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THE ANALYST.

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

EFFECT OF MORPHINE CONCENTRATION ON THE B.P. METHOD OF MORPHINE ESTIMATION.

By H. E. ANNETT, B.Sc. (Lond.), F.I.C., and HARDAYAL SINGH, B.Sc.

In the course of work we have been carrying out on Indian opium we have met with samples which have shown a wide range in morphine content. Some have yielded over 18 per cent. morphine, whereas some samples have contained no morphine. We understand that the B.P. method has been worked out for the analysis of opium containing about 10 per cent. morphine. In our work we have been studying the effects of various factors on morphine content in opium; and since we have mainly used the B.P. method we have been interested in studying the conditions required to make that method reliable. We have found nothing of this nature in the literature, and it seems worth while to record our experimental results.

Analysis of Pure Morphine by the Method of the B.P. 1914.—A weighed quantity of crystalline morphine was in each case triturated with 2 grms. of freshly slaked lime and 20 c.c. of distilled water. A further 60 c.c. of distilled water was subsequently added, the whole was stirred at intervals during half an hour, and the morphine precipitated in 50* c.c. of the filtered liquid. The morphine was filtered off and weighed, after drying at 60° C., and subsequently for two hours at 115° C.

Weight of Crystalline Morphine taken.	Weight of Amorphous Morphine in 50 c.c. taken for Precipitation (calculated).	Weight of Amorphous Morphine found (corrected by addition of 0·050 grm.).	Per Cent. Morphine recovered.
Grm.	Grm.	Grm.	
0·25	0·147	0·1228	83·54
0·50	0·294	0·2818	95·85
0·75	0·441	0·4244	96·23
1·00	0·588	0·5860	99·66
1·25	0·735	0·7344	99·92
1·50	0·882	0·8934	101·30

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* The B.P. 1914 method states that 51 c.c. should be taken. In these experiments with pure morphine we only took 50 c.c. of lime solution, and we added 0·050 instead of 0·051 grm. as a correction for the morphine remaining in solution.

The results indicate that with quantities of 1 grm. of crystalline morphine and upwards the B.P. method gives accurate results. When only 0·25 grm. of crystalline morphine was taken, the method gave low results. Since 8 grms. of opium are taken in the B.P. 1914 method, 0·25 grm. morphine would correspond to an opium of less than 3 per cent. amorphous morphine content.

Analysis of Samples of an Opium containing Various Proportions of Starch.—A sample of moist opium was well mixed and varying amounts, recorded below, were then weighed, and the morphine estimated by the B.P. process.

Sample.	Weight of Amorphous Morphine obtained.	Weight of Amorphous Morphine (corrected +0·051 grm.).	Per Cent. Morphine in Sample.	Per Cent. calculated.
	Grm.	Grm.		
8 grms. opium	0·2675	0·3185	6·37	6·37
6 grms. opium + 2 grms. starch	0·1592	0·2102	4·20	4·78
4 grms. opium + 4 grms. starch	0·0347	0·0857	1·71	3·18
2 grms. opium + 6 grms. starch	0·0012	0·0522	1·044	1·59
8 grms. starch	0·0046	—	—	0·00

It would seem from these figures that with opium low in morphine the B.P. method gives results for the morphine content which are considerably below the truth.

Quantities Taken.	Sample Number.	Weight of Anhydrous Morphine.	Weight of Anhydrous Morphine (corrected + 0·051 grm.).	Per Cent. Morphine in Opium.
(*a*) 8 grms. ...	I.	0·3149	0·3659	7·32
(*b*) 4 ,, ...		0·1368	0·1623	6·49
(*c*) 2 ,, ...		0·0727	0·0854	6·84
(*d*) 12·75 c.c. ...		0·0840	0·0967	7·74
(*a*) 8 grms. ...	II.	0·4780	0·529	10·58
(*b*) 4 ,, ...		0·2308	0·2563	10·25
(*c*) 2 ,, ...		0·1268	0·1395	11·16
(*d*) 12·75 c.c. ...		0·1168	0·1296	10·37

Estimations by B.P. 1914 Method, using Reduced Quantities of Opium and Proportionately Reduced Quantities of Reagents.—In the course of our work we found it necessary to get an idea of the morphine content in samples of opium too small to be analysed in the usual way. We therefore wished to see if we could use 4 grms. and even 2 grms. of opium instead of the 8 grms. laid down in the B.P. method. We tested this point on two samples (I. and II.). In each case we weighed out (*a*) 8 grms., (*b*) 4 grms., (*c*) 2 grms., of the well-mixed powdered opium sample.

(*a*) was analysed in the ordinary way (B.P. 1914); (*b*) was analysed using half the quantities of the reagents recommended. The correction used was 0·0255 grm., and 25·5 c.c. of the lime solution were taken for precipitation. (*c*) was analysed using quarter quantities of the reagents. The correction used was 0·0127, and 12·75 c.c. of the lime solution were taken for precipitation.

After taking 51 c.c. of the lime filtrate from (*a*) for analysis, enough liquid was left to take a further 12·75 c.c. This was then precipitated as described under (*c*) and is designated (*d*) in the table.

The error of the B.P. 1914 method is stated to be 0·5 per cent. morphine either way. In view of this it must be concluded that the results here set out show that useful results can be obtained by analysing opium samples when only 4 grms., or even 2 grms., of the sample are available.

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METHODS OF CALCULATING ADDED WATER IN MILK.

BY LESLIE J. HARRIS.

IN a previous communication dealing with the calculation of added water in milk (ANALYST, 1918, **43**, 345) I proposed the use of the formula,

$$W = 100 - \frac{10{,}000\ N}{8{\cdot}5\ (100 - F) + 3\ N},$$

as being specially applicable to samples containing an excess of cream. By this formula the added water is calculated on the assumption that the original milk contained the *minima* of both fat and solids-not-fat, it being postulated that an accumulation of fat from F to F′ is accompanied by a diminution of solids-not-fat from N to $N \times \frac{100 - F'}{100 - F}$

The formula above involves a rather long and unwieldy calculation. This may be obviated by having resource to one of the methods below:

1. *Calculation of Solids-not-Fat in the Fat-Free Milk.*—If the sample contains N per cent. of solids-not-fat and F per cent. of fat, the amount of solids-not-fat in the fat-free milk will be $\frac{N \times 100}{100 - F}$, and from this we may determine the extraneous water by reference to the table below.

For example, "milk" containing 10 per cent. of fat and 3·88 per cent. of solids-not-fat, contains in the fat-free portion $3{\cdot}88 \times \frac{100}{90} = 4{\cdot}31$ per cent. of solids-not-fat, which is seen to be equivalent to a mixture of 50 parts of water with 50 parts of milk of the minimum standard

In the table, the values for *solids-not-fat in the fat-free milk* are calculated from

$$\frac{(100-\text{added water})}{100} \times 8{\cdot}5 \times \frac{100}{100-\frac{(100-\text{added water})}{100}\times 3}.$$

Mathematical reasoning will show that the results obtained from this expression are identical with those given by the formula.

Added Water.	Solids-not-Fat in Fat-Free Milk.	Corresponding to Solids-not-Fat in Milk of Minimum Standard.	Added Water.	Solids-not-Fat in Fat-Free Milk.	Corresponding to Solids-not-Fat in Milk of Minimum Standard.
Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
0	8·76	8·50	19	7·06	6·88
1	8·67	8·41	20	6·97	6·80
2	8·58	8·33	21	6·88	6·71
3	8·49	8·24	22	6·79	6·63
4	8·40	8·16	23	6·70	6·54
5	8·31	8·07	24	6·61	6·46
6	8·22	7·99	25	6·52	6·37
7	8·13	7·90	26	6·43	6·29
8	8·04	7·82	27	6·34	6·20
9	7·95	7·73	28	6·26	6·12
10	7·86	7·65	29	6·17	6·03
11	7·77	7·56	30	6·08	5·95
12	7·68	7·48	40	5·19	5·10
13	7·59	7·39	50	4·31	4·25
14	7·50	7·31	60	3·44	3·40
15	7·41	7·22	70	2·57	2·55
16	7·32	7·14	80	1·71	1·70
17	7·23	7·05	90	0·85	0·85
18	7·15	6·97			

RULE.—To find the "solids-not-fat in fat-free milk," multiply the *solids-not-fat* by $\frac{100}{100-\text{fat}}$; then find the corresponding figure for added water in Column I.

2. *Alignment Chart.*—Those who prefer a graphical method may make use of an alignment chart. The chart is read by means of a straightedge, stretched thread, or line engraved on a celluloid cursor, etc. The line is made to coincide with the values for fat and solids-not-fat on the respective scales, and the percentage of added water is read off at the intersection with the third scale. A chart of moderate size will indicate the added water with an accuracy of about 0·5 per cent.

The chart is constructed on the principle of a nomogram ("Traité de Nomographie." Par Maurice d'Ocagne. Paris, 1899). The left-hand scale is divided logarithmically in an upward direction from 1 to 100, the figures for fat being complementary, and therefore at a distance from the base proportional to log (100 – F). The scale for solids-not-fat is divided directly on the same logarithmic scale, but in a negative sense. The third scale is midway between and parallel to those for fat and solids-not-fat. If, now, the upper half of this scale be divided logarithmically from 1 to 100, a straight line joining the points representing F per cent. fat and N per cent. solids-not-fat will give a reading of $\frac{(100-\mathrm{F})}{\mathrm{N}}$ in the central scale. By lowering the scale a distance log. 8·5, the readings will be multiplied by 8·5 $\left(i.e.,\ 8\cdot5\,\frac{(100-\mathrm{F})}{\mathrm{N}}\right)$; and by adding 3 units to each of the readings, converting these into their reciprocals, and multiplying by 10,000, the readings will be of the value $\frac{10{,}000}{8\cdot5\,\frac{(100-\mathrm{F})}{\mathrm{N}}+3}$. Since this is equivalent to $\frac{10{,}000\ \mathrm{N}}{8\cdot5\,(100-\mathrm{F})+3\ \mathrm{N}}$, the scale readings will now give the percentage of milk (of the minimum standard) in the mixture, the complementary numbers given in the chart therefore representing the added water.

A further use of the chart is to determine the change in composition of any sample of milk under the action of gravity or centrifugal force; for example, given the fat and total solids in whole milk to find the amount of solids-not-fat which the skim milk would contain, and the composition of the cream for any concentration of fat. For this purpose, all that is required is to place the straight edge coincident with the figure for fat and solids-not-fat, and rotate it around the point of intersection in the central scale until the new conditions are reached.

3. *Slide Rule.*—The principle of the alignment chart may be embodied in the form of a calculating rule. The reading for solids-not-fat on the lower scale is made to coincide with that for fat on the sliding scale; an arrow points to the percentage of added water on the upper scale. The rule is constructed in a precisely similar way to the alignment chart, except that the scale for added water is relatively twice as large, the percentages of added water being placed immediately opposite the figures for solids-not-fat in fat-free milk, as given in the table above. With the sliding scale in a given position, all possible corresponding combinations of fat with solids-not-fat are coincident.

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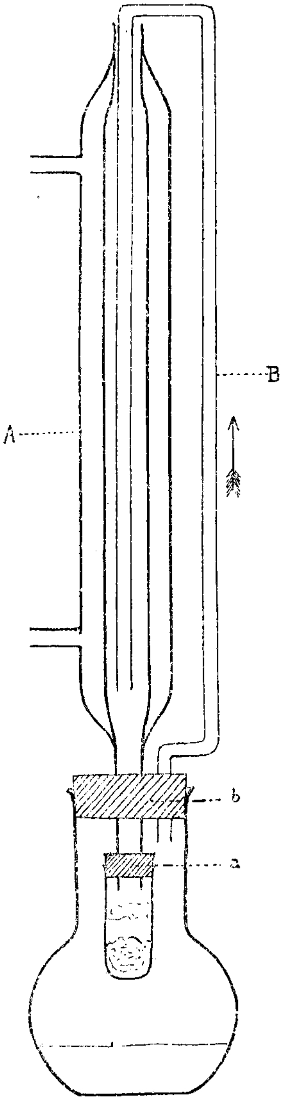
FAT EXTRACTION APPARATUS.

By E. GRIFFITHS-JONES.

The apparatus shown in the accompanying illustration was designed for use when the ordinary Soxhlet apparatus was not available. The apparatus is very simple in construction, and has been found to work satisfactorily, and to give a very rapid extraction.

It consists of an ordinary vertical condenser (A), the lower end of which passes through two corks, the upper one (*b*) to serve as a connection for the extraction flask, and the lower one (*a*) for the reception of the extraction thimble. The side tube (B) passes just through the upper cork, upwards and parallel with the condenser, and then down the condenser tube to approximately the level of the water intake. The material to be extracted is placed in the thimble, covered with a plug of cotton-wool, and firmly fitted over the lower cork (*a*).

The condenser with thimble attached is then connected up with a flask of suitable size containing the solvent. In this laboratory the extraction is carried out over an electrically heated sand bath.



The solvent vaporises and passes up through the side tube (B), and is carried down the neck of the condenser. The condensed solvent drops into the thimble and percolates through the thimble into the flask. It has been found that with 10 grms. of a fairly closely packed material, such as flour, the solvent is never retained, but passes through at a regular rate as soon as a very slight head of pressure of condensed solvent has been reached.

It has been found in practice that the extraction is more rapid with this apparatus than when the ordinary type of Soxhlet is employed.

Other points worthy of consideration are, firstly, that only a small quantity of solvent is required; secondly, there is no danger that, if a partial loss of solvent occurs through evaporation from the top or the condenser, the extraction will cease. This occasionally happens with the ordinary form of Soxhlet, especially in hot climates, where the condenser water is often only a few degrees in temperature lower than the boiling-point of the solvent.

For the estimation of fat in milk by the Adams's method, the coil of paper is tightly rolled and placed inside the thimble.

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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.

FINE CHEMICALS.

The expression "Fine Chemicals" is often loosely employed. One illustration is given in the November Analyst, where Dr. Jowett, in his review of Dr. May's book on "The Chemistry of Synthetic Drugs," p. 396, mentions that "substances used in medicine" are "often referred to as Fine Chemicals."

Medicinal chemicals are but one sub-group of the heterogeneous group of substances comprehensively termed "Fine Chemicals." Analytical reagents and other laboratory chemicals are another sub-group of Fine Chemicals. In their paper on "The Manufacture of Fine Chemicals in Relation to British Chemical Industry," read before the Society of Chemical Industry at their annual meeting in July. 1916, C. A. Hill and T. D. Morson used the term "Fine Chemicals" in this comprehensive sense, and classified Fine Chemicals into three sub-groups: (1) Reagents, (2) Pharmaceutical, (3) Technical. The third group is capable of considerable subdivision, and a number of instances were given in the paper showing how far-reaching is the Fine Chemical industry.

When the Association of British Chemical Manufacturers was founded, a group known as the Fine Chemical Group was formed, and the terminology adopted was that used by Hill and Morson, slightly extended, and appears thus under the general title:

Grouping of Chemical Industries ... ... Group VI.
Fine Chemicals: Analytical; Pharmaceutical; Photographic; Rare Earths; Synthetic Essences and Perfumes.

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EXPERIMENTS ON WASTE PRODUCTS.

The Munitions Inventions Department of the Ministry of Munitions has instituted inquiries with the object of ascertaining whether waste products of various chemical manufactures can be utilised for industrial purposes, and the Waste Products Committee of this Department has carried out experiments on the waste products set out below. Particulars as to the results of their experiments will be furnished to chemical manufacturers interested, upon application being made to the Controller of Munitions Inventions, 10, Princes Street, Westminster, S.W. 1.

1. Sulphide of arsenic residues from the purification of sulphuric acid.
2. Residues containing appreciable quantities of selenium.
3. Waste hydrochloric acid from pickling.
4. Waste chromium sulphate liquors resulting from the oxidation of organic substances.
5. Residues from the manufacture of acetic anhydride.
6. Residues suitable for the purification of coal gas from sulphuretted hydrogen.
7. Maize residues from the manufacture of butyl alcohol.
8. Chrome leather scrap.
9. Mimosa bark residues.

The Committee has also investigated various methods of de-rusting.

GERMAN (PRISONERS OF WAR) BREAD.

The sample described below was handed to a medical man by a prisoner of war returned from Germany, with the statement that it had formed part of their diet, and that it contained sawdust.

The bulk of the sample consists of rye flour, but a number of small yellowish particles embedded in the crust show the structure of finely pulverised wood.

The following figures were obtained on analysis :

		Per Cent.
Moisture		13·7
Ash soluble in water	1·0	
Ash insoluble in water	1·1	
Total ash	—	2·1
Reducing sugar, as glucose		2·4
Crude fibre		4·9
Cold water extract		14·4
Residue (dry) left after salivary digestion		18·3

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ZEISS BUTYRO-REFRACTOMETER: THE CONVERSION OF SCALE READINGS TO REFRACTIVE INDICES.

In a paper before the Society (Analyst, 1916, **41**, 376) C. C. Roberts gave the following formula:

$$1{,}000[n]_D = 1{,}422 + 0{\cdot}817x - 0{\cdot}00142x^2$$

While this formula is satisfactory for the calculation of a refractive index from the scale reading, it is inconvenient for the converse operation of calculating a scale reading from a refractive index.

The following formula calculated from the above is more convenient:

$$\text{Scale reading} = 287{\cdot}3 - \sqrt{97{,}996 - 703{,}235([n]_D - 1{\cdot}4)}.$$

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Composition of Canadian Bran and Shorts. F. T. Shutt and R. R. Dorrance. (*Trans. Roy. Soc.*, Canada, 1918, **12**, 27-29.)—In the manufacture of Canadian "Government Standard Flour" since April, 1918, 196 pounds of flour must be milled from 258 pounds of spring wheat, whereas prior to this date this weight of flour was obtained from about 270 pounds of wheat. This rise in the percentage of extraction, as would be expected, is reflected in the chemical composition of the bran and shorts, and analyses are recorded comparing flours of 1903 and 1917 with present-day samples, which go to show that the 1918 bran is approximately 0·75 per cent. richer in protein, 0·5 per cent. richer in fat, and contains 1·5 per cent. more fibre, while the 1918 regulation shorts are about 1·75 per cent. richer in protein, and contain 2·5 per cent. more fibre. No digestion experiments have been made with the 1918 products, but so far as cattle are concerned the differences will probably prove but slight. The limits of variation in the 1918 regulation bran and shorts are less than those found in previous years for this class of material.

The (Canadian) legal standards for bran and shorts are as follows:

	Bran, Per Cent.	Shorts, Per Cent.
Protein not less than	14	15
Fat not less than	3	4
Fibre not more than	10	8

(*C.f.* Analyst, 1918, **43**, 53.) H. F. E. H.

Estimation of Morphine in Complex Products. A. Tingle. (*Amer. J. Pharm.*, 1918, **90**, 788.)—The author's object is to estimate morphine, not only in simple powders or tablets, but in pills of complex composition. Much material for examination has been of Asiatic origin (pills, etc.), and could not be dealt with by simple extraction methods such as that of Williams (*Amer. J. Pharm.*, 1914, **86**, 308-312). The difficulty of getting a filterable solution in the case of pills cannot be overcome when alcohol, acids, lime, or lead acetate are employed; but in barium hydroxide the author has found an effective reagent which facilitates filtration and completely dissolves any morphine, no matter in what form it is present. The subsequent separation of morphine in a pure form suitable for titration can then be affected by Williams's method (*loc. cit.*). Errors inseparable from the precipitation of morphine by ammonium hydroxide in presence of alcohol render such a procedure undesirable. About 6 grms. of the finely ground sample in a 100 c.c. flask are mixed with about 2 grms. of calcium carbonate, and 20 c.c. of water are then added and warmed till the formation of a uniform thin paste; the mixture is then cooled and 60 c.c. of cold saturated solution of barium hydrate are added, well mixed, and allowed to stand for half an hour. The whole is then diluted to 100 c..c, shaken, and filtered.' The residue on the filter is acidified with hydrochloric acid, warmed nearly to boiling, and filtered, the filtrate being all collected and concentrated to about 15 c.c. If, on being tested with ferric chloride, meconic acid is found, then any morphine found in the pills was there as opium, and the method about to be described cannot be employed. In the absence of meconic acid, 50 c.c. of the original filtrate are freed from barium with sulphuric acid (diluted 1 : 5), the solution being left faintly acid to litmus, diluted to 55, and mixed, allowed to settle, and filtered. The filtrate is then in a suitable condition for further treatment by the titration method already referred to, and the presence of morphine can conveniently be confirmed in the titrated liquid. Should the original pills contain soap, sulphates, or substances that react with barium hydroxide, special measures must be taken. Most of the materials which render filtration of aqueous solutions difficult are amenable to barium hydroxide, which is very helpful when such substances as starch, gum tragacanth, or pure acacia are present.

Many test samples of known composition are analysed and described with special details as to working processes. H. F. E. H.

Opium Analysis. D. B. Dott. (*Pharm. J.*, 1918, **101**, 318.)—The author criticises and discusses the paper by Annett and Singh (ANALYST, 1918, **43**, 205), in which these writers contend that the B.P. method of morphine estimation in Indian opium gives low results, mainly owing to the presence of codeine exerting a solvent action on the morphine and preventing its precipitation by ammonia from a solution of the lime compound. The author contends that it is hardly correct to speak of precipitation by ammonia, the morphine being really precipitated, because the chlorine of the ammonium chloride combines with the calcium, and the morphinate of lime, being decomposed, causes the precipitation of the morphine in the saline solution, in which it is very slightly soluble. Whatever may be the solvent action of codeine on morphine in aqueous solution, assay conditions are different, in that sufficient ether is present to hold all the codeine in solution. Annett and Singh's procedure of shaking the lime solution with toluene before treating with ether and ammonium chloride was tried, using benzene in place of toluene, with the resulting production of a frothy emulsion which makes extraction troublesome. This emulsion probably contains a small quantity of a basic lime compound which is readily separated by filtration, but is greater in quantity and more impure than the trace which always forms when using the B.P. process, where benzene is not employed. On comparing the two precipitates (the B.P. and the benzene treated), the latter, although heavier, shows on ultimate titration no more, and it may be slightly less, than the former, and it is concluded that there is no sufficient reason for altering the process in the direction suggested by Annett and Singh. H. F. E. H.

Effect of Heating Opium on its Morphine Content. H. E. Annett and H. Singh. (*J. Soc. Chem. Ind.*, 1918, **37**, 315-316T.)—When opium is heated in a water-oven at 97° to 98°C., the morphine content decreases gradually. The loss, however, does not become apparent until after four days' heating, but from this time up to 264 hours there is a steady loss of morphine. For instance, a sample containing 6·78 per cent. of morphine showed only 4·26 per cent. after being heated for 264 hours. The loss of morphine does not coincide with the loss of volatile constituents, since no further loss in weight takes place after the seventh day. The physical character of the heated opium is very different from that of opium which has been dried *in vacuo;* the heated opium absorbs moisture very slowly, whilst opium dried *in vacuo* is deliquescent. W. P. S.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

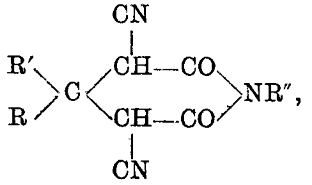
Detection of Small Quantities of Arsenic. O. Billeter. (*Helv. Chim. Acta*, 1918, **1**, 475-498.)—For the detection of small quantities of arsenic in toxicology, it is recommended that the substance be treated with nitric and sulphuric acids to destroy organic matter, the sulphuric acid solution then distilled with the addition of sodium chloride and potassium bromide, the distillate evaporated with the addition of hypochlorous acid, and the residual solution then tested in the Marsh apparatus. The distillation part of the process is essential to insure removal of heavy metals, particularly mercury, traces of which completely mask the presence of relatively large quantities of arsenic in the Marsh apparatus. The addition of hypochlorous acid during concentration of the hydrochloric acid solution prevents loss of arsenic. W. P. S.

ORGANIC ANALYSIS.

Estimation of Aldoses by Iodine in Alkaline Solution. H. Colin and O. Liévin. (*Bull. Soc. Chim.*, 1918, [iv], **23**, 403-405.)—The method depends on the fact that, under certain conditions, dextrose is oxidised quantitatively to gluconic acid by iodine in alkaline solution. The alkalinity must be sufficiently faint to eliminate risk of oxidising the alcohol groups. The author uses a solution containing 35 grms. of sodium phosphate and 50 c.c. of $\frac{N}{1}$ sodium carbonate in a litre. To the solution to be analysed at least three times as much $\frac{N}{10}$ iodine are added as will suffice to oxidise the dextrose believed to be present, and then a volume of the alkali solution about double that of the $\frac{N}{10}$ iodine. After an hour the mixture is made faintly acid with sulphuric acid, and the excess of iodine titrated with thiosulphate. Proteins, tannin, and colouring matters which may react with iodine must first be removed. Usually, careful treatment with the least possible excess of lead acetate will suffice to remove the interfering substances occurring in the inulin-bearing roots, tubers, and the like, for the analysis of which the method was worked out. G. C. J.

Analysis of Percussion Cap Compositions. Marqueyrol and P. Loriette. (*Bull. Soc. Chim.*, 1918, [iv], **23**, 401-403.)—The following routine enables one to identify the constituents of a cap composition and to estimate them with sufficient exactness for most practical purposes. The composition is extracted with anhydrous ether, and the ethereal extract is dried at 60° C., weighed, and examined for the presence of trinitrotoluene, picric acid, tetryl, and other organic nitro-compounds which may enter into cap compositions. Fulminate of mercury and lead azide are nearly insoluble in ether; at its boiling-point, 100 c.c. ether dissolves less than 3 mgrms. The residue insoluble in ether is treated with a few drops of cold water, the aqueous extract is dried at 60° C., weighed, and examined for the presence of chlorates, nitrates, etc. The residue insoluble in ether or water is treated for two hours with 10 c.c. of 5 per cent. alkali cyanide solution. The cyanide solution which has dissolved any fulminate of mercury is electrolysed, and the weight of fulminate calculated from the weight of mercury deposited. The residue insoluble in cyanide solution is distilled with a few c.c. of water and 1 c.c. of acetic acid, and the distillate allowed to pass into a solution of silver nitrate. If lead azide was originally present, silver azide will be precipitated. This is washed, dried, weighed, and identified by its behaviour when subjected to shock or brought in contact with a flame. G. C. J.

General Reaction of Ketones. J. Guareschi. (*Gazz. Chim. Ital.*, 1918, **48**, 83-98.)—Very many ketones react more or less rapidly with cyanacetic ether in the presence of ammonia or amines to form condensation products of the nature of cyclodicyanglutaric or piperidinic derivates of the general formula:



where R and R′ represent CH_3, C_2H_5, $CH_2C_6H_5$, etc., and R″ represents H, CH_3, etc. Aldehydes also react with cyanacetic ether to form cyclodicyanglutaconic derivatives. The action is more generally applicable than the bisulphite reaction, which is not given by certain ketones. The test is applied by mixing the ketone with acetic ether in the proportion of one molecule to two, and adding 11·5 per cent. alcoholic ammonia. The crystalline mass which gradually forms is left for twenty-four to thirty-six hours, then diluted with water, extracted with ether to remove the ketone which has not reacted, and the clear liquid treated with hydrochloric acid. The crystalline precipitate of the ammonium salt is recrystallised from boiling alcohol to obtain a product of constant melting-point. The phenylacetone derivative melts at

255° to 257° C., and the corresponding compound from ethylbenzylketone at 222° to 226° C. C. A. M.

Examination of Organic Photographic Developing Agents. H. T. Clarke. (*J. Ind. and Eng. Chem.*, 1918, **10**, 891-895.)—The more common developing agents may be classified into the following groups, in accordance with their behaviour on treating 0·1 grm. of the sample with 5 c.c. of different solvents: I. *Insoluble in Cold Water:* *p*-Hydroxylphenylglycine. II. *Soluble in Ether:* Hydroquinone, chlorhydroquinone, catechol, pyrogallol. III. *Soluble in Alcohol:* *p*-Aminophenol base, *p*-aminophenol hydrochloride, 5-amino-2-cresol hydrochloride, 2-4-diaminophenol hydrochloride, *p*-dimethylaminophenol oxalate. III*a*. *Insoluble in Alcohol*; *p*-Aminophenol sulphate, 5-amino-2-cresol sulphate, *p*-dimethylaminophenol sulphate, *o*-methylaminophenol sulphate, *p*-phenylenediamine hydrochloride. (*a*) *Groups II., III., and IIIa.*, are decidedly acid to litmus when dissolved. The aqueous solutions should be tested for chlorides, sulphates, etc. (*b*) One or two drops of 10 per cent. sodium carbonate solution are added to 0·1 grm. of the sample in 1 c.c. of hot water, and the mixture allowed to stand for a few minutes. *Group I.:* *p*-Hydroxyphenylglycine dissolves with effervescence. *Group II.:* Hydroquinone, chlorohydroquinone, and catechol do not cause effervescence; the solution darkens gradually. Pyrogallol, no effervescence; solution darkens rapidly. *Groups III. and IIIa.* all cause effervescence, except pure *p*-aminophenol base. The following form crystalline precipitates on cooling: Salts of *p*-aminophenol, 5-amino-2-cresol, and *o*-methylaminophenol. No precipitates are formed by the salts of the other developers mentioned. (*c*) A few drops of 10 per cent. ferric chloride solution are added to 0·1 grm. of the sample in 2 c.c. of water. *Group I.:* No colour reaction is given by *p*-hydroxyphenylglycine, odour of quinone on boiling. *Group II.:* Hydroquinone gives dark green precipitate of quinhydrone, strong quinone odour on boiling. Chlorohydroquinone gives red-brown coloration; odour on boiling resembles quinone. Catechol gives a green coloration with one drop of ferric chloride solution, and a nearly black precipitate with an excess; whilst pyrogallol gives an intense red-brown coloration. *Groups III. and IIIa.:* Salts of *p*-aminophenol and 5-amino-2-cresol give purple colorations not destroyed on boiling; odour of quinones. *p*-Methylaminophenol sulphate behaves in a similar way, but the colour is developed more slowly. Salts of *p*-dimethylaminophenol give no coloration in the cold; on boiling, the solution darkens, and emits odour of quinone. With 2-4-diaminophenol hydrochloride, an intense red coloration is produced; no odour on boiling. A dark purple coloration, changing to red-brown on standing, is produced with *o*-methylaminophenol sulphate; no odour on boiling. *p*-Phenylenediamine hydrochloride gives a deep green colour, changing rapidly to purple, and, on boiling, to dull reddish-brown; odour of quinone on boiling. (*d*) Two c.c. of 5 per cent. silver nitrate solution are added to 0·1 grm. of the sample in 1 c.c. of water. *Group I.*. *p*-Hydroxylphenylglycine in suspension yields a black deposit, changing to light brown on boiling, whilst the liquid becomes purple. *Group II.:* Hydroquinone gives a silky white precipitate; odour of quinone on boiling. Chlorhydroquinone hardly reduces silver nitrate in the cold, but does so rapidly on boiling. Catechol causes slow reduction in the cold, whilst pyrogallol reduces the reagent instantly. *Groups III. and*

Ma. : Salts of *p*-aminophenol, *p*-methylaminophenol, and 5-amino-2-cresol, give purple colorations and quinone-like odours on boiling. Salts of *p*-dimethylaminophenol give no coloration in the cold, but a brown-red colour on boiling; odour of quinone. With 2·4-diaminophenol hydrochloride an intense red coloration is produced; no odour on boiling. *o*-Methylaminophenol sulphate gives a yellowish-brown colour, changing to red-brown on heating, whilst *p*-phenylenediamine gives a pale green colour, changing immediately to deep purple; no odour or change of colour on boiling. Further tests of identity include the formation of the acetyl and benzoyl derivatives, which in many cases have distinctive melting-points. *Quantitative Examination.*—The solubility, ash, and amounts of chloride, sulphate, sulphite, and total nitrogen, should be estimated. For estimating the amount of salts of *p*-aminophenol or *p*-aminocresol, 10 grms. of the sample are dissolved in about 150 c.c. of water (or dilute hydrochloric acid in the case of a free base). A slight excess of sodium acetate is added, followed by about 10 c.c. of benzaldehyde (or less when a relatively small amount of aminophenol is present). After standing twelve hours, the liquid is filtered with the aid of suction, and the solid washed with water, dried at 100° C., and weighed. The following factors are used for calculating the results: As *p*-aminophenol hydrochloride, 0·738; *p*-aminophenol sulphate, 0·802; as *p*-aminophenol base, 0·554; as 5-amino-2-cresol hydrochloride, 0·756; as 5-amino-2-cresol sulphate, 0·815; as 5-amino-2-cresol base, 0·584. In some cases it is necessary to estimate the amount of alkali carbonate by difference, direct estimation being impracticable. Adulterants which have been found in photographic developers include starch, sugar, citric acid, sodium formate, potassium ferrocyanide, boric acid, various salts of sodium and potassium, lead chloride and sulphate, ammonium chloride and sulphate, sodium hydroxide, and magnesium sulphate. C. A. M.

Estimation of Phenol and the Three Isomeric Cresols in Mixtures of these Substances. H. M. Dawson and C. A. Mountford. (*J. Chem. Soc.*, 1918, **113**, 935-944.)—It is shown that freezing-point determinations may be used for the complete analysis of the technically important mixtures: (*a*) Phenol and *o*-cresol; (*b*) *m*-cresol and *p*-cresol; (*c*) *o*-cresol, *m*-cresol, and *p*-cresol; (*d*) phenol, *o*-cresol, *m*-cresol, and *p*-cresol. In the case of the quartenary mixture, a preliminary fractionation is necessary. The quarternary mixture is mixed with about one-half its weight of *o*-cresol, and this mixture is then submitted to slow fractional distillation, using a still-head of the Raschig type. Two fractions are thus obtained, one of which, *A*, consists of a mixture of phenol and *o*-cresol, and the other, *B*, of a mixture of *o*-cresol, *m*-cresol, and *p*-cresol. These fractions are then analysed as follows: *Fraction A.*—If the mixture contains phenol or *o*-cresol in considerable excess (sp. gr. at 25°/4°C. is greater than 1·061 or less than 1·050), the composition may be ascertained directly from the freezing-point of the mixture, the phenol branch of the following table being used for mixtures of higher sp. gr., and the *o*-cresol branch for those falling within the lower range:

Weight per cent. phenol ...	100	95	90	85	80	75	70
Freezing-point (degrees C.)	40.5	37·9	35·2	32·4	29·5	26·5	23.4

Weight per cent. *o*-cresol ...	100	95	90	85	80	75	70
Freezing-point (degrees C.)	30·45	29·0	27·6	26·25	24.95	23.7	22·6

If the sp. gr. lies between the above limits, the mixture *A* should be mixed with a definite quantity of pure phenol (m.p. 40·5° C.) so as to raise the sp. gr. of the resulting mixture, *Y*, above 1·061; the freezing-point of *Y* is then determined. Assuming that *a* parts of *A* are mixed with 100 – *a* parts of phenol, and that this mixture, *Y*, contains, according to the freezing-point determination, *y* per cent. of phenol, then the percentage, *x*, of phenol in *A* is given by the equation: $x=(y-100+a)100/a$. *Fraction B.*—The method depends on the fact that equal weights of *o*-cresol and *m*-cresol depress the freezing-point of *p*-cresol to the same extent, and on the further fact that equal weights of *m*-cresol and *p*-cresol depress the freezing-point of *o*-cresol to nearly the same extent. A portion of the fraction *B* is mixed with a known quantity of pure *p*-cresol, giving a mixture, *Y*, the freezing-point of which is determined. From this, the percentage of *p*-cresol in *Y*, and hence in *B*, can be obtained from the figures given in the following table :

Weight per cent. *p*-cresol ...	100	95	90	85	80	75	70	65
Freezing-point (degrees C.)	34.15	30·7	27·2	23·6	20·0	16·3	12·3	7·8

Similarly, another portion of *B* is mixed with a known proportion of pure *o*-cresol, giving a mixture *Z*, the freezing-point of which is also taken. From this, the percentage of *o*-cresol in *Z*, and therefore in *B*, can be derived from the freezing-points recorded in the table below :

Weight per cent. *o*-cresol ...	100	95	90	85	80	75	70	65
Freezing-point (degrees C.)	30·45	28·05	25.6	23·1	20·4	17·5	14·4	11·0

W. P. S.

Quantitative Estimation of Acetone in Smokeless Powder. A. Pieroni.

(*Atti R. Accad. Lincei*, 1918, **27**, II., 52-57; through *J. Soc. Chem. Ind.*, 1918, **37**, 749A.)—The discordant results obtained in the estimation of acetone are due to the difficulty of converting acetone quantitatively into iodoform, to the ease with which the latter is attacked by excess of alkali, to the loss of iodine owing to secondary reactions, and of the volatility of iodoform. The following procedure gives satisfactory results with smokeless powders, which contain between 3 and 0·25 per cent. of acetone. From 35 to 50 grms. of the powder in lumps are weighed to the nearest 0·01 grm. and ground in a special apparatus in which the powder is kept washed by a stream of distilled water, so as to prevent evaporation of the solvent. The ground mass, together with the water, is either collected in a large beaker, or passed directly through a funnel into a distillation flask of about 2 litres capacity; 100 c.c. of dilute sulphuric acid (1 : 1) are then added to prevent emulsification of the distillate. Steam is passed into the flask, and the latter also heated with a burner. The distillate passes from the condenser into a bulbed adapter, reaching almost to the bottom of a test-tube which is full of water, and is situate in a conical flask. From the latter unabsorbed gases pass through a washer which retains any traces of acetone still unabsorbed. When 250 to 300 c.c. of distillate—*i.e.*, rather more than one half of the volume of liquid originally in the distilling flask—have been collected, the adapter is removed and a little fresh distillate collected in a test-tube and tested for acetone by means of potash and iodine. When acetone ceases to distil over, the whole of the liquid in the collecting flasks, together with the washings of these and of the adapter, is made up to 500 c.c. in a measuring flask. Exactly 100 c.c. of the mixed solution is pipetted into a cylinder with a ground stopper of about 300 c.c. capacity. From two burettes are then run in a solution containing 112 grms. of potassium hydroxide per litre, and another containing 257 grms. of iodine and 330 grms. of potassium iodide per litre; the two solutions should drop at the same time, and at the rate of about 20 drops per minute, the cylinder being kept shaken meanwhile. From time to time the addition is stopped for a time until the iodoform settles and the supernatant yellow liquid clarifies; if further addition of the potash and iodine solutions produces no turbidity the transformation of the acetone is complete. After a rest of a few minutes, such excess of sodium chloride is added that 10 grms. or so remain undissolved, 50 c.c. of ether kept over sodium being then added, the cylinder tightly closed, and the liquid shaken energetically three or four times. As soon as the two layers have separated completely, 25 c.c. of the ethereal iodoform solution are pipetted into 25 c.c. of saturated alcoholic caustic potash solution. The volume of alcoholic potash to be used varies with the volume of iodine used in the reaction with the acetone, the former being, in general, double the latter. The liquid is diluted with alcohol until clear, heated in a water-bath to evaporate the ether, and boiled for fifteen minutes; the alcohol is then distilled off, the liquid diluted with water, cooled, and acidified with dilute nitric acid, and the potassium iodide formed is determined volumetrically by Volhard's method. If a be the number of c.c. of $\frac{N}{10}$ silver nitrate solution used, and b the weight in grms. of the powder taken, the percentage of acetone in the latter will be $2a/b$.

Volumetric Estimation of Reducing Sugars. W. B. Clark. (*J. Amer. Chem. Soc.*, 1918, **40**, 1759-1772.)—The method is a development of that of Scales (*J. Biol. Chem.*, 1915, **23**, 81), in that it depends on the reduction of a copper solution, the solution of the cuprous oxide without removal from its mother liquor by the addition of hydrochloric acid, and treatment of the resulting cuprous chloride solution with excess of standard iodine solution, the excess of which is determined by titration with thiosulphate. Unlike Scales, however, who used Fehling's solution, the author uses a modified Benedict solution (ANALYST, 1907, **32**, 367; 1911, **36**, 298), and carries out all the operations in one vessel.

The copper solution contains 16 grms. crystallised copper sulphate, 150 grms. sodium citrate, 130 grms. anhydrous sodium carbonate, and 10 grms. sodium hydrogen carbonate per litre; the function of the last constituent is to insure a non-oxidising atmosphere in the reduction flask. Reduction is carried out in a 200 or 250 c.c. conical flask carrying a two-hole rubber stopper, through one hole of which a thistle funnel passes. This thistle funnel is drawn out so that 50 c.c. of water will flow from it in twenty-five to thirty seconds, and is cut off so that with 80 c.c. of liquid in the flask, and the tip of the funnel just below the surface, the body of the funnel will come within one or two centimetres of the stopper.

Any analyst can standardise the method under conditions to suit his work, but the author makes up his test solutions so that they will probably not contain more than 30 mgrms. reducing sugar in 10 c.c., and takes 10 c.c. of this solution and 20 c.c.

of the copper solution for the test. The mixture is heated under standard conditions, which are best realised with an electric hot-plate. The author prefers the heating to be such that the mixture begins to boil in three or four minutes, and he maintains it in ebullition for precisely two minutes. Meanwhile, 60 c.c. distilled water have been measured into a cylinder and 4·7 c.c. hydrochloric acid (sp. gr. 1·19) into another. Just enough acid is added to neutralise the alkaline carbonate, to dissolve the cuprous oxide, and render the resulting solution distinctly acid to litmus. When the reduction mixture has boiled for two minutes, the flask is removed from the source of heat, and a few drops of the hydrochloric acid are added. The mixture is then cooled, and the rest of the acid added slowly. When the cuprous oxide has dissolved, about half the 60 c.c. of water is added. The tip of the funnel should now be below the surface of the liquid. An accurately measured quantity of iodine solution, equivalent to 25 c.c. of $\frac{N}{25}$ solution, is next added through the funnel, which is finally rinsed with the rest of the 60 c.c. of water. The stopper is now removed, and the excess of iodine titrated with $\frac{N}{25}$ thiosulphate, using starch towards the end of the titration. The difference between the volume of thiosulphate used and the volume required in a blank test, in which water instead of reducing sugar solution is boiled with the copper solution, gives the thiosulphate equivalent to the iodine consumed in oxidising the copper reduced by the sugar. The thiosulphate solution may be standardised by carrying out an exactly similar test in which a known quantity of invert sugar is used. From this one test, if made with about 20 mgrms. of invert sugar, a factor may be calculated which is very nearly a constant for quantities of invert sugar from 14 to 29 mgrms. of invert sugar—that is to say, for readings in the final titration with thiosulphate ranging from about 1 to 13 c.c. For smaller quantities of reducing sugars, the thiosulphate factor varies appreciably; tables and a curve in the paper show this, and a similar table or curve could be constructed by any analyst to serve for the estimation of very small quantities under his particular conditions. Stress is laid on the importance of each analyst standardising the method for himself. Even the substitution of a new flask may materially affect the results if the flask be thinner or thicker than the last one, as this affects the time required to bring about boiling.

It is stated that cane sugar has no measurable effect unless present in great excess as compared with the reducing sugar to be determined. With a cane-sugar invert ratio of 4 : 1 this error is negligible ; when it becomes 10 : 1, the error is 1·5 per cent. No experiment is recorded where the ratio was 50 : 1 or 90 : 1 : a common circumstance in practice. G. C. J.

Analysis of Distilled Tars and Pitches. J. M. Weiss. (*J. Ind. and Eng. Chem.*, 1918, **10**, 817-823.)—Methods for the estimation of water in distilled tar, for the estimation of matters insoluble in benzene, and for the determination of sp. gr. and viscosity, are indicated by reference to other publications; whilst two methods for the determination of consistency—by Schutte's penetrometer and by the float tester—are described in detail with illustrations. The distillation of distilled tar under standard conditions is also described in great detail.

The pitch tests described in detail include a method for the determination of sp. gr., a method for the estimation of free carbon, the determination of the "water melting-point" and "air melting-point," loss of weight by evaporation under standard conditions (seven hours at 163° C. ± 2°), and a method for the determination of "slide" or elongation of a half-inch cube resting on an inclined plane at 40° C. for seven hours. G. C. J.

Methods of Analysis used in the Coal-Tar Industry. III. Heavy and Middle Oils. J. M. Weiss. (*J. Ind. and Eng. Chem.*, 1918, **10**, 911-916.)—Heavy Oils—*Water.*—Two hundred c.c. of the oil are distilled in a copper still of the dimensions previously given (Analyst, 1918, **43**, 419) until the temperature of the vapour reaches 205° C. The volume of water is measured, and any light oil is returned to the residue in the still. *Sp. Gr.*—This is determined, by means of a calibrated hydrometer, at 38° C., a correction of 0·00075 being added to the observed reading for each 1° C. above that temperature when the oil has required heating. The sp. gr. is also determined by means of the Westphal balance at 15·5° C. *Substances Insoluble in Benzene* are estimated on at least 10 grms. of the material by the method previously described (*loc. cit.*), the results being accurate within 0·2 per cent. *Retort Distillation.*—The bulb of the retort has a capacity of 250 to 290 c.c., the length of the stem is 25 to 30 cms., and the outlet about 1·25 cms. in diameter. The condenser tube is 360 mm. long (±4 mm.), and has a diameter of 12·5 mm. (±1·5 mm.) at the small end, and of 28·5 mm. (±3·0 mm.) at the large end. During the distillation the retort is protected from air currents by means of an asbestos screen. The thermometer is passed through a cork in the tubulure, and the bottom of its bulb is fixed ½ inch above the surface of the oil. In the distillation, 100 grms. of the oil are distilled at the rate of at least one drop, and not more than two drops, per second, and the distillate is collected in weighed receivers. Fractions are collected at 210°, 235°, 270°, 315,° and 355° C. Any water present is separated and separately reported. The sp. gr. of the fractions is determined, and the "float test" adopted by the Amer. Wood. Pres. Assoc. (*A.S.T.M. Proceedings*, 1917, Part I., 826) is applied to the residue. *Coke.*—Hard glass bulbs with a capacity of about 15 c.c. are used. About 1 grm. of oil is introduced, the bulb weighed again, and the oil distilled off over a very low flame as fast as possible without frothing. Finally, the bulb is heated strongly in the Bunsen flame until the evolution of gas ceases and any carbon on the exterior of the outlet has been completely burned off. *Tar Acids (Contraction Method).*—One hundred c.c. of the oil are distilled until at least 95 per cent. of distillate has been obtained. The distillate is transferred to a special graduated separating funnel, which is immersed in hot water at 60° C. until no further change in the volume of the liquid takes place. It is then extracted with successive portions of 50 c.c. each of 10 per cent. sodium hydroxide solution until no further reduction occurs. After settling at 60° C. the alkali is drawn off, and the volume of residual oil measured. The diminution shall be considered as tar acids, though the results are slightly too high. *Tar Acids (Liberation Method).*—The distillate is extracted with sodium hydroxide solution as described, and the separated carbolate is acidified with 40 per cent. sulphuric acid, care being taken to prevent heating. The liberated tar acids and sulphate solution are poured several times through a layer of naphtha in the tar-separating funnel. The sulphate layer is drawn off, and the increase in volume of the naphtha through the absorption of the tar acids is measured, and taken as dry tar acids present. The results are expressed in terms of the anhydrous oil. *Hempel Distillation.* — The Forest Service Hempel flask is used (*Forest Service Circular*, **112**, U.S. Dept. Agric.). The oil is distilled at the rate of one drop per second and fractions are collected in tared flasks at the following temperatures: Up to 170°, 170° to 205°, 205° to 225°, 225° to 235°, 235° to 245°, 245° to 255°, 255° to 285°, 285° to 295°, 295° to 305°, 305° to 320°, and, if possible, 320° to 360° C. The refractive index (Abbé-Zeiss) of each fraction at 60° C. is determined. *Sulphonation Residue.*—Ten grms. of the fraction of oil are weighed into a Babcock milk-bottle, and treated with four successive portions of 10 c.c. of 37N sulphuric acid (total $SO_3 = 80{\cdot}07$ per cent.), the bottle being shaken for two minutes after each addition. It is then kept for an hour at 98° to 100° C., being vigorously shaken every ten minutes, and is subsequently cooled, filled up to the graduation with ordinary sulphuric acid, and centrifuged for five minutes. The unsulphonated residue is then read. It should be a clear, transparent oil, any dark or gummy appearance indicating incomplete sulphonation. *Tar Bases.*—These are extracted in the same way as tar acids (*supra*), except that 20 per cent. sulphuric acid is used instead of the sodium hydroxide solution. *Dry Salts at* 4·5° *C.*—The residue of oil left after extraction of the tar acids is cooled in a copper beaker, with stirring, to 4·5° C., and kept at that temperature for fifteen minutes. The contents of the beaker are then quickly filtered through a Buchner funnel, and the solid cake repeatedly pressed between filter-paper in a letter press, until only a trace of oil is taken up, and weighed. The weight in grms. divided by the sp. gr. of the oil gives the amount of dry solids. The results are accurate within 1 per cent. *Limpid Point.*—Fifty c.c. of anhydrous oil are distilled to dryness, and 30 c.c. of the thoroughly mixed distillate transferred to a test-tube, 5 inches long and 1 inch diameter, which is cooled in a freezing mixture. During cooling the oil is stirred with a thermometer until a large quantity of crystals has separated. The tube is then removed and heated at the rate of 2° C. per minute, while its contents are constantly stirred until the crystals disappear. If no crystals separate when the oil is chilled to about 20° C. a very small amount of naphthalene is added, and if this does not cause any separation the report should be made, "no separation obtainable." MIDDLE OIL TESTS.—These are made in the same manner as the corresponding tests for heavy oils.

C. A. M.

INORGANIC ANALYSIS.

Arsenite Titrations of Permanganate Solutions. A. Bose. (*Chem. News*, 1918, **117**, 369.)—Study has been made of the anomalous results obtained when solutions of permanganic acid containing free nitric acid are titrated with sodium arsenite (ANALYST, 1918, **43**, 234). The free nitric acid is shown not to be responsible for the high reducing value (33 per cent. in excess of its true value) of the arsenite solution, nor is the formation of manganic compounds, as suggested by Ibbotson (*loc. cit.*), proved to be a valid explanation. Finally, the "definite compound" proposed by Ibbotson is shown to have no existence either in the arsenite solution or in the permanganate. No final explanation of the anomaly is, so far, forthcoming, and it is possible that some complicated reactions take place during the titration, as, up to that point, it is clear, from experiments made with ferrous ammonium sulphate titration, that all the manganese is in the permanganic condition, while nitric acid as the disturbing factor has also been ruled out.

H. F. E. H.

Retention of Carbon Dioxide by Erbium Oxide at High Temperatures. E. Wickers, B. S. Hopkins, and C. W. Balke. (*J. Amer. Chem. Soc.*, 1918, **40**, 1615-1619)—In the course of a research on the separation of yttrium from erbium, the authors found that erbium oxalate was not completely converted into oxide even by heating to 885° C. for four hours. The product still contained some carbonate. This observation brings into question the present accepted value for the atomic weight of erbium, and since the authors did not determine under what conditions the last traces of carbon dioxide could be expelled, analysts wishing to estimate erbium as oxide must satisfy themselves in every case that no carbonate is present.

G. C. J.

Quantitative Analysis of Small Quantities of Gases. H. M. Ryder. (*J. Amer. Chem. Soc.*, 1918, **40**, 1656-1662.)—Apparatus is described and illustrated which serves for the analysis of very small quantities of gas, from a few cubic millimetres to perhaps 1 c.c. It was designed for the analysis of the gases given off by metals when heated by the passage of an electric current through the metal specimen itself, a method which eliminates possible error due to heating the containing vessel, but which makes it necessary to use small quantities of metal, and subsequently to analyse a few cubic millimetres of gas. In general, the method provides for the separation and measurement of water vapour and carbon dioxide, using liquid air and solid carbon dioxide, the addition of oxygen or carbon monoxide in excess to the remaining gases, the combustion of this mixture, the separation and measurement of the products of combustion, and the adding of carbon monoxide or oxygen to the residue, and the repeating of the combustion. The measurement of the pressure in a system of known volume at different stages of the process provides data for calculating the composition of the original gas. Test analyses of gas mixtures of known composition (about equal proportions of oxygen, hydrogen, and carbon monoxide) show that any constituent can be estimated within about 3 per cent. on the sample (10 per cent. on the constituent) when the volume available for analysis is between 10 and 100 cubic millimetres.

G. C. J.

Iodimetric Estimation of Hypophosphites and Phosphites. Boyer and Bauzil. (*J. Pharm. Chim.*, 1918, **18**, 321 334.)—The method proposed is based on the fact that hypophosphorous acid is oxidised by iodine in acid solution to phosphorous acid; the latter is not further oxidised in acid solution, but if an alkali (sodium hydrogen carbonate) is added the oxidation proceeds a stage further to phosphoric acid. *Hypophosphites.*—Ten c.c. of the solution containing 0·1 grm. of the hypophosphite (hypophosphites which are insoluble in water may be dissolved in dilute sulphuric acid) are placed in a stoppered flask, and treated with 10 c.c. of 25 per cent. sulphuric acid and 30 c.c. of $\frac{N}{10}$ iodine solution; the mixture is kept in a dark place for ten hours, and the excess of iodine is then titrated with $\frac{N}{10}$ thiosulphate solution. Each c.c. of $\frac{N}{10}$ iodine is equivalent to 0·00328 grm. of hypophosphorous acid (H_3PO_2). *Phosphites.*—Ten c.c. of the solution, containing about 0·1 grm. of the salt, are treated with 10 c.c. of 5 per cent. sodium hydrogen carbonate solution and 20 c.c. of $\frac{N}{10}$ iodine solution; after two hours, 10 c.c. of 10 per cent. acetic acid are added, and the excess of iodine is titrated. Each c.c. of $\frac{N}{10}$ iodine is equivalent to 0·00407 grm. phosphorous acid. The method may be applied to mixtures of the two salts. W. P. S.

Oxidising Action of Potassium Dichromate. C. R. McCrosky. (*J. Amer Chem. Soc.*, 1918, **40**, 1662-1674.)—Wagner has stated (*Zeitsch. anorg. Chem.*, 1899, **19**, 427) that dichromate liberates about 0·3 per cent. more iodine from potassium iodide than it should do according to theory. When the solutions are freed from dissolved oxygen, normal results are obtained. Bruhns, on the other hand (*J. prakt Chem.*, 1917, **93**, 73, 312), found that solutions of dichromate behaved normally, and he found that the removal of dissolved oxygen by bubbling carbon dioxide through the solutions did not reduce the amount of iodine liberated. The author of the present paper reconciles these conflicting views by his discovery among a number of samples of potassium dichromate of one which behaves normally as stated by Bruhns, whereas his other samples behaved as did those of Wagner, even after he had recrystallised them four times. The important point is that, whereas pure dichromate does behave normally even in presence of dissolved oxygen, pure dichromate is difficult to procure or prepare, but normal results may be obtained with any recrystallised sample provided the solutions are freed from dissolved oxygen. The author has not succeeded in identifying the impurity, which appears to exercise a catalytic action, and which is so difficult to eliminate by recrystallisation. G. C. J.

Volumetric Determination of Selenious and Selenic Acids. L. Moser and W. Prinz (*Zeitsch. anal. Chem.*, 1918, **57**, 277-305; through *J. Soc. Chem. Ind.*, 1918, **37**, 730A.)—The most accurate results in the determination of selenious acid are obtained when the substance is distilled with hydrochloric acid and an excess of potassium iodide (four times the quantity required by theory), and the liberated iodine then titrated both in the receiver and in the residue. The indirect iodimetric method described by Gooch and Pierce (*Zeitsch. Chem.*, 1896, **11**, 249), in which the selenious acid is heated with a definite and known excess of potassium iodide in the presence of potassium hydrogen arsenic and sulphuric acid, and the arsenious acid formed then titrated with iodine solution after neutralisation, also gives accurate results if carried out as a distillation process. In the oxidation of selenious acid by permanganate in alkaline solution there is always some loss of oxygen when the hot mixture is acidified previous to the titration of the excess of permanganate, and the results obtained are, therefore, inaccurate. There is formation of hydrogen selenide when selenious acid is titrated with titanium trichloride solution, and this method is, consequently, unsatisfactory. The best method for the determination of selenic acid consists in reduction with hydriodic acid. The selenic acid solution (100 c.c.) is treated with 10 c.c. of concentrated hydrochloric acid and an excess of potassium iodide (5 grms. for each 0·1 grm. of selenium), the mixture distilled in a current of carbon dioxide, and the iodine titrated in the receiver and also in the residue.

Volumetric Estimations of Sulphates. R. Howden. (*Chem. News*, 1918, **117**, 484.)—The solution containing a soluble sulphate (heavy metals and calcium can be removed previously by means of sodium carbonate) is neutralised, using methyl-orange as indicator, a few drops of phenolphthaleïn solution and an excess of barium carbonate are added, and the mixture shaken. Barium sulphate is precipitated, and an equivalent quantity of sodium carbonate is formed. The solution is now titrated with $\frac{N}{10}$ hydrochloric acid until the red coloration disappears; this titration is necessary in order that the reaction may be complete. The mixture is then filtered, and the titration of the filtrate continued until it is neutral to methyl-orange. The method gives approximately correct results. W. P. S.

APPARATUS, ETC.

Calorimetric Lag. W. P. White. (*J. Amer. Chem. Soc.*, 1919, **40**, 1858-1872.)—The lag effects of bodies external to calorimeters are threefold: One, equivalent to a change in the heat capacity of the calorimeter; this can be eliminated if a calorimeter is directly calibrated. Another, much smaller, depends on the amount of thermal leakage; this can be avoided, if necessary, by using the adiabatic method. And a third, dependent on the jacket temperature, which disappears for constant jacket temperature. This one causes the calorimeter to have a different heat capacity when used adiabatically. Of two errors peculiar to the aneroid calorimeter, one comes from improper distribution of the thermojunctions (or equivalent devices) which measure the surface temperatures. This error becomes zero if the surface temperature distribution does not change from one experiment to another. The other special source of error is inconstancy of final temperature, due to inconstancy of final jacket temperature. On account of lag effect, the effective heat capacity of a shield midway between calorimeter and jacket is only one-fourth the actual capacity, and, with due regard to the possibility of change, such shields may often, with great advantage, be used to reduce the thermal leakage. Used as a cover, such a shield has a specially small error, and offers an easy method of dealing with evaporation. Ordinary, non-metallic, covers, on account both of their lag and their heat capacity, are undesirable. Although a normal lag effect characterises even thick and heterogeneous packings round a calorimeter, the loss of time due to the lag is a sufficient objection to the practical use of anything which is not either very small or in very close thermal connection with calorimeter or jacket.

G. C. J.

Conditions of Calorimetric Precision. W. P. White. (*J. Amer. Chem. Soc.*, 1918, **40**, 1872-1886).—In a calibrated calorimeter practically all the errors come in temperature measurement, and the most, though often not the greatest of these, come in the cooling correction—that is, the determination of the effect of the thermal leakage between calorimeter and environment. This leakage effect is equal to KQ_xT, where T is time, Q_x is the thermal head (difference between calorimeter and environment temperatures) for the experimental period, and K the thermal leakiness or leakage modulus of the calorimeter. If any of these three quantities is diminished, its own errors are usually little changed, but the errors of the others now have a smaller multiplier. Thus, by diminishing K (as by means of a vacuum), one diminishes the effect of errors in Q which lie either in getting the environing (jacket) temperature sufficiently uniform or in getting the calorimeter temperature uniform without too great an error from heat of stirring. This latter difficulty grows in importance as measurements become more delicate. Usually errors in Q may easily be made negligible, and great diminution of K is then of secondary importance.

A diminution of Q, as by the adiabatic method, diminishes the effect of errors in K, such as variations from Newton's law. But owing to the peculiarities of the computation, it does not much affect the main error connected with K, that in determining K by means of the cooling rate, unless methods can be used which presuppose an accurate knowledge of the heat of stirring. For this and other reasons the diminution of Q, although the commonest of operative devices, is in practice largely illusory as far as concerns the end chiefly sought. The determination of K generally results in about doubling the accidental thermometric error, but this is usually the largest thermal leakage error in well-planned work. This error can be diminished by devices, some of which are adapted to commercial work and which save time as well as error.

Lags have a law of their own ; they usually vary with K, are independent of T and Q, and can usually be made to cause little or no error.

Stirring introduces a possible error from irregular heat production, due to irregular speed. Since the heat varies as the cube of the speed, the error tends to vary as the cube of the efficiency of stirring, and thus may become very important in delicate measurements. A remedy can usually be found by controlling the speed or, more effectively, by diminishing K. The type of stirrer and the design of the calorimeter with reference to stirring may be very important. The paper also deals with the error due to evaportion and that from heat flow along wires and rods. G. C. J.

Calorimetric Methods and Devices. W. P. White. (*J. Amer. Chem. Soc.*, 1918, **40**, 1887-1898.)—In this paper the general rules for calorimetric precision, given in the paper which forms the subject of the preceding abstract, are applied to certain structures—namely, jacket covers and stirrers—and to the following special devices—namely, vacuum-jacketed vessels, the adiabatic method, aneroid or dry calorimeters, double or differential calorimeters, and measured-shield calorimeters.

It is shown that vacuum-jacketed vessels are mainly useful either in relatively crude or in very exacting work. They are convenient and simple in crude work, but cause difficulty and complication if the work demands more elaborate arrangements. The adiabatic method does not diminish either of the two main sources of thermal leakage error, but has many other advantages. The Pfaundler twin calorimeter method enables high precision to be obtained with relatively simple apparatus, but is most successful when applied to liquids and to the comparing of two things that are nearly alike. Aneroid calorimeters work quicker and, within limits, also more accurately, the smaller they are. The measured-shield method, where a connection shield supplied with thermo-couples is used, greatly diminishes the difficulties of thermal head measurement and stirring, which are among the greatest in work of high precision, and is more convenient than glass vessels. G. C. J.

Effects of Heat on Chemical Glassware. R. G. Sherwood. (*J. Amer. Chem. Soc.*, 1918, **40,** 1645-1653.)—When a glass vessel, designed for vacuum purposes, is being highly exhausted, it is common practice simultaneously to heat it to 200° to 400° C. This heating assists exhaustion by accelerating the removal of adsorbed gases from the inner surface of the vessel. As the result of a series of experiments described in this paper, the author concludes that the comparatively large quantities of these gaseous products obtained on heating the walls of an exhausted vessel cannot be ascribed entirely to surface adsorption. When glass is heated, there is evidence of two distinct kinds of gaseous evolution—that resulting from adsorbed products, which are readily removable below 300° C., and that resulting, in all probability, from a chemical decomposition of the glass itself. The latter effect becomes more important above 400° C. There is some evidence of a definite, characteristic rate of gaseous evolution for each temperature to which the glass is subjected, increasing with the temperature, and extending over a considerable period, one sample of hard glass showing a small continuous evolution even after twenty hours heating at 500° C. Adsorption products are confined to quantities of the order of a layer of gas one molecule deep. The most important of the products obtained at higher temperatures is water, which, at the highest temperatures tried, constitutes almost the entire quantity of the evolution. G. C. J.

Gas Bubbler for Gas Analysis. O. D. Burke. (*Chem. News*, 1918, **117**, 368.)—When sulphur dioxide in gaseous mixtures is estimated by bubbling the gas through $\frac{N}{10}$ or $\frac{N}{100}$ iodine solution, errors up to 1 per cent. may arise through rushing the gas too fast and in too large bubbles. To obviate this, the passage of the gas into the iodine solution is effected through a narrow glass tube drawn out to a very fine capillary, which is ground level at the point. Another limb of glass rod is fused on to the side of the straight limb, and extends downwards parallel to the gas tube to the level of the orifice, where it is turned under just beneath the capillary opening, which it touches, the point of contact being flattened out slightly and ground level, thus fitting tightly to the small opening. In this way the gas on issuing from the minute hole is broken up into extremely fine bubbles, and complete absorption is insured, together with a uniform rate of flow. The general design resembles the letter **b**. H. F. E. H.

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GOVERNMENT REPORT.

Report of the Government Chemist upon the Work of the Government Laboratory for the Year ending March 31, 1918. Cd. 9205. **1918.** Price 2d.—The number of samples examined in the course of the year was 200,453, as compared with 258,456 in the previous year, exclusive of those dealt with at the chemical stations. This diminution in number is principally in customs and excise samples, those taken on importation having been reduced by more than one-third of the number dealt with in the previous year. The restrictions affecting the home wine and spirit trades similarly caused a reduction in the number of samples taken in connection with the enforcement of the home and export regulations. Work for several new departments, including the Air Board, the Ministry of Food, and the Coal Controller, has been undertaken during the year, and there has been a large increase in the number of samples from the Admiralty. The samples of food examined in connection with the supply of the Expeditionary Forces numbered 20,020. This work has been carried out partly at Clement's Inn, and partly by officers of the department stationed at the various supply bases. The chemical work of the following departments and other public bodies is now performed wholly or in part in the Government laboratory: Board of Customs and Excise, Admiralty, Air Board, Board of Agriculture and Fisheries, Central Control Board (Liquor Traffic), Department of Agriculture and Technical Instruction for Ireland, Colonial Office, Crown Agents for the Colonies, Ministry of Food, Foreign Office, Geological Survey, Home Office, India Office, Public Record Office, Stationery Office, Board of Trade, Trinity House, War Office, War Trade Department, Office of Woods and Forests, Office of Works (London and Dublin). Numerous questions have been submitted by the War Trade Department, the Foreign Office, the Treasury Solicitor, and the Admiralty Marshal, relating to contraband trading with the enemy (Prohibition Orders); and the services of the laboratory have also been extensively utilised in connection with the work of various departmental committees.

Customs and Excise—*Beer.*—The number of samples examined was 394, of which 209 were malt, corn, brewing sugars, and exhausted grains, and 185 yeast foods and miscellaneous substances used, or proposed to be used, in the preparation of beer. Two hundred and seventy-five samples of finished beer from brewers premises were examined for saccharin, saponin, and other prohibited ingredients, but except occasional traces of arsenic, as mentioned below, nothing of a deleterious or illegal character was found to be present. The percentage number and extent of the under-declarations were practically the same as last year.

Non-alcoholic Beers, Herb Beers, and Beer Substitutes.—The original gravity and percentage of alcohol were determined in 206 samples. Seventy-two of the samples were submitted as non-alcoholic beers under Section 8 of the Finance Act (Sess. 2), 1914, which exempts from beer duty beers which are not brewed on licensed premises if the original gravity and proof spirit do not exceed 16 degrees and 2 per cent. respectively. Eleven of the samples were outside both limits, and in 19 other cases either the original gravity or the percentage of proof spirit exceeded the limits allowed ; in 26 samples the proportion of proof spirit was found to be between 2 and 3 per cent., and in 3 samples between 3 and 4 per cent.; 1 contained over 4 per cent. In 125 of the herb beer samples the proof spirit did not exceed 2 per cent.; in 7 samples it was between 2 and 3 per cent.; and in the remaining 2 samples from 4 to 5 per cent.

Imported Beers.—Owing to the restrictions caused by the war the importation of beer has ceased.

Examination of Beer and Brewing Materials for Arsenic.—The total number of samples tested, including beer, worts, malt, sugar, and other materials used in brewing, was 1,469. Of these, 75 were found to contain arsenic in excess of the limits suggested by the Royal Commission on Arsenical Poisoning—namely, the equivalent of $\frac{1}{100}$ grain of arsenious oxide per pound in the case of solids, or per gallon in the case of liquids. Of 249 samples of malt and sugar tested, none contained arsenic in excess of the limits. Of 1,037 samples of beer and wort examined, one contained $\frac{1}{72}$ grain per gallon. The other 74 cases in which excess of arsenic was found all occurred in salts and solutions proposed to be used for treating brewing waters, but in no case was beer made from a treated water found to contain arsenic in excess of the Royal Commission limits.

Cider and Perry.—Seventeen samples of imported cider were examined, all of which were of normal character. Of the 6 samples purchased by customs and excise officers in the United Kingdom 2 were factitious — that is to say, did not consist solely of fermented apple-juice. The 12 genuine ciders examined contained 3 to 12 per cent. of proof spirit.

Table Water Duty.—The Finance (New Duties) Act, 1916, and the Finance Act, 1916 (6 Geo. V., c. 11, and 6 and 7 Geo. V., c. 24) imposed excise and customs duties on "table waters" at rates varying from 2d. to 8d. per gallon. In order to determine whether the waters from certain British and foreign spas and other liquids fall within the scope of these duties, and if so at which rate of duty they were chargeable, 10 samples of mineral and spa waters, 175 samples of cordials, syrups, fruit juices and essences, and 5 samples of "non alcoholic" wines, were examined. The 5 so-called "non-alcoholic" wines all contained spirits, the percentage at proof-spirit strength varying from 7·0 to 17·3. One of the "cordials" contained 15·9 per cent. of proof spirit. Sixty-one samples of herbal preparations, which the vendors claimed were not beverages, but *bona fide* medicines, were analysed. In the majority of instances this claim was substantiated by the analysis.

Spirits—"Groggings."—During the year 6 samples of "grog," and a sample of water connected with one of the "grogs," were examined. In 4 of the cases evidence was obtained that the spirit had been extracted from the wood of the cask.

Duty-free Spirits, including Methylated, specially Denatured Spirits, and other Manufacturing Spirits.—Eight hundred and seventy-seven samples of wood naphtha and of mineral naphtha, intended for use in the preparation of methylated spirit, were examined. The naphtha represented by 874 of these samples was approved as fit for methylating purposes, whilst that represented by the other 3 samples was found to be unsuitable. The number of samples examined shows a considerable decrease as compared with those of last year, which numbered 1,045. The decrease is largely due to the falling off in the quantity of "mineralised methylated spirit" which was formerly available for general use. The quantity of "industrial" or manufacturing methylated spirit, on the other hand, shows a small increase as compared with last year. There has also been an increase in the use of pure and specially denatured alcohol in manufacturing operations.

Tobacco—Manufactured Tobacco for Home Consumption.—To insure observance of the regulations as to moisture and oil, which are based upon the provisions of the Customs and Inland Revenue Act, 1887, the Oil in Tobacco Act, 1900, and the Finance Act, 1904, samples are taken from the factories and from the premises of retail dealers by customs and excise officers, and examined in the Government laboratory. The number of certificates of analysis issued for offal tobacco was 8,750, or 1,596 less than the number for the previous year. These decreases are due partly to the fact that less tobacco leaf was available for manufacture in the year 1917-8, but are also partly to be attributed to the efforts of the manufacturers to reduce the amount of refuse tobacco as much as possible.

Sugar, Glucose, and Saccharin—Sugar.—The great bulk of the revenue from sugar is derived from refined sugar, the duty being charged when the sugar is removed from the refinery warehouse. There are many articles for which it has not been found practicable to fix a special rate of duty, and, which, therefore, have to be tested on each importation or where drawback of duty is claimed on exportation. Amongst these may be mentioned egg yolk, gelatin, glue, honey, manna, meat extracts, parchment paper, printer's roller composition, and tanning extracts. Honey, although not liable to duty when genuine, is sometimes adulterated with invert sugar, upon which duty is payable. The number of samples of sugar and articles containing sugar examined for assessment of duty or drawback was 23,095. Of these, 36 samples consisted of molasses from the British beetroot sugar factory at Cantley, and 41 of molasses and solutions of molasses used in a new yeast-making process. *Saccharin.*—The presence of saccharin is searched for in all preparations in which there is any probability of its occurrence, and 140 samples of substances imported were specially examined with this object. On exportation of saccharin products 28 samples were drawn for examination for the purpose of assessing the amount of drawback payable. The manufacture of saccharin in the United Kingdom, which had ceased for some years, was resumed during the year and 15 samples of saccharin and of the materials used in its production were examined in connection with the assessment of duty.

Tea.—All consignments are usually examined at the port of entry by tea inspectors under the provisions of the Food and Drugs Act, 1875, doubtful samples only being sent to the Government laboratory: 8,556 of these having been examined. Of the total number of samples submitted, 1,485, representing 101,573 pounds, were condemned as containing sand or other foreign matter, and, in addition, some 7,000 pounds were reported as unfit for human consumption. The quantity rejected, though large, is quite insignificant in relation to the total amount of tea imported—namely, approximately 311 millions of pounds.

Cocoa and Chocolate.—By the Finance Act, 1911, the old fixed rate of duty of 2d. per pound was replaced by duties calculated upon the actual proportion of dutiable ingredients (cocoa, sugar, and cacao-butter). It is necessary, therefore, to estimate the proportion of each ingredient contained in such articles both on importation and exportation. No comment is made upon any of the samples examined.

Matches.—Only 4 samples of imported matches were submitted under the "White Phosphorus Matches Prohibition Act, 1908," and in no instance was any evidence of the presence of white phosphorus discovered.

BOARD OF AGRICULTURE AND FISHERIES.—Some 1,500 samples, as compared with 2,000 for the previous year, were examined for this department. During the past year no fresh milk has been imported, but 14 samples of imported sterilised milk were examined. In addition to the usual condensed milks, dried milks and milk powders are now frequently received for analysis (see special Report: ANALYST, 1918, **43**, 239-248). The percentage of fat in the samples of imported condensed milk ranged from 6·5 to 10·9. The samples with the low percentage of fat were taken from consignments of evaporated milk from America. A considerable quantity of this class of milk has been imported during the year, and it is worthy of note that it contains a lower percentage of milk solids than that of the condensed milk usually prepared for this country. The American product is usually described as "evaporated" milk, and consists of whole milk which has been evaporated to about half its bulk. All the samples of imported condensed milk were free from preservatives. The fat content of 14 samples of imported sterilised cream ranged from 23 to 30 per cent. Four hundred and thirty samples of imported butter, coming from eleven different countries, all contained less than the legal limit of 16 per cent. of water. Some of the samples from every country, with the exception of Denmark, contained boron preservative, and most of them contained added colouring matter. There was no evidence of the presence of fat other than butter in any sample, nor did the Reichert-Wollny number in any case fall below 24.

Imported Margarine.—Six hundred and seventeen samples were examined, all of which were imported from Holland; in no case did the content of butter-fat exceed that legally permissible. Water in excess of the limit was found in 10 cases.

MINISTRY OF MUNITIONS.—Amongst the 41 samples examined for this department were a so-called substitute for platinum—which proved to be an alloy of nickel, chromium, and tungsten—petrol substitutes of the usual kind, peat, and fire-extinguishing fluids, mainly of the chlorinated hydrocarbon type.

HOME OFFICE.—For this department, chocolates given to children and others under suspicious circumstances were examined, also drugs taken in connection with the regulations limiting the use of cocaine and opium, and various samples of liquors sold under conditions constituting a breach of the regulations made under the Defence of the Realm Act.

WAR DEPARTMENT.—Amongst other work involving the examination of 20,000 food samples, investigations were carried out with reference to the waxed-paper containers now being used considerably in place of tin for packing supplies to the Forces.

WAR TRADE DEPARTMENT.—Under the Exportation and Importation Prohibition Regulations it was required that disinfectants exported must not contain more than a certain proportion of pure carbolic acid or phenol. As no satisfactory method was available for making this estimation, a method was worked out in the Government laboratory (ANALYST, 1917, **42**, 329).

SALE OF FOOD AND DRUGS ACTS.—Examination of samples referred by magistrates under the Sale of Food and Drugs Acts of 1875 and 1899. When proceedings are taken under these Acts either the prosecutor or the person charged may request the justices before whom the case is heard to forward a portion of the sample in question to the Government laboratory for analysis. Under these provisions 102 samples were received, as compared with 69 in the previous year. These included 81 samples of milk, 3 each of nut lard, Epsom salts, and cream of tartar, 2 each of butter, dripping, rum, sweets, and vinegar, and 1 each of whisky and baking-powder. Six samples could not be examined owing to their condition on receipt. The results of the examination differed from those of the public analyst in 13 out of the 96 cases, or 13·5 per cent., as against 10·3 per cent. in the previous year. The disagreements in respect of the milk samples were 6 out of 51, as against 7 out of 59 in 1915-6.

The following are the particulars of some of the most important cases: *Butter.*—One sample contained excess water, but the evidence as to the presence of foreign fat in the other case was not conclusive. *Sweets.*—It was alleged that the samples contained lead, but the results as to the amount did not agree with those of the public analyst. *Spirits.*—The results as to strength confirmed those of the public analyst in 1 case, but differed in 2 cases. *Dripping.*—In 1 case the sample contained cotton-seed oil; and in the other case the material had not the character of dripping. *Cream of Tartar.*—Two samples contained lead, and 1 sample arsenic, as alleged by the public analyst. With respect to the nut lard alleged to contain water, baking-powder alleged to contain arsenic, vinegar alleged to be deficient in strength, and Epsom salts alleged to contain lead, the results agreed with those of the public analyst. *Milk.*—In the 75 cases in which certificates were issued the charges were as follows: In 42 cases, added water; 25, abstraction of fat; 6, added water and abstraction of fat; and 2, presence of dirt. Boric acid and artificial colouring matter were also alleged to be present in 2 of the samples.

H. F. E. H.

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THE INSTITUTE OF CHEMISTRY.

CHEMISTS EMPLOYED IN H.M. FACTORIES UNDER DEPARTMENT OF EXPLOSIVES SUPPLY.

IT is anticipated that many of the chemists engaged in the factories under the Department of Explosives Supply, Ministry of Munitions, will shortly be available for employment in private works or elsewhere.

In view of the impetus which has been given to many branches of chemical industry during the war, and the necessity for furthering the development and extension of chemical manufacturing industry, it is important that the public and those interested in chemical manufactures should realise how great a national asset the services of these chemists may prove.

These men have had advantages which rarely fall to the lot of chemists in works, since, owing to the lack of workmen competent to conduct complicated and often dangerous chemical operations, many of the chemists themselves have been required to carry out all the manual work involved until ordinary workmen or women were sufficiently trained. In this way they have gained an excellent first-hand practical experience in running the plants and in overcoming technical difficulties. In addition, they have had quite exceptional opportunities of learning the principles on which the plants are constructed and the various operations carried on.

Great pains have been taken by the Factories Branch of the Department of Explosives Supply to give the chemists full opportunities of thoroughly understanding the plant and the processes with which they were working, not only in regard to purely technical matters, but also in regard to working costs. They have thus always had before them the incentive to reduce the working costs as much as possible by the economy of raw materials, the recovery of waste products, and the skilful employment of plant and labour. In other words, they have been taken into the confidence of the management of the factory, have had these aims clearly presented to them, and have been shown the best means to attain these.

Again, many of them have carried out on the large scale valuable investigations relating to the difficulties that have arisen in the course of manufacture and in the recovery of waste products. They have all been taught to consider the plant operations in the form of a balance-sheet, and to account as far as possible for the yields and losses involved.

There is no doubt that many such chemists can be usefully and profitably employed in the scientific management of factories, and that they will prove extremely valuable to our chemical industry. The crying need in this country is for careful, thorough, scientific control of its industries, and these chemists afford the chemical manufacturers the opportunity of getting reliable aid in that essential work.

CHEMISTS: PIVOTAL MEN.

With regard to the release of pivotal men from the forces for employment as chemists, the Demobilisation and Resettlement Department of the Ministry of Labour has authorised the Institute of Chemistry to assist in the selection of names of chemists —professional, consulting, analytical, and research—whose prompt return to civil life will prove of the most value to the country for the purposes of reconstruction and absorption of unemployed labour. The Institute is asked to select officers and men who can be classed as pivotal, preference being given to those with the longest service and to married over single men.

Works chemists are not to be included in the selection, as their employers are required to apply for them under the industry in which they are engaged.

The names of selected men will be forwarded by the Registrar of the Institute to the Department of Demobilisation.

REVIEW.

A TREATISE ON APPLIED ANALYTICAL CHEMISTRY. Vol. II. By VITTORIO VILLAVECCHIA, Director of the Chemical Laboratories of the Italian Customs. Translated by T. H. Pope, B.Sc., A.C.G.I., F.I.C. Pp. xv + 536. London: J. A. Churchill, 1918. Price 25s. net.

This book forms the second volume of Professor V. Villavecchia's "Applied Analytical Chemistry," and deals in sixteen chapters with the application of chemistry to the analysis of the commoner industrial and food products.

The Director has been assisted by several well-known Italian collaborateurs, who have done their best to compress the very large subject into one volume. This has been rendered more difficult inasmuch as mineral colours, which should have been dealt with in the first volume, occupy 35 out of the 528 pages. This volume contains an index to both.

The first chapter is devoted to meat and its preparation, and, after dealing with the usual physical observations, gives in full detail methods for determining preservatives, colouring matter, horseflesh, casein, starch, and nitrogen in its various forms. The portion on meat extracts is particularly well done, and collects in a small space the various analytical standardising processes which the analyst must ordinarily obtain with great expenditure of labour from the more lengthy textbooks on the subject. The chlorine and bromine determinations for gelatin are omitted, as is also the Gunning-Arnold-Kjeldahl modification.

Milk and its products is the subject next dealt with. The more antiquated methods of analysis are entirely omitted, and great prominence is given to Gerber's volumetric estimation of fat, which is now the standard commercial process adopted for this estimation. Special tests, such as that for reductases, cryoscopic index, Cornalba's constant (total soluble matter), and others, are very briefly, but concisely, treated, as well as their modifications, which are required when testing butter, cheese, condensed milk, and margarine.

The table on the variation in the composition of milk in various towns in Italy is scarcely of interest to the British student.

The third chapter treats the very debatable subject of flour, starch, and their derived products. Here the same definiteness is maintained as with the other chapters, and very useful hints on the microscope and its use in this connection are given. Methods for the detection of foreign and injurious seeds, wood-meal, mineral substances, chemical treatment, and baking tests are also given.

The next chapter—that on sugars and products containing them—is one of the longest in the book. Besides the usual sugar estimations, the processes used in testing the prime materials of the sugar industry are described, as well as the modifications of these processes which are necessary when an opinion is required on such products as jams, preserved fruit, chocolate, and other sweetmeats.

Beverages are very exhaustively treated. Three chapters—one for beers, one for wine, and another for spirits and liqueurs—give all the processes commonly required for their manufacture and standardisation, together with the most recent tables which are necessary in this connection. The chapter on beer has been mainly rewritten for this English edition, as this beverage is not the natural drink of Italy, and its manufacture there is not so highly developed as it is in this country. The new Original Gravity Tables for Beer is included.

Another very important chapter in the book is that dealing with colouring matters. These are divided into two sections—inorganic and organic. The inorganic colours are collected under the individual colours—*i.e.*, all the whites are treated together, then all the reds, yellows, greens, blues, and greys. With each particular colour the method of analysis is given, together with the estimation of the more common impurities present. The organic colours are further subdivided into the natural and synthetic colours. Various methods of identifying the latter are given with tables.

The remainder of the chapters in the book are short, and deal with essential oils, turpentine and its products, varnishes, rubber and gutta-percha, tanning products, inks, leather and textile fibres, yarns, and fabrics.

The book, like its predecessor, Vol. I., is one for reference, and not for study. It contains information which is indispensable for the analyst, and for this reason should find a place in every laboratory bookshelf. S. Rideal.