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THE ANALYST.

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

ORDINARY MEETING HELD MARCH 5, 1919.

An ordinary meeting of the Society was held on Wednesday, March 5, in the Chemical Society's Rooms, Burlington House. The President, Dr. S. Rideal, F.I.C., occupied the chair.

The minutes of the previous meeting were read and confirmed.

Certificates of proposal for election to membership in favour of Messrs. R. O. Bishop, A.I.C.; H. W. Bywaters, D.Sc., Ph.D., F.I.C., A.R.C.Sc.; B. S. Evans, M.C., B.Sc., F.I.C.; F. S. Fowweather, M.Sc., A.I.C.; R. A. Oddy; W. B. Pollard; and C. K. Tinkler, D.Sc., F.I.C., were read for the second time; and certificates in favour of Mr. Madanlal Jekisandas Gajjar, M.A. (Bombay), Director of the Gwalior State Laboratory; and Mr. James Sorley, F.I.C., partner in the firm of Tatlock and Thomson, Analytical and Consulting Chemists, Glasgow, were read for the first time.

Mr. C. W. Wood was elected a member of the Society.

The following papers were read: "The Estimation of the Approximate Quantity of Meat in Sausages and Meat Pastes," by G. Stubbs, O.B.E., F.I.C., and A. More, A.R.C.S., F.I.C.; "Analysis of Sausages, Meat Pastes, and Army Rations," by A. W. Stokes, F.I.C.; and "The Estimation of Small Quantities of Acetone, Alcohol, and Benzene in the Air," by Major S. Elliott, B.Sc., F.I.C., and Captain J. Dalton, M.C.

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REGENERATION OF NESSLER SOLUTION.

BY D. PULLMAN.

(*Read at the Meeting, February* 5, 1919.)

As a result of the recent high prices of iodides and mercury salts the following method of regeneration of the Nessler solution has been found economical and efficient in a laboratory where a large number of samples of water are examined daily.

The problem resolves itself into two phases: The recovery of all the mercury and iodine, and the conversion of the products thus formed into a Nesslerising solution.

1. If, after neutralising the Nessler residues, a solution of a soluble mercuric salt be added in amount equivalent to the mercury already present, all the iodine and double the mercury in the original residues will be precipitated as mercuric iodide.

2. Experiments have shown that a Nesslerising solution, in which the potassium of the double iodide is replaced by zinc, is in all respects as good as the one described by Nessler. If, therefore, zinc be added to a hot suspension of mercuric iodide in water, in the proportion of one equivalent of zinc to two of mercuric iodide, the following reaction will take place: $Zn + 2HgI_2 = ZnHgI_4 + Hg$.

The double iodide of mercury and zinc will remain in solution, and will only require the addition of the requisite amount of sodium hydroxide to produce the finished reagent. The mercury formed may be filtered off, dissolved in a slight excess of nitric acid, and used to precepitate further residues.

In practice the Nessler solution is prepared as follows: 431 grms. of mercuric iodide are heated to boiling-point on a water-bath with 500 c.c. of water; 31 grms. of zinc (old zinc-copper couples, from which most of the copper has been removed, answer the purpose excellently) are quickly added, and the mixture stirred and heated for a further five minutes, the reaction being allowed to take place as violently as possible. The hot solution of zinc mercuric iodide is filtered, the precipitated mercury washed with boiling water, and the total filtrate made up to about 3,000 c.c. with warm water.* To this, after cooling, a saturated solution of mercuric chloride sufficient to produce a very distinct permanent red precipitate is added, a few c.c. at a time. Finally, 540 grms. of sodium hydroxide dissolved in about a litre of water are quickly stirred in, and the volume made up to 5 litres. After any sediment has been allowed to settle, the solution is ready for use. It is found that the best results are obtained by adding about 3 c.c. to every 50 c.c. of liquid to be Nesslerised.

As is well known, its sensitiveness will depend on the amount of permanent precipitate formed above; in rare cases, where this has been pushed too far, the Nessler solution, when used, will throw down a red precipitate. Should the finished solution be found to act in this manner, a small quantity of potassium iodide dissolved

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* Should a precipitate appear at this stage the solution is not filtered.

in it will correct the fault, but no more than is absolutely necessary should be employed.

To recover the mercuric iodide, the metallic mercury obtained above is dissolved, with the aid of heat, in 100 c.c. of strong nitric acid, and the solution made up to a litre.

The liquid from the Nessler glasses is collected in a large bottle, and, when sufficient has been obtained, neutralised or slightly acidified with sulphuric acid. For every 5 c.c. of original Nessler solution present, 1 c.c. of the mercuric nitrate solution is now added, and the precipitated mercuric iodide washed and dried for further use.

NOTE.—In the particular case where 3 c.c. of the reagent have been added to every 50 c.c. Nessler glass, it will be found most convenient to make up the above mercuric nitrate solution to 883 c.c. instead of a litre.

Every 10 litres of residues will then require the addition of 40 c.c. of strong sulphuric for neutralisation, and then 100 c.c. of the mercuric nitrate solution to precipitate the mercuric iodide.

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THE ESTIMATION OF THE APPROXIMATE QUANTITY OF MEAT IN SAUSAGES AND MEAT PASTES.

BY GEO. STUBBS, O.B.E., F.I.C., AND A. MORE, A.R.C.S., F.I.C.

(*Read at the Meeting, March* 5, 1919.)

THE Meat Rationing Order, 1918 (No. 404), of the Ministry of Food fixes the minimum quantity of meat in first quality and second quality uncooked sausages at 67 per cent. and 50 per cent. respectively. Specifications for sausages, issued by Navy and Army authorities, and also those issued by some public institutions, specify minimum limits for the amount of meat that must be present. It is therefore desirable that the analyst should be in a position to state, irrespective of the food value of the article, the quantity of meat entering into its composition. The following method of analysis has been devised with this object in view.

Sausages and meat pastes generally consist of a mixture of minced meat and "filler," with salt, preservatives, and seasoning substances. Bread was at one time almost the only filling substance used, but, in addition to bread, cooked rice, potatoes, or maize meal are now frequently employed, and in some cases cooked soya meal has been found to occur.

The method for the estimation of the amount of meat present is based on the following considerations:

1. Meat (beef, mutton, or pork) is free from carbohydrate and crude cellulose matter, and contains a fairly uniform percentage of nitrogen, calculated on the fat-free meat, the average percentage in beef and mutton being 3·75 and in pork 4·0.

2. The substances used as "fillers" contain, when in a condition suitable for

mixing, about 40 per cent. of water, and, with the exception of soya meal, about 50 per cent. of carbohydrate and crude cellulose matter, and 1 per cent. of nitrogen.

It is therefore possible, having estimated the amount of carbohydrate and crude cellulose matter, to allocate the quantity of nitrogen which is associated with it, the balance of nitrogen being then calculated to the quantity of defatted meat which it represents. To this quantity is added the fat for the purpose of arriving at the total meat present. The amount of fat in the filling material is not likely to be great, and is given to the credit of the meat, although in the presence of much maize meal an adjustment may in some cases be necessary.

The details of the examination are as follows:

1. PREPARATION OF SAMPLE FOR ANALYSIS.—In the case of sausages, the meat is removed from the skins, and the skins and the outside of the meat in contact with the skins are closely examined for evidence of insufficient cleaning.

A few pieces of the meat are selected for general examination, and the remainder is quickly and thoroughly mixed by passing it at least twice through a mincing machine. In the case of pastes or mixtures not in skins, the sample is mixed in a mortar or on a slab.

The sample thus prepared is at once placed in a stoppered or screw-capped bottle, from which the portions are taken for analysis.

2. ANALYSIS—(*a*) *Water.*—Five grms. are weighed into a flat porcelain dish containing a glass rod and about 20 grms. of ignited sand, mixed thoroughly with the sand, and dried at 100° C. to constant weight.

(*b*) *Fat and Non-Fatty Solids.*—Five grms. are weighed into a flat-bottomed dish containing a glass rod with flattened end, and dried on the steam-bath for thirty minutes, being rubbed down with the rod at intervals. It is then macerated with ether until the fat is extracted, the ether solution being filtered through a dried, weighed filter-paper, which is finally washed free from fat. The residue in the dish, together with that on the paper, dried to constant weight, gives the *non-fatty solids.*

The *fat* is obtained by evaporating the ethereal solution, and drying and weighing the residue: $100 -$ percentage of (fat $+$ non-fatty solids) $=$ percentage of water, which should agree with the direct estimation.

(*c*) *Nitrogen* is determined by the Kjeldahl process on a suitable quantity of the sample.

(*d*) *Ash* is estimated in the usual manner.

(*e*) Salt, boric acid, sulphites, and other preservatives are tested for, and the quantity estimated if present.

(*f*) The portion of the original sample removed before mincing is examined microscopically as to the nature of the "filler," and generally as to the character and quality of the meat.

CALCULATION.—(1) The percentage of non-fatty solids, less the sum of proteins and ash, gives the amount of carbohydrate and crude cellulose material. If this amount is multiplied by 2, the approximate percentage of bread or cereal filler containing 40 per cent. of its weight of water is obtained.

(2) One per cent. of the "filler" is taken as the nitrogen due to the "filler." If this quantity is deducted from the total nitrogen, the balance due to the meat is

obtained. To obtain the percentage of defatted meat, the meat nitrogen is multiplied by $\frac{100}{3\cdot75}$ in the case of beef or mutton, or by $\frac{100}{4\cdot0}$ in the case of pork, or by $\frac{100}{3\cdot87}$ in the case of mixed meats.

(3) The total percentage of meat in the sample is the sum of the percentages of the fat and of the defatted meat obtained under (2). (It should be noted that the percentage of chemical fat is not the same as that of fatty tissue, which contains, associated with the fat, both fibre and water.)

(4) The difference between 100 and the sum of the percentages of "filler" and total meat gives the additional water—that is, water used in the sausage or paste other than that which is natural to the meat and that which is present in "filler" containing 40 per cent. of water.

A check on the analysis can be obtained as follows :

The total percentage of water found by direct estimation should be equal to the sum of—(*a*) 40 per cent. of the filler as found above under (1); (*b*) 75 per cent. of the defatted meat as found above under (2) ; and (*c*) the additional water (if any) as found above under (4). The discrepancy should not exceed 2 per cent., unless a meal richer in nitrogen than ordinary cereal meals—*e.g.*, soya—has been used in preparing the "filler."

We are indebted to Sir James J. Dobbie for permission to publish the above method, which is in use at the Government Laboratory.

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THE ANALYSIS OF SAUSAGES, MEAT PASTES, AND ARMY RATIONS.

By A. W. STOKES, F.I.C.

(*Read at the Meeting, March* 5, 1919.)

Sausages.—While the "Food Control Regulations" lay down qualities and prices for sausages, requiring that the meat should be, for one price, 50 per cent. and, for another price, 67 per cent. by weight, it is necessary to be able to determine these limitations. Having analysed over sixty samples, a description of the method I have used may be of interest.

The main constituents of sausages are meat, bread, and water. A little salt and spice are usually added. Whilst the meat may be derived from any part of the animal, it is, of course, in most cases, impossible to determine either the animal or the part used.

The water is the chief difficulty. In the following analyses of all meat products I have assumed that meat naturally contains 70 per cent. and bread 40 per cent. of water. The meat is dissolved by boiling in alcoholic soda solution, with formation of a soap from the fat. The insoluble matter is then placed in an aqueous caustic soda solution to dissolve the starch, which is then precipitated by strong alcohol.

The actual method used is as follows: The total solids, water, and ash are estimated in the usual way on 5 grms. of the material. Boric acid may be looked for in the ash. Ten grms. are boiled for one hour in alcoholic (methylated) solution of caustic soda (the alcohol being of at least 90 per cent., and the caustic soda of 5 per cent. strength) under a reflux condenser. This treatment should dissolve the meat, form a soap with the fat, and leave the starch unaltered. This is filtered, while hot, through a small plug of slag-wool, and washed with strong hot alcohol. The filtrate is cooled and made up to 250 c.c. with water and 25 c.c. evaporated to about 15.c.c. (to remove the alcohol). The fatty acid is determined in this, using the Schmid method as in milk analysis. The filter-plug and residue are returned to the original flask; about 100 c.c. of cold aqueous 5 per cent. soda solution are added and left for one hour, with occasional shaking. This will dissolve the starch. The volume is made up to 500 c.c. with cold water and filtered through a small slag-wool plug. Fifty c.c. are collected and about 100 c.c. of strong alcohol (90 per cent.) are added to precipitate the starch; after stirring and allowing to stand for half an hour the starch is filtered through counterpoised filter-papers, washed with alcohol, dried and weighed. The filtrations are sometimes difficult, but may easily be managed without a filter-pump if a filter-flask be used to which is attached a short piece of rubber tube; mouth suction is applied to the end, the tube doubled over, and a clip applied to the part doubled.

Calculation.—The fatty acids may be assumed to equal 95 per cent. of actual fat. From the total solids are subtracted the starch and fat to get, by difference, the dry meat. To this "dry meat" is added 2·33 times its weight of water; this will give the fat-free flesh (called "flesh" in the following tables). To the starch is added 0·66 times its weight of water to form bread.

Frequently it will be found that the water actually present is above the quantities allowed by this calculation. This is due to the fact that in the manufacture of sausages the crusts and the bread used are soaked in water to soften them, and this water is only partially squeezed out before being chopped up with the meat. Very rarely the bread or the meat may contain less than the amounts I have allowed for water. The ash consists mainly of salt, though in twelve out of sixty samples analysed boric acid was also present, but in small amounts. The total ash varied from 1·2 to 3 per cent., with an average of 2 per cent.

The results obtained from the five following samples may be of interest; they are selected as showing mainly the extremes of each ingredient:

SAUSAGES.

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	Average of 60 Samples.
Fat, per cent. ...	0·9	20·2	9·5	6·9	20·9	11·2
Bread, per cent.	9·5	16·7	5·3	32·2	29·4	20·3
Flesh, per cent. ...	89·1	72·2	71·0	51·1	26·0	60·2
Water present, per cent.	66·8	48·1	66·0	58·4	53·6	58·6
Water allowed, per cent.	66·3	57·2	51·8	48·6	29·9	50·3
Water excess, per cent.	0·5	—	14·2	9·8	23·7	8·3

The smallest amount of water found to be present was 48 per cent. (No. 2); this sample, it will be observed, sums up to 109·1 per cent. This and three other samples were the only specimens showing that the meat and the bread contain less than the calculated allowances of water. No. 5, which contained the largest excess of water (23·7 per cent.), was seen by microscopical examination to consist largely of tripe.

In only a few cases were these samples examined microscopically; usually the indications were not particularly useful. Some few were analysed before and after cooking; generally the amounts of water and of fat diminished after cooking. But, as no standard for water could be assumed for these, the results are of little interest, and are not recorded here.

MEAT PASTES.—Meat pastes, variously described as ham and chicken, tongue and chicken, veal and ham, ham and tongue, ham, tongue, and chicken, turkey and tongue, and wild duck, prepared by various makers, were analysed in the same way. The results of fourteen of these show for the extreme percentages of each of the ingredients (Nos. 1 to 5), and the averages for all as follows:

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	Average of 14 Samples.
Fat, per cent.	3·6	16·6	15·4	13·4	11·1	13·2
Bread, per cent. ...	11·0	3·0	6·7	13·8	13·6	8·7
Flesh, per cent. ...	61·0	85·3	63·2	45·1	41·1	63·6
Water present, per cent.	71·6	55·9	61·6	65·0	69·0	62·6
Water allowed, per cent.	47·0	60·9	46·9	37·3	34·2	48·1
Water excess, per cent.	24·6	—	14·7	27·7	34·8	14·5

The only sample out of these fourteen that summed up to over 100 was No. 2, which, on the allowances for water in the flesh and bread, totalled to 104·9 per cent. This was in every respect far above all the other samples, containing the least amount of bread and no excess of water. On reference to the maker, I was informed that my results were within 2 per cent. of the materials used.

Of the samples the highest ash was 5·28 per cent., but this was mainly due to common salt: the lowest was 1·48, the average 2·9 per cent. No boric acid was present in any of these.

To see how the process would work in various hands, Messrs. G. A. Stokes and E. W. Wright kindly analysed a few samples of sausages for me. Their results were very similar to mine, as shown by the following example:

	G. A. S. and E. W. W.	A. W. S.
Fat, per cent. ...	14·5	14·2
Bread, per cent. ...	28·0	27·8
Flesh, per cent. ...	39·5	41·0
Water excess, per cent.	18·0	17·0

Army Rations.—A number of army rations were examined to see if they complied with the contract specifying them to contain not more than 12 per cent. fat, or 70 per cent. water, or more than 2 grains of tin per pound. They were not to have a greater acidity than the equivalent of 72 c.c. of $\frac{N}{10}$ soda in 100 parts. They consisted of mixtures of meat, potato, haricot beans, peas, carrots, and onions, cooked in sealed tins.

It was found easiest to pick out all the meat separately and to shred this finely. The vegetables could readily be mashed and mixed in the tin, the meat then added, and the whole well incorporated in the same tin so as to absorb any moisture therein. The analyses of these were performed as in the case of the sausages, omitting, however, any estimation of starch.

For the acidity, 5 grms. were shaken with 100 c.c. of cold water for one hour; an aliquot part was then titrated, after filtration, with $\frac{N}{10}$ soda. The results of five samples are appended:

	No. 1.	No. 2.	No. 3.	No. 4.	No. 5.	Average.
Fat, per cent. ...	11·7	4·9	8·2	8·2	8·1	8·2
Water, per cent.	67·5	73·0	67·4	68·0	68·4	68·8
Acidity, per cent.	16·0	32·0	48·0	32·0	30·0	31·6
Ash, per cent. ...	1·2	1·3	1·4	1·3	1·6	1·4

Only in one case (No. 2) was the amount of water slightly beyond the limit. In no case was any tin or boric acid detected.

A review of the results of the analyses of the sausages and meat pastes shows that an excess of water, not natural to the meat and bread, may in the sausages reach about 24 per cent., and in the meat pastes 35 per cent. This high excess is usually due to the use of much bread; this soaked in water enables a large amount to be incorporated.

In the United States the use of any starchy matter in sausages is forbidden. It is to be regretted that such is not the case in England. A sample like sausage No. 5, which contains about equal parts of fat (21 per cent.), meat (26 per cent.), bread (29 per cent.), and excess water (24 per cent.), would not be possible were it illegal to use bread.

I am greatly indebted to Mr. G. W. Bender for his assistance in this work.

Discussion.

The President, in opening the discussion, said that the Ministry of Food had consulted the Society in reference to the carrying out of their Sausage Orders under the following circumstances. On January 21, 1918, the Department found it necessary to introduce the meatless days, and in that Order, known as the Public Meals Order, the expression "meat" was deemed to include sausages. In February the Meat Licensing Order was issued which forbade any person after March 15 from dealing in dead meat without a licence, and in this Order sausages were included as dead meat. This was revoked in Ireland on June 17, but later in the year the Ministry again issued an Order in which they then fixed the price of sausages according to their meat content, ignoring the fact that sausages were supposed by the previous regulations to be all meat. Pork sausages had also not been included in their Pig and Pig Products (Prohibition of Export) Order, as only bacon, ham, and lard were included in it. The Government Laboratory had tried to solve the mystery of what is now meant by a sausage of different grades, and the Society was glad to welcome their contribution to the discussion.

Dr. Voelcker suggested that it would be better if the Ministry of Food and other Government Departments, before making their Orders and putting out regulations as to how mixed foods should be compounded, would consult analysts generally and the Government Laboratory in particular as to whether it was possible to ascertain by analysis whether the composition could be ascertained, and not wait, as was here done in the instance of sausages, etc., to hear the difficulties expressed at a subsequent meeting of this Society.

It seemed to him that the authors of the papers they had heard read relied mainly upon the amount of nitrogen shown in the analysis, and reckoned this as representing the meat present. He would like to ask what would happen if he sprinkled a little sulphate of ammonia over a sausage or mixed some leather powder with it—would this be reckoned as "meat"?

He had heard no mention of microscopical analysis in this connection, and thought that it should be employed. The inclusion of soya bean meal, for instance, could be so detected.

Mr. Cribb inquired if the Food Controller had drawn up any definition of "meat," as the value of the methods proposed in the two papers under discussion must largely depend on what was meant by that word.

In pre-war days he had had occasion to examine some sausages which consisted almost entirely of fat and bread, with a little connective tissue and only a trace of muscle fibre. Would such a mixture comply with the "Order"?

An ingenious manufacturer might put a large proportion of gristle and connective tissue into his sausage meat. This, owing to the low nitrogen factor for gelatin as compared with that for proteins, would seriously affect the figure for "flesh" obtained by the method recommended by Messrs. Stubbs and More. The presence of anything like a substantial proportion of liver, owing to the glycogen present, would give trouble in other directions.

In view of these difficulties he would like to see a number of analyses, by the methods now put forward, of sausages of known composition, as by this means only would it be possible to decide whether the problem set by the Ministry of Food could be satisfactorily solved. In any case, he thought that the analytical figures obtained would have to be interpreted with great caution, and it would be quite unsafe to take action in cases anywhere near the border-line.

With regard to sausage skins, to which the President had referred, some years ago he was consulted about a large consignment of sausages which on being sent to America were refused admission to that country, on the ground that they contained boric acid. The manufacturers had put in none, and he was quite unable to find any in the sausages as a whole; but it transpired that the authorities in America had actually examined the skins separately, and had found an extremely minute trace of boric acid, which on examining a number of the skins he afterwards ascertained to be between one and two parts per million.

The skins before use were kept in a saturated salt solution, and this was the only apparent source from which the boric acid could have been derived. Skins kept in this way would presumably be sterile.

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THE ESTIMATION OF SMALL QUANTITIES OF ACETONE, ALCOHOL, AND BENZENE IN AIR.

BY MAJOR ELLIOTT, B.Sc., F.I.C., AND CAPT. J. DALTON, M.C., DIPL. CHEM.

(*Read at the Meeting, March* 5, 1919.)

THE authors were requested to undertake an investigation of the quantity of acetone, alcohol, and benzene in the air of certain factories. As it was found that the amount of the substances would be less than three parts per million by weight in a million volumes of air, the usual methods of absorption and measurement of the diminution of volume were not applicable; therefore it was necessary to draw large quantities of air through various absorbents and determine these by ordinary volumetric methods.

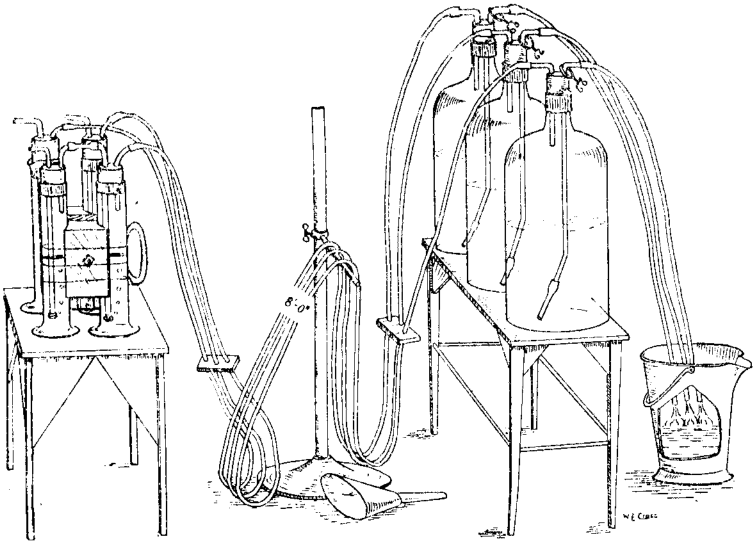
A general account of the impurities of air in factories and workshops has been given in Dr. Legge's translation of "Ramsboucks' Industrial Poisoning," but methods for the determination of the above substances are not given.

The air was collected as near to the level of the mouths of the operatives as convenient, in order to obtain a sample representative of the air breathed by them, and the time taken to collect the sample was between one and two hours. The air was drawn through the absorbent liquids arranged in parallel by means of Winchester quart bottles fitted with syphons, the amount of water syphoning over giving a measurement of the air drawn through the apparatus.

The apparatus used for the absorption consisted of four narrow measuring cylinders of about 50 c.c. capacity fitted with Folin tubes (*J. Bio. Chem.*, 1912, **11**, 493) and rubber stoppers, with one exception, when a cork soaked in paraffin was used. These cylinders were clamped together by means of wooden blocks for convenience in carrying, and were a modification of Drechschmidt's absorbing cylinder referred to in Lunge's "Technical Gas Analysis"; and experiments showed that when the air was drawn through at the rate of about 10 litres an hour, only one cylinder was necessary for complete absorption in each case. Three syphon bottles were connected separately to three of the cylinders by three lengths of tubing 8 to 10 feet long, and this allowed the syphons to remain in a stationary position, while the cylinders could be moved to any desired part of the room. In each case it was found necessary to draw 10 litres of air through the apparatus in order to obtain a sufficient quantity of the vapours for an accurate analysis.

The absorbent solutions were placed in the cylinders before leaving the laboratory, and the apparatus was carried to the factory in an upright position, no trouble being experienced by splashing of the liquids during the journey. After the collection the cylinders were brought back to the laboratory and the contents transferred to suitable apparatus. This method was found to be very convenient, and obviated the difficulty of fitting up apparatus in the factory. Solutions were chosen which only reacted with the particular substance to be absorbed.

1. THE ESTIMATION OF ACETONE.—Denigès gives a method (*Compt. rend.*, 1898, **126**, 1868, and 1898, **127**, 963) by which the acetone is precipitated as a mercuric sulphate compound and weighed as such; the small amounts to be dealt with, however, precluded this method. But a modification of this method given by Scott Wilson (*J. Physiol.*, 1911, **42**, 444; ANALYST, 1911, **36**, 548), in which the keto mercury compound is estimated by titration with standard potassium thiocyanate, gave very satisfactory results, and could be used instead of Messinger's method.



Another method (Jolles, *Ber.*, 1906, **39**, 1306; ANALYST, 1906, **31**, 236) consists in the formation of a bisulphite compound with acetone and the titration of the excess of sodium bisulphite, but, owing to the large quantity of air drawn through the solution and the consequent oxidation of the bisulphite, this method was rejected as unsuitable in this case.

Auld (*J. Soc. Chem. Ind.*, 1906, **25**, 100; ANALYST, 1906, **31**, 235) suggests the use of bromine in potassium bromide for absorption of the acetone, but the large amount of air drawn through, and the consequent loss of bromine, renders this method unsuitable.

The method finally adopted was that of Messinger (*J. Soc. Chem. Ind.*, 1889, **18**, 138), so largely used in medical work for this purpose. It was considered that the modification suggested by Jitendranath Rakshit (ANALYST, 1916, **41**, 245), in which lime-water is used instead of soda, could not be well adapted, owing to the large bulk of lime-water required; and, under the conditions of the experiment, the error due to the formation of iodoform by the alcohol vapour in the air was found to be inappreciable so long as the temperature was kept low.

Twenty-five c.c. of $\frac{N}{10}$ iodine and 5 c.c. of 20 per cent. sodium hydroxide were placed in one of the absorption cylinders of the apparatus, the measured volume of air drawn through, and the excess of iodine titrated back after acidification with 2N hydrochloric acid by means of $\frac{N}{10}$ sodium thiosulphate, starch being used as indicator. One c.c. $\frac{N}{10}$ iodine corresponds to 0·000964 grm. of acetone.

As will be seen from the following table, the presence of alcohol can be neglected for all practical purposes:

TABLE A.

Amount of Acetone Taken.	Amount of Alcohol Taken.	Difference in c.c. between Blank and Experiment.	Quantity of Acetone Found.	Error.
0·010 grm.	Nil	9·8 c.c.	0·0094 grm.	6 per cent.
0·010 grm.	Nil	10·0 c.c.	0·0096 grm.	4 per cent.
0·0008 grm.	Nil	1·0 c.c.	0·0009 grm.	11 per cent.
Nil	0·0104 grm.	0·2 c.c.	0·00018 grm.	—
Nil	0·0052 grm.	0·1 c.c.	0·00009 grm.	—
Nil	0·0026 grm.	Nil	Nil	—
0·010 grm.	0·0026 grm.	9·7 c.c.	0·0093 grm.	7 per cent.
0·010 grm.	0·0052 grm.	9·7 c.c.	0·0093 grm.	7 per cent.
0·010 grm.	0·0104 grm.	9·8 c.c.	0·0094 grm.	6 per cent.

2. THE ESTIMATION OF ALCOHOL.—Various methods are given, the basis being the oxidation of alcohol to aldehyde or acetic acid by means of potassium bichromate or permanganate, the excess being titrated.

Benidict and Norris (*J. Amer. Chem. Soc.*, 1890, **12**, 302) oxidise to acetic acid and then titrate back the excess of bichromate with ferrous ammonium sulphate.

Argerson (*Büll. Soc. Chim.*, 1902, **18**, 302) oxidises to aldehyde, which is distilled over and determined colorimetrically with Schiff's reagent.

Syerbenyi (*Zeitsch. Anal. Chem.*, 1915, **45**, 409) titrates back the excess of bichromate iodimetrically after oxidation of the alcohol to acetic acid, and a modification of this method was adopted.

A mixture of 100 c.c. of $\frac{N}{2}$ potassium dichromate, 100 c.c. strong sulphuric acid, and 50 c.c. distilled water, was cooled to normal temperature. Twenty-five c.c. were placed in another absorption cylinder, and the air drawn through, a plug of cotton-wool being interposed to prevent access of dust to the bichromate. The liquid was transferred to a flask, the residue being washed in with 45 c.c. of water in order to make up the bulk of the liquid to 70 c.c. Having boiled the solution under a reflux condenser for exactly fifteen minutes and cooled, 100 c.c. of 2 per cent. sodium iodide were added to the mixture, and the liberated iodine titrated with $\frac{N}{10}$ sodium thiosulphate, using starch as indicator.

After the boiling a faint odour of acetic acid, but none of aldehyde, was detected. A blank experiment was carried out as a control.

Each c.c. of $\frac{N}{10}$ sodium thiosulphate corresponds to 0·00115 grm. alcohol. Experiments were made to determine the exact effect of acetone on this estimation, and as will be seen from the following table, no appreciable error was introduced if the proper precautions were adhered to :

TABLE B.

Amount of Acetone Taken.	Amount of Alcohol Taken.	Difference in c.c. between Blank and Experiment.	Quantity of Alcohol Found.	Error.
Nil	0·0150 grm.	11·5 c.c.	0·0132 grm.	12 per cent.
Nil	0·0050 grm.	4·3 c.c.	0·0049 grm.	2 per cent.
Nil	0·0040 grm.	3·5 c.c.	0·0040 grm.	Nil
0·010 grm.	Nil	0·1 c.c.	0·0001 grm.	—
0·010 grm.	Nil	Nil	Nil	—
0·020 grm.	Nil	0·3 c.c.	0·0003 grm.	—
0·010 grm.	0·0052 grm.	4·5 c.c.	0·0052 grm.	Nil
0·020 grm.	0·0052 grm.	4·6 c.c.	0·0053 grm.	2 per cent.

Experiments were carried out in which the alcohol was oxidised to acetic acid by means of potassium bichromate or potassium permanganate, and after distillation the acid was titrated, but the results were not so satisfactory as in the method adopted. The same applies to a modification of Messinger's method, the solution of alkaline iodide being heated to 60° C. for several minutes in order to form iodoform.

3. THE ESTIMATION OF BENZENE.—A method is given for the detection of benzene (*J. Pharm. Chem.*, 1900, **118**, 373) by nitration, reduction, diazotisation, and coupling up the product with *a*-naphthol to form a dye, but was considered more complicated than Pfeiffer's method (*Chem. Zeit.*, 1909, **28**, 884), which was finally adopted.

A differential pressure method (Davis and Davis, *J. Ind. and Eng. Chem.*, 1918, **10**, 719; ANALYST, 1918, **43**, 417) seems to give satisfactory results for large quantities of benzene, but the authors considered that the vapour pressure given by the quantities in the air under investigation would be too small for measurement.

The air containing the benzene was passed through 15 c.c. of a mixture of equal volumes of fuming nitric and strong sulphuric acids, contained in the cylinder fitted with a paraffin cork; and the fourth cylinder, containing 20 to 25 c.c. of 20 per cent.

sodium hydroxide, was interposed between this cylinder and the syphons, to act as a trap for the acid fumes, and to prevent corrosion of the rubber tubing.

The mixture of acids was heated on the water-bath for about fifteen minutes to ensure complete nitration, cooled, diluted with water, made just alkaline to litmus with about 50 c.c. of 40 per cent. sodium hydroxide, and then just faintly acid with dilute hydrochloric acid. After dilution to about 500 c.c. to prevent crystallisation of sodium sulphate, the liquid was extracted with three successive portions of ether, and the ether treated with about 0·5 c.c. caustic soda. After separation of the ether from the soda and distillation, the residue of dinitrobenzene was heated on the water-bath to remove any oxidation products of the alcohol and acetone, and then dissolved in 10 c.c. of alcohol. The alcoholic solution was generally coloured a yellow-brown, and was heated with 10 c.c. of a standardised solution of stannous chloride for ten minutes, and after cooling the excess was titrated back with $\frac{N}{10}$ iodine, using starch as indicator. A blank experiment was carried out as a control. Each c.c. of $\frac{N}{10}$ iodine corresponds to 0·00065 gr. benzene.

TABLE C.

Substance Taken.	Blank Experiment.	Experiment.	Difference.	Quantity Found.	Quantity Taken
	c.c. of $\frac{N}{10}$ Iodine.			Grms.	Grms.
Benzene	16·8	14·6	2·2	0 00143	0·00167
	16·8	14·2	2·6	0·00169	0·00167
	16·8	14·2	2·2	0·00169	0·00167

The following figures indicate the amount of these substances per million c.c. of air found in the factories:

	Acetone.	Alcohol.	Benzene.
	Grms.	Grms.	Grms.
Minimum	0·02	0·02	0·01
Maximum	1·86	3·00	0·90
Mean	0·31	0·65	0·26

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NOTES.

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

RAPID DETERMINATION OF SOLUBILITY.

THE solubility of a solid substance in a solvent frequently serves as a means of identification of the solid or as a criterion of its purity. This constant may be rapidly and accurately determined on small quantities of the material by the following method:

About 3 c.c. of the solvent in a test-tube is immersed in a beaker of water from 10° to 20° C. above the temperature at which the solubility is to be determined, and the solid added with occasional shaking until a portion remains undissolved. The test-tube is then transferred to another beaker containing water at the temperature at which the solubility is required for ten minutes with occasional shaking. A small thimble made of folded filter-paper (similar to a miniature Soxhlet thimble) is then pushed down into the liquid, and filtration allowed to proceed from exterior to interior. A definite volume (0·5 to 2·0 c.c.) of the clear solution is removed by a pipette and transferred to a weighed covered dish, which is again weighed. The solvent is evaporated in a well-ventilated oven at a suitable temperature, and the residue cooled and weighed.

The following results obtained by this method were each completed in less than one hour:

Solid.	Solvent.	Volume of Solution weighed.	Temperature at which Solubility was determined.	Temperature at which Solvent was evaporated.	Solubility—Grms. per 100 c.c. of Solvent.	Published Figures.
Sodium chloride	Water	0·25 c.c.	15·5° C.	100° C.	36·3	35·9
Potassium chlorate	Water	1·0 c.c.	20° C.	100° C.	7·20	7·22
Strychnine	Benzene	2·0 c.c.	20° C.	90° C.	0·679	0·680
Sulphur	Carbon bisulphide	1·0 c.c.	15·5° C.	50° C.	44·97	44·25

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BRITISH PHARMACOPŒIA, 1914: CANCELLATION OF ALTERATIONS AND AMENDMENTS.

By resolution on February 25, 1919, the Executive Committee of the General Council of Medical Education and Registration decided that the temporary alterations in the British Pharmacopœia arising out of the scarcity during the war of sugar, glycerol, and some oils and fats should be withdrawn on April 30, 1919.

An Order of the Food Controller, dated February 14, 1919, removes the restrictions on the use of lard for purposes other than the preparation of foodstuffs. Lard is now allowed to be used in the manufacture of ointment.

The schedule of preparations withdrawn from the British Pharmacopœia because of their content of sugar or of glycerol was given in the *London Gazette* for July 27, 1917. Substitutes for them were published in an *Addendum* (*War Emergency Formulary*) *to the British Pharmaceutical Codex*, 1911. The Alterations and Amendments to the British Pharmacopœia arising out of the prohibition of the use of lard for medicinal purposes and permitting the use of substitutes for olive oil were given in the ANALYST (1918, pp. 185 and 378), and also a full list in the President's Annual Address (*ibid.*, 1919, 86).

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Burma Beans as a Food. Rothea. (*Ann. Falsific.*, 1918, **11**, 361-369.)—In view of the fact that Burma beans contain a cyanogenetic glucoside and yield hydrocyanic acid when the glucoside is hydrolysed, the author has carried out a number of experiments to determine the rate of hydrolysis and the elimination of hydrocyanic acid from the beans. It was found that with beans yielding a total of 0·029 per cent. of hydrocyanic acid, the hydrolysis was complete when the beans were steeped for twenty-four hours in water at 15° C. After being boiled in a fresh quantity of water for three hours, the beans contained 0·019 per cent. of hydrocyanic acid, the remainder of the hydrocyanic acid having passed, in almost equal quantities, into the portions of water used for the steeping and boiling. Direct boiling of the beans, without preliminary steeping, results in the removal of about one-half of the hydrocyanic acid present. It is suggested that the permissible maximum limit of hydrocyanic acid in these beans should be 0·010 per cent., and that they should never be consumed unless after being steeped and well boiled, the water used being rejected. In any case, the beans should not be eaten by invalids and children. (See also ANALYST, 1919, 97.)

W. P. S.

Tentative Standard Methods for the Sampling and Analysis of Commercial Fats and Oils. (*J. Ind. and Eng. Chem.*, 1919, **11**, 69-71.)—In addition to methods described previously (ANALYST, 1918, **43**, 60, 270), the procedure recommended for determining the melting-point and cloud-test is now given. *Melting-Point.*—The melted fat is drawn into a capillary tube 1 mm. in diameter (three tubes are used for each determination), the filled tube is cooled at 4° C. to 10° C. for eighteen hours, and the melting-point then observed by attaching the tube to a thermometer and heating it in a water-bath. The heating is continued until the contents of the tube become uniformly transparent. *Cloud Test.*—The oil is heated at 150° C. and stirred with a thermometer. As soon as it can be done with safety, about 45 c.c. of the oil is poured into a 4 ounce oil bottle and the latter cooled in a suitable bath. The oil is stirred constantly with a thermometer, and the point is noted at which the first permanent cloud appears in the oil. The oil must be heated just previously to making the determination, and the cloud-point should be approached as quickly as possible, yet not so fast that the oil is frozen on the sides of the bottle before the cloud-point is reached. W. P. S.

Edible Flours and their Products. E. Collin. (*Ann. Falsific.*, 1918, **11**, 372-384.)—The characteristic microscopic features of wheat, rye, barley, maize, rice, buckwheat, and potato flours are described and illustrated. The different starches are best identified by an examination of the wash-water resulting from the separation of the gluten in the usual way; if this wash-water is first passed through a fine sieve, the starches are found in the liquid portion, whilst particles of rice, maize, cellular tissue, etc., remain on the sieve and may be examined separately. The character of the gluten is of importance; if it has a soapy feel, the presence of rye flour is indicated. The gluten has often a granular feel when rice or maize flours are present.

W. P. S.

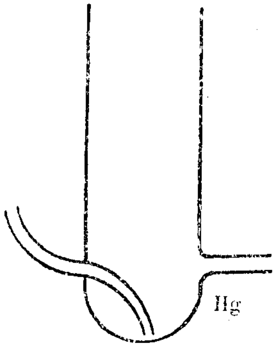
Analysis of Kaolin Soaps. W. D. Cohen. (*Chem. Weekblad*, 1919, **16**, 144-145.)—According to the Dutch regulation, kaolin alumina soaps must contain at least 20 per cent. of fatty acids and 66 per cent. of kaolin and alumina, whilst the saponification value of the fatty acids must be not less than 225. For the rapid analysis of such soaps 50 grms. of the sample in fine shavings are dried at 105° C. until constant in weight. The dry residue is extracted for about eight hours with 96 per cent. alcohol in a Soxhlet tube, which is meanwhile immersed in a bath of boiling water. The extract is evaporated, and the fatty acids separated and weighed, whilst the residue of kaolin, etc., in the extraction thimble is dried at 100° C and weighed. Four commercial samples thus examined gave the following results: Moisture, 6·6 to 11·0; kaolin, etc., 65·1 to 72·1; fatty acids, 17·8 to 22·2 per cent.; and saponification value of fatty acids, 242·5 to 248·7. C. A. M.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Shark and Ray Liver Oils. M. Tsujimoto. (*J. Chem. Ind.*, Tokyo, 1918, **21**, 1015-1042; through *J. Soc. Chem. Ind.*, 1919, **38**; 109A.)—The oil from the liver of the shark, Imori-zame (*Pristiurus pilosus*, Garman), had the highest iodine value of the liver oils of the thirteen species of Japanese sharks examined. It had the following characters: Sp. gr. at 15°/4° C., 0·8664; acid value, 0·32; saponification value, 28·2; iodine value (Wijs), 309·0; refractive index (20° C.), 1·4912; unsaponifiable matter, 85·5 per cent.; insoluble bromides of fatty acids, 7 per cent.; and squalene, about 79 per cent. The other shark-liver oils had iodine values ranging from 91·3 to 236·6. The oils of low sp. gr. (below 0·9 at 15°/4° C.) invariably contained the hydrocarbon squalene. Both the liver and egg oils of the sharks known as the frill shark (*Chlamydoselachus anguineus*, Garman) and kinbei-zame (*Lepidorhinus kinbei* Tanaka—a new species) also contained squalene. It was not present in the liver oils of the five species of Japanese rays examined, nor in the liver oils of an elephant fish (*Chimæra mitsukurii*, Dean).

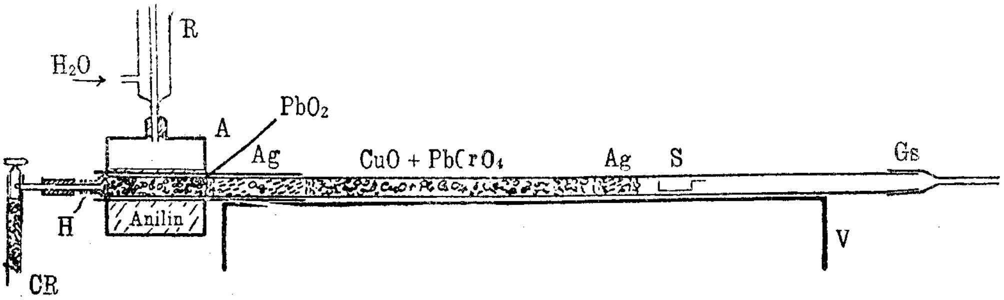
ORGANIC ANALYSIS.

Elementary Micro-Analysis of Organic Substances. J. V. Dubsky. (*Helv. Chim. Acta*, 1919, **2**, 63-75.)—*Gas-Volumetric Estimation of Nitrogen:* The use of Kipp's apparatus for the preparation of air-free carbon dioxide has the drawback that, owing to the adsorption of the air on the sides of the flask and the marble fragments, the gas is unsuitable for the purpose until after one or two days. A more simple and rapid method is to prepare the gas by heating sodium bicarbonate for six to eight minutes in a micro-bicarbonate tube 8 cms. long by 10 to 15 mm. in diameter, closed by a cork through which passes a capillary tube. This must fit perfectly into the perforation, or air will be drawn into the tube. A new form of micro-nitrometer is described. It has a capacity of 1·9 cms., and is sealed by a small amount of mercury, whilst the carbon dioxide is introduced directly at the base (see diagram). The combustion tube is charged in the manner previously described (*Chem. Zeit.*, 1916, **40**, 201), but care must be taken that the reduced copper spiral is carefully heated in the oven to expel the last traces of methyl alcohol. When once reduced, the same spiral may be used for a series of analyses, provided that the combustion tube is always allowed to cool in a current of carbon dioxide. It is essential to have a sufficiently long layer of copper oxide in the tube, and for this reason a tube 43 cms. long is now used. A series of analyses of different substances by the micro-Dumas method gave results agreeing within 0·1 to 0·3 per cent. with the theoretical values. In the absence of a micro-balance an ordinary balance may be used in the estimation of nitrogen by this method, the weighing being effected by Treadwell's swinging method (*Lehrb. anal. Chem.*, [1913], 9.)



Micro-Estimation of Carbon and Hydrogen: The combustion tube, 43 cms. in length, charged with a silver spiral and copper oxide, is suitable for most organic compounds, provided that the substance is ignited at the beginning of the combustion in a slow current of air or in a closed tube. In the latter case it is advisable to have an oxidised copper spiral behind the boat containing the substance. The results for hydrogen and carbon thus obtained with explosives such as picric acid, or with compounds poor in hydrogen such as trichlorodinitrobenzene, are too high, but in many cases good results can be obtained with such compounds by using a layer of lead peroxide in place of silver, and keeping this layer at a constant temperature of 180° to 200° C. Or a simple method of reducing nitrogen oxides, such as the simultaneous combustion of a weighed quantity of benzoic acid, naphthalene, or sugar in a separate boat, may be used. The burning benzoic acid first reduces the adjacent copper oxide to copper, which then reduces the nitrous vapours subsequently evolved. In the case of nitrogenous compounds rich in hydrogen the use of benzoic acid is unnecessary, since, when slowly burned, these themselves reduce a part of the copper oxide. The use of reduced copper spirals is inadmissible in the estimation of carbon and hydrogen, since, when the reduction is effected by means of methyl alcohol, the spiral invariably contains traces of formaldehyde, whilst when it is reduced with hydrogen correct values are obtained for the carbon, but the hydrogen results are too high owing to the presence of occluded hydrogen. For the micro-analysis of explosive compounds the substance is mixed with ignited sand or lead chromate. The tube is best charged with a layer of 5 cms. of pieces of fine silver wire, and a layer of 16 cms of a mixture of copper oxide and lead chromate, kept in position by plugs of asbestos or platinised asbestos. It is also advisable to have a second layer of silver immediately in front of the platinum boat, whilst in the case of compounds containing sulphur or halogens the substance may also be covered with silver powder. C. A. M.

Elementary Micro-Analysis of Compounds containing Sulphur, Halogens, or Nitrogen. C. Gränacher. (*Helv. Chim. Acta*, 1919, **2**, 76-84.)—It is doubtful whether the specific action of silver upon the higher oxides of nitrogen invariably prevents the results for carbon and hydrogen being too high in the case of nitro compounds. The use of lead peroxide as an absorption agent for nitrogen oxides gives good results, even in the case of compounds also containing sulphur and halogen, but requires the temperature of the lead peroxide to be maintained between 170° and 180° C. This may be effected by means of the device shown in the diagram. It consists of a copper tube 12 cms. in length and 12 mm. in diameter, on which at a distance of 1 cm. from one end is soldered a copper drum 5 cms. in length and 4·5 cms. in diameter. On the top of the drum is a tube 15 mm. in diameter, which is connected with a small reflux condenser, the cooling tube of which need not exceed 10 cms. in length. Sufficient pure dry aniline is introduced into the drum to cover the inner tube, which is then passed over the combustion tube as shown. If two combustions are made in parallel combustion tubes, the aniline drum is made in a larger size and contains two copper tubes instead of one. C. A. M.



Estimation of Free Carbon in Rubber Goods. A. H. Smith and S. W. Epstein. (*J. Ind. and Eng. Chem.*, 1919, **11**, 33-36.)—The following method may be used for the estimation of free carbon which is sometimes present in rubber articles; it depends on the oxidation of the rubber substances with nitric acid, the carbon being only slightly affected under the conditions given. One grm. of the sample is extracted for six hours with acetone and then for three hours with chloroform or carbon disulphide. After the sample has been dried to remove adhering solvent it is treated with a few c.c. of hot concentrated nitric acid, allowed to stand in the cold for ten minutes, 50 c.c. of hot concentrated nitric acid are then added, and the mixture is placed on a boiling water-bath for one hour or until all foam has disappeared from the surface. The hot liquid is filtered through asbestos and the insoluble portion washed with hot concentrated nitric acid; the filtrate is removed, and the filter and its contents then washed successively with acetone, benzene, hot 15 per cent. sodium hydroxide solution, hot ammonium acetate solution (this is necessary if lead sulphate is present), and 5 per cent. hydrochloric acid. The filter and its contents are dried at 150° C. for ninety minutes, weighed, the carbon burned off at a dull-red heat, and the filter reweighed. The difference in the two weights represents approximately 105 per cent. of the carbon present originally as lampblack. Experiments with lampblack alone showed that the treatment increased its weight by about 5 per cent. W. P. S.

Preparation of β-phenylhydroxylamine and Cupferron. C. S. Marvel and O. Kamm. (*J. Amer. Chem. Soc.*, 1919, **41**, 276-282.)—This is the third paper of a series dealing with the preparation of organic substances (see ANALYST, 1918, **43**, 386). Cupferron is the ammonium salt of *n*-nitrosophenylhydroxylamine, and is prepared from β-phenylhydroxylamine, ammonia, and amyl nitrite; the β-phenylhydroxylamine is prepared by the reduction of nitrobenzene with zinc dust.

β-Phenylhydroxylamine.—Eight litres of water, 500 grms. of nitrobenzene, and 250 grms. of ammonium chloride are placed in a 4-gallon earthenware jar. The mixture is stirred thoroughly by means of a mechanical stirrer, and 670 grms. of zinc dust (75 to 80 per cent.) are added during twenty minutes. As the reaction proceeds the temperature of the mixture rises to about 60° C., and is prevented from rising higher by the addition of ice from time to time. About 1 kilo of ice is required. After the zinc has been added the stirring is continued for fifteen minutes. The reaction is complete when there is no further rise in the temperature, and, after stirring for five minutes longer, the phenylhydroxylamine solution is filtered from the zinc oxide, the latter being washed with 1 litre of warm water. The filtrate is collected in an enamelled pan, saturated with sodium chloride, and cooled to 0° C. The β-phenylhydroxylamine begins to crystallise out even at 30° C. The yield, after filtration by suction, amounts to from 330 to 390 grms. The average yield of dry product is 290 grms., which corresponds with 64 per cent. of theory. For the preparation of cupferron, it is, however, unnecessary to use dry β-phenylhydroxylamine.

Cupferron.—The moist β-phenylhydroxylamine obtained, as described, from 725 grms. of nitrobenzene is dissolved in 3 litres of ether, the insoluble portion (sodium chloride) is filtered off and weighed so as to obtain an approximate measure of the β-phenylhydroxylamine in solution, and the ethereal solution is cooled to 0° C. in a 5-litre flask provided with a stirrer. A rapid current of dry ammonia gas (from a cylinder) is now passed into the solution for about sixteen minutes, and the theoretical quantity of freshly-distilled amyl nitrite (107 grms. for every 100 grms. of β-phenylhydroxylamine) is then added slowly through a tapped funnel. The addition requires about thirty minutes, during which time the current of ammonia gas is maintained.

The temperature must not be allowed to rise above 5° C., and is controlled by the rate at which the amyl nitrite is added. The mixture is stirred for a further ten minutes, the cupferron then collected on a filter, washed several times with ether, spread on paper until all ether has evaporated, and the substance is stored in bottles, where it is exposed to the vapours of ammonium carbonate; this may be done by protecting each cork with a double layer of filter-paper and placing a lump of ammonium carbonate between the cork and the paper. The yield of cupferron from a given weight of β-phenylhydroxylamine averages from 80 to 90 per cent. of that theoretically possible. The greater part of the ether used and of the amyl alcohol produced in the reaction may be recovered. W. P. S

Estimation of Cyanogen Compounds in Concentrated Ammonia Liquor. P. E. Spielmann and H. Wood. (*J. Soc. Chem. Ind.*, 1919, **38**, 43-45 T.)—The authors have worked out a method for the colorimetric estimation of cyanogen compounds in ammonia liquor by means of the Lovibond tintometer, after conversion into ferric thiocyanate. The thiocyanate originally present in the liquor is first estimated in the following manner: Ten c.c. of the sample are run into 40 c.c. of water, and then acidified with dilute sulphuric acid while cooling. Iron alum is added until the liquid becomes red, the mixture is warmed to coagulate the precipitate, allowed to stand for half an hour, the precipitate is removed by filtration and washed, 10 c.c. more of saturated iron alum solution acidified with nitric acid are added to the filtrate, which is then made up to 100 c.c. and examined in the tintometer. In another portion of the sample the ammonium cyanide is estimated after conversion into thiocyanate. If ferrocyanide is absent, 10 c.c. of the sample are diluted slightly, ammonium polysulphide solution is added until the liquid is distinctly yellow, and it is then diluted to 100 c.c. The liquid is then heated to boiling-point to drive off ammonia, treated with iron alum as before, and examined in the tintometer. The difference between the two estimations gives the thiocyanate produced from the cyanide. When ferrocyanide is present, 25 c.c. of the sample are added to an excess of polysulphide solution. After standing for twenty minutes the liquid is acidified with dilute sulphuric acid, keeping it well cooled, and iron alum is added. The solution is warmed not above 60° C. to coagulate the ferrocyanide, which process is assisted by a little sodium sulphate. After standing for half an hour the precipitate is filtered off and washed, and 50 c.c. of iron alum with a little nitric acid are added to the filtrate. The different procedure recommended when ferrocyanide is present is due to the necessity of avoiding heating the ammonium ferrocyanide with polysulphide. For the estimation of the ferrocyanide, the precipitate of ferrocyanide and sulphur is treated according to the method described in the Alkali Inspector's 46th Report, 1909, with the exception that after decomposing by sodium hydroxide it is distilled according to Williams's method. In using the Lovibond tintometer a half-watt lamp with frosted glass bulb is employed. Standard solutions of ammonium thiocyanate are examined in cells of three thicknesses—$\frac{1}{8}$ inch, $\frac{1}{2}$ inch, and 1 inch—and the colours recorded in terms of red units only, and the source of light is standardised against a solution of potassium bichromate saturated at 20° C. in order that a permanent record may be kept for comparison. J. F. B.

Estimation of Oil in Seeds. R. Biazzo. (*Annali Chim. Applic.*, 1918, **10**, 130-133.)—To prevent oxidation of the oil and loss of volatile constituents during the preliminary drying of oil seeds the finely divided substance is kept in contact with a dehydrating agent such as anhydrous copper sulphate. C. A. M.

Estimation of Benzene and Toluene in Petroleum. F. B. Thole. (*J. Soc. Chem. Ind.*, 1919, **38**, 39-42 T.)—In the author's scheme for the estimation of benzene and toluene in petrols the aromatic hydrocarbons are removed by sulphonation, for which purpose 98 per cent. sulphuric acid has been found most suitable, since with this acid there is no tendency to charring or development of sulphur dioxide such as occurs when "oleums" are employed. The hydrocarbon is vigorously shaken in a stoppered 50 c.c. cylinder with three times its volume of 98 per cent. sulphuric acid, which is sufficient for the sulphonation of mixtures containing up to 50 per cent. of aromatic hydrocarbons. With richer mixtures the supernatant layer should be treated a second time with fresh acid. The principle of estimating the volume of unattacked hydrocarbon for obtaining the proportion of non-aromatic constituents is discarded as not sufficiently accurate. The result is obtained very much more closely by determining the specific gravity of the spirit before and after the removal of the aromatic constituents. Thus, the percentage of aromatic hydrocarbons $= \frac{\text{initial sp. gr.} - \text{final sp. gr.}}{\text{sp. gr. of aromatic} - \text{final sp. gr.}} \times 100$. The results are calculated on the following data for the sp. gr. at 15°/4°: Benzene, 0·8841; toluene, 0·8712; *o*-xylene, 0·8807; *m*-xylene, 0·8680; *p*-xylene, 0·8618. The results should be corrected for the error due to the fact that the sp. gr. of a mixture of hydrocarbons is not the arithmetical mean of those of its constituents. The correction factor can be obtained from "deviation curves" which can only be constructed experimentally with known mixtures. A set of four such curves will suffice, two prepared with a petrol consisting mainly of paraffins and two with a petrol consisting mainly of naphthenes. These two petrols are distilled in two fractions, 40° to 95° C. and 95° to 122° C., which are mixed with benzene and toluene respectively in known proportions and the specific gravities determined. In the case of normal petrols free from admixture with cracked spirit, the influence of the olefines may be neglected; the presence of these may be detected, even when less than 1 per cent., by the immediate decolourisation of dilute permanganate. The influence of sulphur compounds in commercial petrol is necessarily so small as to be negligible. For the estimation separately of benzene and toluene it is necessary to concentrate the benzene as far as possible into one fraction and the toluene into another. As the result of several experiments with a Young-Thomas five-section column, the following are the cutting temperatures for the fractionation of the petrol containing these two aromatic hydrocarbons—namely, 40° to 95° C. for the estimation of the benzene, and 95° to 122° C. for the estimation of the toluene. Owing to the different specific gravities of the three xylenes, the results for this constituent cannot be obtained with the same accuracy unless the proportions of the three isomerides are known. J. F. B

Study of the Test for Tartrates Depending on the Formation of the Copper-Tartrate Complex. L. J. Curtmann and B. R. Harris. (*J. Amer. Chem. Soc.*, 1919, **41**, 207.)—The statement made in a previous paper (ANALYST, 1918, **43**, 96) that relatively small amounts of phosphates or borates, when treated by the Bottger procedure, respond in the same way as do tartrates, is now corrected. A large number of experiments has demonstrated that neither of these anions, even in amounts as high as 0·5 grm., give a blue filtrate. W. P. S.

INORGANIC ANALYSIS.

Chemical Standards for Iron and Steel and their Bearing on Unification of Analysis. C. H. Ridsdale and N. D. Ridsdale. (*J. Soc. Chem. Ind.*, 1919, **38**, 15-26 T.)—The Iron and Steel Institute having put in hand the preparation of analytical steel standards, the authors review the whole history of the movement in favour of such standards, and offer suggestions with a view to eliciting such discussion as may tend to help those entrusted with the preparation of standards, and thus increase the authority of any standards ultimately issued. Means are described for obtaining a large quantity (several hundredweight) of iron or steel in condition suitable for analysis and possessing homogeneity in a high degree. An account is given of co-operative work initiated by the authors, which led to the preparation of twelve standards, of which four are now exhausted. More than twenty chemists and firms took part in this work, including five referee analysts, three railway chemists, the chemists attached to the Admiralty and Ministry of Munitions, and the chemists to a number of the largest steel works. The mean and extreme values found by these workers are published by the authors, who further describe in some detail the methods they recommend for the analysis of iron and steel. G. C. J.

Purification and Analysis of Gallium Chloride. T. W. Richards, W. M. Craig, and J. Sameshima. (*J. Amer. Chem. Soc.*, 1919, **41**, 131-132.)—The method described depends on the fact that gallium chloride sublimes and distils at a relatively low temperature, whilst other chlorides likely to be associated with it are much less volatile. The apparatus used is made wholly of glass and all the joints are fused. Three successive distillations of the gallium trichloride are made in chlorine at 220° to 230° C., three more at about 175° C. (the melting-point of gallium dichloride), three in nitrogen at 90° to 110° C., and five sublimations under diminished pressure at 65° to 80° C.—fourteen operations in all. The resulting product shows no trace of other substances when examined spectroscopically.

W. P. S.

Extraction and Estimation of Dissolved Gases in Water. F. W. Richardson. (*J. Soc. Chem. Ind.*, 1919, **38**, 32-33 T.)—The apparatus of Thresh, Hoppe-Seyler, and Petterson for the extraction of gases from water is criticised adversely. By the use of a simplified form of eudiometer, already described by the author (*J. Soc. Chem. Ind.*, 1910, **29**, 198), he has been able to extract with relative ease the gases dissolved in natural waters. The first extractions were made by the aid of a pear-shaped thistle funnel of 250 c.c. capacity which had been completely evacuated. By immersing the lower tap in a measured volume of the water and by opening the tap, about 200 c.c. of the sample was drawn into the funnel, the upper stoppered tube of which was connected with the first burette of the eudiometer.

A simpler and more satisfactory method has now been adopted. A stout glass quart bottle is completely filled with the water. The lower tube of a bulb or thistle funnel head of 50 c.c. capacity and provided with two stoppers, as described in the earlier paper (*loc. cit.*), is passed through a rubber stopper, the bulb is evacuated and the stopper inserted in the bottle, thereby connecting the tube with the water. When the lower tap of the bulb is opened, a little of the water rushes up, and it becomes possible to press the cork firmly into the bottle neck. Evolution of gas at once begins. If the bottle is placed in a vessel of water at about 40° C. and the gases occasionally removed from the small bulb, the water will continue to boil until all gases have been removed. Carbon dioxide is tenaciously held by water, and when it is present alone repeated evacuations of the small bulb are necessary, but carbon dioxide can be completely removed in the apparatus. When the water contains much dissolved air in comparison with the amount of carbon dioxide, the latter is more easily removed, as the other gases help the evacuation. G. C. J.

Determination of Active Oxygen in Sodium Peroxide. J. Milbauer. (*J. prakt. Chem.*, 1918, **93**, 1-8.)—Methods depending on the liberation of hydrogen peroxide by water, followed by titration with potassium permanganate, and on the treatment of sodium peroxide with potassium iodide and potassium bicarbonate and titration of the liberated iodine with sodium arsenite give low results, whilst measurement of the oxygen liberated by water in the presence of cobalt nitrate gives high results. The following processes yield accurate results: (1) Water (100 c.c.) is mixed with concentrated sulphuric acid (5 c.c.) and chemically pure boric acid (5 grms.); sodium peroxide (0·5 grm.) is added gradually to the mixture, which is kept briskly shaken, and the liberated hydrogen peroxide is titrated with potassium permanganate. The low results given by the older permanganate method are to be attributed to the catalytic decomposition of a portion of the hydrogen peroxide by the manganese sulphate formed during the titration. (2) Sodium peroxide is introduced gradually into a solution of potassium iodide (2 grms.) in dilute sulphuric acid (1 in 20; 200 c.c.); the iodine is titrated with standard sodium thiosulphate. The results agree with those obtained by the permanganate method. (3) Sodium peroxide (0·2 to 0·3 grm.) is mixed with about 10 c.c. of copper sulphate solution (0·05 per cent.) in a small flask connected to a nitrometer; the flask is shaken, and decomposition is completed within a minute, when the liberated oxygen is measured. The gas evolved contains about 0·32 per cent. of carbon dioxide and 0·08 per cent. of hydrogen. With cobalt nitrate as catalyst, the results are invariably high; the author considers that this may indicate the presence of an oxide higher than the peroxide. The action of the atmosphere on sodium peroxide has also been investigated; moisture appears to be more active than carbon dioxide in causing decomposition.

Estimation of Phosphorus in Steel and Iron. N. D. Ridsdale. (*Chem. News*, 1919, **118**, 100-101.)—It is stated that the following quick method yields as accurate results as a method involving an arsenic separation, provided the prescribed conditions are rigidly adhered to. The steel (2 grms.) is dissolved in 55 c.c. nitric acid of sp. gr. 1·2, the solution is simmered to expel brown fumes, but not appreciably evaporated, and 5 c.c. of 5 per cent. permanganate solution are added. The solution is boiled for two minutes and enough 4 per cent. ammonium oxalate solution (10 to 13 c.c.) added to destroy the excess of permanganate and clear the solution. After simmering for two minutes, 15 c.c. of 60 per cent. ammonium nitrate solution are added, the mixture is heated to boiling, removed from the source of heat, and 50 c.c. cold, neutral 5 per cent. ammonium molybdate solution are added. The mixture is shaken for exactly two minutes, and allowed to stand at the temperature of the laboratory for 5 to 15 minutes. The phospho-molybdate should now be completely precipitated free from silicon and arsenic, and can be filtered and estimated by any of the usual methods. The author strongly recommends titration by means of $\frac{N}{6 \cdot 72}$ sodium hydroxide and nitric acid. The number of c.c. of sodium hydroxide required $\times 0 \cdot 01 =$ percentage of phosphorus in the steel. G. C. J.

Estimation of Phosphorus and Silicon in Cast Iron. A. Cavazzi. (*Annali Chim. Applic.*, 1918, **10**, 137-149.)—The following simplified modification of the author's method (ANALYST, 1917, **42**, 371) is described: Five grms. of the finely-divided sample of iron are washed with ether, and treated with 60 c.c. of dilute (1 : 1) nitric acid, until only the silica and carbon remain undissolved. The contents of the beaker are then transferred to a porcelain basin and slowly boiled, after the addition of 9 c.c. of sulphuric acid, with frequent stirring, until a paste-like residue is obtained and white acid fumes appear. The temperature is then raised, but not above 210° to 220° C., and the heating continued until a slightly calcined mass consisting of ferric sulphate, silica, and carbon is left. This is cooled, finely pulverised, and again heated and stirred until all free sulphuric acid has been expelled, after which it is boiled in a beaker with 50 c.c. of water and 10 c.c. of strong nitric acid. During the boiling the beaker is covered with a small retort filled with cold water, to act as a reflux condenser. After thirty to forty-five minutes the whole of the ferric sulphate will have dissolved, and the phosphorus have been converted into orthophosphoric acid. The liquid is cooled and filtered from the carbon and silica, and the residue washed six times with water acidulated with a few drops of nitric acid; the filtrate and washings are treated with 15 grms. of crystalline ammonium nitrate, and heated to boiling, after which 40 c.c. of a solution containing 5 grms. of ammonium molybdate are added, and the mixture vigorously stirred and kept for thirty minutes at 70° to 80° C. It is then cooled, the liquid filtered, and the precipitate washed four times by decantation with 20 c.c. each time of a boiling solution of 25 grms. of ammonium nitrate and 20 c.c. of nitric acid in 500 c.c. of water. It is next dissolved in ammonia solution (1 : 2), and the solution treated with 3 grms. of sodium chloride, and boiled for ten minutes, the effect of which is to separate as ferric phosphate any iron remaining in the phosphomolybdate. The precipitate is separated, washed with boiling water containing 1 grm. of sodium chloride in 50 c.c. and ignited in a platinum crucible. The ash is fused with about 0·3 grm. of a mixture of sodium and potassium carbonates with a little potassium nitrate, the mass taken up with boiling water, the liquid filtered, and the filtrate acidified with nitric acid and boiled for ten minutes. A slight excess of ammonia is then added, the solution united to the original solution, and the phosphoric acid precipitated with magnesia mixture, and weighed as pyrophosphate. The residue of carbon and silica is washed free from iron, as described above, dried in the oven, and ignited, apart from the filter-paper, in a platinum crucible, and the residual silica is weighed.

C. A. M.

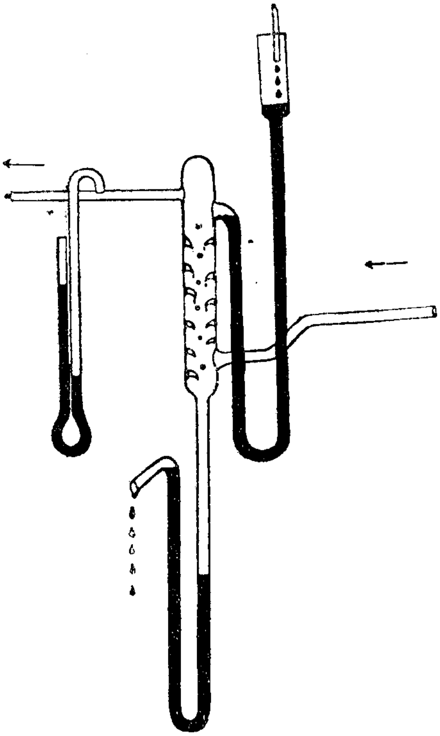
Rapid Method for the Estimation of Sulphide Sulphur in Pyrites. A. Bartsch. (*Chem. Zeit.*, 1919, **43**, 33-34.)—The iron sulphide in pyrites does not readily yield hydrogen sulphide when treated with acids, but the author has found that, in contact with mercury, hydrobromic acid will decompose the whole of the sulphide with evolution of hydrogen sulphide. This observation forms the basis of a rapid method for the quantitative valuation of pyrites, employing an apparatus similar to that used in metallurgical laboratories for the estimation of sulphur in iron and steel. An evolution flask of Franke's pattern with water-cooling is preferably used, and a delivery tube leads directly from this to a Philipps' beaker of 600 c.c. capacity, half filled with the absorption reagent. This reagent consists of 25 grms. of cadmium acetate and 200 c.c. of acetic acid diluted to 1 litre, or, for economy, 20 grms. of zinc acetate and 5 grms. of cadmium acetate may be substituted for the pure cadmium salt. Zinc acetate alone will not serve, giving results considerably too high. The bottom of the evolution flask is covered with mercury (about 20 c.c.); 0·25 grm. of the finely powdered pyrites is introduced with a little water, and the air is expelled from the apparatus by a strong current of carbon dioxide which has been washed with some of the above absorption liquid. Fifty c.c. of hydrobromic acid, sp. gr. 1·49, free from chlorides, are now introduced, and the contents of the flask are warmed, but not boiled, until the end of the reaction, which is recognised by the disappearance of the colour of the acid. The liquid is then boiled, the flame is removed, and a strong current of carbon dioxide passed through the apparatus in order to sweep out the hydrogen sulphide, which adheres obstinately to the walls of the flask. This scrubbing operation cannot be omitted nor replaced by mere boiling. The liquid in the absorption vessel is treated with 50 c.c. of standard iodine solution, acidified with 50 c.c. of dilute hydrochloric acid (1 : 1), titrated with a small excess of thiosulphate, and then accurately back with the iodine solution. The results agree well with the gravimetric oxidation process; the presence of copper does not interfere with the accuracy of the estimation.

J. F. B.

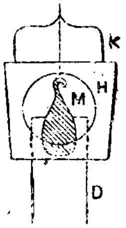
APPARATUS, ETC.

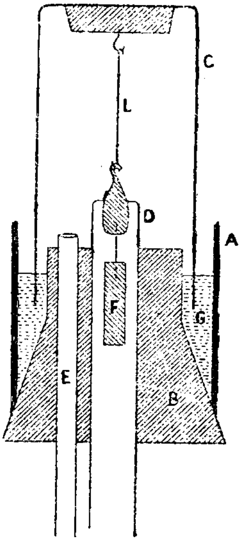
Limits of Separation by Fractional Distillation. A New Still-Head. S. F. Dufton. (*J. Soc. Chem. Ind.*, 1919, **38**, 45-46 T.)—The author describes new type of still-head (see figure), consisting of a glass tube 120 to 150 cms. long, of 17 mm. internal diameter, in which is fitted a closed inner tube, 13 mm. external diameter, so that a narrow annular space is formed between the two. A spiral of wire of 1 mm. diameter is wound round the inner core. When the still is in action the greater part of the descending liquid is attracted to the wire and travels down the spiral track, sealing the annular space between the tubes so that the ascending vapour is forced to traverse the same path in the opposite direction and come most effectively into contact with the liquid. A column of this type was found to be extremely efficient, the pitch of the wire spiral being made 18 mm. at the top, increasing to 22 mm. at the bottom. If the distillation flask be fitted at floor level, the delivery tube reaches a convenient height above the working bench. The column is protected by a covering of 2 inches of cotton wadding bound with asbestos yarn. A further development of the principle has been adopted in order to make the still capable of the most complete separation of small quantities of liquid. A still-head was constructed 120 cms. long of glass quill tubing in sections increasing in diameter from top to bottom. The top section contained a bicycle spoke 1·4 mm. in diameter, round which was wound a spiral of wire 0·7 mm. in thickness; the second spiral was made of wire 1·0 mm. thick, the third 1·2 mm., and the last 1·4 mm., the cores all being similar and the pitch increasing from 6 mm. at the top to 12 mm. at the bottom. With this still-head a mixture of 5 c.c. of benzene and 10 c.c. of toluene yielded 4·6 c.c. of pure benzene and 9·4 c.c. of pure toluene. The cut-point at 94° C. corresponded to the distillation of a volume of liquid equal to the actual volume of benzene present in the mixture. The quantity of liquid running back into the distillation flask was only 1·4 c.c. The still-head is more easily constructed of a single piece of tubing of approximately uniform bore, the gradation of the annulus being obtained by winding the spirals upon cores of decreasing diameter. These still-heads have the great practical advantage that the distillation, having been started at the proper rate, slows down and stops when the limit of each pure constituent is reached. It is possible with this apparatus to analyse mixtures of benzene, toluene, and xylene by direct distillation. The main portions are obtained pure and the intermediates are so small in amount that there is no appreciable error in the use of a midway cut-point for estimation of the total quantity of each constituent. The cut-point for benzene is 94° C., and that for toluene is 124° C. An apparatus of similar type is extremely effective for the purpose of washing a gas with a small quantity of liquid. In this case the annulus is of uniform size throughout, and the pitch of the wire spiral is kept as small a practicable in order to reduce the speed of descent of the liquid. J. F. B.

Apparatus for Continuous Testing of Gases with Special Reference to Acid or Alkaline Constituents. C. A. King. (*J. Soc. Chem. Ind.*, 1919, **38**, 33 T.) —The apparatus illustrated was designed to detect ammonia in the gases leaving an ammonia recovery plant, but it is suggested that it might serve equally well for other purposes—*e.g.*, the detection of acid vapours in chimney gases. A current of the gas from the outlet main is caused to pass upwards through a small glass absorption chamber about 7 cms. long and $1\frac{1}{2}$ cms. in diameter, provided with projections from the inner wall to effect more efficient contact with a solution of a suitable indicator, which flows from a U-shaped tube into the upper end of the extraction chamber. The indicator solution drains away from the lower end of the absorption chamber through a similarly shaped tube in which any change of colour of the solution is noted. The inlet and outlet bends are of such a length that the solution forms a liquid lute against the pressure of gas in the apparatus. The indicator used by the author was a solution of methyl red with sufficient acid added just to develop the true red colour of the acid indicator. Complete absorption of ammonia takes place with a flow of gas through the apparatus at the rate of 5 cubic feet a minute, and with the indicator solution set at a fast dropping rate a very sensitive indication can be obtained. The arrangement as shown is made specially to obviate siphoning of the entering liquid, but under certain conditions siphoning at regular intervals might be an advantage. G. C. J.



Gas Pressure Governor for the Bunsen Burner. G. R. Clare. (*J. Soc. Chem. Ind.*, 1919, **38**, 38 T.)—Sometimes it is necessary to have the pressure of gas for one burner controlled, but not desirable to have the gas supply of the whole laboratory reduced by a governor fitted to the main. The governor illustrated was designed to regulate the supply to a single burner. The inlet valve is made by grinding a piece of glass rod into the glass tube *D*, suitably shaped to act as a seating, and is suspended from a cork fastened into the bell with glue. *A* is a brass tube, 2 × 2 inches; *B* a rubber stopper; *C* a glass weighing bottle, $1\frac{5}{8}$ inches





diameter and 3 inches long; *D* the gas inlet tube, $\frac{7}{16}$ inch diameter; *E* the gas outlet tube, $\frac{5}{16}$ inch diameter; *F* a lead weight attached to the valve by platinum wire, to prevent the valve from sticking due to occasional high pressure; and *G* is a mercury seal. The bell is kept vertical by means of the arrangement shown in the smaller diagram. *H* is a $\frac{7}{8}$ inch cork fixed on to the gas inlet tube, *D*, and holding a glass tube, *K*, drawn out to a small hole which serves as a guide for the wire, *L*. The gas passes from *D* into the bell, *C*, by the hole, *M*. The required controlled pressure can be varied by means of the weight, *F*, or by placing an additional weight centrally on the top of the bell. G. C. J.

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REVIEWS.

COAL AND ITS SCIENTIFIC USES. By WILLIAM A. BONE, D.SC., PH.D., F.R.S. London: Longmans, Green and Co., 1918. Price 21s. net.

The subject of fuel and its efficient utilisation claims an early place in a series of monographs on Industrial Chemistry, and this work of Professor Bone is accordingly one of the earliest published in the series edited by Sir Edward Thorpe.

A large amount of information drawn from scattered sources is compressed into the 22 pages of the introductory statistical chapter, dealing with output, coal reserves, analyses of the uses of coal in the United Kingdom, and cost. After a short sketch of the theories of the origin and formation of coal, and a description of the British coalfields, follow seven chapters on the chemical composition of coal. In these chapters, occupying over a quarter of the book, Professor Bone gives a critical account of recent investigations on the composition of coal, its carbonisation products at low and high temperatures, and the phenomena of weathering and spontaneous oxidation of coal. These chapters form very interesting reading; the facts are summarised in clear and logical sequence, and impartially criticised. Chapter X. deals with the combustion of carbon and of coal. A large amount of information of value to boiler users is contained in these 18 pages, but the matter is very compressed. Thus, the important work of Kreisinger, Ovitz, and Augustine is summarised in a dozen lines without name or reference. A description is given of the "Underfeed" stoker in this chapter, and the context might lead the reader to suppose that this is a type of the usual mechanical stoker. The use of powdered fuel is also mentioned here, although the dust-fired Bettington boiler is described in a later chapter. (A search for this boiler disclosed some deficiencies in the index: there is no entry in the name index under "Bettington," or in the subject index under "Coal dust." It appears under "Boilers," wrongly paged 313 instead of 213.) The next chapter, dealing with the principles governing combustion and heat transmission in boilers, gives a summary of the valuable researches of Constam and Schläpfer on the efficiency of two types of boiler with different varieties of fuel, a short discussion of the evaluation of chimney losses, and an account of the principles of heat transmission in boilers. Some of the opening remarks of this chapter are not happily worded, and unduly depreciate the efficiency of a modern power plant. One-fifth of a pound of coal of 13,000 B.T.U. is not "the theoretical figure" for one H.P. hour; the comparison should be with the thermodynamical heat engine working between the same limits of temperature. The subjects of domestic heating and smoke abatement are briefly dealt with, and then follow five chapters dealing with the carbonisation and gasification of coal. This section is not quite so satisfactory as the remainder of the work. It is not so up-to-date, and does not give the impression of first-hand knowledge which is characteristic of the rest of the book. The effect of war conditions upon the gas supply is barely mentioned, most of the data stopping short at 1913. A very important section is that dealing with the possibilities of fuel economy in the manufacture of iron and steel. In the year 1913, the author estimates that the total consumption of coal in connection with the manufacture of iron and steel was something under 30 million tons, rather less than 3 tons per ton of iron or steel. The organisation of a modern iron and steel plant with a view to fuel economy is then discussed, and suggestions are put forward which, if carried out generally in this country, would undoubtedly result in a very marked economy of coal. Chapter XXI., on power production from coal, opens with the remark that the subject is "so many-sided that any brief review of it in these pages is bound to be inadequate." One could only wish that space could have been found for a comparison of capital outlay and cost of upkeep of a large boiler and steam turbine plant and a producer gas and gas engine plant of the same output. Comparisons of efficiency are numerous, with a result usually in favour of gasification, but the discussion of capital costs, and especially of running costs, is usually left severely alone. The final chapter, on surface combustion, is a reprint of a lecture given before the Royal Institution of Great Britain five years ago, supplemented with an account of some more recent developments. The accounts of the temperatures reached on the Bonecourt gas muffles will arouse feelings of envy in chemists whose laboratory muffles, fed with war gas, can hardly be persuaded to reach 1,000° C. Perhaps the author will later turn his attention to the design of small laboratory muffles on the surface combustion principle.

In a work dealing with the utilisation of fuel the definition and measurement of calorific value is fundamental, and it is to be regretted that Professor Bone has given the weight of his authority to the use of the net as opposed to the gross calorific value in calculating efficiencies. After stating "that for scientific purposes the *gross* value is invariably employed," the, or rather, *a* net value is used throughout the book. If the calorific value is to be regarded as a property constant for a given material, and not as a variable dependent on its mode of use, then the gross value, the actual experimental figure, should alone be employed. Once started on the slippery slope of correcting the observed value according to the mode of utilisation, there is no logical stopping-point until the heat carried away by the nitrogen and carbon dioxide is also deducted. Several definitions of net calorific value are in use. Professor Bone gives one, which is doubtless used throughout in the heat balances based on his own work. The net values used by Constam and Schläpfer, quoted on p. 196, are based on another definition, and there are others. The fourth significant figure in calorific values is usually uncertain, even in the best determinations, to two or three units. Throughout the book superfluous figures in heat balances are usually suppressed, but there are some slips, as on p. 464, where the product of a gas volume, measured with an accuracy certainly less than 1 in 1.000, and a calorific value of the same order of accuracy, the total net heat supplied is given as 52,003,996 B.T.U.'s. This is from an American report, but another example occurs on p. 398, in the heat account of a blast furnace, where the total outgoing energy is stated as 35,908,000

B.T.U.'s, in which the carbon in the coke is credited with its full heat of combustion, although a portion of it, representing over 1,000,000 B.T.U.'s, remains unburnt in the cast iron. In the table of calorific values on p. 50 the maximum figure for anthracites (9,000 K.C.U.'s per kgrm.) is certainly too high.

The analysis of coal is sketched without detail in a dozen pages. Under the heading, "The Special Sources of Error in the Estimation of Carbon and Hydrogen in Coals," the effects of the presence of calcium carbonate and of hydrated minerals are considered : the loss of carbon due to the difficulty of completely burning methane with copper oxide is not mentioned. The author does not use the international method for the estimation of the volatile matter, and gives an alternative method of his own. The American method, the description of which is quoted textually as the international method, was modified before receiving international recognition. In a purely arbitrary determination such as this is, there are many advantages in an international agreement as to method.

Professor Bone states in the preface that the book has been written during a period of quite exceptional stress of academic and professional duties. He has, at any rate, produced a book which must be read by all who are interested in the economic utilisation of solid and gaseous fuel. G. NEVILL HUNTLY.

HIGH EXPLOSIVES—A PRACTICAL TREATISE. By CAPTAIN E. DE W. S. CALVER. London : Crosby Lockwood and Son, 1918. Price £3 3s. net.

The author and compiler of this large work intimates very rightly that little attention has been given, in the past, to the chemistry of explosives. Long periods have certainly elapsed between what may be termed epoch-making discoveries of explosive substances.

These long periods of quiescence may be due to absence of, or lack of, interest in the subject on the part of chemists; but it may also be asked, What stimulus or incentive has the chemist had, even of our day, or prior to 1914, to arouse his interest in this class of substance? Gunpowder was thought good enough to fill all the requirements of the military authorities, and the introduction of anything new may possibly have been regarded as unnecessary. Of course it needs a *good deal* of trouble and patience and some hope of justification of expense to try new things, but it looks as if in the past a very little prospective trouble and extra work prevented the employment of new things which might have been foreseen by a properly educated authority to have promised improvement on the old, and which have actually, on eventual trial, proved themselves to be superior. Mining has been the chief encourager of the explosive industry, and there also improvements have been made as a result of trial and failure.

After a mere mention of the old black powder, and a very short historic sketch of the invention and introduction of substances such as fulminate, picric acid, trinitrotoluene, and the like, chapters follow containing very full descriptions of the plant employed, as well as many proposed and patented for the manufacture of the specific explosives. There are also complete details on the preparation of acids and reagents employed in the explosives industry and in the recovery of waste products. These are very fully illustrated by diagrams, plans, and in some cases by photos of the apparatus. The book at first glance, from the large amount of detail crowded around each item, seems a little confusing. There is, however, a contents to each chapter which is of material assistance. The specifications, or as much as is desirable, for "passable" purity of the various explosives, and their raw materials, are given, and particular care seems to have been taken to collect methods of analytical examination as used abroad in addition to our officially recognised methods of examination.

The consideration of trinitrophenol takes about fifty pages—not so much as some more recently introduced substances—commencing with the hexagon representation of the three modifications. In the formation of the sulphonic derivative of phenol this elaboration is not continued, the diagram of the 1 : 2 sulphonic acid only being given. Surely it would have been instructive to put in all three, and also to mention why the phenol and sulphuric acid are heated, or should be, to a higher temperature than mentioned on p. 294. When the 1 : 4 sulphonic acid is formed, the nitration product contains very little, if any, β or γ modification.

Following the physical and chemical properties of the explosive substance—taking picric acid as an example—is a section on testing and analysis. It is typical of the methods of testing given of other explosives, acids, and raw products. After describing the methods of determining the setting and melting points, follows the examination for foreign matters, ash, acids, other carbon compounds, moisture, nitrogen, and trinitrophenol content. In this connection several of the methods of carrying out the "Nitron" process of nitrogen determination are given. The setting-point determination is described, with a diagram of the apparatus, in the chapter on trinitrotoluene; but the official method of taking melting-points does not seem to be mentioned excepting in the appendix.

The chapters on the testing of acids used for nitration purposes and waste or recovered acids are very clear and quite practical. They contain also several useful tables.

The chapters on the valuation and testing of benzene, naphtha, phenol, etc., are excellent, and bear out the author's opinion expressed on p. 63 "that analytical control at all stages of the processes is most desirable and is *most necessary* if a factory is to be run on sound economical principles." Your reviewer would like to have "most necessary" written "absolutely necessary."

Chapter XLVIII., pp. 600 to 670, are taken up with description of methods of ascertaining the pressure of explosive gases, velocity of detonation, sensitivity, energy, and other questions which, if not chemical, are in a sense analytical.

There are several appendices: Patents dealing with nitro compounds; specifications for raw materials and explosives; bibliography; a patent register; name index and subject index.

W. R. HODGKINSON.

INDIGENOUS DRUGS OF INDIA. By J. C. GHOSH, B.Sc., F.C.S. Calcutta : Butterworth and Co. Price 1s.

This small monograph, one of a series of six on allied subjects by the same author, contains much suggestive information relating to the future development of

India as a source of crude vegetable drugs. Owing to the war European supplies were largely cut off, and steps have recently been taken in that country to develop the cultivation of belladonna, ipecacuanha, and other plants. The results obtained indicate that a great and promising field is here open to pharmaceutical manufacturers from both a scientific and commercial standpoint, especially when it is realised that the greater proportion of medicinal substances used by the inhabitants of that vast country are derived from the vegetable kingdom.

The information contained is accurate, and although the style is somewhat quaint at times, in no case is the reader left in doubt as to the author's meaning.

A useful feature is an Appendix consisting of a table giving the names in eight Indian languages of the official vegetable drugs recognised by the British Pharmacopœia.

The pamphlet should be of much use to pharmacists, technical chemists, and others interested in the scientific cultivation and manufacture of plant products.

T. J. Ward.