alcoholic solution, but, apart from that, water causes a dissociation or hydrolysis of soap, so that hydroxide is liberated.

The literature relating to the hydrolysis of soaps due to the action of water is considerable, but it need not be recapitulated here, since a full account of the work of Chevreul, Krafft and Stern, Lewkowitsch and others will be found in Lewkowitsch's *Chemical Technology and Analysis of Oils, Fats and Waxes*, Vol. 1, pp. 127–136, and the later investigations of Prof. McBain and others in the *3rd Report of the British Association Committee on Colloid Chemistry*, pp. 2–31. Prof. Wilder D. Bancroft has also a few remarks on the subject in the 2nd Report of the same Committee, p. 15.

Without entering into details, it is sufficient to state that when a soap is dissolved in water the solution gives with phenolphthalein a pink colour which deepens with further dilution; this shows that free hydroxide is liberated, and that apparently the amount increases when more water is added.

The author has found, however, that castor oil soap forms an exception, since a solution in water gives with phenolphthalein only a very slight pink colour which does not become appreciably deeper on dilution. When castor oil soap is compared with any other soap in its behaviour to this indicator the difference is so marked as to excite attention, and the author has felt that it is worthy of investigation.

In the estimation of the alkalinity of soap solutions with standard acid the difficulty is likely to be met with that, as soon as a portion of the free alkali has been neutralised, the equilibrium may be affected and more alkali liberated, and this might go on till the soap was entirely destroyed; but this was found not to be the case. With slight dilutions this does appear to occur to some extent, since a faint pink colour persists over a somewhat large range, and, in some cases, it reappears after a few seconds, rendering the titrations somewhat difficult. But one point was clear, *viz.* that the amount of acid required to neutralise the solution and bring about equilibrium increased with the dilution, up to a certain point, but that at dilutions of about 1 in 160 or more, the end-point could be much more easily distinguished and the results were more concordant; further dilution had little effect.

With the soaps of oleic, palmitic and stearic acids, and of oils and fats in which one or more of these acids predominate, the amount of acid required to bring about neutrality at such dilutions was approximately half the total combined alkali present; in some cases rather less, in others rather more than this quantity.

Several samples of commercial soaps and soaps made in the laboratory were examined in this way, the results being fairly uniform, but castor oil soaps behaved quite differently, the amount of acid required to effect neutrality in this case being very small, not more than 3 to 11 per cent. of the total alkali. Some of these results have already been published, (Hursts' *Soaps*, 3rd Edition, 1922, p. 8).

The following recent determinations with solutions containing 1 part of soap in 320 parts of water will illustrate this:

Soap	Total Na_2O found Per Cent.	"Free" Na_2O found Per Cent.	Per Cent. of total.
Stearic acid*	0·111	0·056	50·5
Palmitic acid*	0·119	0·057	48·0 (1 part in 160)
Oleic acid*	0·103	0·049	47·5
Yellow bar soap	0·082	0·037	45·1
Olive oil soap	0·083	0·043	52·3
Castor oil fatty acids*	0·093	0·004	4·3

Strong solutions (10 or 20 per cent.) were made, and the requisite quantity taken and further diluted with water. The soaps marked with an asterisk were made by dissolving the calculated quantity of fatty acid in sodium hydroxide solution to form neutral soap solutions. Solutions of the solid fatty acid soaps were difficult to deal with, and had to be made up and measured hot, since they solidified at comparatively high temperatures. The solutions of the first two were slightly stronger than required.

A consideration of these figures led the author to believe that this difference in behaviour between castor oil soap and other soaps offered another means of discriminating between castor oil and other oils, and might also be employed as a test for the purity of castor oil.

With this idea in view, mixtures in definite proportions of castor oil and arachis oil were prepared, and the following procedure was followed:—Five grms. of the oil were taken and mixed with 40 c.c. of approximately 0·5 *N* alcoholic potassium hydroxide (*i.e.* rather more than 1 grm. of KHO) in a wide-mouthed flask, which was connected with an upright condenser and heated on a sand bath, with gentle shaking from time to time; the time allowed was 30 minutes, after which in every case the oil was completely saponified.

The liquid was exactly neutralised to phenolphthalein by the addition of *N* hydrochloric acid, and evaporated to dryness in a dish. The soap was dissolved in hot water and washed into a 100 c.c. flask, and, after cooling, it was diluted to 100 c.c. Ten c.c. of the solution (=0·5 grm. of oil) were then taken and added to 250 c.c. of neutral distilled water, mixed, phenolphthalein added, and the solution titrated with 0·1 *N* hydrochloric acid.

The results obtained are given in the following table:

Castor Oil Parts.	Arachis Oil Parts.	0·1 *N* HCl required c.c.	"Free" KOH per 100 of oil Parts.	Arachis Oil Calculated Per Cent.
100	0	0·5	0·56	
90	10	1·4	1·57	9·9
80	20	(1) 2·3, (2) 2·3	2·58	19·8
70	30	(1) 3·2, (2) 3·3	3·58	29·7
60	40	4·3	4·81	41·7
50	50	5·0	5·60	49·5
40	60	6·1	6·83	61·5
30	70	6·95	7·78	70·9
20	80	7·8	8·73	80·2
10	90	8·7	9·74	90·1
0	100	9·6	10·75	—

The saponification values of these oils were:—Castor oil, 178·4; arachis oil, 189·6. The amount of free alkali found by titration, compared with the total combined alkali, was therefore 3·1 per cent. in the case of the castor oil and 56·7 per cent. in that of the arachis oil; these are very similar figures to those obtained in dealing with soaps.

Before passing on, there is one other point worthy of mention. Castor oil is not pure ricinolein, but contains small quantities of stearin and palmitin. Lewkowitsch (1914, Vol. II., 398) calculated from the mean acetyl value that castor oil contains 93 or 94 per cent. of ricinolein; therefore, we may assume that the other glycerides amount to 6 or 7 per cent. If, then, the potassium salt of ricinoleic acid does not dissociate under the conditions of this test, and the slight hydrolysis shown by castor oil soap is due to other fatty acids, then the approximate amount of other glycerides present in castor oil can be calculated; the alkali combined with these fatty acids being approximately double that shown above, or 1·12 per cent., and the amount of glycerides 5·7 per cent., taking the mean molecular weight at 860.

Subsequently a number of oils and fats were examined by this method, which was slightly modified, the *N* hydrochloric acid being replaced by ordinary dilute acid, and, in case of slight excess, a drop or two of *N* sodium hydroxide solution being added to bring it to exact neutrality (it must not be pink) and, instead of the solution being washed into a dish, the alcohol was removed from the neutralised soap solution by evaporation in the flask. The solution becomes pink as the alcohol evaporates, but no more acid should be added.

In the following experiments two or more tests on the same solution were made in each case, the solution being titrated first in presence of phenolphthalein, methyl orange then added, and the titration continued to a faint orange tint in the cold. The total amount of acid required represented approximately the saponification value, but in several cases the figures were slightly higher than those usually accepted, owing, probably, to the presence of a trace of carbonate. The calculations were, however, made on these figures, since any errors arising

	0·1 *N* HCl		KOH on 100 of oil		
	With phenolphthalein c.c. A.	Total, with methyl orange c.c. B.	Parts A.	Parts B.	$\frac{A}{B} \times 100.$
Castor oil:—Medicinal	0·6	16·5	0·672	18·48	3·6
(Original sample)	0·7	16·6	0·784	18·59	4·2
Old Solution of KOH.					
	0·7	16·0	0·784	17·92	4·4
	0·7	16·0	0·784	17·92	4·4
Fresh solution in absolute alcohol.					
Medicinal	0·8	16·7	0·896	18·70	4·8
	0·8	16·9	0·896	18·93	4·7
(10 c.c. *with* 500 c.c. *water*)	0·8				
Medicinal	0·8	16·7	0·896	18·70	4·8
	0·8	16·6	0·896	18·59	4·8
Best seconds	0·8	16·7	0·896	18·70	4·8
	0·8	16·8	0·896	18·81	4·8
Madras	1·2	16·3	1·34	18·25	7·3
	1·3	16·5	1·45	18·48	7·9
Arachis oil	9·5	17·8	10·64	19·94	53·7
(Original sample)	9·6	17·8	10·76	19·94	54·0
	9·3		10·42		52·3
Olive oil, medicinal	8·4	17·7	9·41	19·82	47·5
	8·5	17·4	9·52	19·49	48·8
	8·2	—	9·18	—	—
Linseed oil	5·5	17·9	6·16	20·05	30·8
	5·6	17·9	6·27	20·05	31·3
Rape oil (crude)	8·0	15·7	8·96	17·58	51·0
	7·7	15·8	8·62	17·69	48·7
Rape oil (adulterated)	3·7	11·8	4·14	13·21	31·4
	3·6	11·9	4·03	13·33	30·3
	4·3	12·2	4·81	13·66	35·2
	3·8	12·0	4·26	13·44	31·7
Cottonseed oil	7·3	18·0	8·17	20·16	40·6
	7·4	18·0	8·29	20·16	41·1
Palm oil	8·5	17·8	9·52	19·93	47·7
	8·6	18·0	9·63	20·16	47·7
	8·2	—	9·18	—	—
Lard	9·4	18·1	10·53	20·27	51·9
	9·2	18·2	10·30	20·38	50·6
Tallow	8·7	17·5	9·74	19·60	49·7
	8·8	17·7	9·85	19·82	49·7
Butter fat	7·4	20·5	8·29	22·96	36·1
	7·9	20·7	8·85	23·18	38·1
Coconut oil	4·6	22·5	5·15	25·20	20·4
	4·4	22·9	4·93	25·65	19·2
(100 c.c. *with* 500 c.c. *water*)	4·2				
Palm-nut oil	5·3	23·1	5·93	25·87	22·9
	5·2	22·9	5·82	25·65	22·7
Rosin	8·5	15·9	9·52	17·81	53·5
	8·5	16·1	9·52	18·03	52·8

from this cause do not appreciably affect the conclusions to be drawn. The figures are in general agreement with those previously obtained, but some oils, *e.g.* linseed and cottonseed oil, give lower results than olive oil for instance; the behaviour of butter fat, coconut oil and palm nut oil is also interesting. With these fats 55 c.c. of the alcoholic potassium hydroxide solution were used. The figures in these cases are all much lower than with most oils and fats, and there are appreciable differences between them. It must be mentioned, however, that the coconut oil and palm nut oil were ordinary commercial samples, not the edible kinds, and further investigation will be necessary to determine whether the distinction would hold with the edible oils. One sample sold as rape oil was evidently largely adulterated with mineral oil. It gave a turbid solution, and the relationship between the "free" and total alkali was not the same as that of the other sample. The results obtained are given on page 594. In each case 5 grms. of oil or fat were saponified, the solution made up to 100 c.c., and 10 c.c. diluted to 250 c.c. with neutral distilled water.

DISCUSSION.

Mr. W. H. SIMMONS raised the question of hydrolysis by saying he had noticed that the author had found coconut oil showed very much lower alkalinity than other fats. He mentioned that coconut and palm kernel soap were known to irritate the skin, which action was attributed by some authorities to greater hydrolysis. Professor McBain, however, by electrical experiments, contradicted this, with which conclusion, the speaker presumed, Mr. Stocks agreed.

Mr. R. G. Pelly said that the difference between castor oil and other oils observed by the author was most interesting, and was, presumably, associated with the constitution of the characteristic hydroxy-fatty acid—ricinoleic acid—of castor oil. It would be interesting to know if the author had examined the behaviour of other hydroxy acids, e.g. dihydroxystearic acid.

Mr. STOCKS, in reply, remarked that the method described in the paper was purely a chemical one; Professor McBain and his collaborators employed mostly physical methods, electrical conductivity with the use of a hydrozen electrode, etc. In comparing castor oil soap with other soaps, in their behaviour to water, it was very early apparent that there was a marked difference in the amount of hydrolysis. With regard to the influence of lipase on the hydrolysis, he was not sure whether castor oil contained lipase, though the seeds certainly did. Lipase worked better in an acid medium than in an alkaline one, and it certainly played no part in this test, as there was very little hydrolysis with castor oil soap. Castor oil soap in this respect behaved like a crystalloid.

With regard to the enquiry as to whether he had carried out work with other hydroxy-acids, he had not had an opportunity of doing so up to the present.

Notes.

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

SEPARATION OF ARSENIC.

WHEN the residue of the treatment of an organic mixture, such as is obtained by treating viscera, etc., with nitric acid, or with hydrochloric acid and potassium chlorate, is filtered, and the solution, containing any arsenic present, is neutralised with ammonia, and excess of ammonium sulphide added, the dissolved arsenic sulphide is not entirely precipitated on adding hydrochoric acid, even after heating and allowing the solution to stand for some time; on the contrary, a considerable proportion remains in solution, in a form, however, which is easily precipitated by hydrogen sulphide, especially if the solution be warmed before passing the gas. Fresenius says that the whole of the sulphide comes down, under the above circumstances, on neutralisation, and says nothing about subsequent treatment with hydrogen sulphide. The facts here stated have been verified by a considerable number of experiments, spread over some time, directed towards shortening the estimation of arsenic in toxicological analyses. This end may be served by following the procedure sketched above.

An objection raised against the valuable Reinsch's test, an exhaustive examination of which has been recently made by Dr. Evans, is that it fails to detect arsenates, although some obscure indications are obtained in the presence of organic matter. I have found that if a crystal of ferrous sulphate be added, a good coating is obtained with arsenic acid in a few minutes. As there are few, if any, cases of poisoning recorded with arsenates, it is well to apply the test in the usual form first, and, if the result is negative, to add then about 0·5 grm. to 1·0 grm. of pure ferrous sulphate, and boil again for a short time. Dr. Evans has noticed that the test is much less sensitive in the case of mercury than of arsenic; a doubtful film is more easily identified if a drop of nitric acid is put upon the copper slip; the characteristic "silvering" then comes out much more prominently.

THOS. P. BLUNT.

THE WYLE COP, SHREWSBURY.

CONDENSED MILK REGULATIONS 1923.*

UNDER these Regulations the tin containing the condensed milk has to bear a label stating the number of pints of milk to which the contents of the tin are equivalent. The method of arriving at these "equivalent pints" from the analytical results thus becomes of importance.

For the purpose of the Regulations "milk" means milk which contains not less than 12·4 per cent. of milk solids (including not less than 3·6 per cent. of milk fat) and "skimmed milk" means milk which contains not less than 9 per cent. of milk solids other than milk fat. These milks are referred to here as "standard milks."

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* *Cf.*. ANALYST, 1923, 271, 550.

It is assumed that the analytical results will be expressed in percentages by weight, and that the percentages prescribed in the standard milks are also percentages by weight.

Inasmuch as the amount of milk to which the contents of the tin are equivalent has to be expressed in pints, it is necessary to know the specific gravity of the standard milks.

The specific gravity of a milk containing 3·6 per cent. of fat and 8·8 per cent. of solids-not-fat, calculated from the various formulæ which have been put forward, lies between 1031·6 and 1032·3, whilst that of a skimmed milk containing 9 per cent. of solids-not-fat or 9 per cent. of total milk solids would be about 1035·5. There can be no very strict value attaching to the specific gravity of either of the standard milks, for in neither case is the percentage of both fat and solids-not-fat laid down.

It is suggested that, for the purpose of the calculation of the equivalent pints, 1032 be taken as the specific gravity of full cream milk, and 1035·5 as that of skimmed milk. In an ordinary case of a full cream milk 1031·5 gave 1·854, and 1032·5 gave 1·852 for the equivalent pints—a negligible difference.

Simple general formulæ for calculating the equivalent pints from the analytical figures can be found as follows:

Let T.M.S. be the percentage of total milk solids in the condensed milk.

S.N.F. be the percentage of milk solids other than milk fat in the condensed milk.

F be the percentage of fat in the condensed milk.

W. be the total weight of condensed milk in the tin (in grms.).

1032 be the specific gravity of standard full cream milk.

1035·5 be the specific gravity of standard skimmed milk.

Then in the case of full cream milk:

$$\text{The total milk solids in the tin} = \frac{\text{T.M.S.} \times \text{W}}{100}.$$

Now 1 pint = 20 fluid ounces.
= 20·64 ounces by weight.

12·4 per cent. of this = 2·5594 ounces of total milk solids in the tin, or 2·5594 × 28·3495 grms.

Therefore the equivalent pints is given by the expression—

$$\frac{\text{T.M.S.} \times \text{W.}}{100 \times 2{\cdot}5594 \times 28{\cdot}3495}, \ \textit{i.e.} \ \frac{\text{T.M.S.} \times \text{W.}}{7255}.$$

Similarly, equivalent pints is given by

$$\frac{\text{F} \times \text{W.}}{2106}, \text{ and also by } \frac{\text{S.N.F.} \times \text{W.}}{5149}$$

(No significant difference is obtained if the denominators in these formulæ are "rounded off" to 7260, 2110 and 5150 respectively).

Now, the equivalent pints calculated from these formulæ will not, in general, be the same; they could be the same only if the original milk from which the condensed milk was made contained either exactly 3·6 per cent. of fat and 8·8 per cent. of milk solids other than milk fat, or fat and milk solids other than milk fat in the ratio of 3·6 to 8·8.

Which requirement must the declaration of equivalent pints satisfy, or must it satisfy all three?

The differences may be considerable; for instance, three makes taken at random gave the following results:

Equivalent pints:	No. 1.	No. 2.	No. 3.
Calculated on T.M.S.	1·85	1·85	1·87
Calculated on F.	1·88	1·70	1·77
Calculated on S.N.F.	1·84	1·92	1·91

In No. 2 the equivalent pints calculated from the total milks solids and the fat respectively differed by more than the "unit difference" allowed, namely, one-eighth of a pint; calculation from the S.N.F. differed still more.

As I read the regulations, it would be necessary to satisfy the calculation from total milk solids and that made from the fat, but not that from the solids-not-fat.

In the case of a skimmed milk the equivalent pints is given by the formula—

$$\text{Equivalent pints} = \frac{\text{S.N.F.} \times \text{W.}}{5284}.$$

It would appear that in this case the equivalent pints must satisfy the calculation from the solids-not-fat only, though it is to be noted that the condensed milk itself has to contain not less than a certain percentage of total milk solids including fat.

E. Hinks.

16, Southwark Street, S.E. 1.

Notes from the Reports of Public Analysts.

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

CITY AND COUNTY OF KINGSTON-UPON-HULL.

Annual Report of the Public Analyst and Bacteriologist for 1922.

During the year 1922 the total number of samples examined was 6253, of which 993 were foods and drugs, 4924 pathological specimens (bacteriological), 91 waters and sewage for chemical examinations, and 43 waters for bacteriological examination.

Of the samples examined under the Food and Drugs Acts, 519 were official samples, 454 informal samples, and 15 private samples. Of these 870 were genuine, 52 suspicious, and 66 (=6·7 per cent.) adulterated.

Milk.—Twenty-nine samples (6·3 per cent.) contained visible "dirt." Fifty-three samples were also taken specially for examination for "dirt," with the following results: Fourteen contained from 2·7 to 9·5 parts per 100,000 of dirty sediment (in 5 cases dung was present). Eleven contained 2·1 to 2·5 parts (sand and vegetable fibres). Twenty-eight contained not more than 2 parts per 100,000 (clean). Eight of the vendors of the first fourteen samples were cautioned, and in 4 cases proceedings were taken and fines of £3 3s. to £7 7s. were imposed.

Dripping.—Six of 10 samples examined were genuine, whilst 4 were adulterated, containing from 1·2 to 20·8 per cent. of water, with 1·4 to 7·1 per cent. of mineral and organic impurities. A fine was imposed in the case of the sample containing 20·8 per cent. of water. Although the Sale of Food Order, 1921, referring to dripping, has been revoked, the standard laid down by that Order is a reasonable one.

Cakes.—The amount of boron preservative (as boric acid) in 5 samples varied from 0·25 to 0·48 per cent. A circular letter was sent to the traders concerned, warning them against the use of liquid egg containing excessive quantities of preservative.

Canned Foods.—Twenty-three samples were examined, but in most cases the metallic contamination was trivial. Six samples of preserved vegetables contained 0·09 to 0·35 grain of copper per lb. In three instances the samples had no declaratory label and were therefore condemned as adulterated.

Drugs.—Of 105 samples (official B.P. preparations), only two, of lime water, which were both deficient in caustic lime and contaminated with lead to the extent of 9·0 parts per million, were found to be adulterated.

Atmospheric Pollution.—A table of monthly analyses is given showing the total solid matter collected in the rain and soot gauge, and the proximate composition of the insoluble and soluble constituents.

Arnold R. Tankard.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

COPPER IN BEANS: DISCLOSURE ON TIN.

On October 2, G. Fidler was summoned at Kensington for selling canned beans which were injurious to health by reason of containing copper equivalent to 2·125 grains of crystallised copper sulphate per lb. A label on the tin stated that the beans contained a small amount of copper sulphate, and it was urged for the defence that the defendant had no knowledge that the beans contained an excessive quantity of copper.

A fine of £3, with 10s. 6d. costs, was imposed.

"SYRUP TREACLE": DISCLOSURE OF CORN SYRUP.

On September 28th, Abraham Altham, Ltd., Burnley, were summoned at Salford for the sale of syrup not of the nature, substance and quality demanded.

The Inspector, Mr. W. S. Bowker, stated that he had asked for "syrup treacle," and was supplied with a carton on which was printed "Vernon's Delicious Cane Sugar," and, in smaller type, "With a percentage of corn syrup to prevent granulation."

The Borough Analyst, Mr. G. D. Elsdon, certified the sample to contain 65 per cent. of corn syrup (glucose syrup), and only 35 per cent. of cane syrup, or syrup treacle; whereas syrup treacle should be made from beet or cane sugar and not contain any glucose.

Mr. E. C. Burgis, for the defence, said that there was substantial agreement as to the analyst's certificate. He contended that "syrup treacle" was not a product recognised in the trade or to be found in trade catalogues, and suggested that if "golden syrup" had been asked for, a product derived from pure cane or pure beet sugar ought to be supplied, but if "syrup" or "syrup treacle" was asked for, any of the many other syrups which existed might legitimately be supplied. He also contended that the disclosure on the carton brought the defendant under the protection of Section 8 of the Sale of Food and Drugs Act. It was analogous to a case decided in 1894, in which a mixture sold as cocoa, but containing 30 per cent. of cocoa and 70 per cent. of starch, was held to be protected by the Section.

The Stipendiary held that the defendants had managed to keep within the Section 8, but considered that the label was deceptive and unfair to the public. As he was unwilling, however, to put the defendants to the expense of fighting a case in the House of Lords, he dismissed the summons.

"CRYSTAL SYRUP."

In the case of a summons against F. Fallon, Manchester, also heard at Salford on September 28th, there was no disclosure on the label. An article described as "Superfine Crystal Syrup, and containing 70 per cent. of glucose syrup, had been supplied instead of the "syrup treacle" demanded.

For the defence, Mr. Burgis urged that the prosecution had asked for something unknown commercially, and that the defendant had given them something corresponding as nearly as possible to that unknown thing.

Mr. W. H. Roberts, Public Analyst for Liverpool, said that he drew a sharp distinction between "syrup" and "golden syrup," and that "golden syrup" was the only term known to him which was applied to a syrup made from cane or beet sugar.

The Stipendiary, in dismissing the case, said that he hoped the press would warn people that if they wanted pure syrup they must ask for "golden syrup," and that if they accepted "crystal syrup" they would get a mixture containing corn syrup. The refinements of the trade had tended to work against the interests of the public as regards sweetness, though not as regards food value. He would not allow costs, since he considered that people were being deceived.

DEFINITION OF BRANDY.

On September 26, H. B. Fearon, Block & Co., were summoned at Liverpool for the sale of "brandy" containing 45 per cent. of spirit other than brandy. It was stated that the article, which was labelled "Pale Brandy," with one star, had in fact been made from Spanish brandy, and that, if the word "Spanish" had been on the label the purchaser would have expected a less value.

Mr. Roberts, Public Analyst for Liverpool, said that he had found the sample to contain only 45 parts of esters per 100,000 of absolute alcohol, as against the minimum of 80 parts.

Mr. L. Kelly, for the defence, urged that the invoice constituted a warranty. If the brandy had been distilled from grape juice, the origin of the grapes was immaterial. This brandy had been bought from a firm of good repute, and as it was above the strength usually sold to the public, a portion of water had been added. He asked the Magistrate to say that the ester test was not trustworthy.

A director of the defendant firm agreed that "British brandy" was not necessarily a distilled product of the grape; whereas "Spanish brandy" indicated brandy distilled from grapes grown in Spain.

The Stipendiary held the invoice to be a warranty, and dismissed the summons, without expressing any opinion on the scientific question of the nature of brandy.

Sale of Food and Drugs Acts.

EXTRACTS FROM THE ANNUAL REPORT OF THE MINISTRY OF HEALTH FOR 1922-1923 AND ABSTRACT OF REPORTS OF PUBLIC ANALYSTS* FOR THE YEAR 1922.

APPOINTMENT OF ANALYSTS.—During the year the Department approved of 48 appointments of Public Analysts in England.

PERCENTAGE OF ADULTERATION.—The total number of samples purchased for analysis in 1922 was 113,860, of which 7106 were reported as not being genuine or up to standard. In 1921 the number of samples taken was 113,664, of which 7582 were reported against. The proportion of samples reported against shows a decrease from 6·7 per cent. in 1921 to 6·2 per cent. in 1922.

MILK.—Of 60,274 samples of milk purchased and submitted for analysis, 4624 were reported as adulterated or not up to standard. These represent a proportion of 7·7 per cent. as against 8·6 per cent. in 1921 (5290 out of 61,439).

In London the proportion of adulterated milk samples was 4·5 per cent. (593 out of 13,179), as compared with 5·1 per cent. in 1921 and 6·7 in 1920.

In the 40 largest provincial towns in England and Wales the proportion of adulterated samples was 8·0 per cent., and in the remainder of the country, excluding London, the proportion was 8·2 per cent.

Of the total number of milk samples examined in the whole country, 135 of whole milk and 48 of separated milk contained colouring matter.

Prosecutions were instituted successfully in the majority of cases in which adulteration was found, with the exception of cases in which the sample was purchased informally.

As many as 166 samples contained excessive amounts of sediment, and 18 of these samples were also otherwise adulterated. Another 33 samples were condemned by reason of containing boric acid, and 7 as containing formaldehyde. The number of milk samples adulterated by reason of containing preservatives indicates a substantial reduction as compared with the figures for 1921.

CREAM.—Reports for 1922 indicate that 902 samples of cream and 976 of preserved cream were examined under the Sale of Food and Drugs Acts, or the Public Health (Milk and Cream) Regulations, 1912 and 1917.

There is no standard of composition for cream, but it is fairly generally accepted in this country that cream should contain about 40 or 50 per cent. of butter fat, the remainder being mostly water with about 5 per cent. of the non-fatty solids of milk. The Public Analyst for Kent reports that in seven samples

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* H.M. Stationery Office. 1923. *Price* 1*s*. 6*d*. *net*.

of cream analysed by him in one quarter the percentage of fat varied from 22·2 to 57·2, and he adds that tinned creams invariably contain a low percentage of fat, and usually contain an unwarrantable amount of other solids.

Of the 902 samples sold as cream (without declaration of preservative) 279 were found to be preserved, the preservative in nearly all the cases being boric acid. Of the 976 samples sold as preserved cream, 37 were reported against for some contravention of the Regulations, the contravention in most cases being the omission or incompleteness of the required declaratory label.

BUTTER AND MARGARINE.—The number of samples of butter examined during the year was 11,234, and of these 189 were reported against, the percentage being 1·7 as compared with 2·4 in 1921.

Of the samples reported against, 92 contained foreign fat (margarine), and 63 contained an excess of water. A number of samples were also found to contain an excessive amount of preservative.

In two samples the percentages of water were found to be as high as 29·9 and 25·3, as compared with the statutory limit of 16. On the other hand, the percentage of water found in a sample of Dorset factory butter was as low as 11·8.

As a means of checking the quality of food sold in restaurants and tea-shops, 55 of the above samples were purchased as bread-and-butter, and in 16 cases the "butter" was found to consist of margarine. In one case the proprietor and manageress of a restaurant were together fined £40.

Of 3700 samples of margarine submitted for analysis, 27 were reported against, including 4 cases in which the sample was found to contain more than the permissible limit of 10 per cent. of butter.

LARD AND DRIPPING.—During the past year 3214 samples of lard were reported upon by Public Analysts, and 6 were regarded as adulterated. Of 400 samples of dripping analysed, 19 were condemned. In both cases the principal defects were the presence of water and of free fatty acids.

COCOA.—Many types of tinned, packet, and loose cocoas have been examined at the instance of a number of Authorities, and of a total of 1851 samples taken during the year, 26 were condemned as containing more than one-hundredth of a grain of arsenic per pound, this being the limit suggested by the Royal Commission on Arsenical Poisoning. Five other samples of cocoa were found to be adulterated with foreign starches or sugar or cocoa shell.

VINEGAR.—The number of samples of vinegar submitted during the year was 1968, of which 110, or 5·6 per cent., were reported against as not being genuine. Included in these totals are two samples of genuine wine vinegar and 475 samples of malt vinegar, of which 38 were adversely reported upon. One sample was condemned because it contained one-ninetieth of a grain of arsenic per gallon. There is no evidence indicating that glucose had been used in the preparation of this sample, but contamination by impure glucose might account for the presence of arsenic.

The sale of vinegar containing "vinegar eels" was reported in one case. The common vinegar eel is a nematode, *Anguillula aceti*, and there is no record of any case of illness having been attributed to it.

MISCELLANEOUS ARTICLES OF FOOD.—The practice of using talc for the facing of rice appears to be on the increase, 52 samples out of 1129 having been reported against. A vendor of one sample containing extraneous mineral matter was fined £5 and £5 5s. costs.

Cornflour was found in one sample of arrowroot to the extent of 50 per cent., and the vendor was fined £5.

Eight samples of aerated waters were condemned, being contaminated with copper, lead, or zinc.

Exception was taken in a few cases to the small percentage of meat in sausages. A fine of £2 inclusive of costs was recovered in respect of one sample containing only 42 per cent. of meat.

Dyes, salicylic acid, and apple pulp are still used in the adulteration of jams.

In one case it was suspected that an attack of vomiting affecting several children was due to the consumption of lemonade made from a popular brand of lemonade powder, and a sample of the powder was therefore submitted to analysis. The analyst reported that this powder, like other lemonade powders, sold under proprietary names, consisted of tartaric acid with the addition of colouring matter and flavouring, and he expressed the view that the illness might have been caused by the tartaric acid.

Salicylic acid has been reported in samples of coffee extracts, but it is understood that the trade has undertaken to discontinue its use.

DRUGS.—The percentage of adulteration of drugs has increased from 6·7 per cent. during 1921 to 7·3 per cent. during 1922, 426 samples out of 5863 being reported against.

Of 429 prescriptions analysed 96, or 22·4 per cent., were found to be inaccurate, as compared with 29 out of 178, or 16·3 per cent., during 1921.

A number of these prescriptions were made up in accordance with the procedure for supplying drugs to insured persons under the National Health Insurance Acts.

Federated Malay States.

ANNUAL REPORT OF THE CHEMICAL LABORATORIES, INSTITUTE FOR MEDICAL RESEARCH, FOR THE YEAR 1922.

THE total number of samples examined was 2702, as compared with 5321 in 1921. The decrease is principally due to samples of liquors and counterfeit coins.

MEDICAL DEPARTMENT.—The work consisted chiefly in the examination of milks (657), water (331) and toddy (451). Of the milks 73 were deficient in non-fatty solids and 9 in fat. Seven of the samples of toddy contained more than 0·8 per cent. of acetic acid. None contained more than 10 per cent. of absolute alcohol. During the year 2350 fluid ozs. of vitamin *B* extract were prepared for the treatment of beri-beri.

TRADE AND CUSTOMS DEPARTMENT.—The total number of samples was 719, chiefly in connection with assessment of duty and examination of samples collected under the Chandu and Deleterious Drugs Enactment. Twenty-eight samples of chandu contained no morphine.

POLICE DEPARTMENT.—Of 261 exhibits, 131 were counterfeit coins. Only 25 of 76 exhibits examined for blood gave a positive reaction.

TOXICOLOGICAL ANALYSES.—Nine cases of human poisoning and 6 of animal poisoning were investigated. The poisons detected were potassium cyanide (3), hydrochloric acid (2), caustic soda (1), and corrosive sublimate (1).

A plant termed "Siak-Siak" by the Malays, and stated by them to be used as a rat poison, was identified as *Dianella ensifolia*. The roots of this plant contained numerous crystals of calcium oxalate.

MISCELLANEOUS EXHIBITS.—Three samples were found to contain Ganja, *i.e. Cannabis indica*.

R. W. BLAIR (Chemist-in-Charge).

Ministry of Health.

DRIED MILK. Circular 452.

THE following Circular has been sent to the Clerks of Authorities (England and Wales) administering the Food and Drugs Acts:

SIR,

I am directed by the Minister of Health to forward for the information of the Local Authority the enclosed copy of the Public Health (Dried Milk) Regulations, 1923, prescribing the labelling and composition of dried milk. The Regulations will come into operation on the 1st May, 1924.

It will be seen that the provisions of the Regulations are generally similar to those of the Regulations recently issued with regard to condensed milk (*cf.* ANALYST, 1923, 271), and the remarks contained in Circular 393 will, therefore, apply with the necessary modifications to the new Regulations.

The following points of difference may, however, be specially mentioned:—(i) The sole responsibility for the enforcement of the Regulations is entrusted to Local Authorities, no duties being assigned to the Officers of Customs and Excise.

(ii) Under Article 5 (3) of the Regulations the person who procures a sample for the purpose of testing the equivalent quantity of liquid milk is required to send a part of the sample to the seller after the net weight has been ascertained. No definite procedure is laid down for ascertaining the weight, but it will of course be necessary that this should be done in such a manner as to ensure that sufficient proof is available as to the accuracy of the estimation. In some cases it may be convenient for the person who takes the sample to weigh the contents in the presence of the seller, and where this course is adopted there would be no difficulty in following the ordinary procedure for the division of the sample into three parts.

(iii) It will be observed that Article 4 (2) of the Regulations, which deals with the composition of dried milks, prescribes minimum percentages only of milk fat. But the requirements as to labelling contained in the Schedule provide that the equivalent amount of liquid milk contained in dried milks is to be calculated by reference to a definition of milk similar to that adopted in the case of the Condensed Milk Regulations, and comprising minimum percentages of both milk fat and total milk solids.

The Regulations (Order, 68,576) and this Circular will be placed on sale, and copies may be obtained through any bookseller or directly from H.M. Stationery Office.

I am, sir, your obedient servant,

A. K. MACLACHLAN (Assistant Secretary).

November 8, 1923.

Gas Regulation Act, 1920.

ABSTRACT OF MEMORANDUM BY THE GAS REFEREES ON THE APPOINTMENT, QUALIFICATIONS AND DUTIES OF GAS EXAMINERS.

THE Gas Regulation Act, 1920, provides that the charges made by Gas Undertakings for the public supply of gas shall be based on its calorific value, and that official tests shall be made by Gas Examiners for the purpose of ascertaining the calorific value, purity and pressure of the gas.

Under the Act the Gas Examiners are appointed by local authorities, or where they fail to appoint an examiner, or the testing of the gas is imperfectly attended to, by Quarter Sessions (or in Scotland, the Sheriff). (Sec. 4 (3).

The gas referees shall prescribe (*a*) the places and times for the tests; (*b*) the method by which the apparatus shall be verified; and (*c*) the time and form of the reports to be made by the Gas Examiner to the Gas Referees (Sec. 5–(1).

As regards "competence" of Gas Examiners, the Gas Referees consider that it is important that a Gas Examiner should have a sufficient knowledge of physics and chemistry to enable him to understand thoroughly the tests to be applied to the gas. It is also important that he should be a person accustomed to making quantitative measurements, the accuracy of which he is prepared to sustain under cross-examination. A person of good professional standing as an analyst or engineer, and with experience of the physics and chemistry of gas, should be suitable.

As regards "impartiality," it is obviously undesirable that the Gas Examiner should be in the employ of, or should have any financial interest in the gas undertaking. (*Cf. Report to the Board of Trade of the Therm Charges Committee*, Cmd. 1825, price 6d. net.)

When a gas examiner is appointed, a deputy, similarly qualified, should, where practicable, also be appointed to act in his temporary absence.

The apparatus and method by which tests are to be made are described in the "General Notification of the Gas Referees" (H.M. Stationery Office, Kingsway, W.C.2. Price 1s. net).

The times and places at which tests shall be made are prescribed by the Gas Referees in a Special Prescription for each undertaking, the times varying with the number of million cb. ft.

The Board of Trade Annual Returns of Gas Undertakings, Part I., 1922, show the quantities of gas supplied by all authorised gas undertakings in Great Britain. (H.M. Stationery Office, price 5s. net.)

Economy and efficiency in gas testing will be promoted if local authorities enter into arrangements for the joint appointment and employment of Gas Examiners, or, in suitable cases, arrange for the appointment to be made by the County Council.

66 VICTORIA ST., LONDON, S.W.1.
June, 1921. *Revised Sept.*, 1923.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Detection of Dried Egg in Liquid Egg. C. Hohenegger. (*Zeits. Nahr. Genussm.*, 1923, **46**, 91–93.)—When made into a paste with water, dry egg does not yield a uniform emulsion, except when either liquid egg-white or yolk (or both) is also added. Unless water is introduced, dried egg cannot be regularly distributed throughout liquid egg. Added water may be detected by shaking 10 c.c. of the material vigorously in a test-tube with ether and allowing the mixture to stand; three distinct liquid layers separate, the uppermost of which consists of an ethereal solution of the fat coloured with lutein, the middle of a whitish yellow, milky layer of albumin, and the bottom of clear, aqueous solution. The last collects at first immediately below the ethereal layer and sinks only after the lapse of some time.

Dried egg may be detected by the appearance of the film formed when six or seven drops of the material are allowed to dry on a glass plate in the air. Under these conditions (1) liquid egg gives a mass showing rectilinear radii, (2) dried egg mixed with water yields a uniform, amorphous mass without characteristic appearance, and (3) a mixture of liquid and dried egg gives rise to a film exhibiting curved veinings resembling those of a leaf, the divergence of the appearance from that given by the liquid egg varying with the proportion of dried egg present. Further, centrifugation effects no change in liquid egg, but separates a mixture of liquid with dried egg into two distinct layers; under the microscope liquid egg reveals a regular appearance similar to that of a colloidal solution in the ultramicroscope, whereas a mixture of dried egg and water exhibits small and large particles of albumin, which are coloured yellow by iodine solution. T. H. P.

Abnormal Cells in Cocoa Powder. A. Nestler. (*Zeitsch. Nahr. Genussm.*, 1923, **46**, 86–91.)—A sample of cocoa, with an odour recalling star anise, in addition to the normal smell, and with no abnormality in its chemical composition, exhibited: (1) a number of large or small thick-walled sclerenchyma cells, either colourless or with reddish-brown contents and both separate and in fragments of tissue; (2) groups of more or less long, thick-walled, spotted cells, with or without reddish-brown contents; (3) very large, lignified cells; (4) colourless fragments of large sclereids; and (5) round, mostly spined pollen grains, both isolated and in groups of 18 or more. The unusual sclerenchymatous elements and the high proportion of normal husk constituents are found to be derived from twinned and tripled cocoa seeds. When such seeds are roasted, dehusking fails to eliminate portions of husk lying at the surface of contact of the seeds, and also hard portions of sclerenchymatous tissue occurring at the edges of the seeds. These abnormal constituents may be observed in small quantities in normal cocoa powders of good quality. The pollen grains mentioned owe their presence to faulty cleaning of the raw material. T. H. P.

Influence of Starch on the Strength of Wheat Flour. J. H. Buchanan and G. G. Naudain. (*J. Ind. Eng. Chem.*, 1923, **15**, 1050–1051.)—Measurements of the sizes of starch grains in seven samples of flour indicated a relation between the size of the grains and the baking strength of the flours. The samples contained from 78·0 to 83·2 per cent. of small grains (diameter less than 8 microns), and the flour with the largest percentage of these small grains yielded a loaf having the largest volume. W. P. S.

Presence of Maltase in Germinated and Ungerminated Barley. A. R. Ling and D. R. Nanji. (*Biochem. J.*, **17**, 593–596.)—Glucose may be produced from starch and some of its hydrolytic products by diastase preparations not containing maltase, but, when precipitated malt diastase is the hydrolysing agent for starch, glucose is only formed when the diastase has been heated to 65° C. in aqueous solution. Under no conditions is glucose produced by the action of malt diastase on maltose. It is produced, however, by the action of malt extract on maltose. Experiments described and results given show that green or kilned malts obtained by germinating barley contain an enzyme capable of hydrolysing maltose. The power of the enzyme depends, *ceteris paribus*, on the temperature and the way in which the malt has been heated on the kiln. Diastase preparations obtained by precipitating a cold water extract of malt with alcohol do not contain maltase, as this enzyme is destroyed by the alcohol. Ungerminated barley contains an insoluble enzyme capable of converting maltose into glucose. It cannot be extracted with water, but its activity is demonstrated by allowing ground barley (grist) to act on maltose. It is rendered partially soluble during germination. Green malts contain more maltase, as the enzyme is probably partially destroyed during the kilning process. P. H. P.

Separation of Dextrose and Lævulose. F. Lucius. (*Zeitsch. Nahr. Genussm.*, 1923, **46,** 94–95.)—It has been shown (*Zeitsch. Nahr. Genussm.*, 1919, **38,** 177) that dextrose and lævulose, when present together, may be estimated by polarisation before and after destruction of the lævulose. It is now found that doubling the concentration of the acid permits of the reduction of the duration of heating to 3 hours; with still higher concentrations, however, the dextrose also is attacked. The estimation is carried out by heating 50 c.c. of the sugar solution in a 100 c.c. measuring flask with 20 c.c. of 5 *N*-hydrochloric acid for 3 hours in a boiling water-bath. The cold liquid is treated with 5 *N*-sodium hydroxide solution until it shows only a faintly acid reaction, and is then made up to 100 c.c. and filtered, the brownish-yellow filtrate being treated with a little animal charcoal in the cold and the rotation determined at 20° C. in a 200 mm. tube in the polarimeter. This reading represents the dextrose, the lævulose being given by the change in rotation produced by the acid treatment. T. H. P.

Formation of Formic Acid by Caramelisation of Cane Sugar. S. G. Simpson. (*J. Ind. Eng. Chem.*, 1923, **15,** 1054–1055.)—Appreciable quantities of formic acid are produced when sugar or substances containing sugar are heated at temperatures above 160° C., the amount of acid formed depending on the temperature and time of heating. For instance, cane sugar heated at 243° C. for five minutes yielded 0·16 per cent. of its weight of formic acid, and 0·548 per cent. after the heating had been continued for thirty minutes. A sample of caramel examined contained 0·404 per cent., and a sample of roasted coffee 0·221 per cent. of formic acid. W. P. S.

Deterioration of Edible Fats. J. F. Bevis. (*J. Soc. Chem. Ind.*, 1923, **42,** 417–420T.)—The results of experiments on beef and mutton fats from the caul and kidneys of the animals, rendered at 54–71° C., show that these fats decompose more rapidly when kept at 100° C. than when aerated at a lower temperature for a similar period. The increase in the free fatty acid is gradual at first, but increases in rate with the time of aeration. The increase occurring in the saponification value and the diminution in the iodine value show some agreement with the results of rancidity tests. For the detection of rancidity, the Kreis test, depending on the production of a red coloration when an ethereal solution of phloroglucinol is shaken with a mixture of the fat and concentrated hydrochloric acid, appears to be more sensitive than the Issoglio test (ANALYST, **1917, 42, 301**), which measures the amount of permanganate required to oxidise the water-soluble material in 100 grms. of the fat.

The decomposition of fat at ordinary temperatures is greatly accelerated by the influence of light, and the presence of free oleic acid increases the rate of formation of the compounds causing the Kreis reaction, but free glycerol is virtually without effect in this direction. The intensity of this reaction is not related to increase in the free acidity. The latter increases when mould appears in a fat. The colouring matter of fat appears to play a part in its decomposition.
T. H. P.

Quantitative Aspects of the Kreis Test. G. E. Holm and G. R. Greenbank. (*J. Ind. Eng. Chem.*, 1923, **15**, 1051–1053.)—In the case of an oxidised fat the intensity of the coloration obtained in the Kreis test is proportional to the amount of oxygen absorbed by the fat, but the intensity of the coloration is not proportional to the rancidity. A rancid fat will yield a coloration, but many fats which have absorbed large quantities of oxygen exhibit only faint rancidity, although they yield an intense coloration with the test. There is some evidence to show that oleic acid may be the only unsaturated acid in fats, which, when oxidised, gives the Kreis reaction. Absorption of free oxygen is not necessary for the production of a coloration by a fat, since exposure of a fat to light in the absence of free oxygen will cause the fat to yield a coloration when submitted to the Kreis test. W. P. S.

Rapid Method for the Estimation of Carbon Dioxide in Beverages. J. Cannizzaro. (*J. Ind. Eng. Chem.*, 1923, **15**, 1074–1075.)—The bottle containing the beverage (*e.g.* beer or the like) is placed in a mixture of ice and salt until cooled thoroughly; 25 c.c. of the cold beverage are then transferred by a pipette to a beaker containing 30 c.c. of 0·2 *N* sodium carbonate solution, 400 c.c. of water (boiled previously and then cooled to 5° C.) are added, and the excess of sodium carbonate is titrated with 0·2 *N* hydrochloric acid, with the use of phenolphthalein as indicator. Twice the volume of hydrochloric acid used is subtracted from the 30 c.c. of sodium carbonate solution in order to obtain the volume of the latter actually used for the titration of the carbon dioxide and fixed acids. A correction is made for these acids by boiling another portion of the beverage to expel carbon dioxide, cooling the solution to 5° C., and titrating it with 0·2 *N* sodium carbonate solution, with the use of phenolphthalein as indicator. W. P. S.

Estimation of Para-acid (*p*-Sulphaminobenzoic Acid) in Commercial "Saccharin." O. Beyer. (*Chem. Zeit.*, 1923, **47**, 744.)—According to the method of the Reichgesundheitsamt, *p*-sulphaminobenzoic acid in "saccharin" is estimated by decomposing either 0·6 grm. of the substance with 10 c.c. of 25 per cent. hydrochloric acid, or 5 or 10 grms. with 20 c.c. of this acid, leaving the mixture for two days at 17 to 18° C., and separating and drying the acicular crystals of the para-acid; the corrected melting point of the latter lies between 287° and 289° C. The results of the author's investigations show that this method is satisfactory if 10 grms. of the material are employed. Hefelmann's method for the same estimation is less accurate. T. H. P.

Estimation of Formaldehyde in Formalin. P. Borgstrom. (*J. Amer. Chem. Soc.*, 1923, **45**, 2150–2155.)—Formaldehyde was estimated in presence of certain substances that may occur in formalin (methyl and ethyl alcohols, sodium formate, acetone and methylal), by the three methods previously studied (ANALYST, 1923, **48**, 457). The iodimetric method can be used in presence of methyl alcohol and formic acid; acetone and ethyl alcohol interfere. In the hydrogen peroxide method, acetone gives rather high results, the other four substances not interfering. The neutral sulphite method, too, is correct except in the presence of acetone, which reacts with the sulphite. W. R. S.

Potassium Ferrocyanide as a Microchemical Reagent for some Alkaloids. H. I. Cole. (*Phillipine J. Sci.*, 1923, **23**, 97.)—Tests have been carried out with potassium ferrocyanide on forty of the common alkaloids, of which thirteen give characteristic crystalline precipitates. These are addition-compounds, and probably of the type $B_2H_4Fe(CN)_6xH_2O$. The test is carried out as follows:—An aqueous solution of the alkaloid is rendered acid with dilute hydrochloric acid. One drop of this (not exceeding 2 to 3 mm. diameter) is placed on a slide. Close to it is placed a smaller drop of a 5 per cent. solution of potassium ferrocyanide. A tiny channel is caused to flow from the reagent to the test-drop by means of a finely-pointed glass rod. The resulting precipitate, if not at once crystalline, can be made so by scratching with the glass rod, and is then examined under a low power for colour, shape, crystal angles, polarisation, extinction angles and behaviour with reagents. The sensitiveness in each case is determined by testing solutions of the alkaloid in decreasing concentration till one is reached that fails to give crystals in five minutes. Allowance must be made for the dilution of the drop by the reagent solution

By this test the precipitates show the following characteristics:

β-Eucaine. Sensitiveness, 1:200. Colourless thin elongated or rhombic plates. Under crossed Nicols they are weakly polarised, exhibiting parallel and symmetrical extinction.

Brucine. Sensitiveness, 1:2500. Highly refractive prisms with chisel-shaped ends, usually in rosettes. They are strongly polarised, exhibiting parallel extinction.

Cinchonidine. Sensitiveness, 1:300. If the concentration of the alkaloid is over 0·5 per cent., rosettes of yellow, curving, hair-like needles; if 1:300 long, thin rectangular yellow plates, exhibiting parallel extinction.

Cinchonine. Sensitiveness, 1:1000. Yellow irregular, trapesium-shaped crystals polarising strongly. Distinction from cinchonidine and quinine. Quinine does not yield a crystalline precipitate.

Cocaine. Sensitiveness, 1:500. Colourless six-sided plates and prisms of irregular shape. They polarise much more strongly than those from β-eucaine and are more irregular in shape.

Coniine. Sensitiveness, 1:50. Rosettes of colourless needles and long thin square-ended prisms. They polarise weakly, exhibiting oblique extinction. The extinction angle is about 30°.

Heroine. Sensitiveness, 1:50. Hexagonal plates only appear after vigorous scratching.

Hydrastine. Sensitiveness, 1:700. White spheroids showing polarisation.

Quinoline. Sensitiveness, 1:800. Lemon-yellow rhombohedra, showing parallel and oblique extinction.

Sparteine. Sensitiveness, 1:2000. Characteristic colourless rhombs exhibiting symmetrical extinction.

Stovaine. Sensitiveness, 1:300. Rosettes of needles, showing parallel extinction.

Strychnine. Sensitiveness, 1:20,000. Long slender needles or spear-shaped crystals with serrated edges, showing oblique extinction; sometimes hemimorphic triangular plates. When the concentration of the strychnine is high, but very little reagent is added; the true form of the compound sometimes comes out on scratching. These are small rhombic plates, showing symmetrical extinction under crossed Nicols.

Veratrine. Sensitiveness, 1:100. Crystals are only formed with great difficulty and are imperfect. They polarise strongly.

Photomicrographs are given of the crystals formed with each alkaloid, except veratrine.

R. F. I.

Estimation of Nicotine in Tobacco and Tobacco Extracts. O. M. Shedd. (*J. Agric. Res.*, 1923, **24**, 961–970.)—A source of error in the standard methods of Kissling and of Chapin (*cf.* ANALYST, 1911, **36**, 544) is the steam distillation. This is obviated by the following method: To one grm. of tobacco extract or 5 grms. of air-dry powdered tobacco are added 2·5 to 5 c.c. of alcoholic sodium hydroxide solution (6 grms. in 100 c.c. of 50 per cent. alcohol) and the mixture extracted with ether for 5 hours under a reflux condenser; in the case of tobacco extracts sufficient calcium carbonate is added first to make a pasty mass. It is important that the temperature of the cooling water should not exceed about 25° C., and that the volume of ether in the extraction flask should not, at any time, be lower than about 25 c.c. After the extraction the ether is distilled off to a volume of about 25 c.c., which should suffice to keep the extracted matter in solution at room temperature, and the residue washed four or six times with dilute hydrochloric acid (1:4). At this point an emulsion may be prevented by the addition, if necessary, of 1 c.c. of alcohol. The acid extracts are filtered through glass wool, diluted to 100 c.c., and 25 c.c. further diluted to 400 or 500 c.c.; a further quantity of acid is then added, and the nicotine precipitated by adding, with constant stirring, 1 c.c. of silicotungstic acid solution for each 0·01 grm. of nicotine present. The reagent is a 12 per cent. solution of silicotungstic acid which must be of the formula $SiO_2.4H_2O, 12WO_2.22H_2O$. After standing a few minutes the precipitate is filtered through a Gooch crucible, washed with 1:1000 hydrochloric acid and ignited, and the weight of the residue, multiplied by 0·114, gives the nicotine in the aliquot part taken. Direct precipitation without ether extraction yields satisfactory results with most tobaccos, but not with all.

H. E. C.

Conductometric Titration of Alkaloids. W. D. Treadwell and S. Janett. (*Helv. Chim. Acta*, 1923, **6**, 734–743.)—The curve of the titration of a weak base with a strong acid shows at first a moderate increase in current density, which slows down as the end-point is approached. The first drop of free acid shows a steep, almost rectilinear increase. The end-point lies at the intersection of the two members of the curve, and is best determined by extrapolation; the curve should preferably be plotted some distance beyond the end-point. Brucine, quinine, narcotine, morphine, and caffeine were dissolved in alcohol, and the solutions titrated with 0·1 *N* hydrochloric acid at a potential of 8 volts. The first four alkaloids gave satisfactory results, but caffeine was found to be too weak a base for alkalimetric estimation. W. R. S.

Properties of Colloidal Gum Benzoin. H. D. Wright and W. O. Kermack *Biochem. J.*, 1923, **17**, 635–657.)—The authors investigated the colloidal properties of benzoin sol in order to get uniform suspensions for the precipitation test of Guillain, Laroche and Lechelle (*Compt. Rend. Soc. Biol.*, **1920**, **83**, **1077**) for detecting pathological variations in the cerebrospinal fluid. Precipitation of colloidal gum "benzoin" occurs when it is added to an equal volume of gelatin of very dilute concentration, *viz.* about 1/500,000. The actual concentration of gelatin required varies with the P_H of the solution. No precipitation occurs on the alkaline side of the isoelectric point of gelatin, and the charge on the particles there is always negative. If, due to insufficient gelatin being added, no precipitation occurs, the charge is negative, but if due to excess being added, then the charge is positive. Similar results are obtained with oxyhæmoglobin in place of gelatin, used because it has a markedly different isoelectric point from gelatin. The power of gelatin to protect "benzoin" against precipitation by sodium chloride depends on the P_H and is a minimum at the isoelectric point. Precipitation by an acid depends essentially on the concentration of hydrogen ions. That the amount of salt required to precipitate "benzoin" increases with increase in the P_H largely accounts for the differing amounts of salts of univalent kations required to effect precipitation. The effect of tannin was tried in precipitating mixtures of gelatin and "benzoin." P. H. P.

Biochemical, Bacteriological, etc.

Growth-promoting Value and Vitamin Content of Palm-kernel Meal. A. J. Finks. (*J. Agric. Res.*, 1923, **25**, 165–169.)—Feeding experiments on rats show that the proteins of palm-kernel meal are adequate for their growth when other dietary factors supplying vitamins are present to a normal extent. The commercial solvent-extracted meal does not contain a sufficiency of vitamin *A* to prevent xerophthalmia, neither is it a satisfactory diet in respect of vitamin *B*. No experiments on untreated kernels are recorded. H. E. C.

Presence of Vitamin *B* in Frozen Flesh Food. A. M. Wright. (*J. Soc. Chem. Ind.*, 1923, **42**, 403–404T.)—The work was carried out on young Leghorn cockerels, 2–3 weeks old, at the beginning of the experimental feeding, in which they received a diet where the protein was entirely derived from the meat, the fat from the meat supplemented by lard, with or without butter fat, and the carbohydrates from purified starch. In the controls the protein was derived from purified casein, and compressed yeast was used to supply vitamin *B*. All animals received a mixture of inorganic salts and orange or swede juice to supply vitamin *C*, and water. As no data were available with regard to the effect, on the animals used, of artificial diets or deficiency in vitamin *B*, a considerable amount of preliminary work was necessary. It was found that in 60 days the average increase in weight with all three vitamins present was from 161 to 333 grms.; with vitamin *B* absent, the weight remained fairly constant, death supervening about the 21st day; with vitamin *A* absent, the weight again remained more or less constant, the animals dying in about 35 days, while with both vitamins *A* and *B* absent, weight was lost, and death resulted in less than a fortnight. The following results were obtained with fresh and frozen meats:

	Controls. Grms.	Lamb fresh. Grms.	3 yrs. cold storage. Grms.	Mutton fresh. Grms.	Beef fresh. Grms.	2 yrs. cold storage. Grms.	Pork 9 yrs. cold storage. Grms.
Original wt.	161	180	169	197	153	169	173
7–10 days	165	178	168	187	154	175	201
10–14 ,,	177	170	171	211	151	156	209
21 ,,	214	220	200	217	183	185	249
28 ,,	247	237	229	265	194	209	284
35 ,,	300	256	267	298	227	222	286
42 ,,	302	302	304	—	247	245	295
49 ,,	321	305	308	—	264	268	343
60 ,,	333	349	344	—	313	315	352

The steady increase in weight with pork is noteworthy, and opposed to the results obtained with other meats, with which the weight either begins by decreasing or remains stationary for the first fortnight. The explanation is not at present forthcoming. It will be seen that these experiments do not disclose any loss of vitamin *B* in the frozen meats used. D. G. H.

Relation Between the Growth-promoting and Anti-rachitic Functions of Certain Substances. H. Goldblatt and S. S. Zilva. (*Lancet*, 1923, **205**, 647–649.)—Experiments, described in detail, show that the growth-promoting and anti-rachitic properties of cod liver oil are rendered inactive at different rates when heated in the presence of air. A great difference was also observed between the growth-promoting and anti-rachitic potency of spinach. A sample of hydrogenated cod liver oil was tested for the two functions, and it was found that a daily dose of about 33·7 mgrms. of the melted fat was capable of affording protection to rats against the development of rickets, whilst 1·8 mgrms. of the fat promoted growth. These observations give further support to the hypothesis of McCullum, Simmonds, Shipley and Park (*Johns Hopkins Hospital Bull.*, **1922, 33, 296**), that the anti-rachitic and the fat-soluble vitamin *A* factors are different principles.

Identity or Non-Identity of Antineuritic and Water-Soluble *B* Vitamins. P. A. Levene and M. Muhlfeld. (*J. Biol. Chem.*, 1923, **57**, 341–349.)—The problem was investigated after it was incidentally observed, in an effort to organise the work on the chemical nature of the water-soluble vitamin *B*, that certain samples of yeast which were sufficiently active to maintain normal growth in white rats failed to protect pigeons fed on polished rice, from polyneuritis. Three samples of bakers' yeast and one sample of brewers' yeast were employed, and all were effective in maintaining normal growth in daily doses of 0·2 grm. The water-soluble vitamin *B* of each varied comparatively little, the proportional difference between the best and poorest being not greater than 2 to 1. Brewers' yeast and bakers' yeast No. 3 protected pigeons from polyneuritis in doses of 0·5 grm. per day, whilst bakers' yeast No. 9 did not, but it gave irregular results in doses of 1 grm. Bakers' yeast No. 2 failed to protect in doses four times as large as the protective dose of No. 3. A previous impression that an extract of a certain sample of yeast contained the protective power when the untreated yeast failed to afford protection, was proved erroneous, since the Osborne and Wakeman fraction II. obtained from yeast No. 2 was inactive in doses of 0·5 grm., whereas a similar fraction from brewers' yeast completely protected in doses of 0·125 grm. The concentration of the protective principle was higher in yeast No. 3 than in brewers' yeast, but the concentrations of it in the extracts from the samples were reversed. All samples of the Osborne and Wakeman fraction II. in daily doses of 0·02 to 0·05 grm. maintained the normal growth of rats fed on the basal diet of Osborne and Mendel. Experimental details are described. These results, given in tables and a chart, are additional evidence on the non-identity of the antineuritic and growth-promoting principles, but the chemical nature of these must be known for a final solution of the problem. P. H. P.

Insulin. F. A. Hocking. (*Pharm. J.*, 1923, **57**, 392–394.)—The quantity of insulin producing in 2 hours a reduction in the blood sugar of a rabbit weighing about 2 kilos. from the normal 0·1 per cent. to about 0·04 per cent., is termed three units, and in order to fix the correct dose for the patient, *i.e.* the dose that will bring the blood sugar to the normal figure of 0·1 per cent., it is necessary to know the exact content of sugar in the blood. Lynch (*Lancet*, 1923, June 9th) discusses methods of carrying out this estimation. Protein must be precipitated, and in Folin's method tungstic acid is used for the purpose. The sugar is then estimated in the clear solution in the usual way by means of alkaline copper sulphate solution, and the amount of cuprous oxide found by means of a colorimeter, a solution containing a known quantity of dextrose being used as a control.

The active principle of insulin has not so far been isolated in a chemically pure state, and there is some confusion as to its properties. It is possibly a protein derivative with a highly complex molecule. It may be of the nature of an albumose, or perhaps merely an adherent of the protein, itself of a simpler chemical structure. It is soluble in water and alcohol except at its iso-electric point $P_H = 5$ or $P_H = 6$. Boiling for 3 minutes in aqueous solution completely destroys it, although it has been stated to be stable for several hours at 100° C., in strongly acid solution; and, if this is so, extracting the pancreas with acidulated hot water might be a possible method of preparation. Its activity is rapidly destroyed in alkaline solution, and also in the presence of trypsin and pepsin. It is readily adsorbed by such substances as kaolin and charcoal, and for this reason filtration through paper is to be avoided in the course of its preparation. Some workers state that the potency is considerably reduced by passing through a Berkefeld filter, whilst others advocate the use of the filter as a means of sterilising insulin solutions. Half saturation with ammonium sulphate, picric and benzoic acids, and barium salts of weak acids causes precipitation. Precipitation also occurs at the iso-electric point. D. G. H.

Action of Proteolytic Enzymes upon Insulin. E. J. Witzemann and L. Livshis. (*J. Biol. Chem.*, 1923, **57**, 425–435.)—Insulin preparations obtained by the Collip method show the typical qualitative reactions of proteins. They must be contaminated with alcohol-soluble protein, unless insulin itself is a protein or polypeptide. A series of experiments is described in which Lilly and Co.'s iletin and the authors' own insulin (prepared by the Collip method) were subjected to the action of proteolytic enzymes (trypsin, papain and pepsin). Tables of the results are given. The insulin preparations were completely inactivated by the enzymes, thereby indicating that insulin itself is protein-like in nature and that the protein reactions are not entirely due to impurities. Certain interpretative experiments, *viz.* Influence of digestion products on Insulin; and Formaldehyde Titration of a Trypsin Digestion of Insulin, failed to invalidate this conclusion. Boiled trypsin and pepsin did not change the activity of insulin, and digested insulin did not inactivate undigested insulin. The results of Best and Macleod (*J. Biol. Chem.*, 1923, **55**, 29) contradict the conclusion that insulin is protein-like in nature, but Doisy, Somogyi and Shaffer (*J. Biol. Chem.*, 1923, **55**, 31) describe a solid insulin preparation containing **14** per cent. of nitrogen, which is undoubtedly a protein, if all insulin. The available information upon the nature of insulin is discussed, and it is suggested that insulin is the "effect" of a certain group or grouping which persists in an effective form in several stages of protein breakdown. The "effect" appears to be associated with a protein, on the one hand, and with peptones and polypeptides, on the other. This interpretation concerning insulin, if correct, conforms with the new facts, and does away with the existing contradictions about its nature. P. H. P.

Species of *Rhizopus* Responsible for the Decay of Sweet Potatoes. J. I. Lauritzen and L. L. Harter. (*J. Agric. Res.*, 1923, **24**, 441–456.)—*Rhizopus tritici* and *R. nigricans* are the species of fungi mainly responsible for soft-rot in potatoes; the former is the more active at temperatures from 6° C. to 20° C., and the latter from 30° C. upwards; between these temperatures the activities of the two species overlap. Other species—*R. oryzae*, *R. reflexus*, and *R. artocarpi*—can also cause some decay, but the two first-named cause very much more and predominate in a mixed infection. H. E. C.

Production of Acid by *Rhizopus tritici* in decaying Sweet Potatoes. H. A. Edson. (*J. Agric. Res.*, 1923, **25**, 9–12.)—The fermentative decay of sweet potatoes by *Rhizopus tritici* is of the usual alcohol and acetic acid type. Ethyl alcohol and acetic acid are the principal products, together with small quantities of formic, butyric, and lactic acids, and, possibly, a little succinic acid, acetone, and an unidentified aldehyde. In addition, there is some nitrogenous decomposition, giving rise to ammonia and other products. It is shown that these changes are produced by the fungus, and not by bacteria associated with, or following it.

H. E. C.

Spores in the Upper Air. E. C. Stakman and Others. (*J. Agric. Res.*, 1923, **24**, 599–606.)—In order to investigate the distribution of spores of "rust" and other pathogenic fungi in the upper air, spore-traps were exposed from aeroplanes at various altitudes and then examined microscopically. The spore-traps simply consisted of microscope slides covered with a thin film of vaseline; these were exposed, some by hand and some by mechanical devices, from the fuselage of an aeroplane. Many spores, condiophores, pollen grains, glumes, and small insects were caught on the slides; spores and pollen are very abundant in the air up to 11,000 feet, but are much rarer at higher altitudes, though *Puccinia* was found at 16,000 feet. Those caught at 7000 feet germinated well; although the germinating power of the spores cannot be accurately determined on account of the film of grease, a fair percentage developed readily. It is evident that spores and fungi are carried great distances by air currents.

H. E. C.

Micro-Estimation of Magnesium in Serum. St. Gadient. (*Helv. Chim. Acta*, 1923, **6**, 729–733.)—The serum need not previously be freed from albumin. One c.c. is precipitated with magnesia mixture in a centrifuge tube. After standing, the tube is centrifuged for 5 minutes; the clear liquid is pipetted off and the precipitate washed three times with 2 per cent. ammonia, 5 c.c. at a time; each portion is centrifuged and pipetted off. The precipitate is dissolved in a few drops of 2 *N* sulphuric acid and rinsed quantitatively into a graduated 25 c.c. tube with not more than 10 c.c. of water. This solution is treated with 1 c.c. of molybdate solution (50 grms. of ammonium molybdate in 1000 c.c. of *N* sulphuric acid), 2 c.c. of hydroquinone solution (20 grms. of hydroquinone in one litre of water and 1 c.c. of strong sulphuric acid), and, after 5 minutes, with 10 c.c. of a solution made by mixing 2 litres of 10 per cent. sodium carbonate with 500 c.c. of 15 per cent. sodium sulphite solution. The standard solution contains 0·4387 grm. of potassium dihydrogen phosphate per litre (1 c.c. = 0·0001 grm. P; factor for converting P into Mg = 0·78350). Quantities of 0·5, 1·0, and 2·0 c.c. of standard solution are measured into graduated cylinders and treated like the assay. After addition of the carbonate and sulphite solution the volumes are made up to 25 c.c. with water, and the comparison made without delay, as the coloration is not very stable.

W. R. S.

Estimation of Ammonia and Urea in Urine. S. Levy-Simpson and D. C. Carroll (*Biochem. J.*, 1923, **17**, 391–402.)—Experimental details are given of an investigation into the conditions required for obtaining accurate results by the following method: A distillation flask of 1 litre capacity is arranged with the neck at an angle of 45° with the horizontal, and the side tube is connected with the upper end of a vertical condenser. A rubber cork fitting the neck of the flask supports a tap funnel containing alcoholic potassium hydroxide solution coloured with phenolphthalein, and a glass tube connected with a supply of steam. The lower end of the condenser dips into standard hydrochloric acid solution contained in a flask. Five or 10 c.c. of the sample under examination are mixed with 90 c.c. of 96 per cent. alcohol and transferred to the distillation flask and, after the cork has been replaced, a vigorous jet of steam is led into the liquid, and sufficient of the alcoholic potassium hydroxide solution is added to render the liquid just alkaline to phenolphthalein. Distillation is continued until the liquid begins to foam, after which the steam inlet tube is rotated out of the liquid and a current of steam is passed for a further 1½ minutes to rinse the last traces of ammonia out of the flask and condenser. The ammonia is estimated by titrating the standard acid with sodium hydroxide solution free from carbon dioxide, alizarin red being used as indicator. The urea solution remaining in the flask is brought to a P_H of 7·02 by the addition of potassium hydroxide and acid potassium phosphate buffer solution, and the urea present is converted into ammonium carbonate by treatment with a standard urease solution for 20 mintues at 40° C., after which distillation is carried out as before, and the urea present is calculated from the volume of standard hydrochloric acid neutralised by the distillate. The method yields results in excellent agreement with those given by the van Slyke and Folin aeration method, and the complete estimation of ammonia and urea may be carried out in 30 minutes.

T. J. W.

Quantitative Colour Reaction given by Adrenalin and Urine. H. Friend (*J. Biol. Chem.*, 1923, **57**, 497–505.)—Hitherto there has been apparently no satisfactory quantitative chemical estimation for adrenalin either in blood or urine. The reaction here described depends on the fact that epinephrine reacts with sulphanilic and nitrous acids in the presence of an alkali (ammonia) to give a red colour which is quantitatively proportional to the adrenalin concentration, and is also given by the phenols (which cannot be removed) present in urine. If an iron suspension (ferric chloride) be added, to destroy the adrenalin, to one of two estimations running simultaneously, the decrease in the colour of this one gives the amount of adrenalin present. None of the urinary constituents tested has given a positive reaction before ferric chloride treatment and a negative reaction after it. Possibly the urinary constituent giving the colour before, but not after, the ferric chloride treatment is adrenalin. A table is given of the estimations of this constituent in a number of normal and pathological urines. The method is as follows:—Add 37·5 c.c. of a filtered 25 per cent. solution of lead acetate and 37·5 c.c. of 15 per cent. ammonium sulphate solution to 300 c.c. of urine in a precipitating flask, stir and decant through a filter paper after leaving for 15 minutes, thus getting a clear filtrate. Place 125 c.c. of this filtrate in a 300 c.c. Erlenmeyer flask fitted with a capillary opening, pinch-cock arrangement and a Centigrade thermometer. Add 20 c.c. of the iron suspension (0·025 grm. of ferric chloride in 1 litre of water), heat slowly to 50° C., cool and clamp the capillary tube.

Add 20 c.c. of distilled water to another 125 c.c. of filtrate. To each add 20 c.c. of sulphanilic acid (4·0 grms. of sulphanilic acid crystals mixed with 90 c.c. of 37 per cent. hydrochloric acid and made up to a litre with distilled water), 5 c.c. of a 1 per cent. sodium nitrite solution, and 10 c.c. of ammonium hydroxide (28 per cent.) Shake and leave for 15 minutes; then compare the two in a Duboscq or Kober colorimeter (125 c.c. of the filtrate are equivalent to 100 c.c. of the original urine). Since 1 mgrm. of adrenalin gives a reading of 5 mm., the reading multiplied by 0·2 gives the amount present. P. H. P.

Refractometric Estimation of Hæmoglobin. J. L. Stoddard and G. S. Adair. (*J. Biol. Chem.*, 1923, **57**, 437–454.)—The refractometric method, unlike those based on oxygen capacity, gives the absolute value of hæmoglobin. The refractive constant "a" for human hæmoglobin, determined by the authors on pure hæmoglobin solutions, the concentration of which was determined by getting the dry weight at 110° C., was found to be $194{\cdot}2 \times 10^{-5}$. This compares favourably with 183×10^{-5} found by Howard with crystalline horse hæmoglobin, and, if the substances are comparable, would indicate 5·7 per cent. of water of crystallisation. Subtraction from the refractive index of the hæmoglobin of the refractive index of water at the same temperature gives a figure proportional to the hæmoglobin concentration over a wide range. The method for hæmoglobin in blood is essentially a separation of the refractive index of hæmoglobin from those of the other blood constituents. It is, briefly: (1) Wash the red cells from a measured quantity of blood. (2) Hæmolyse with distilled water and saponin, add salt to throw down the stromata, make up to volume and centrifuge. (3) Determine the refractive index of the solution. (4) Determine the refractive index of the filtrate obtained by heat coagulation of the hæmoglobin. (5) Subtract (4) from (3) and divide by "a" for hæmoglobin. The following are discussed:—Separation from plasma; methods of hæmolysis; possible effects of saponin on the refractive index; possible effects of solution of the stromata; possible effects of dissolved leucocytes; and separation of hæmoglobin from non-protein constituents by heat coagulation. In the presence of sodium chloride and at a P_H close to the isoelectric point of 6·8, hæmoglobin can be coagulated in 3 minutes at 100° C., giving a clear filtrate. The refractive effect of hæmoglobin is easily separated from that of non-protein constituents under suitable conditions, since the refractive indices of the filtrates from the heat coagulation of hæmoglobin correspond closely to those of the ultra-filtrates from the same solutions. For obtaining the concentration of hæmoglobin in terms of dry weight of the substance applicable to blood, a detailed refractometric method is given, which, compared with the oxygen capacity of the same blood obtained by the methods of van Slyke and Haldane (which do not agree), gives a ratio which definitely supports the former. A short method is also described, the use of which is advised with a small correction, for it checks closely with the longer and more conclusive method and shows no variation in the degree of solution of the stromata. P. H. P.

Toxicological and Forensic.

Normal Arsenic Content of the Human Body. O. Billeter and E. Marfurt. (*Helv. Chim. Acta*, 1923, **6**, 780–784.)—Using an analytical method, not described in the paper, by which they claim to be able to estimate arsenic in organic tissues with an accuracy of ±0·00003 mgrm., the authors found appreciable quantities in all the organs examined. The contents are of the same order of magnitude, and average 0·0103 mgrm. per organ; the thyroid gland contains 0·0032 mgrm. more, the spleen 0·0019 less. The amount of arsenic increases with age. The body of an adult person contains probably about 0·1 mgrm. of arsenic.

W. R. S.

The Fate of Arsenic in the Fermentation of Arsenical Fruit Juices. H. Bosselmann and A. Koch. (*Zeitsch. Unters. Nahr. Genussm.*, 1923, **46**, 10–33.) When fruit juices containing small quantities of arsenic undergo fermentation there is a diminution in the arsenic content of the liquor. This is due to the activity of the yeast cells; there is no liberation of gaseous arsenic compounds, but hydrogen sulphide is generated by the yeast, which forms colloidal arsenious sulphide which is then adsorbed by the plant. The relative amount of arsenic adsorbed in this way depends upon the quantity present, the higher the arsenic content the less the proportion adsorbed. The presence of copper facilitates this adsorbtion of arsenic by the yeast, provided that sufficient hydrogen sulphide is generated to form copper sulphide, which is also adsorbed. Isinglass tannin readily carries down arsenious sulphide, but arsenious oxide is not precipitated by isinglass or adsorbed by yeast. The presence of arsenic exerts slight inhibition on the action of the yeast, but this antifermentative action also is lessened by any trace of copper present.

H. E. C.

Agricultural Analysis.

Oxygen-supplying Power of the Soil as indicated by Colour Changes in Alkaline Pyrogallol Solution. L. M. Hutchings. (*J. Agric. Res.*, 1923, 25, 133–140.)—An apparatus is described which is designed to measure the actual oxygen-supplying power of a soil by diffusion, which is the real condition determining the health of roots, so far as oxygen is concerned. It consists essentially of a thin porous pot, soaked in melted paraffin wax, which is closed by a rubber stopper and connected by glass tubes with an indicator bottle containing alkaline pyrogallol. Arrangement is made whereby the potassium hydroxide solution and the pyrogallol are actually mixed in the bottle after all oxygen has been displaced, so that a colourless solution is obtained. On placing the pot at a suitable depth in the soil under exactly prescribed conditions the rate of oxygen absorption through its walls is indicated by the time taken for the pyrogallol solution to change in colour from that of a pale standard solution of copper-cobalt-iron chloride to that of a darker solution made from the same salts. The apparatus is standardised in air and has an oxygen-absorbing capacity of about 0·02 c.c. per hour. The arrangement and manipulation is described in exact detail for which the original must be consulted.

H. E. C.

Microscopic Estimation of Colloids in Soil Separates. W. H. Fry. (*J. Agric. Res.*, 1923, **24,** 879–883.)—Samples of soil are repeatedly treated by agitation with distilled water containing a trace of ammonia, rubbed with a rubber pestle, and the supernatant liquor decanted and centrifuged. As many as 40 or 60 such treatments only remove about 60 per cent. of the colloid particles present; the fine and coarse residues are therefore examined under a petrological microscope, with the use of an eye-piece micrometer, and the relative space occupied by the mineral particles and the colloid aggregates is calculated. This is easily possible by reason of the fact that the colloid aggregates are transparent or translucent, whereas the mineral particles are opaque. The method is exact but tedious, as 10 or 12 counts of four slides are necessary to ensure reliable results. H. E. C.

Organic Analysis.

Detection of Pentoses, Formaldehyde and Methyl Alcohol. J. B. Sumner. (*J. Amer. Chem. Soc.*, 1923, **45,** 2378–2381.)—A stable modification of Bial's reagent for pentoses is recommended: Six grms. of orcinol and 40 drops of a 10 per cent. solution of ferric chloride are dissolved in 200 c.c. of alcohol, without the addition of acid. Fifteen drops of this reagent and 5 c.c. each of the sugar solution and of concentrated hydrochloric acid are heated in boiling water. In the presence of 1 mgrm. of arabinose or xylose a clear blue colour is developed, and with a larger quantity a precipitate forms. Under these conditions formaldehyde also reacts; 2 mgrms. give a white precipitate, 0·2 mgrm. yields a precipitate only on scratching the sides of the tube, and a less quantity gives a yellow coloration which, on adding excess of sodium hydroxide, turns pink, with an intense green fluorescence. The following modification is suitable for the detection of methyl alcohol down to 0·5 per cent. in alcoholic liquors. To 1 c.c. of the concentrated alcohol are added, in a wide-mouthed test tube 2 c.c. each of a 6·7 per cent. solution of potassium dichromate and of sulphuric acid (62 per cent.); after the mixture has stood for about 10 minutes 15 c.c. of water are added, and the solution heated for 10 minutes in boiling water, after which 5 mgrms. of orcinol in 1 c.c. of water are added, and the heating continued for 30 minutes. In the presence of 5 per cent. of methyl alcohol a brown precipitate forms in 5 minutes, and with 1 per cent. in about 15 minutes, whilst 0·5 per cent. only produces a precipitate after the liquid has been cooled and allowed to stand for half an hour. Formic acid, amyl alcohol, acetone or furfural do not interfere with the reaction. H. E. C.

Estimation of Tolidine. S. Palkin. (*J. Ind. Eng. Chem.*, 1923, **15,** 1045.) —The method depends on the insolubility of tolidine dihydrochloride in sodium chloride solution. About 1 grm. of the sample is dissolved in 10 c.c. of warm 3·5 per cent. hydrochloric acid, and 100 c.c. of saturated sodium chloride solution are added. After thirty minutes the crystalline precipitate is collected on a small filter and washed with saturated sodium chloride solution until all free acid has been removed. The filter and precipitate are then transferred to a beaker, the precipitate is dissolved in hot water and 0·1 *N* ammonia solution is added until the solution is slightly alkaline to methyl-red. The precipitated tolidine is collected on a filter, washed with water, and the excess of ammonia in the filtrate is titrated with 0·1 *N* hydrochloric acid. Each c.c. of 0·1 *N* ammonia solution is equivalent to 0·0106 grm. of tolidine. Sodium hydroxide solution may be used for the titration, provided that it is free from carbonate. W. P. S.

Analysis of Triphenylmethane and Azo Dyes by means of Titanium Trichloride Solution. W. S. Calcott and F. L. English. (*J. Ind. Eng. Chem.*, 1924, **15,** 1042–1044.)—Dyes of the triphenylmethane class, such as pontacyl green S, crystal violet, victoria green, etc., may be titrated with titanium trichloride solution in the presence of sodium tartrate solution. The titanium trichloride solution should be about 0·05 *N* in concentration and be standardised against pure metanil yellow which has been crystallised twice from water and three times from alcohol and dried at 120 C. For the titration a weighed portion of the sample is dissolved in a mixture of 50 c.c. of water and 50 c.c. of alcohol, 50 c.c. of 25 per cent. sodium tartrate solution are added, the mixture is boiled for five minutes, a current of carbon dioxide is passed into the flask, and the hot solution is titrated with titanium trichloride solution, the end-point being denoted by the last distinct change in colour. The results obtained are not affected by considerable changes in the conditions of the titration (excess of sodium tartrate, temperature, etc.) In the application of the method to azo dyes it is necessary to select a solvent (water, alcohol, acetic acid, or mixtures of these) which will decrease the high colour of the reduced solution so that the end-point can be observed; the choice of a solvent depends on a preliminary trial. W. P. S.

Properties of Dyed Materials. H. Wales. (*J. Amer. Chem. Soc.*, 1923, **45,** 2420–2430.)—Study of the absorption spectra of the light reflected from dyed materials shows that reflection spectra more nearly explain the variations in colour between two dyes than the transmission spectra of the solutions. It is also shown that dyeings on wool, paper, or gelatin really consist of a solution of the dye in the material. Many sulphonated azo dyes may be extracted from their aqueous solutions by diluting them with an equal volume of hydrochloric acid and shaking out the solution with amyl alcohol; repeated washing with dilute (1:1) hydrochloric acid will not remove the dye from the alcohol, but with a lower concentration of hydrochloric acid the dye passes to the aqueous layer. In investigating the dyes it is important not to use any acids or salts as mordants, since these cause secondary reactions on the fibre—a point which has been often overlooked. H. E. C.

Chemical Analysis of Cotton. I. Absorption of Methylene Blue by Cotton. C. Britton, D. A. Clibbens and B. P. Ridge. (*J. Textile Inst.*, 1923, **14,** T.297–314.)—A quantitative investigation of the absorption of methylene blue by a large number of samples of unbleached cotton either as loose fibre, yarn, or cloth, shows that there are a number of factors affecting the value. The concentration of the methylene blue solutions was determined by a colorimetric method where the concentration was about 0·05 and 0·25 millimole per litre, and a titrimetric method for concentrations of about 2·5 to 3·5 millimoles per litre, the latter method depending on the fact that an acid dye, such as naphthol-yellow-S, when run into a basic dye like methylene blue, forms a reddish brown precipitate, the blue colour of the solution disappearing and a yellow colour forming. It may be noted that for a normally bleached American cotton the absorption of the methylene blue was of the order of 0·1 grm. for 100 grms. of cotton.

Since an increase in the alkalinity of the ash content results in an increased absorption of methylene blue, other factors being equal, the disturbing influence of such variations was eliminated in all samples under treatment by washing with dilute acid. It was found that American cottons bleached by a normal efficient process showed the same absorption power within narrow limits, whilst Egyptian cottons, again agreeing among themselves, invariably showed higher absorption power. Further, the quality of the cotton being the same, the absorption was found to be influenced by the efficiency of the bleaching. In particular, non-cellulose impurities insufficiently removed by the scouring process caused a higher absorption, unaffected by the subsequent chemic. Any addition of soap or olein to the keir liquor had a similar result if left in the cotton in the form of insoluble soaps. Processes affecting the surface properties or degree of dispersion of the cotton cellulose had no effect on the absorption. It is thus possible to follow the progressive purification of the cotton cellulose during bleaching by the methylene blue absorption power, and the data given by the whole investigation form a basis for the systematic study of the relations between extent of oxidation (or over-bleaching) of cotton and its absorption of methylene blue. D. G. H.

Wearing Qualities of Shoe-leathers. (*Bull.* No. 1168., *U. S. Dept. Agric.*, 1923.)—Details are given of an investigation on this subject under military conditions, with the object of testing the effect on the wearing qualities of the kind of tannage, loading materials, and grease. The shoes were all worn in a very dry climate where the soil was alkaline. In upper leathers, those which had been bark-tanned were decidedly harder and stiffer than those chrome-tanned, but the problem centred itself largely on the sole-leathers, as these always wore out at a much greater rate than the uppers.

The number of pairs of soles tested in each case varied from 31 to 126. There was very little difference between the four vegetable leathers, as shown by the following figures, which give the average number of days' wear per standard thickness:—Oak bark, 78·1; chestnut-wood extract, 80·0; hemlock bark, 78·5; and oak belting, 86·5. The waxed chrome-tanned soles wore longer (102 days), and unwaxed chrome-tanned longest of all (125·8 days). Shoes with fibre soles, if not ripped or broken, wore very well (120 days), but these imperfections were numerous. Duration of period of tanning was not a material factor. There was no indication that leathers loaded with 9 per cent. of glucose and Epsom salts wore more quickly in a dry climate than those containing only 2 per cent. Soles containing the usual oil content appeared to wear slightly longer than those of high oil content. Under damp-wearing conditions this result might have been different. Leathers plumped with sulphuric acid before tanning wore a slightly shorter time than those not so treated. In the case of rolled leathers the period of wear was 16 per cent. longer than in the case of unrolled leathers, thus supporting the usual tannery practice. R. F. I.

Seeds and Fruit of Certain Field Weeds. J. Greger. (*Zeitsch. Unters. Nahr. Genussm.*, 1923, **45**, 357–361.)—The structure and microscopic characteristics of the fruit of the three varieties of knot-grass, *Polygonum aviculare, P. perspicaria* and *P. convolvulus*, and of three varieties of dock, *Rumex crispus, R. acetosa* and *R. acetosella*, are described in detail. Although the differences in the varieties are not great, they are readily distinguishable even in fragments. The best diagnostic feature of knot-grass is the characteristic large warty epicarp cells of the fruit; in *P. convolvulus* these cells are of a dark brown colour and have thick walls irregularly grooved and twisted; they are of about 100μ in length and 25μ diameter. In *P. perspicaria* the length of these cells is about 85μ long, and they taper at the ends; they are of yellowish brown colour in section resembling pallisade cells. *P. aviculare* resembles *P. convolvulus*, but the cells are smaller, being about 60μ by 14μ, do not taper so much at the end, and are of a golden yellow colour. The fruits of the three varieties of *Rumex* are almost identical in structure, the leading feature being the epidermal cells of the pericarp; these are of average widths 17μ, 20μ, and 50μ in *R. crispus, acetosa* and *acetosella* respectively. The seed coat of *R. crispus* contains dark reddish brown pigment cells which yield characteristic colours. The pigment is soluble in hot water or glycerin; sulphuric acid changes it to a deep golden yellow; ammonia yields a red colour changing to carmine, then through brown to yellow. In *R. acetosella* the cells are only a pale yellow, and the pigment does not give the above colour reactions, and in *R. acetosa* the pericarp cells are almost identical in size and appearance with those of *R. crispus*, but contain a light yellow pigment which does not give the colour reactions described for this variety. H. E. C.

Inorganic Analysis.

Bromoxylenol Blue. A True-Neutrality Point Indicator. A. Cohen. (*Biochem. J.*, 1923, **17**, 535.)—The author has previously synthesised xylenol blue (*Biochem. J.*, 1922, **16**, 31), and shown that its double working ranges of utility coincide with those of thymol blue; thus the pair of indicators share with certain other pairs of homologous indicators the property of anomalous unshifted P_H ranges. Owing to this, a solution of xylenol blue of twice the intensity of thymol blue was obtained. Similar investigation in buffer solutions of $P_H=6{\cdot}0$ to $7{\cdot}6$ shows that the analogy of these two indicators in respect of range of P_H is continued on bromination. Xylenol blue is the more easily brominated. To 1 part of xylenol blue, suspended in 10 parts of glacial acetic acid, 3·1 parts of bromine are added, in the cold, with constant shaking. The pale pink crystals, after standing overnight, are thoroughly drained by suction and recrystallised from anhydrous boiling toluene. On cooling, a white crystalline product separates, which is, as analysis has shown, a dibromoxylenol sulphonephthalein. Bromoxylenol blue, like bromothymol blue, can have its working range halved by the addition of bromocresol purple, as the author has shown (*J. Amer. Chem. Soc.*, **44**, 1851); and the resulting mixed indicator can then be used where a blue end-point of Sörensen value about 6·8 is desired in a titration. Dibromoxylenol sulphonephthalein has the formula $C_{23}H_{20}O_5Br_2S$. P. H. P.

Estimation of Sulphur, Arsenic, and Antimony in Inorganic and Organic Compounds. F. Feigl and R. Schorr. (*Zeitsch. anal. Chem.*, 1923, **63**, 10–29.)—The method is termed "oxidising sintering," and is somewhat similar to Eschka's sulphur estimation in coal. The very finely powdered substance is intimately mixed in an iron crucible with 2 grms. of potassium permanganate and 2 grms. of sodium carbonate (both finely powdered and quite dry), and the mixture is covered with 3 grms. of equal parts of the two salts. The covered crucible is heated 20 to 30 minutes over a small flame, then 30 to 40 minutes at a stronger heat until the bottom of the crucible is red hot. The cold mass is transferred to a beaker, and the crucible cleaned with hot water. Any permanganate (manganate) is destroyed by addition of alcohol. After digestion on a water-bath the liquid is filtered, and the residue well washed with hot water containing a little sodium hydroxide. The sulphate in the acidified filtrate is precipitated as usual with barium chloride solution. The method gave very good results when applied to various sulphide ores, phenylthiourea, sulphanilic acid, and sulphonal. In the estimation of sulphur in organic substances, the raising of the temperature must take place very gradually, and the oxidising mixture must be very thoroughly dehydrated. The above method is applicable to the estimation of arsenic in ores and organic substances (*e.g.* atoxyl, various substituted phenylarsinic acids, and neosalvarsan); the filtrate containing arsenate is precipitated as usual with magnesia mixture. The precipitate may be ignited and weighed, or dissolved on the filter in 25 per cent. hydrochloric acid, and the solution is treated with 2 to 3 grms. of potassium iodide, and titrated, after 15 minutes' standing, with thiosulphate. For the estimation of arsenic, sulphur, and antimony in ores, the sintered mass is leached with 30 per cent. alcohol, the extract filtered after 24 hours, and the residue washed with alcohol of the same strength. The filtrate is made up to 500 c.c., and the arsenic and sulphur estimated in aliquot portions. The residue is dissolved in dilute hydrochloric acid, and a little oxalic acid; this solution contains the whole of the antimony. The method is being extended to the estimation of chlorine in organic substances. W. R. S.

Systematic Procedure for the Detection and Separation of Acids. Z. Karaoglanov and M. Dimitrov. (*Zeitsch. anal. Chem.*, 1923, **63**, 1–10.)—The solution is freed from heavy metals by the usual method and tested for acetic acid by the ester or cacodyl reaction. From 20 to 50 c.c. of the neutral solution are acidified with 5 to 10 c.c. of 2 *N* acetic acid; a current of air is passed through the liquid, the gases being made successively to pass tubes containing solutions of silver nitrate in nitric acid, barium hydroxide, and potassium iodide with starch. A precipitate in the first tube indicates cyanide, sulphide, or hypochlorite; in the second, carbonate, sulphite, or thiosulphate (in which case, the original solution treated with strong sulphuric acid becomes milky); and in the third the blue colour shows hypochlorite or nitrite. A larger quantity of original solution is evaporated with acetic acid for the elimination of the above acids before testing for the remainder. The residual solution is diluted with water, and a small portion tested with diphenylamine. If a blue colour is obtained, the presence of nitrate is definitely proved by boiling with excess of potassium hydroxide until any ammonium salts present are decomposed, and continuing the boiling after addition of zinc dust. Evolution of ammonia proves the presence of nitrate. Another part of the solution is tested for borate as usual. The bulk of the diluted residual solution is used for the separation of the other acids as follows:—Barium acetate is added in portions, followed by calcium acetate until no further precipitate forms. After two hours' standing, with occasional stirring, the precipitate is filtered off, digested with dilute nitric acid for 15 minutes, and the liquid again filtered; the precipitate is tested for sulphate (silver coin test), fluosilicate, and iodate, and the filtrate for fluoride, oxalate, chromate, and iodate. The main filtrate is made feebly ammoniacal and treated with barium acetate (precipitate: phosphate, arsenate, silicate). The filtrate is diluted, neutralised with ammonia, and treated with lead acetate, and the precipitate filtered off after one hour (precipitate: iodide, arsenite, tartrate, ferrocyanide). The filtrate is treated with excess of lead acetate, made strongly ammoniacal, boiled for a long time, and filtered while hot (precipitate: ferricyanide precipitated as ferrocyanide, and arsenite). Part of the filtrate is tested for thiocyanate; the remainder is treated with excess of nitric acid and silver nitrate (precipitate: chloride, bromide, thiocyanate, traces of iodide). The filtrate from the silver precipitate may contain chlorate, perchlorate, and bromate. Directions are also given for the identification of each anion in the various fractions. W. R. S.

Separation of Metals of the Ammonium Sulphide Group. G. Luff. (*Zeitsch. anal. Chem.*, **1923, 63,** 32–39.)—The separation of ferric iron and aluminium from manganese, zinc, and nickel (cobalt) was tested by (1) single precipitation with ammonia, (2) double precipitation with ammonia, (3) precipitation with pyridine, and (4) precipitation with pyridine followed by ammonia. The ammonia solution was added, drop by drop, to the boiling neutral solution containing 10 grms. of ammonium chloride per 100 c.c., until a spot test showed a feeble alkaline reaction to litmus paper. In method (1), only manganese was separated almost quantitatively. Method (2) separated manganese, zinc, and nickel quantitatively from iron and alumina, but cobalt required at least 3 precipitations. In the pyridine tests, 5 c.c. of the precipitant were added, drop by drop, to the almost neutral, cold solution containing ammonium chloride as before; the solution was filtered after 15 minutes' digestion at boiling heat, filtered, and the precipitate washed with hot water containing a little pyridine. The separation of manganese was good, of nickel fair, of zinc and cobalt incomplete. Method (4) gave good results, except for cobalt, which required another ammonia separation.

W. R. S.

Estimation of Carbon Monoxide in Air. V. Andriska. (*Zeitsch. Unters. Nahr. Genussm.*, 1923, **46,** 43–46.)—The tannin reaction described as a qualitative test by Weltzl (*Ver. Phys. Med. Gesellschaft, Wursburg.*, 1889, **33,** 3) is modified to form an exact quantitative method. The air is shaken out with blood solution (1 part of defibrinated ox blood to 4 parts of water), and to 5 c.c. of this are then added 15 c.c. of 1 per cent. tannin solution. Experiment shows that the colour developed in 24 hours is just detectable when the blood solution contains one per cent. of carbon monoxide. The method is therefore to shake 5 litres of the air with 1/100 of its volume of blood solution for three hours, and then prepare a series of test tubes with 0·5, 1·0, 1·5—5·0 c.c of this blood solution, to which are added respectively 4·5, 4·0,—0·0 c.c. of the fresh blood solution. To each tube are added 15 c.c. of tannin solution, and the mixtures, after shaking, allowed to stand for 24 hours. The tube which then only just shows the reaction contains one per cent. of carbon monoxide in the 5 c.c., and from this it is a simple matter to calculate the amount in the sample of air.

H. E. C.

Separation of Arsenic and Phosphorus from Vanadium. H. A. Doerner. (*J. Ind. Eng. Chem.*, 1923, **15,** 1014.)—A method described for the separation of pure vanadium pentoxide from vanadium ores containing arsenic and phosphorus depends on the precipitation of vanadic acid from an acid solution. Five hundred grms. of the sample (*e.g.* vanadinite concentrate) are fused in a graphite crucible with 150 grms. of sodium carbonate, 150 grms. of sodium hydroxide and 10 grms. of powdered charcoal; the lead button obtained is separated, the slag is powdered and extracted with hot water, and the solution filtered. The filtrate is oxidised with sodium peroxide and diluted to 2 litres, and sulphuric acid is added until the concentration of the free acid is 0·05 *N* as determined by titration, with the use of methyl-orange as indicator. The mixture is boiled for several hours by the introduction of steam, kept hot for eighteen hours, and the precipitated vanadic acid then collected and washed.

W. P. S.

Reduction of Bismuth by Glucose. H. Cousin. (*J. Pharm. Chim.*, 1923, **28,** 179-181.)—In order to determine whether bismuth, in the subnitrate for example, could be estimated by reduction with glucose by a method analogous to that of Vanino and Treubert (*Ber.*, **31,** 1303), who reduce the bismuth salt with formaldehyde and weigh the precipitate, 1·231 and 1·704 grms. of the subnitrate were dissolved in nitric acid, and to the diluted solution 10 grms. of glucose and 30 c.c. of sodium hydroxide were added. After being heated on a water bath the precipitate was filtered off and weighed. For the above weights of subnitrate the precipitates weighed, respectively, 0·884 and 1·227 grms., corresponding to 71·81 and 72·01 per cent., respectively, of bismuth in the salt. It is preferable, however, to use a simpler method for estimating bismuth in such salts. D. G. H.

Separation of Tin from other Metals and Estimation of Tin by means of Cupferron. N. H. Furman. (*J. Ind. Eng. Chem.*, 1923, **15**, 1071-1073.)—The cupferron method described by Kling and Lassieur (ANALYST, 1920, **45**, 275), yields accurate results; the tin should be weighed as stannic oxide. It is essential in the separation of tin from antimony in dilute hydrofluoric acid solutions that the antimony should be present in the trivalent state. The cupferron method also affords a means of separating tin from zinc, manganese, cobalt and nickel.

W. P. S.

Detection and Estimation of Didymium in Cerium Salts. L. Dede. (*Chem. Zeit.*, 1923, **47**, 82.)—When the light from mercury vapour is made to pass through crystals or solutions of didymium salts, the absorption band covers the two yellow lines of mercury, and the solution or crystals appear blue. The reaction is sensitive; thus crystals of cerium nitrate in which the didymium band can hardly be detected in a 20 mm. layer of 5 per cent. solution, appear distinctly blue in mercury vapour light. In dilute solutions the absorption of the mercury lines is proportional to the concentration of the didymium, which can therefore be estimated colorimetrically without a spectral photometer. W. R. S.

Separation of Uranium from Thorium and Rare Earths in Pitchblende. W. Riss. (*Chem. Zeit.*, 1923, **47**, 765-766.)—The mineral is dissolved in nitric acid and the silica removed in the usual manner. The filtrate is repeatedly evaporated to dryness for the elimination of free acid. The residue is dissolved in water; the solution in a beaker is neutralised exactly with a few drops of very dilute ammonia, strongly diluted, and boiled for 10 minutes with excess of thiosulphate. After standing 24 hours the precipitate is filtered off, well washed with water containing a little thiosulphate, dissolved in hydrochloric acid, and re-precipitated, after elimination of free acid, as before. If properly performed, the second precipitation removes the last of the cerium. The precipitate, which contains part of the uranium, is dissolved in hydrochloric acid, and the thorium precipitated as oxalate. The filtrate is evaporated to dryness in a porcelain dish, and the residue cautiously heated with strong sulphuric acid for the decomposition of the oxalic acid; the acid is finally heated until strong fumes appear, and the uranium precipitated with ammonia after cooling and diluting the solution. The filtrate from the thiosulphate precipitate, containing the balance of the uranium, is freed from rare earths by means of oxalic acid. The excess of precipitant is destroyed as before, and the iron separated from uranium by precipitation with ammonium carbonate, uranium being again precipitated with ammonia. The united uranium precipitates are ignited and weighed. W. R. S.

Indirect Estimation of Alkalis in Rocks. F. Walker. (*J. Chem. Soc.*, 1923, **123**, 2336-2341.)—The Lawrence Smith method was used with the following modifications: (1) The ignition was carried out in an ordinary platinum crucible with a ventilated lid, and placed on a perforated silica plate beneath which two-fifths of the crucible projected. (2) The bulk of the calcium was removed by

double precipitation with ammonium carbonate in a porcelain basin on a water-bath; two drops of ammonium oxalate solution were added, the solution left on the bath for 5 minutes, and decanted off; the precipitate was dissolved in the minimum quantity of hydrochloric acid, the solution re-precipitated and filtered into a platinum basin, and the precipitate thoroughly washed with hot water. (3) After the first evaporation to dryness on a water-bath, the platinum basin was heated in an oven to 140° C. while covered with a previously heated clock-glass. The moisture was thus expelled in 15 minutes, and the particles which decrepitated did not adhere to the cover. In the final precipitation of the calcium, 1 drop of ammonia and 5 drops of ammonium oxalate solution were used. (4) In presence of sulphur in the rock, 2 to 3 drops of barium chloride solution were added before the final precipitation of calcium, the excess of barium salt being precipitated with a few drops of ammonium carbonate solution. In the check analyses by the platinum chloride method, absolute methyl alcohol was used. A blank estimation was made with all the reagents used, which were purified if necessary. The calcium carbonate (Kahlbaum) used (4 grms.) left a residue of 0·0015 grm.; this was dissolved in 1 c.c. of water and titrated with silver nitrate in presence of chromate; the amount consumed was deducted from the reading in the actual estimation, while the weight of the residue was deducted from that of the mixed chlorides. In the analysis of rocks sufficient powder should be taken to yield at least 0·04 and not more than 0·06 grm. of mixed chlorides, *i.e.* 0·6 to 0·35 grm. This is mixed with 4 grms. of calcium carbonate and 0·5 grm. of ammonium chloride. The weighed mixed chlorides are dissolved in 20 c.c. of water in a porcelain dish; 0·25 c.c. of 0·2 *N* potassium chromate solution are added, and the liquid titrated with 0·02 *N* silver nitrate solution, added in portions of 0·5 c.c., until the pale yellow tinge changes to very pale salmon pink. The average time for an indirect estimation is 4½ hours. When the purest reagents are used, the indirect method is as accurate as the platinochloride method. This involves the following sources of error: Some impurities in the mixed chlorides are wholly or partly insoluble in alchohol, and remain with the potassium precipitate, causing high potassium and low sodium results. The impurities in the reagents estimated by a blank assay are assumed to be sodium chloride, but, besides this, at least calcium chloride and carbon are present. Some potassium platinochloride is dissolved by the alcohol, which tends to lower the potassium and raise the sodium figure.
W. R. S.

Separation of Potassium from Sodium by the Perchlorate Method. G. F. Smith. (*J. Amer. Chem. Soc.*, 1923, **45**, 2072–2080.)—The process consists in precipitating potassium perchlorate by the gradual addition of a large quantity of normal butyl alcohol containing a little perchloric acid. The solubility of sodium perchlorate under these conditions is about 5 per cent. at 25° C. The solubility of potassium perchlorate is materially diminished by addition of perchloric acid, being 0·0016 grm. per 100 c.c. of the mixture obtained by the procedure. The weighed mixed chlorides, free from sulphate, are evaporated to dryness with excess of perchloric acid; a second evaporation is desirable if the quantity is large. Two or three c.c. of water are then added, and the salts dissolved by heating. Sixty-five or 100 c.c. (according to the volume of water used) of butyl alcohol containing 1 per cent. of perchloric acid are heated to boiling and gradually added to the warm perchlorate solution, with vigorous stirring. The solution is gently boiled for half a minute, cooled, filtered through a Gooch crucible, and the precipitate washed with 8 to 10 portions of 1 to 2 c.c. of precipitant; it is dried at 150° to 250° C., and weighed as $KClO_4$. The filtrate and washings are diluted considerably and evaporated on a water-bath; if the residue is not colourless, the beaker is heated on a hot plate till fumes of perchloric acid are evolved; a little more acid may be added, if necessary. When colourless, the residue is heated with 0·5 c.c. of strong sulphuric acid until fumes appear, and then transferred to a platinum crucible for the estimation of sodium as sulphate. The results of test analyses show an average negative error of 0·0002 grm. for KCl, and a positive one of 0·0006 for NaCl, due to a compensation of errors: in the absence of sodium, the correction is 0·0011 grm. for the solubility of potassium perchlorate per 100 c.c., equivalent to 0·00038 grm. of K_2O. On account of the slow evaporation of butyl alcohol at water-bath temperature, the direct estimation of sodium is rather inconvenient, and it is preferably estimated by difference from the weight of the mixed chlorides. W. R. S.

Estimation of Fluorine in Zinc Blende. L. da Rocha-Schmidt and K. Kruger. (*Zeitsch. anal. Chem.*, 1923, **63**, 29–32.)—One grm. of the dry, finely powdered ore is intimately mixed with 2 grms. of ignited fine quartz powder, 2 grms. of sea-sand (previously boiled with strong sulphuric acid, washed, and ignited), and 5 grms. of dehydrated copper sulphate. The mixture is transferred to Treadwell's distillation apparatus (*Treadwell's Quantitative Analysis*) and covered with 10 grms. of chromic acid. All the parts of the apparatus must have been dried over phosphorus pentoxide. A current of carefully dried air is passed through the system, and 20 c.c. of strong sulphuric acid admitted through the funnel. The acid is dehydrated by boiling off one-third of its bulk in a beaker, after which it is left to cool in a well-dried, empty desiccator; this should contain no phosphorus pentoxide, otherwise a certain amount of sulphur trioxide is formed by condensation of acid vapours on the drying agent. The decomposition vessel is heated gradually in an oil-bath until, after 2 hours, the temperature reaches 130° to 140° C. After about 3 hours' heating the apparatus is allowed to cool for half-an-hour in the air current. The liquid in the receivers (containing a saturated solution of potassium chloride in 50 per cent. alcohol) is titrated with 0·1 *N* sodium hydroxide solution in presence of freshly-made cochineal tincture (1 c.c. 0·1 *N* alkali $=0{\cdot}0057$ grm. F). W. R. S.

Titration of Hydrofluoric Acid and Hydrofluosilicic Acid in Mixtures containing Small Amounts of Hydrofluosilicic Acid. P. H. M. P. Brinton, L. A. Sarver and A. E. Stoppel. (*J. Ind. Eng. Chem.*, 1923, **15**, 1080–1081.)—In a method described by Scott the sample of the mixed acids is treated with potassium nitrate and titrated at 0° C. with sodium hydroxide solution, phenol phthalein being used as indicator—$2HF+2NaOH=2NaF+2H_2O$ and $H_2SiF_6+2KNO_3+2NaOH=K_2SiF_6+2NaNO_3+2H_2O$. The mixture is then heated to about 80° C., and the titration continued to the second end-point—$K_2SiF_6+4NaOH=2KF+Si(OH)_4$. Investigation of this method showed that trustworthy results can be obtained only when the alkali solution used for the titration is free from silica, or when an allowance is made for the amount of silica present. The sodium hydroxide should be prepared by the action of water vapour on sodium metal, the hydroxide collected in a platinum vessel and the solution stored in bottles lined with ceresin wax. If use is made of sodium hydroxide solution stored in plain glass bottles, the silica content must be estimated; the quantity dissolved from the glass increases rapidly as the solution is kept. Each molecule of silica reacts with six molecules of hydrofluoric acid to form one molecule of hydrofluosilicic acid; therefore the weight of silica added (in the alkali solution) during the cold titration must be multiplied by the factor 2·393 to obtain the weight of hydrofluosilicic acid to be deducted from the weight of this acid found, and by the factor 1·991 to obtain the weight of hydrofluoric acid to be added to the weight found. The sodium hydroxide solution should also be free from carbonate. The method of weighing out the portion of the mixed acids for the titration may introduce an error; if the acid is weighed in a platinum vessel, there is some loss of acid vapour when the vessel is opened. It is recommended that the sample be weighed in a rubber bottle which has been nearly filled with crushed ice and weighed. To overcome the change in weight, due to condensation of moisture on the cold bottle, the latter is weighed in a stoppered weighing bottle well lined with asbestos paper and counterpoised by a weighing bottle of similar external form on the opposite balance pan. After weighing, the bottle is shaken to absorb any free acid vapours, and the cold dilute solution may then be rinsed out into the alkali solution without loss.

W. P. S.

Physical Methods, Apparatus, etc.

Porous Glass Filter Plates for Laboratory Use. G. F. Hüttig. (*Chem. Zeit.*, **1923**, **47**, 782.)—Porous glass plates are made by Schott and Gen., Jena, by sintering together more or less finely powdered and screened glass. The plates are made in different sizes, thicknesses, and degrees of porosity, and of various kinds of glass; they can be fused on to filters, crucibles, and other apparatus made of glass of the same composition.

W. R. S.

Surface Tension Balance. Apparatus for the Rapid Measurement of Surface Tension. R. G. Green. (*J. Ind. Eng. Chem.*, 1923, **15**, 1024–1025).—The apparatus consists, essentially, of three parts—namely, a dropping pipette, a balance beam mounted on a torsion wire, and an adjustable scale. The balance beam carries at one end a pivoted silver cup and the torsion of the wire is indicated by a pointer moving over a scale on which the readings indicate surface tension in dynes per cm. Two types of the balance are described in detail.

W. P. S.

Transparent Preparations of Coal for Microscopic Investigations. J. and J. R. Lomax. (*Bull.* 14, *Lancashire and Cheshire Coal Research Association*, pp. 24. London: H. F. and G. Witherby. 1923. Price 2s. net.)—This bulletin deals only with the technique of preparation of transparent slices. The authors, by the use of the method described, have succeeded in obtaining transparent sections of coal up to 8 by 4 in. without previous treatment with softening agents, the use of which is condemned because it renders the photographic record incomplete by the removal of certain constituents. To prevent disintegration of the coal in cutting, the block is treated with a binding solution (pale shellac in methyl alcohol), which renders it sufficiently compact to resist grinding and polishing; the cementing material is invisible. The surface from which it is proposed to prepare a section is then cut and ground perfectly flat and smooth. After being washed in a stream of water, the specimen is heated in a water-jacketted oven during 4 or 5 hours at a temperature slightly below 100° C. The heat treatment prevents the appearance of gas bubbles in the subsequent mounting. The prepared surface is immersed in shellac solution at a slightly lower temperature than was used in the drying, cooled, given a final polish, and mounted on glass with a mixture of copal gum and liquid Canada balsam (1:3). The glass slip is previously heated, covered with the mountant, and cooled to a suitable temperature before the block of coal is pressed upon it. A thin slice (1/8 in.) is next cut by means of a hack saw, the block being held in a vice. This section is ground down on revolving cast iron discs fed with different grades of carborundum powder. The final reduction must be carried out carefully by hand, first on a thick glass plate covered with the finest carborundum powder, then with oilstone. Soft coals are, as a rule, sufficiently translucent at a thickness of 1/1500 in., while hard coals must be reduced to about 1/2000. The sections are finished by being cut to a uniform size, washed with soap and water, and dried; they are covered with a thin cover glass attached with Canada balsam. Strict adherence to all the details of the procedure described is essential for success in the preparation. W. R. S.

Apparatus for Determining the Viscosity of highly Viscous Substances. G. V. Derfeld. (*Chem. News*, 1923, **127**, 228–229.)—The time taken for a brass or aluminium ball to fall through a perpendicular column of the liquid under definite conditions is compared with the time taken for the ball to fall through a similar column of glycerin. The apparatus consists of a vertical tube in an outer jacket resembling a Liebig condenser. Connected with the centre of the inner tube is a short elbow piece bearing a thermometer. At the lower end are two lead wires arranged as a contact device for use in noting the time taken by the ball in falling when dark coloured liquids are being tested. The apparatus is set up vertically by means of a plumb line; the outer jacket is filled with water at 70° C. and allowed to cool to 62° C., the inner tube filled with the liquid to be tested (previously warmed to 70° C.), is allowed to cool until the thermometer at the side indicates 58° C., while the water in the outer jacket is at 62° C. The time taken for the ball to drop is noted, and the apparatus is standardised with glycerin under similar conditions. By using balls of various sizes the apparatus may be modified for application over a large range of viscosities, and is specially useful for the examination of lacquers, varnishes and similar materials. H. E. C.

Reviews.

Dizionario di Merceologia e di Chemica Applicata. Vol. I., A to Cuscuta. By Professor G. Vittorio Villavecchia and Others. Fourth Edition, revised and enlarged. Milan: Ulrico Hoepli. 1923. Price 35 lire.

With this volume, the first of four, begins the fourth edition of Villavecchia's well-known Dictionary, which was first published in 1896. The third edition, which appeared in two volumes in 1911 and 1913, contained altogether 2730 columns of subject matter, whereas the present volume, covering only the letters A to C, comprises 872 columns. Nearly all the articles have been re-cast to bring them up to date, and many new ones have been introduced.

The substances dealt with include the raw materials and finished products of the chemical, pharmaceutical, metallurgical, foodstuffs, agricultural, dyeing, textile, and analogous industries. After the usual Italian name of each product are given the chemical formula, the Italian synonyms, the French, German, English, and Spanish names, the origin, description, preparation, commercial qualities, properties, composition, adulterations, characteristic tests, methods of storage and packing, and uses. Then follow references to regulations imposed on the use and manufacture of the product (in Italy) from considerations of health or public safety, and to the conditions under which transport by rail is permitted. The amount of the Italian customs' duty for each material is given, as well as statistics relating to production, importation and exportation.

Although the ground covered is, in some directions, more restricted than is the case with Thorpe's Dictionary, Villavecchia's work renders readily accessible to the reader of Italian a large amount of information useful, not merely to the chemist, but to a large portion of the business community also. The statistics of production, etc., are the latest available, and the modifications of the customs duties imposed by the Italian Government in October and November, 1922, are added as appendices. An alphabetical index of about 60,000 names—Italian and foreign—is promised when the work is complete, reference to any desired subject being facilitated by numbering, not the pages, but the columns, of the letterpress.

As regards the English equivalents of the Italian names, few actual errors are detectable, although such expressions as oxenhides, coccons, deadlynightshade, woodroot (for woodruff), cotton west (*i.e.* waste), bliste-ring flies (cantharides) succedaneous of the coffee (coffee substitutes), etc., call for attention.

T. H. Popnal

A Systematic Organic Chemistry. By W. M. Cumming, B.Sc., F.I.C., I. V. Hopper, B.Sc., F.I.C., and T. S. Wheeler, B.Sc., A.I.C. Pp. xxii. + 535. London: Constable & Co. 1923. Price 25s. net.

In writing this book the authors have produced so useful a volume that no apology is needed for the appearance of yet another work on organic chemistry. Although in many respects it goes far towards being "a complete Laboratory Guide to the Preparations and Estimations of Organic Chemistry," there are many things which with advantage might be included. For instance, in common with most existing text books, no mention is made of the highly interesting hydroaromatic compounds, which could be easily represented by dimethyldihydroresorcinol and its derivatives (Crossley, *J. Chem. Soc., Trans.* 1899, **75**, 772). These compounds are the more worthy of inclusion in view of the possible use, as a unique brominating agent, of dibromodimethyldihydroresorcinol (Gupta and Thorpe, *J. Chem. Soc., Trans.*, 1922, **121**, 1896), and also on account of the recent work of Thorpe and Ingold (*J. Chem. Soc., Trans.*, 1920 onwards) on intra-annular tautomerism and the structure of benzene. In the book itself we find the usual sections, arranged, however, in a somewhat unusual manner which, although very detrimental to the value of the book from the point of view of the elementary student, is by no means without merit.

The opening chapters are devoted to a clear, concise and well illustrated account of the apparatus and methods of organic chemistry. Several points deserve special commendation. The subject of fractional crystallisation is rarely understood by students, and still more rarely is a clear explanation to be found in books. Here we have the subject treated very fully, and the average reader can scarcely fail to understand the method described. The note on costing is invaluable, and teachers of chemistry everywhere would do well to insist on the cost of each preparation being worked out, for so many students leave our Universities and Colleges with absolutely no idea of this important aspect of chemistry.

The arrangement of Part II. is based on the Richter "alphabet," and, although very useful for reference, is in consequence such that the student has great difficulty in finding for himself a logical sequence of reactions. This defect is somewhat remedied by the suggested courses mapped out in Chapter I. The preparations described cover a very wide range, and supply the teacher with a mass of material from which by judicious selection many instructive practical courses can be evolved. The general discussions which are placed before each set of reactions greatly enhance the value of the book, and go far to bridge the gulf which, unfortunately, so often exists between the lecture room and the laboratory.

In the chapter on ultimate quantitative analysis the authors recommend the use of sulphuric acid and caustic potash for absorbing water and carbon dioxide, respectively, and point out that the carbon result is always low owing, in part, to the loss of water from the potash bulbs, yet no mention is made of the absorption combination of fused calcium chloride and soda lime. The present writer has always experienced difficulty in getting a satisfactory blank when working with potash bulbs and sulphuric tubes, owing to the slow but continuous decrease in weight of the former, but has always obtained good results when using calcium chloride and soda lime in the absorption apparatus. This method has also the advantage of avoiding dangerous liquids and fragile glassware. To find included a chapter on group estimations, with its implied demand for accuracy, is very refreshing now that there is gradually springing up a tendency among students to imagine organic chemistry as something empirical, and that only in the realm of physical chemistry is accuracy to be expected or obtained. While the authors bring the book up-to-date by including estimations based on the use of titanous chloride and give details for such a specialised estimation as that of anthracene, no mention is made of the technically important determinations of iodine and saponification values.

Of the inorganic section little need be said beyond the fact that the importance of "oleum" is realised, and a useful section on this comparatively new reagent is included. That water is the chief impurity in anhydrous aluminium chloride makes rather queer reading.

The book deserves a place in all chemical libraries, and although the arrangement of this book, since it runs parallel with no possible scheme of elementary lectures, renders such books as Gattermann's *Praxis des organischen Chemikers* preferable for beginners in the subject, it might, by a skilled teacher, be used with advantage from the very outset of a course. Nevertheless, the book under consideration is nearly ideal for honours students, and even those engaged in research will find within its pages much that is helpful. In fact, the book is probably one of the best of its kind in our language. HAROLD TOMS.

LA TECHNIQUE INDUSTRIELLE DES PARFUMS SYNTHÉTIQUES. By RENÉ SORNET, with a Preface by Prof. MARCEL DELÉPINE. Pp. 136. Paris: Gauthier-Villars et Cie. Price 10 francs.

This is a very concise little book, and contains much valuable information in regard to the preparation of various organic compounds of importance to the perfumery industry. The author has evidently an exact but rather limited personal knowledge of the manufacturing details of some of the substances dealt with in the text, and these parts are very well done indeed, but elsewhere the working directions are so very meagre as to be almost useless. Still, the book covers a great deal of ground in a thoroughly interesting way, and the author is quite modest in his claims as set forth in the Introduction.

It is most certainly a book to be bought by all interested in the manufacture of such compounds, and, moreover, students of organic chemistry might read this book with advantage and learn much valuable technique by making a few carefully selected preparations following the directions given within its pages.

L. G. RADCLIFFE.

WAVE-LENGTH TABLES FOR SPECTRUM ANALYSIS. By F. TWYMAN. Pp. 116. London: Adam Hilger, Ltd. 1923. Price 7s. 6d. net.

Undoubtedly the best form of advertisement in scientific work or professional practice is good work well and carefully done. The author and Messrs. Hilger may be congratulated on the compilation of this little book, which has been produced with care and accuracy, and will be of distinct service to all who use the spectrometer. The volume commences with a simple account of wave-length units, numbers and tables for their correction and interpolation; then follows a table of standard wave-lengths and lists of the most distinctive lines for each element. From these it is easily possible to identify elements in an unknown mixture. There is also a chapter on wave-lengths useful for calculating stellar radial velocities, an appendix on the condensed spark, and a complete list of references. The descriptive matter consists almost entirely of transcription or translation from the original papers of leaders in this branch of science.

The book is well bound, clearly printed and, so far as the reviewer has been able to check it from other tables, free from errors. It will help to make the spectrometer a useful instrument, even in a commercial laboratory.

H. E. Cox.

INK. By C. AINSWORTH MITCHELL. Pp. ix.+128. London: Sir Isaac Pitman & Sons, Ltd. 1923. Price 3s. net.

This little book is an interesting addition to the publishers' well-known series on Common Commodities and Industries, the main object of which is to give trustworthy information to all who have to deal with the various products in commerce.

Those who are not familiar with Mr. Mitchell's large hand-book will be surprised to learn how much chemistry is involved in the manufacture of such an apparently simple product as ink, and this point is well brought out, without undue technicality, in the pages of this book.

After a short outline of the origin and development of writing inks, the author gives concise, though readable, descriptions of the nature of the raw materials and the modern processes of making iron-gall inks, and these are followed by a brief account of the methods of testing inks, in which sufficient detail is given to enable those who have not specialised in the subject to understand the meaning of an analysis. The following chapters deal with logwood, vanadium, coloured inks and copying inks, and then comes a section on the characteristics of ink in writing, in which the author refers to a good deal of his own original work.

This is followed by a section on marking ink which, in passing, it might be pointed out should not be placed under the page heading of "Characteristics of Ink in Writing." Indian ink, sepia and similar preparations are dealt with in a chapter on "Inks for Artists," and then comes a chapter on printing inks, which includes an account of the recently-introduced rotagravure inks, and the book concludes with chapters on typewriting inks and carbon papers, and on inks for various special purposes.

There are 24 well-chosen illustrations, and there is a sufficiently full index.

The author does not profess to go very deeply into the subject, but it is remarkable how much scientific information has been compressed into a very small space, and the book may be recommended to all who wish to have ready to their hand a preliminary guide to knowledge which is essential for the chemical examination of inks.

A. BAKER.