MAY, 1916. Vol. XLI., No. 482.

THE ANALYST.

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

An ordinary meeting of the Society was held on Wednesday evening, April 5, in the Chemical Society's Rooms, Burlington House. The President, Mr. G. Embrey, F.I.C., occupied the chair.

The minutes of the previous ordinary meeting were read and confirmed.

A certificate of proposal for election to membership in favour of Mr. M. S. Salamon, B.Sc., was read for the second time.

Mr. F. T. Alpe was elected a member of the Society.

The following papers were read: "On the Alkalimetric Estimation of Certain Divalent Metals in the Form of Tertiary Phosphates," by W. R. Schoeller, Ph.D., and A. R. Powell; "Note on a Specimen of Ancient Russian Oak," by P. A. Ellis Richards, F.I.C.; and "The Estimation of Potassium in Presence of Other Substances," by A. H. Bennett.

An exhibit of laboratory glass and porcelain of English manufacture was shown by Messrs. Baird and Tatlock (London), Ltd., and an improved polariscope, also of English manufacture, by Messrs. Bellingham and Stanley, Ltd., of 71, Hornsey Rise, N.

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A RAPID METHOD FOR THE ESTIMATION OF FAT IN POWDERS.

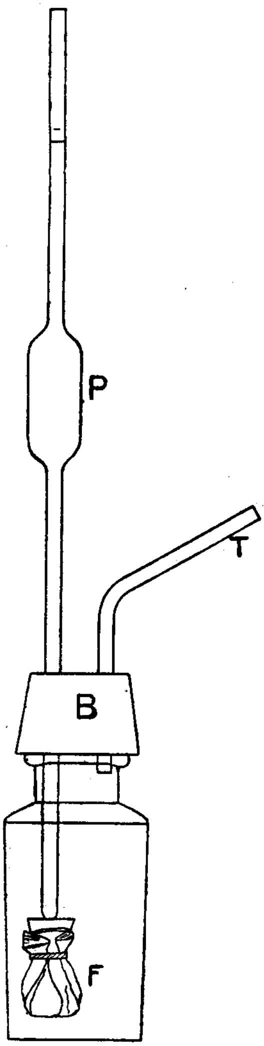
By S. B. PHILLIPS.

(*Read at the Meeting, March* 1, 1916.)

THE method consists in shaking a weighed quantity of the substance with a measured quantity of solvent at room-temperature, and of estimating the fat in an aliquot part of the filtered solution. This is similar in principle to Neumann's process (ANALYST, 1911, **36**, 542), but the apparatus devised facilitates the process.

An apparatus is employed which enables a result to be obtained in about an hour. This rapidity is attained by a new device for filtering the solvent directly into the measuring pipette. The solvent used is trichlorethylene, as recommended by L. Gowing-Scopes (ANALYST, 1910, **35**, 238; 1914, **39**, 18), which has the advantage over ether that the loss due to evaporation is negligible. Filtration is hastened by pressure in preference to suction, as the latter would cause loss by evaporation.

The substance is weighed in a counterpoised weighing-tube. Such a weight of substance is taken as is likely to contain 1·5 to 3·5 grms. of fat. This is transferred to a 6-ounce wide-mouthed stoppered bottle, with an accurately fitting stopper, the last traces of the substance being removed from the tube with the aid of a little sand; 100 c.c. of trichlorethylene are now added from a pipette, the stopper replaced, the bottle thoroughly shaken for a few seconds, and allowed to stand for about a quarter of an hour.



The filtering apparatus consists of a pipette, *P*, graduated for the delivery of 20 c.c. of liquid, passed through one hole of a double-bored bung, *B*. The other hole of the bung carries a short piece of glass tubing, *T*, through which the pressure is applied. The filtering arrangement is fixed to the delivery end of the pipette. This consists of two filter-papers, 9 cm. diameter, folded together over the end of a test-tube so as to resemble a Soxhlet thimble. This is tied firmly by means of string on to a small perforated cork, and fixed on to the end of the pipette as in *F*.

The bottle is again well shaken, the stopper is removed, and the filter is lowered into the solution. The bung is pressed with the hand against the bottle mouth and, as described below, air pressure is applied to the tube *T*, the filtered solution is forced into the pipette, and 20 c.c. removed and run into a tared flask. The solvent is evaporated off on an oil-bath at a temperature of 150° C. The fat is almost free from traces of the solvent, but it is generally advisable to dry for about an hour in a water oven, removing the last traces of solvent by means of blowpipe bellows. The fat is then cooled and weighed.

The solution of fat in the solvent causes an increase in volume. As the exact change in volume, which occurs when fats dissolve in trichlorethylene, has not been published, it became necessary to find a factor which, when multiplied by the weight of fat in 20 c.c. of solution, would give the total weight of fat present, from which figure the required percentage can be calculated. The following factors were determined for the estimation of cacao butter in cocoa, chocolate, etc.:

Weight of Fat in 20 C.C. Solution.	X.	Factor =	Percentage of Fat Present.
0·30 grm.		5·040	
0·35 ,,		5·055	
0·40 ,,		5·085	
0·45 ,,		5·100	
0·50 ,,		5·110	
0·55 ,,		5·122	
0·60 ,,		5·133	
0·65 ,,		5·148	
0·70 ,,		5·165	

Application of Pressure.—A convenient working pressure was found to be about 100 mm. of mercury. All of the following methods of applying pressure were found successful: (1) A force pump and aspirator; (2) an aspirator of water in an elevated position connected to one on the bench; (3) water pressure (direct); (4) a filter pump fixed in the mouth of an aspirator.

The following comparative figures were obtained with cocoa

BUTTER PER CENT.

	Soxhlet Process.	New Process.
(1)	25·90	26·07
(2)	27·55	27·59
(3)	27·95	27·70

In conclusion, the author wishes to thank Mr. A. W. Knapp for his valuable suggestions and criticisms.

RESEARCH LABORATORY,
MESSRS. CADBURY BROTHERS, LTD., BOURNVILLE.

Mr. A. W. KNAPP writes: The factors given in this paper apply to a hard fat (cacao butter), but it was found that if the factors were used for olive oil, the error was not more than 0·1 per cent. However, it is probably advisable for anyone who proposes to use the process for mustard, finely powdered feeding cake, or dried milk, to prepare a set of factors for the oil or fat to be estimated. It may be pointed out that the device for filtering an aliquot portion is capable of general application in analytical processes.

ON THE ALKALIMETRIC ESTIMATION OF CERTAIN DIVALENT METALS IN THE FORM OF TERTIARY PHOSPHATES, WITH ESPECIAL REFERENCE TO THE VOLUMETRIC DETERMINATION OF COBALT AND NICKEL.

By W. R. SCHOELLER, Ph.D., and A. R. POWELL.

(*Read at the Meeting, April* 5, 1916.)

The development of technical inorganic analysis has resulted in the gradual replacement of gravimetric by volumetric methods; and if precipitation methods can be superseded by the more scientific processes of oxidation, reduction, or saturation, a further advance will have been accomplished. External indicators and unstable solutions are the rule in the first, the exception in the latter, group of methods. The former are generally empirical, hence assay and standard must be titrated under identical conditions, whereas the latter are amenable to stoichiometric calculation, which allows a variety of elements or compounds to be estimated on the basis of a common standard. This applies especially to saturation methods, the most convenient primary standard for which is sodium carbonate. This mode of analysis has not been utilised to any extent for the determination of metals other than alkalis and alkaline earths. Recently, however, it was applied by Miles (*J. Chem. Soc.*, 1915, **107**, 988) to the determination of lead. Stolba's (*Zeitsch. anal. Chem.*, 1877, **16**, 100) method for the estimation of phosphoric acid, based upon the conversion of magnesium ammonium phosphate into acid phosphate in presence of an indicator of the methyl orange group, as expressed by the equation

$$2MgNH_4PO_4 + 2H_2SO_4 = MgH_4(PO_4)_2 + MgSO_4 + (NH_4)_2SO_4,$$

can be used for the determination of magnesium, and the method has been extended to zinc in zinc ammonium phosphate. We have investigated the alkalimetric determination of some other metals capable of yielding tertiary phosphates of the general type $M''NH_4PO_4$, which are obtained as crystalline precipitates, and therefore easily filtered off and washed. The results obtained are given below, together with brief notes on the published methods for magnesium and zinc.

Magnesium.—Magnesium is the only metal that is always separated as the double phosphate of the type here discussed. The volumetric process obviates the tedious ignition to pyrophosphate. Instead of washing the precipitate with dilute ammonia and partially drying the filter to get rid of the excess of the latter (Low, "Technical Methods of Ore Analysis," 5th edition, 1911, 159), we prefer to displace the ammoniacal washing solution with 50 per cent. alcohol (Classen, "Theorie und Praxis der Massanalyse," 1912, 214), after removing the filtrate and washings from under the funnel and substituting a flask containing acidulated water, so as to prevent re-absorption of ammonia vapour by the wet filter. The precipitate may be rinsed back and titrated direct with $\frac{N}{10}$ or $\frac{N}{5}$ acid in presence of cochineal, or it may be dissolved in a measured quantity of the acid and the excess of the latter titrated back with alkali, adding methyl orange, or preferably cochineal.

Zinc.—We need not enumerate the drawbacks of the ferrocyanide and sodium sulphide methods, both typical examples of precipitation analysis. The alkalimetric determination of zinc has been described by Walker (Sutton, "Volumetric Analysis," 9th edition, 1904, 359); and though the precipitation as phosphate (Treadwell-Hall, "Analytical Chemistry," Part II., 3rd edition, 1912, 140) involves a slightly more elaborate preliminary treatment than the ferrocyanide method, the superiority of the subsequent titration over the ordinary zinc assay will, we trust, lead to its more general adoption.

Cadmium.—What appears to be the only volumetric method so far published is the permanganate titration of the ferrous salt produced by the action of an acid solution of ferric sulphate upon cadmium sulphide (Sutton, *loc. cit.*, 167). In the writers' experience this type of titration, applicable to several metals, has not found general favour in practical metallurgical analysis. In gravimetric work cadmium is often weighed as sulphate (Low, *loc. cit.*, 61). By precipitating a neutral solution of the sulphate with a large excess of di-ammonium phosphate, cadmium ammonium phosphate is obtained as a silky crystalline precipitate, which may be dried at 105° C. and weighed as $CdNH_4PO_4 + H_2O$, or ignited to pyrophosphate (Miller and Page, *School of Mines Quarterly*, 1901, **22**, 301.)

On account of the high atomic weight of cadmium, we used $\frac{N}{10}$ acid (1 c.c. = 0·00562 grm. Cd.) to dissolve the phosphate precipitate, the excess being titrated back with $\frac{N}{10}$ alkali in presence of cochineal, because this indicator is more sensitive to decinormal solutions than methyl orange (Classen, *loc. cit.*, 216), and its colour increases in intensity as the end-point (*i.e.*, alkalinity) is reached. (For the same reason we give preference to methyl orange when titrating to acid reaction.) The results obtained are given in Table I. :

TABLE I.

Number.	Cadmium— Taken.	Cadmium— Found.	Error.
1.	0·0074	0·0075	+0·0001
2.	0·0263	0·0258	−0·0005
3.	0·0295	0·0298	+0·0003
4.	0·0527	0·0529	+0·0002
5.	0·0637	0·0638	+0·0001
6.	0·0779	0·0780	+0·0001
7.	0·0790	0·0787	−0·0003
8.	0·1053	0·1051	−0·0002
9.	0·1147	0·1160	+0·0013
10.	0·1216	0·1234	+0·0018

The figures are in close agreement, except Nos. 9 and 10. This is accounted for by the progressive decolorising effect of increasing quantities of cadmium on cochineal, which renders the end-point less distinct; in presence of about 0·1 grm. of cadmium in 100 to 150 c.c. bulk, the solution, instead of becoming violet, turns to a pale claret colour. With smaller quantities of cadmium the end-point is easily perceptible, hence it is advisable to have less than 0·1 grm. present in the assay. It is interesting to note that, unlike cadmium, a neutral solution of zinc sulphate strikes a deep pink colour on addition of a few drops of cochineal tincture.

Manganese.—The two permanganate methods (Low, *loc. cit.*, 162, 167) for manganese are satisfactory, although as regards Volhard's method the standardisation of the permanganate solution has not yet been quite satisfactorily decided (Classen, *loc. cit.*, 344). The precipitation as manganese ammonium phosphate (Treadwell-Hall, *loc. cit.*, 126), though not advantageous in the cases of ores, is useful in the analysis of manganese salts. By treating the precipitate as described under cadmium we obtained results shown in Table II.

TABLE II.

Number.	Manganese—		Error.
	Taken.	Found.	
1.	0·0124	0·0131	+0·0007
2.	0·0310	0·0307	−0·0003
3.	0·0620	0·0616	−0·0004
4.	0·1240	0·1229	−0·0011
5.	0·1860	0·1846	−0·0014

The results are a trifle low, which we believe to be due to a slight loss in the washing.

Cobalt.—The precipitation of cobalt as cobalt ammonium phosphate was first described by Dirvell (*Chem. News*, **40**, 268). Directions for carrying out the process have been published by Clarke (*Chem. News*, **48**, 262) and by Hope (Beringer, "Textbook of Assaying," 12th edition, 1910, 442). Though for full details these references should be consulted, a brief comparison of the two methods appears desirable, as we found the properties of cobalt ammonium phosphate to vary according to the procedure followed. Clarke adds an amount of ammonium phosphate equal to five times the combined weight of cobalt and nickel, and five parts of hydrochloric acid to one of ammonium phosphate. Ammonia is cautiously added to the boiling solution till the precipitate redissolves, and the liquid vigorously stirred to precipitate the cobalt. Hope directs adding nearly the same excess of ammonium phosphate, but no hydrochloric acid or ammonium chloride. The boiling solution is treated drop by drop with dilute ammonia whilst continually stirring until the blue amorphous precipitate at first produced becomes pink. The essential difference between the two methods lies in the quantity of ammonia added, while the properties of the resulting precipitates differ as shown below :

Clarke's Method.	Hope's Method.
Coarsely granular.	Silky crystalline.
Deep lilac.	Pale lilac.
Precipitate generally nickel-free, but filtrate usually contains a little cobalt.	Filtrate cobalt-free, but precipitate usually carries a little nickel.
Precipitate slowly soluble in $\frac{N}{5}$ acid, which must be added in excess and titrated back (indirect titration).	Precipitate instantly soluble in $\frac{N}{5}$ acid; suitable for direct titration.

Indirect Titration.—The titration with $\frac{N}{5}$ acid and alkali did not prove a complete success. In presence of more than 0·05 grm. of cobalt, methyl orange was useless, the pink colour of the solution masking the end-point; nor was the appearance of the bluish precipitate that marks the end of the reaction sufficiently pronounced to serve as an indicator. Nickel sulphate as a decoloriser proved disappointing, the liquor assuming a dirty-grey tinge, through which the delicate transition-point was difficultly perceptible. With lacmoid the change of colour could not be determined with sufficient accuracy. Cochineal was found to be the most serviceable indicator, but the results were always slightly high, as shown in Table III.

TABLE III.

Number.	Indicator.	Cobalt—		Error.
		Taken.	Found.	
1.	Methyl orange	0·0202	0·0210	+0·0008
2.	„	0·0280	0·0285	+0·0005
3.*	„	0·0493	0·0497	+0·0004
4.	Cochineal	0·0250	0·0256	+0·0006
5.	„	0·0409	0·0414	+0·0005
6.	„	0·0479	0·0490	+0·0011
7.	„	0·0499	0·0511	+0·0012
8.	„	0·0562	0·0575	+0·0013
9.	„	0·0759	0·0755	-0·0004
10.	„	0·0784	0·0799	+0·0015
11.	„	0·1048	0·1065	+0·0017
12.	„	0·1124	0·1135	+0·0011
13.	„	0·1406	0·1424	+0·0018
14.	„	0·1687	0·1699	+0·0012

We ascribe this positive error to the action of cobalt on the indicator, having made the observation that the addition of a few drops of a neutral cobalt solution to distilled water tinted with cochineal produces a deep pink colour which requires

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Mean of four titrations, error varying from nil to +0·0006.

a drop of $\frac{N}{5}$ acid to discharge. Hence in titrating the excess of acid the colour changes to purple before the solution is actually alkaline. Although, roughly speaking, the error tends to increase with the amount of cobalt, we were unable to establish a definite relation between the magnitude of the error and the quantities of metal or indicator used. For this reason, and because the end-point is not easily perceptible in presence of a large amount of cobalt, we prefer the direct method described below.

Direct Titration.—The filter containing the precipitate is spread against the side of the beaker, rinsed down with water followed by a few drops of the acid used in the titration, and again with water to displace the acid, taking care not to use more than about 30 c.c. in all. The filter is now discarded, and the addition of acid continued until the precipitate disappears. No indicator is required, for as long as any but a small quantity of precipitate remains it imparts a lilac colour to the liquid, while the last trace is sufficiently conspicuous by the light reflected on the glittering scales to determine its disappearance within 0·1 c.c., even in presence of suspended filter fibres. The advantage of this method over the preceding one lies in the fact that large quantities of cobalt can be thus determined, provided the cobalt is precipitated in the modification instantly soluble in $\frac{N}{5}$ acid; and as this is easily contaminated with nickel if present to any extent, double precipitation must be resorted to. Table IV. shows results obtained by the direct method.

TABLE IV.

Number.	Cobalt—		Error.
	Taken.	Found	
1.	0·0402	0·0408	+0·0006
2.	0·0954	0·0963	+0·0009
3.	0·1159	0·1160	+0·0001
4.	0·1164	0·1149	−0·0015
5.	0·1447	0·1445	−0·0002
6.	0·1616	0·1598	−0·0018
7.	0·1818	0·1808	−0·0010
8.	0·1902	0·1901	−0·0001
9.	0·3022	0·3023	+0·0001

Nickel.—A method for separating nickel and cobalt, based upon the conversion of cobalt into a cobaltamine salt and precipitation of the nickel as nickel ammonium phosphate, has been worked out by Clarke (*J. Soc. Chem. Ind.*, 1896, **25**, 866). We did not investigate the application of the alkalimetric method to nickel ammonium phosphate, because the precipitate is less insoluble than the other phosphates discussed in this paper, and the accuracy of Moore's cyanide titration (Sutton, *loc. cit.*, 251, 252) could not be exceeded; but we desire to call attention to the fact that by precipitating cobalt as cobalt ammonium phosphate a feebly ammoniacal filtrate is obtained in which the nickel can be titrated by cyanide without further manipulation. This advantage does not seem to have been hitherto realised, for Mellor ("Treatise on Quantitative Inorganic Analysis," 1913, 400) thus sums up the present position of the volumetric determination of the two metals: "The methods which have been suggested up to the present for the determination of cobalt volumetrically are not satisfactory except under special conditions, and hence no volumetric method for cobalt has won a place in general practice. Volumetric methods for nickel also fail in the presence of cobalt, although the cyanide process is quite good when cobalt is absent, or only present in minute quantities." We verified the accuracy of the nickel titration after separating cobalt as the double phosphate, and obtained very satisfactory results (see Table V.). The nickel filtrate, after being left to cool, should be titrated without undue delay, as it gradually deposits a pale green transparent crystalline precipitate, practically insoluble in ammonia, and containing nickel and phosphoric acid. Once formed, it requires a fair excess of hydrochloric acid for its solution, after which the liquid may be made slightly ammoniacal, cooled, and titrated in the usual manner.

TABLE V.

Number.	Nickel Taken.	Cobalt Added.	Nickel Found.	Error.
1.	0·0356	0·0799	0·0357	+0·0001
2.	0·0630	0·0983	0·0630	0·0000
3.	0·0856	0·0574	0·0855	−0·0001
4.	0·1196	0·0843	0·1191	−0·0005

The superiority of the phosphate process over all the other methods of separating nickel and cobalt lies in the fact that it allows a separate volumetric estimation of the two metals to be made, and that both are obtained in the form directly suitable for titration. If a small quantity of cobalt has escaped precipitation, due to the use of an excess of ammonia, it is at once detected by the darkening of the liquid in the nickel titration, while the colour of the cobalt precipitate is a sure indication of contamination with nickel: if bright lilac, the separation has been properly carried out; but if of a dirty pink colour, too little ammonia has been added, and it will be necessary to dissolve in a little acid and repeat the precipitation, which can be done in a few minutes. The atomic weights being practically the same, nickel may be reckoned as cobalt and *vice versa*. Table VI. gives a few results obtained by the process; in carrying out the separation we usually add 5 grms. of ammonium chloride, and always reprecipitate the cobalt, unless present in small quantity, adding more ammonia in the first than in the second precipitation. In this manner a good separation is effected in the first operation, while the second yields a precipitate which can be titrated with acid alone, no indicator being required.

TABLE VI.

Number.	Cobalt—		Error.	Nickel—		Error.
	Taken.	Found.		Taken.	Found.	
1.	0·0130	0·0135	+0·0005	0·0175	0·0178	+0·0003
2.	0·0180	0·0177	−0·0003	0·1168	0·1175	+0·0007
3.	0·0520	0·0524	+0·0004	0·0519	0·0520	+0·0001
4.	0·0520	0·0524	+0·0004	0·0519	0·0517	−0·0002
5.	0·0624	0·0636	+0·0012	0·0554	0·0554	0·0000
6.	0·1043	0·1059	+0·0016	0·0088	0·0092	+0·0004

The greater part of the experimental work recorded above was carried out in the metallurgical laboratory of the Sir John Cass Technical Institute.

DISCUSSION.

Mr. G. N. HUNTLY said that, although the phosphate method was of considerable interest, it seemed not to have come into general use. In the determination of magnesia the phosphate precipitate, after being washed with ammonia, required very thorough washing with alcohol, and provided this was duly carried out the method was quite satisfactory. Like the present authors, he had found cochineal to be the best indicator for the purpose. For the determination of zinc the method was extensively used in America, where, however, it was the practice to weigh the precipitate. The zinc was separated first as sulphide, and redissolved, being then thrown down as the double phosphate, which was weighed, either after drying or (which was more satisfactory) after ignition. If the phosphate precipitate could be readily and accurately titrated, he thought this would be found more satisfactory than weighing the precipitate, but there was some little difficulty in connection with the question of its composition. In America the theoretical factor based on atomic weights was used, while Bell and Ibbotson recommended an appreciably lower factor, and he had found that the lower factor seemed for some reason to give the more correct result. For the determination of manganese it was also the practice in America to weigh the precipitate, but he believed that ice-water had to be used for washing it, which introduced rather a complication. The bismuthate method for medium quantities and the persulphate method for small quantities were so rapid and accurate that even this method hardly seemed likely to displace them. With regard to cobalt and nickel, it was to be noted that the authors had started with pure solutions; one would like to know in what form the cobalt and nickel would be separated from a mixture before the phosphate method was applied, as any other metals that might be present would also be thrown down by the phosphate. For nickel the cyanide titration method was largely used, but the glyoxime method was so easy and accurate that it would probably be difficult to displace it.

Dr. J. T. DUNN said that he had tried the phosphate method some years ago for the determination of zinc, but had given it up on account of the difficulty of seeing the end-point. With regard to nickel and cobalt, he should, like Mr. Huntly, be interested to hear whether the authors had done more than merely apply the method to pure salts or mixtures thereof, and how, in the determination of nickel and cobalt in, for instance, steel, they would prepare for the application of the phosphate method.

The PRESIDENT remarked that the question of separation of nickel and cobalt seemed to be a particularly difficult one. It seemed likely that in the future an important part would be played, not only by nickel steel, but also by cobalt steel, and in that event a simple volumetric method for the determination of cobalt would be a great desideratum.

Dr. SCHOELLER, in reply, said that the manganese precipitate was slightly soluble in water, and to a much smaller extent in ammonium salts. A neutral ammonium salt like ammonium nitrate must, therefore, be used for washing, in default of which the result would be slightly low. It might be mentioned that the application of the method to cadmium was as new as its application to cobalt. He did not think that any other volumetric method except the sulphide titration had yet been proposed in the case of cadmium. The separation of nickel and cobalt from other metals, especially manganese, zinc and iron, was certainly a difficult problem. Clark and Hope had started with pure nickel and cobalt salts, and the present investigation had been commenced at the point where these chemists left off, the volumetric method having been applied to solutions containing nickel and cobalt salts only. In Hope's method the iron was separated as basic acetate, and the nickel and cobalt precipitated as sulphides in a solution weakly acid with acetic acid. The sulphides were dissolved in *aquaregia*, and this solution was used for the phosphate precipitation. That method of separation was not, however, very satisfactory. Mr. Powell and he had been experimenting for some time with a new method of separating cobalt and nickel, but they were not yet able to record any definite results.

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ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

FOOD AND DRUGS ANALYSIS.

Estimation of Alkaloids by the Hydrochloride Method. G. D. Beal and E. Brady. (*J. Ind. and Eng. Chem.*, 1916, **8**, 48.)—The method consists in the extraction of the alkaloid with ether, precipitation from this solution as the hydrochloride by passing in dry gaseous hydrochloric acid, evaporating off the ether and weighing the salt, then dissolving the hydrochloride in water, and titrating the hydrochloric acid with standard alkali, using phenolphthaleïn as indicator. The results obtained were checked in each case by a standard method already in use, that of the U.S.P. viii. being used wherever available. Excellent results having been obtained in case of conium seeds, in which the alkaloid was estimated as coniine monohydrochloride, the method was extended to the analysis of tobacco and colchicum root, with similar results. In all these cases the hydrochloride of the alkaloid separated partly as a crystalline solid and partly as an oil, when the ether extract, colourless and free from fat, was saturated with dry gaseous hydrochloric acid. The nature of this oil is being investigated, but it would appear to be simply a higher hydrochloride. If the oil be washed with cold ether until it is free from free hydrochloric acid it still fumes in moist air; and when placed in a vacuum desiccator with some solid potassium hydroxide it is converted into the monohydrochloride. H. F. E. H.

Estimation of Cane Sugar in Condensed Milk. G. W. Knight and G. Formanék. (*J. Ind. and Eng. Chem.*, 1916, 8, 28-31.) After a critical review of numerous methods for the estimation of cane sugar in condensed milks the author refers to the fact mentioned by Richmond (ANALYST, 1910, **35**, 516) that acid mercuric nitrate fails to remove all the proteins occurring in condensed milk, since on adding phosphotungstic acid to a solution of milk thus clarified a voluminous precipitate is formed. It is therefore recommended that the sample should be first clarified by the addition of 1·7 c.c. of 5 per cent. phosphotungstic acid solution for each 10 grms. of condensed milk, and then, after shaking, an addition is made of 2·1 c.c. of a 25 per cent. neutral lead acetate solution for each 10 grms. of milk. After shaking and filtering, potassium oxalate crystals are added until a curdy precipitate forms, which quickly settles, leaving a clear liquid. The solutions are again filtered and polarised at 20° C. Another portion of the filtrate is inverted by means of concentrated hydrochloric acid all night at the room temperature, which should not be below 20° C., after which it is again polarised. Test mixtures were made up with condensed milks containing known amounts of cane sugar, the whole of the added sugar being accurately returned by the above method, and also by the gravimetric method of Munson and Walker (ANALYST, 1906, **31**, 337) which was employed as a check. H. F. E. H.

Use and Purchase of Feeding-Stuffs. (*Dept. of Agric. and Tech. Instruct. for Ireland J.*, 1915, **15**, 759-774; through *J. Agric. Intell. and Plant Diseases*, 1915, **6**, 1670-1671.)—The following table is abridged from one compiled by Crowther. It refers to feeds of average quality:

Table showing the Total Food Ingredients, Digestible Portion, and Manurial Value, of the Principal Feeding-Stuffs.

Name of Feeding-Stuff.	Total Percentage in Foods.				Digestible Percentage in Foods.			Estimated Manurial Value by Consumption of One Ton.		
	Albuminoids, Amides, etc.	Oil.	Soluble Carbohydrates.	Crude Fibre.	True Albuminoids.	Oil.	Carbohydrates and Fibre.	£	s.	d.
Linseed cake	30	10	34	9	25	9½	32	1	17	3
Decorticated cotton cake	41	9	26	8	34	8½	20	2	14	10
Undecorticated cotton cake	22	5½	34	20	15½	5¼	20	1	13	7
Soya cake (soy bean cake)	43	6	28	4	34	5½	22	2	13	6
Soya meal (soy bean meal) extracted ...	45	2	30	5	36	1¾	24	2	16	0
Coconut cake	22	10	36	15	17	9½	39	1	11	10
Palmnut cake	17	10	36	22	14	9½	36	1	0	1
Beans	25	1½	48	7	19	1¼	48	1	11	11
Peas	23	1½	54	6	17	1	53	1	7	7
Brewer's grains (wet)...	5	1½	12	5	3½	1¼	10	0	5	11
Brewer's grains (dry)...	19	5½	45	19	12½	5	38	1	2	5
Malt coombs	23½	2	44	12½	11½	1½	39	1	14	11
Bran	14	4	56	9	10	3	45	1	6	6
Molasses or treacle (beet)	10	—	60	—	—	—	55	0	19	1
Molasses or treacle (cane)	2	—	66	—	—	—	60	0	19	1
Linseed or flax seed ...	23	36	23	6	17	34	21	1	9	2
Wheat	12	2	69	2	9	1¼	65	0	15	2
Barley	10	2	67	5	7	1¾	64	0	13	10
Oats	12	6	55	10	9	5¼	45	0	15	0
Maize or Indian corn ...	10½	5	70	2	7	4½	68	0	13	2
Maize germ meal ...	14	8	57	5	10	7½	54	0	17	7
Rice meal	12	12	50	8	6	10	42	0	19	10
Meadow hay	10	2½	42	26	4	1	41	0	16	4
Clover hay	13	2½	37	25	5½	1½	38	1	1	8
Pasture grass	3	¾	10	5	1½	½	11	0	5	9
Oat straw	3½	2	38	37	1	½	39	0	9	6
Potatoes	2	¼	21	1	1/10	1/10	19	0	4	6
Mangels	1¼	¼	9	1	1/10	1/10	9	0	3	5
Swedes	1¼	¼	8	1½	¼	1/10	8	0	2	8
Turnips	1	¼	6	1	¼	1/10	6	0	2	7
Carrots	1¼	¼	9½	1½	½	1/10	10	0	2	7
Cabbage	2½	¾	7	2	1½	½	7	0	4	4

H. F. E. H.

Estimation of the Amount of Offals in the Flour of Cereals. Heiduschka and Heinich. (*Zeitsch. für Untersuch. der Nahrungs- und Genussm.*, 1915, **30**, 226-228; through *J. Agric. Intell. and Plant Diseases*, 1915, **6**, 1699.)—With 2·5 grms. of flour are mixed 10 c.c. of water and 20 c.c. of hydrochloric acid (sp. gr. 1·19); the whole is stirred and kept for half an hour at ordinary temperature. The mixture is then centrifuged for ten minutes, the clear liquor decanted, and the insoluble residue retained on an 11-cm. filter of known weight. The precipitate is washed once with 5 c.c. of 25 per cent. hydrochloric acid and twice with 10 c.c. of water, after which it is dried at 100° C. Only the offals are left on the filter, all the hydrolysable contents of the flour being dissolved.

H. F. E. H.

Composition and Nutritive Value of Mutton and Lamb. A. M. Wright. (*J. Soc. Chem. Ind.*, 1916, **35**, 235-236.)—The following results were obtained in the analysis of boneless meat from fresh New Zealand mutton and lamb:

	Mutton.				Lamb.		
	Leg.	Loin.	Shoulder.	Neck and Breast.	Leg.	Loin.	Fore-quarter.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Moisture	49·08	46·64	55·94	43·35	57·92	40·12	44·42
Ash	0·87	0·67	0·76	0·55	0·89	0·49	0·64
Fat	22·60	37·48	27·66	42·56	24·08	48·38	40·54
Total nitrogen	2·956	2·394	2·495	2·112	2·742	1·688	2·283
Cold water extract:							
Total solids	4·75	3·49	3·65	3·06	4·39	2·47	3·31
Ash	0·75	0·52	0·56	0·44	0·71	0·43	0·51
Organic extractives	4·00	2·97	3·09	2·62	3·68	2·04	2·80
Nitrogen	0·596	0·430	0·485	0·375	0·566	0·311	0·421
Coagulable proteins	1·72	1·31	1·36	1·18	1·66	0·86	1·21
Non-coagulable proteins	0·18	0·12	0·23	0·11	0·22	0·11	0·16
Meat bases	0·92	0·62	0·72	0·52	0·78	0·52	0·63
Insoluble protein	14·74	12·24	12·55	10·79	14·87	8·59	11·62
Total determined proteins	16·64	13·67	14·14	12·08	16·75	9·56	12·99
Crude protein ($N \times 6{\cdot}24$)	18·48	14·94	15·56	13·18	17·11	10·56	14·27

No relationship could be traced between the market prices of the different cuts and their palatability as indicated by the amounts of mineral salts, nor between the prices and the proportion of fat, protein, or meat bases.

C. A. M.

Method for the Determination of the Strength and Baking Qualities of Wheat-Flour. C. H. Bailey. (*J. Ind. and Eng. Chem.* 1916, **8**, 53-57.)—An apparatus is described which automatically measures the extent of expansion of dough. A dough made from 150 grms. of flour is placed in a cylindrical vessel, and a disc is suspended in the cylinder attached by a cord to a counterpoised weight, the cord being passed over a light-running pulley. The cord is attached to a device provided with a needle which moves along the face of the scale and shows the extent of expansion. A circle of paper is inserted between the top of the dough and the disc, so that the latter does not stick, and it is accordingly left at the highest point which it reaches during the course of the expansion. Other qualities of the flour are determined by a separate baking test on another portion of the same dough. A standard mixture of flour, yeast, salt, and sugar is always employed, and the dough is made in a motor-propelled mixing machine, a control flour being used to check every determination. As soon as the dough is made, it is allowed first to expand in an electrically-heated and controlled fermentation cabinet at a temperature of 28° C. The length of this fermentation period is made to vary with the nature of the flour, a period of about four and a half hours being the usual time. It is then divided into two portions, a smaller portion being placed in the expansion apparatus already described, while the larger portion is allowed to expand in a thermostat at a temperature of 34° C., until that portion in the measuring cylinder occupies a volume of 710 c.c. It is then placed in an electric oven previously heated to 200° C., and baked at that temperature for about forty-five minutes.

The portion in the expansion cylinder is maintained at 32° C., and requires no further attention until the dough falls, when the maximum expansion attained can be read on the scale. The expansion of the dough in the cylinder is better stated in terms of the increase in volume in c.c., rather than of the total volume of the fermented dough. The total volume of the baked loaf, when cool, is determined by displacement in flax-seed. The completed record of the baking test contains six items: (1) The maximum expansion of the dough in the cylinder; (2) the volume of the baked loaf; (3) absorbtion of water in making the dough in terms of c.c. of water at 28° C. per 100 grms. of flour; (4) colour of crumb; (5) crumb texture; and (6) remarks concerning the flavour and odour. In place of Humphries' and Biffen's definition of "strength," viz., "a strong wheat is one which yields flour capable of making large well-piled loaves" (*J. Agri. Sci.*, 1907, **2**, 1-16), the author prefers the statement that "the strength of flour is determined by the ratio between the rate of production of carbon dioxide in, and the rate of loss of carbon dioxide from, the fermenting mass of dough." H. F. E. H.

Fat of Nux Vomica. H. E. Watt and G. B. Angus. (*J. Soc. Chem. Ind.*, 1916, **35**, 201.)—Nux vomica contains 1 to 2 per cent. of a solid fat consisting principally of the glycerides of capric, caprylic, caproic, butyric, and palmitic acids. A sample examined by the authors was of a dark brown colour, had an unpleasant odour, and gave the following values: Sp. gr. at 100°/15·5° C., 0·892; solidification-point, 60° C.; acid value, 33·7; saponification value, 152; Reichert-Wollny value, 1·0; iodine value (Hübl), 54; and acetyl value, 31·2. C. A. M.

New Method of Analysing Oils. A. Mazzaron. (*Staz. Sperim. Agrar. Ital.*, 1915, **48**, 583-594; through *J. Agric. Intell. and Plant Diseases*, 1915, **6**, 1700-1701.)—In performing the rise of temperature test with oils by Tortelli's method, the reaction of the strong sulphuric acid with the oil is always accompanied by the liberation of gas consisting chiefly of sulphur dioxide. This evolution, which is very small in the case of pure olive oil, is considerable in the case of mixtures of olive and seed oils, and very large with pure seeds oils, especially with soya oil. Experiments have shown that what the author terms the "sulphur dioxide value" may be taken as the basis of a new analytical constant in the examination of oils. This value is defined as the number of c.c. of $\frac{N}{10}$ iodine solution reduced by the sulphur dioxide given off by 20 c.c. of the oil when treated with 5 c.c. of pure sulphuric acid, sp. gr. 1·8417. Table I. shows the sulphur dioxide values of some common vegetable oils.

TABLE I.

Oils.	Temperature Test.	Sulphur Dioxide Value at 20° C.	Sulphur Dioxide Value at 15° C.
Olive	43·2	2·4	1·6
Sesame	72	49·5	29
Cotton-seed	85	137·5	93·5
Maize...	75	65	38
Soya	93·5	223	173
Colza	61	15	9·5
Earthnut	51	7	4

Table II. shows the remarkable constancy of the sulphur dioxide value of pure olive oils from different localities.

TABLE II.

Locality.	Temperature Test.	Sulphur Dioxide Value at 20° C.	Locality.	Temperature Test.	Sulphur Dioxide Value at 20° C.
Florence ...	43·2	2·2	Lucca... ...	43·6	2·4
Lucca	42·6	2·3	"	43·3	2·3
"	43·4	2·4	Florence ...	42·2	2·1
"	43·5	2·5	Molfetta ...	46·1	2·6

It is remarked that the range of variation allowed in the temperature test of olive oil (between 41° and 46°) allows of olive oil being blended with considerable quantities of other oils of different temperature tests, whereas the greater constancy of the sulphur dioxide value, and the great distance separating that of olive oils from those of the others, are reasons for recommending this new method. Further investigation is required, and should be extended to animal oils and fats, as the method may possibly be applicable to butter fat. J. F. B.

Estimation of Starch in Raw Potatoes. E. Ewers. (*Zeitsch. offentl. Chem.*, 1915, **21**, 232-233; through *J. Soc. Chem. Ind.*, 1916, **35**, 432.)—Twenty grms. of the uniformly ground potato are heated for fifteen to seventeen minutes in the water-bath with 50 c.c. of dilute hydrochloric acid (20 c.c. of sp. gr. 1·124 per litre) in a flask containing 100 c.c. at 20° C. The mixture is then diluted to about 90 c.c. with cold water, cooled to 20° C., treated with 1·25 c.c. of sodium molybdate solution (prepared by fusing 30 grms. of MoO_3 with 25 grms. of sodium carbonate, dissolving the melt in water, and diluting to 250 c.c.), made up to 100 c.c. at 20° C., filtered, and polarised in a 200-mm. tube. The percentage of starch is obtained by multiplying the saccharimeter reading in Ventzke divisions by 0·44007.

BACTERIOLOGICAL, PHYSIOLOGICAL, ETC.

Detection and Concentration of Antigens by Ultrafiltration, Pressure Dialysis, etc., with Special Reference to Diphtheria and Tetanus Toxins. A. T. Glenny and G. S. Walpole. (*Biochem. J.*, 1915, **9**, 298-308.)—Since antigens cannot pass through the collodion membranes described in an abstract (see p. 152), these membranes may be used for the purification and concentration of any antigen, so that the latter may be examined and recognised. Mallein and tuberculin can be freed from glycerol and a considerable quantity of nitrogenous substance in this way. A process for the concentration and purification of diphtheria toxin has been worked out. It consists essentially in dialysing under pressure, followed by acidification, centrifugalisation, and re-solution of the precipitate in a very small quantity of alkali. The use of the membranes prevents the periodic recurrence of toxins of low value. If ultrafiltration is followed by pressure dialysis, tetanus toxin may be concentrated, and at the same time freed from disintegration products of broth materials, which are particularly noxious. These membranes are impermeable to enzymes, but allow the co-enzyme of zymase and the toxic constituent of Witte peptone to pass.
W. P. S.

Boron: Its Absorption and Distribution in Plants, and its Effect on Growth. F. C. Cook. (*J. of Agric. Research*, 1916, **5**, 877-890.)—Since both borax and calcined colemanite are largely used on manure heaps as valuable larvicides for the house-fly maggot, their effect on the growth of a variety of plants was investigated.

The material was added to manure at the rate of 0·62 pound per 8 bushels or 10 cubic feet. When the manure was mixed with the soil at the rate of 20 tons per acre, 216 pounds of borax per acre were present. The boron was quantitatively determined both in the soils and in the dried plant colorimetrically, using curcumin. In the case of plants, about 3 grms. of the dried sample were incinerated with lime-water, and an aliquot portion of a hydrochloric acid extract of the ash was tested by means of strips of curcumin-paper, suspended so as to allow them to dip in the solution to the depth of a quarter of an inch. The colour developed was compared with standard solutions at the end of four hours. In the case of soils, the boron soluble in dilute hydrochloric acid, but not the total boron, was determined. Fifty grms. of the soil were shaken with 200 c.c. of a solution of hydrochloric 1 : 20 for one hour; this was then filtered, and 100 c.c. of the filtrate made alkaline with lime-water, evaporated, and incinerated. The ash after acidification with hydrochloric was then tested as before.

Tomatoes, beet, lettuce, potatoes, radishes, corn, oats, and wheat, appear normal when grown in the presence of amounts of boron which produced injury to leguminous plants. When borax is added to manure at the rate of 0·62 pound to each 8 bushels, and the manure is applied to the soil at the rate of 15 tons per acre, 0·0011 per cent. of boron is added to the soil. It was found that this quantity of boron may injure leguminous plants, but it did not injure the other plants tested, although no stimulation was noted.

Wheat and oats absorbed very little boron, whilst leguminous and succulent plants absorbed comparatively large amounts. The yield of wheat from a plot heavily treated with borax was 90 per cent. of the manured control yield and greater than the yield from the unmanured control; the wheat grains were sound, and contained but a trace of boron. The yield of tomatoes in pot tests was unaffected when borax was added in amounts to produce 0·0018 per cent. of boron in the soil, but when the amount was increased to 0·0030 per cent. a reduced yield resulted.

The distribution of boron in the different parts of the plants is also discussed.

H. F. E. H.

Use of Enzymes and Special Yeasts in Carbohydrate Analysis. W. A. Davis. (*J. Soc. Chem. Ind.*, 1916, **35**, 201-210.)—For the estimation of cane sugar in plant material the author used a method of hydrolysis with 10 per cent. citric acid solution, and a method of hydrolysis for twenty-four hours at 38° C. with autolysed yeast. *Hydrolysis with Citric Acid.*—Fifty c.c. of the solution (which has been treated with basic lead acetate, and from which the excess of lead has been removed by means of sodium carbonate) are treated with sulphuric acid, drop by drop, until it shows the faintest indication of pink with methyl orange. Solid citric acid is then added to form a 10 per-cent. solution, the liquid boiled for ten minutes under a reflux condenser, then cooled, neutralised (with phenolphthaleïn as indicator) by means of 10 per cent. sodium hydroxide solution, diluted to 100 c.c., and polarised, or the reducing powder determined. Under these conditions maltose is not hydrolysed. *Hydrolysis of Cane Sugar with Invertase.*—Fresh pressed beer-yeast is washed on a Buchner funnel, and packed into a wide-mouthed bottle. About 30 to 50 c.c. of toluene are allowed to percolate through the mass, and the bottle is then covered with paper and left for about a fortnight in a warm place (25° to 30° C.). The liquefied yeast is filtered through a Buchner's funnel, to obtain a clear solution of invertase free from maltase and zymase. *Method of Hydrolysis.*—Fifty c.c. of the solution, in which reduction and polarisation have been determined, are prepared in the same manner as for the citric acid hydrolysis, treated with 1 c.c. of the autolysed yeast, and left for twenty-four hours at 38° C., after which 5 to 10 c.c. of alumina cream are added, the volume made up to 100 c.c., and the rotation and reducing power determined. Under these conditions the Clerget-Herzfeld constant holds good, after allowance has been made for the concentration by Herzfeld's table. *Alleged Precipitation of Reducing Sugars by Basic Lead Acetate.*—The experiments cited show that reducing sugars are not actually precipitated by basic lead acetate, but that lævulose, for example, is either destroyed by the basic lead acetate or it is transformed into another sugar with a different specific rotatory power and a smaller reducing power. Hence in sugar analysis any considerable excess of basic lead acetate should be avoided, and such excess should be removed before polarisation. *Estimation of Raffinose.*—Autolysed top yeast (as used for cane sugar) converts raffinose into lævulose and melibiose, whereas autolysed bottom yeast contains the enzyme melibiase in addition to invertase, and converts the raffinose into lævulose, galactose, and dextrose. Polarimetric measurement of the difference between the two equations thus enables the raffinose to be estimated. To 95 c.c. of the solution which has been hydrolysed with top yeast are added 5 c.c. of autolysed bottom yeast, and the rotatory power measured each day until constant. If the readings are made in a 200-mm. tube, a 1 per cent. solution of melibiose will change in rotation during hydrolysis 1·45 angular degrees, or 4·18° V. Hence a change of 1° V. is equivalent to 0·239 grm. of melibiose per 100 c.c. of solution, or 0·352 grm. of anhydrous raffinose. Good results were obtained with raffinose in admixture with lævulose, invert sugar, lactose, maltose, dextrose, cane sugar, cellose, and trehalose, and the method is applicable to the estimation of raffinose in low grade beet sugars and molasses. *Estimation of Maltose.*—Fifty c.c. of the solution (purified with basic lead acetate and free from lead), which should be faintly acid to litmus, are mixed with 5 c.c. of yeast-water, and sterilised for twenty minutes in an autoclave at 115° C. to 120° C. The liquid is then inoculated with a trace of a maltase-free yeast (*S. Marxianus*, *S. exiguus*, or *S. anomalus*), and incubated at 25° C. for three to four weeks (*cf.* Baker and Dick, ANALYST, 1905, **30**, 79). When the fermentation is complete 5 c.c. of alumina cream are added, the solution boiled and filtered, and the precipitate washed until the filtrate and washings amount to 100° c.c. The reducing power of an aliquot portion is then determined, and the amount of maltose calculated from the result. When pentoses are present, they, like maltose, are not fermented by maltase-free yeasts. A correction may be made by making another fermentation with brewer's yeast, which ferments away the maltose, but leaves the pentoses. The slight reducing power of such fermented solution affords a measure of the pentoses present. *Estimation of Other Sugars.*—A mixture of raffinose, cane sugar, and monoses, was fermented with *S. cerevisiæ* (bottom yeast U), ordinary pressed yeast, *Torula dattila* and *Torula monosa.* The difference between the results obtained with the first two yeasts gave the raffinose. The cane sugar was estimated from the volume of gas given by *Torula dattila,* after making allowance for the raffinose present; while the gas emitted by *Torula monosa* afforded a measure of hexoses present (dextrose, lævulose, or mannose). Estimation of lactose in presence of other sugars by treatment with ordinary yeasts has been studied by Baker and Hulton (ANALYST, 1910, **35**, 512). *Estimation of Starch.*—The dry material is freed from sugars by extraction with boiling 80 per cent. alcohol, and, if necessary, from gums, amylans, etc., by extraction with water. It is heated with 200 c.c. of water in a 250 c.c. flask for thirty minutes on the water-bath, then cooled to 38° C., treated with 0·1 grm. of taka diastase and 2 c.c. of toluene, and allowed to stand for twenty-four hours. After being boiled to destroy the diastase, the liquid is filtered into a 500 c.c. flask, the residue washed, and the filtrate and washings diluted to about 475 c.c. Sufficient basic lead acetate is then added to precipitate tannins, etc. (a large excess being avoided), and the solution made up to 500 c.c. and filtered. The excess of lead is removed from an aliquot portion of the filtrate by adding solid sodium carbonate, the lead carbonate removed, and the reducing power and polarimetric reading of the filtrate determined (*cf.* Revis and Burnett, ANALYST, 1915, **40**, 429). C. A. M.

Presence of Urease in Soya Beans. T. M. Groll. (*Chem. Weekblad,* 1916, **13**, 254-255.)—Soya beans were immersed for five minutes in 1 per cent. mercuric chloride solution, and then washed and freed from husks, rinsed with sterile water, and cut up. On mixing 150 mgrms. of the material with water, adding 10 c.c. of 1 per cent. urea solution at 25° C., and maintaining the mixture at that temperature for thirty minutes, the amount of ammonia liberated required 10 c.c. of $\frac{N}{10}$ acid for neutralisation. C. A. M.

ORGANIC ANALYSIS.

Simultaneous Estimation of Carbon and Halogen by the Chromic Acid Method. P. W. Robertson. (*J. Chem. Soc.*, 1916, **109**, 215-221.) — The satisfactory results obtained with the chromic acid method in the estimation of chlorine and bromine in organic compounds (ANALYST, 1915, **40**, 413) have led the author to extend the process to the simultaneous estimation of carbon, and his method of procedure may be applied with advantage to the estimation of carbon alone, particularly in compounds which are combustible with difficulty in the dry way. The apparatus, when used for the estimation of carbon only, may be adapted for either gravimetric or volumetric measurement of the carbon dioxide. In either case, in order to complete the combustion of traces of carbon monoxide, a short silica tube containing platinised asbestos is attached to the delivery tube from the reaction flask, and between this and the absorption apparatus is interposed a small U-tube containing glass-wool well moistened with sulphuric acid containing chromic acid, to retain the oxides of sulphur. The substance to be analysed is weighed from a small tube into the reaction flask, and 3 grms. of chromic acid added. For the gravimetric procedure the absorption apparatus consists of a calcium chloride tube and two soda-lime tubes, the latter being previously weighed full of oxygen. A slow current of oxygen, free from carbon dioxide, is passed through the whole apparatus, and about 30 c.c. of redistilled sulphuric acid added to the reaction flask. The reaction is controlled by cooling if necessary, and the combustion completed in seventy to ninety minutes. The results are checked by a blank experiment and corrected for the amount of carbon dioxide produced by the reagents alone. The numbers compare favourably with those obtained by dry combustion; even in the case of trinitrophenol no error is introduced by nitrous fumes. By the volumetric procedure the method may be applied to much smaller quantities of material. In this case the absorption apparatus is replaced by an evacuated flask of 1,500 c.c. capacity containing a measured quantity of standard baryta solution. A pressure regulator is attached to the oxygen inlet tube, consisting of a T-piece with its end dipping in water, and a gentle stream of bubbles is allowed to escape. When the sulphuric acid has been added, the tap of the evacuated flask is slightly opened, and the combustion allowed to proceed for about two hours. The baryta solution is slightly agitated at intervals, and finally titrated with $\frac{N}{20}$ acid. For the simultaneous estimation of carbon and bromine, two absorption tubes are used, the first containing 10 c.c. of 1·3 N-sodium hydroxide and 2 c.c. of 2 N-sodium sulphite, and the second tube 10 c.c. of sodium hydroxide, part of which is retained by glass beads in the exit tube. Oxygen is passed through at the rate of a group of bubbles every two or three seconds, and the reaction is assisted by heat. At the end, the contents of the absorption tubes are washed into a flask, boiled, treated with 5 c.c. of hot saturated barium nitrate solution, and filtered. The filtrate is titrated with $\frac{N}{3}$ nitric acid in presence of phenolphthaleïn, the result being corrected by a blank determination. The solution is further acidified with nitric acid, 10 c.c. of $\frac{N}{10}$ silver solution are added, and the excess is titrated with $\frac{N}{10}$ thiocyanate. The silver solution is previously standardised by pure potassium bromide treated with the reagents in the same apparatus, errors being thus compensated. The value for the bromine, reduced to $\frac{N}{3}$, is subtracted from the total alkalimetric result to obtain the value for carbon dioxide. The estimation of carbon and chlorine is carried out in a similar manner, except that between the small U-tube containing chromic and sulphuric acids and the first absorption tube there must be inserted a second short length of silica tubing, which is heated to decompose any chromyl chloride carried over, chromic oxide being deposited. This is necessary because chromyl chloride is capable of neutralising twice as much alkali as the corresponding amount of chlorine. The results, as in the case of bromine, are sufficiently satisfactory and concordant. Attempts to estimate nitrogen in the residual acid in the reaction flask, on the principle of the Kjeldahl method, did not yield satisfactory results. J. F. B.

Direct Estimation of Carbon Monoxide in Mixtures containing Unsaturated Hydrocarbons. A. Piva. (*Annali Chim. Applic.*, 1916, **5**, 82-93.)—The gaseous mixture is repeatedly passed through a tube containing soda-lime in which the proportion of sodium hydroxide is about 20 per cent., heated in an air-bath to 230° C. Under these conditions carbon monoxide is absorbed and retained by the soda-lime. The tube should be connected with pipette tubes charged with alkaline pyrogallol and sodium hydrosulphite, to remove carbon dioxide and oxygen before absorption of the carbon monoxide. As a typical example, an analysis of the illuminating gas of Palermo is quoted. By Hempel's method this contained —Carbon dioxide, 1·6; unsaturated hydrocarbons, 2·9; oxygen, 0·6; hydrogen, 47·3; methane, 33·2; nitrogen, 6·5; and carbon monoxide, 7·9 per cent. By direct absorption with soda-lime at 230° C. the amount of carbon monoxide found was 7·8 per cent. C. A. M.

Detection of Glycerides by the Magenta-Sulphurous Acid Reagent. M. François. (*J. Pharm. Chim.*, 1916, **13**, 65-77.)—A test for glycerol described recently by François and Boismenu (ANALYST, 1915, **40**, 238) is modified so that it may be used for the detection of glycerides in the presence of paraffins, waxes, resins, etc. About 1 grm. of the substance is mixed in a test-tube with 10 grms. of sand, and then heated over a flame, the vapours produced being conducted into another test-tube containing 3 c.c. of Schiff's reagent (sulphurous acid, 220 c.c.; 0·1 per cent. magenta solution, 30 c.c.; and concentrated sulphuric acid, 3 c.c.). White fumes produced at the commencement of the heating should be prevented from passing into the second test-tube by removing the flame for a few moments at a time. The heating should be continued (nearly to redness) for about ten minutes; the test-tube containing the reagent is then heated in a boiling water-bath for fifteen minutes. If the substance under examination contained a glyceride, the reagent is coloured red while cold, and the colour changes to blue on heating. All glycerides give this reaction; paraffins, fatty acids, waxes, resins, spermaceti, tolu balsam, and caoutchouc, yield vapours which give a red coloration with the reagent while cold, but the colour disappears on heating. The mechanism of the decoloration of magenta by sulphurous acid and the formation of the red and blue colorations is discussed. W. P. S.

Solubility of Naphthalene in Ammonia. S. Hilpert. (*Zeitsch. angew. Chem.*, 1916, **29**, 57-59.)—The solubility of naphthalene in ammonia solution and in liquid ammonia is given in the following table, the figures expressing grm. per 1,000 grms. of liquid:

Per Cent. NH_3:	0.	5.	10.	25.	100.
At 0° C.	0·018	0·030	0·042	0·064	33·0
At 25° C.	0·030	0·044	0·074	0·162	120·0

The presence of 2 per cent. of pyridine in the liquid ammonia increases the solubility of the naphthalene by 0·082 grm. at 0° C., and by 0·245 grm. at 25° C.; the solubility is not influenced by phenol. If carbon dioxide is passed into 25 per cent. ammonia containing dissolved naphthalene, the greater part of the latter is precipitated. When ammonia containing naphthalene is distilled, the condenser tubes are liable to become blocked with naphthalene unless they are maintained at a temperature not lower than 30° C. The picrate method may be used for the estimation of naphthalene in ammonia. A definite quantity of the sample is cooled in ice-water, neutralised by the gradual addition of sulphuric acid, care being taken to keep the mixture quite cold, and then distilled until about 30 c.c. of distillate have been collected. The distillate is extracted with ether, the condenser also being rinsed out with this solvent, and the separated ethereal solution is then treated with an excess of $\frac{N}{20}$ picric acid solution. After the lapse of two minutes the ether is evaporated under reduced pressure, the residual aqueous solution is cooled in ice-water, the precipitate collected on a filter, washed with 5 c.c. of ice-water, and titrated with $\frac{N}{10}$ sodium hydroxide solution, using litmus as indicator (see also Analyst, 1915, **40**, 64). W. P. S.

Estimation of Prussian Blue in Spent Oxide. E. L. Randall. (*J. Gas Lighting*, 1916, **133**, 158; through *J. Soc. Chem. Ind.*, 1916, **35**, 42.)—The process is based on the fact that when titanous chloride is gradually added to a dilute solution of an alkali ferricyanide a light green coloration is produced which deepens to an intense green and then slowly becomes reddish-brown, the reaction being quantitative in the presence of a large excess of ammonium or alkali thiocyanate. Not less than 0·1 grm. of the ferricyanide should be present, a satisfactory degree of dilution being about 0·6 grm. per litre for the potassium and about 2 grms. per litre for the sodium salt. The titanous chloride (1 per cent. solution) is standardised against ferrous ammonium sulphate or potassium ferricyanide. Results within 0·3 per cent. of the theoretical were obtained in estimations made on solutions of the latter salt. Alkali ferrocyanides alone and mixed with ferricyanides were also satisfactorily estimated, the former being oxidised by a dilute solution of potassium permanganate the slight excess of which was removed by the addition of thiocyanate before titration. For the estimation of Prussian blue in spent oxide the following procedure is suggested: After the removal of free sulphur by extraction with carbon bisulphide the material is treated with sodium hydroxide and a little ferrous sulphate (to convert any cyanide into ferrocyanide), the mixture filtered, and the filtrate agitated with lead carbonate to remove sulphides. The solution is then neutralised and diluted to a definite volume, of which an aliquot portion is treated with permanganate and titrated after the addition of sodium thiocyanate, the potassium or ammonium salt not being permissible.

Estimation of Mixtures of Paracetaldehyde and Acetal. K. J. P. Orton and P. V. McKie. (*J. Chem. Soc.*, 1916, **109**, 184-186.)—The method for the separate estimation of paracetaldehyde and acetal is based on the different rates of decomposition of these substances, with formation of acetaldehyde, on heating with dilute aqueous acids. The acetaldehyde is distilled off and estimated by one of the standard methods. Acetal is stable in alkaline or neutral solutions, but is completely hydrolysed in a few minutes on boiling in 1 per cent. solution with a trace of a strong acid at a concentration of $\frac{N}{6000}$, or with a weaker acid at equivalent hydrion concentration. Paracetaldehyde shows no signs of decomposition until the hydrion concentration is increased to $\frac{N}{2000}$, and a rapid action is only realised when the concentration of the strong acid amounts to $\frac{N}{10}$ or $\frac{N}{7\cdot5}$. In the estimation of acetal, the inconvenience of measuring accurately the minute quantity of strong acid necessary for the separation is best avoided by the use of acetic acid. Seven c.c. of $\frac{N}{100}$ acetic acid are diluted to 30 c.c. in a small flask of the Kjeldahl pattern, and 0·2 to 0·4 grm. of the mixture to be analysed is added. The flask is attached to a condenser connected by means of an adapter with a receiver (*e.g.*, a small measuring cylinder) containing 10 c.c. of water or alcohol for the better absorption of the acetaldehyde. The receiver is well cooled and the liquid gently heated until 7 c.c. have been distilled in the estimation of acetal. For the estimation of paracetaldehyde (or of the total acetal and paracetaldehyde in a mixture), 30 c.c. of $\frac{N}{5}$ hydrochloric acid are used and 10 c.c. of distillate collected. The acetaldehyde in the distillate may be determined either by the hydrogen sulphite method (Ripper's modification), or by the neutral sulphite method of Seyewetz; both give concordant results. If the mixture contain acetaldehyde in addition to acetal and paracetaldehyde, it may be estimated by a separate distillation. J. F. B.

New Reaction of Picric Acid, and its Applications. J. Castets. (*J. Pharm. Chim.*, 1916, **13**, 46-49.)—When picric acid is boiled with bromine solution 2-bromo-4 : 6-dinitrophenol is formed ; the test proposed depends on the formation of this substance and the red coloration which it gives with ammonia vapour or potassium cyanide. Ten c.c. of the picric acid solution are treated with 10 drops of saturated bromine-water, the mixture is heated to boiling, cooled, and extracted with ether. The ethereal solution is separated and divided into two portions, one of which is evaporated in a porcelain basin and the residue treated with ammonia vapour ; a red coloration is obtained which is discharged by an excess of ammonia or by the addition of water. The other portion of the ethereal solution is evaporated, drop by drop, on a piece of filter-paper ; a red coloration is obtained if the paper is then treated with ammonia vapour or with a drop of potassium cyanide solution, and again dried. For the estimation of picric acid in urine, 100 c.c. of the sample are mixed with 2 c.c. of hydrochloric acid and 20 c.c. of chloroform, heated on a water bath, cooled, the chloroform layer is separated, and evaporated. The residue thus obtained is dissolved in water, treated with bromine, and the test proceeded with as described. The test will detect the presence of 5 mgrms. of picric acid per litre of urine ; it must be remembered that a positive reaction for picric acid in urine can be obtained only when a quantity of the acid has been taken sufficient to insure that a portion of it shall pass into the urine before it has been converted into picramic acid during its passage through the body. W. P. S.

Estimation of Toluene: Application of the Method to Benzene and Xylene. H. W. James. (*J. Soc. Chem. Ind.*, 1916, **35**, 236-240.)

Naphthas.—The sample is distilled with the aid of a fractionating column at the rate of one drop per second, so as to obtain two fractions—I. and II.—consisting respectively of substantially a mixture of toluene and benzene, and of a mixture of benzene, toluene, and xylenes.

A. 250 c.c. are distilled up to 140° C. If at least 95 per cent. distils below 140° C., this distillation may be omitted.

B. The distillate from *A* (or the original sample) is redistilled, and the following fractions collected: (1) Below 90° C.; (2) from 90° to 100° C.; (3) from 100° to 109° C. ; (4) 109° to 112° C. ; (5) 112° to 120° C. ; (6) residue at 120° C.

C. The fractions 2, 3, 4, and 5, are separately redistilled, all fractions distilling below 110·6° C. being collected in receiver No. 1, and all residues left at 110·6° C. being added to that in receiver No. 6. Should the amount of liquid in any of the flasks become too small for distillation before 110·6° C. is reached, the distillation is stopped, the flask cooled, the contents of the next fraction added to the residue, and the distillation continued.

In the case of crude unwashed naphtha the sample is distilled to 150° C. in stage *A*. The distillate is washed with sulphuric acid in the usual way, and distilled as in *B*, the following fractions being collected : (1) Below 90° C. ; (2) 90° to 100° C.; (3) 100° to 109° C.; (4) 109° to 112° C. ; (5) 112° to 120° C.; (6) 120° to 140° C. In stage *C* all the fractions are separately distilled to 110·6° C. into No. 1, and all residues put together.

Table I.

Mixtures of Toluene and Benzene.

Toluene per Cent.	85° C.	90° C.	95° C.	100° C.	108° C.	Toluene per Cent.	85° C.	90° C.	95° C.	100° C.	108° C.
1	—	—	—	—	—	51	—	—	44·9	—	—
2	—	—	—	—	—	52	—	—	43·0	—	—
3	98·2	—	—	—	—	53	—	—	40·2	—	—
4	94·5	—	—	—	—	54	—	—	38·0	—	—
5	91·0	—	—	—	—	55	—	—	35·2	—	—
6	89·2	—	—	—	—	56	—	—	32·8	—	—
7	87·0	—	—	—	—	57	—	—	30·5	—	—
8	85·0	—	—	—	—	58	—	—	28·0	—	—
9	82·6	—	—	—	—	59	—	—	25·0	—	—
10	80·0	—	—	—	—	60	—	—	22·0	56·5	—
11	77·0	—	—	—	—	61	—	—	—	54·5	—
12	74·2	—	—	—	—	62	—	—	—	52·3	—
13	71·0	—	—	—	—	63	—	—	—	50·1	—
14	68·0	—	—	—	—	64	—	—	—	47·9	—
15	64·0	—	—	—	—	65	—	—	—	45·6	—
16	60·0	—	—	—	—	66	—	—	—	43·2	—
17	56·2	—	—	—	—	67	—	—	—	40·8	—
18	52·4	—	—	—	—	68	—	—	—	38·2	—
19	48·0	—	—	—	—	69	—	—	—	35·7	—
20	42·5	—	—	—	—	70	—	—	—	33·0	—
21	37·0	—	—	—	—	71	—	—	—	30·0	—
22	32·0	—	—	—	—	72	—	—	—	27·0	—
23	28·0	—	—	—	—	73	—	—	—	24·0	—
24	23·0	—	—	—	—	74	—	—	—	21·0	—
25	17·0	71·3	—	—	—	75	—	—	—	18·0	76·0
26	—	69·5	—	—	—	76	—	—	—	—	75·0
27	—	67·2	—	—	—	77	—	—	—	—	73·9
28	—	65·0	—	—	—	78	—	—	—	—	72·6
29	—	63·0	—	—	—	79	—	—	—	—	71·3
30	—	60·7	—	—	—	80	—	—	—	—	70·0
31	—	58·0	—	—	—	81	—	—	—	—	68·4
32	—	55·6	—	—	—	82	—	—	—	—	66·8
33	—	53·0	—	—	—	83	—	—	—	—	65·0
34	—	50·5	—	—	—	84	—	—	—	—	63·0
35	—	48·0	—	—	—	85	—	—	—	—	60·8
36	—	45·0	—	—	—	86	—	—	—	—	58·2
37	—	42·1	—	—	—	87	—	—	—	—	55·9
38	—	39·5	—	—	—	88	—	—	—	—	52·5
39	—	35·5	—	—	—	89	—	—	—	—	49·1
40	—	33·2	—	—	—	90	—	—	—	—	45·0
41	—	30·0	—	—	—	91	—	—	—	—	40·0
42	—	26·8	—	—	—	92	—	—	—	—	35·0
43	—	23·2	—	—	—	93	—	—	—	—	29·0
44	—	19·5	—	—	—	94	—	—	—	—	21·0
45	—	16·0	57·0	—	—	95	—	—	—	—	11·0
46	—	—	55·3	—	—	96	—	—	—	—	6·0
47	—	—	53·5	—	—	97	—	—	—	—	3·0
48	—	—	51·5	—	—	98	—	—	—	—	—
49	—	—	49·5	—	—	99	—	—	—	—	—
50	—	—	47·2	—	—	100	—	—	—	—	—

Table II.

Mixtures of Toluene and Xylenes.

Toluene per Cent.	115° C.	120° C.	125° C.	130° C.	137° C.
1	—	—	—	—	—
2	—	—	—	—	—
3	—	—	—	—	4·0
4	—	—	—	—	6·5
5	—	—	—	—	11·0
6	—	—	—	—	17·0
7	—	—	—	—	23·0
8	—	—	—	—	28·0
9	—	—	—	—	32·3
10	—	—	—	—	37·0
11	—	—	—	—	41·0
12	—	—	—	—	45·0
13	—	—	—	—	48·5
14	—	—	—	—	51·2
15	—	—	—	—	53·5
16	—	—	—	—	55·8
17	—	—	—	—	58·0
18	—	—	—	—	59·8
19	—	—	—	—	61·5
20	—	—	—	—	63·3
21	—	—	—	—	65·0
22	—	—	—	—	66·4
23	—	—	—	—	67·8
24	—	—	—	—	69·2
25	—	—	—	21·7	70·5
26	—	—	—	24·7	—
27	—	—	—	27·5	—
28	—	—	—	30·3	—
29	—	—	—	33·0	—
30	—	—	—	35·7	—
31	—	—	—	38·2	—
32	—	—	—	40·7	—
33	—	—	—	43·1	—
34	—	—	—	45·4	—
35	—	—	—	47·6	—
36	—	—	—	49·8	—
37	—	—	—	51·8	—
38	—	—	—	53·5	—
39	—	—	—	55·3	—
40	—	—	—	57·2	—
41	—	—	—	59·0	—
42	—	—	—	60·7	—
43	—	—	—	62·3	—
44	—	—	—	63·8	—
45	—	—	36·3	65·3	—
46	—	—	38·8	—	—
47	—	—	41·2	—	—
48	—	—	43·4	—	—
49	—	—	45·7	—	—
50	—	—	48·0	—	—
51	—	—	50·2	—	—
52	—	—	52·3	—	—
53	—	—	54·5	—	—
54	—	—	56·6	—	—
55	—	17·0	58·6	—	—
56	—	20·0	—	—	—
57	—	23·5	—	—	—
58	—	26·5	—	—	—
59	—	29·5	—	—	—
60	—	32·7	—	—	—
61	—	35·7	—	—	—
62	—	38·7	—	—	—
63	—	41·5	—	—	—
64	—	44·0	—	—	—
65	—	46·7	—	—	—
66	—	49·5	—	—	—
67	—	52·1	—	—	—
68	—	54·9	—	—	—
69	—	57·2	—	—	—
70	—	59·6	—	—	—
71	—	62·0	—	—	—
72	—	64·5	—	—	—
73	—	67·0	—	—	—
74	—	69·4	—	—	—
75	—	71·6	—	—	—
76	—	73·7	—	—	—
77	—	75·7	—	—	—
78	—	77·5	—	—	—
79	—	79·2	—	—	—
80	41·5	80·9	—	—	—
81	46·0	—	—	—	—
82	50·0	—	—	—	—
83	54·0	—	—	—	—
84	58·0	—	—	—	—
85	62·0	—	—	—	—
86	66·0	—	—	—	—
87	70·0	—	—	—	—
88	73·5	—	—	—	—
89	76·3	—	—	—	—
90	79·3	—	—	—	—
91	82·2	—	—	—	—
92	85·0	—	—	—	—
93	87·7	—	—	—	—
94	90·4	—	—	—	—
95	93·0	—	—	—	—
96	95·5	—	—	—	—
97	98·0	—	—	—	—
98	—	—	—	—	—
99	—	—	—	—	—
100	—	—	—	—	—

The three fractions—viz., No. I., distillate below 110·6° C.; No. II., from 110·6° to 140° C.; No. III., residue at 140° C.—are measured, and No. III. is discarded. The others are distilled as follows—No. I.: The fraction, or 100 c.c. thereof, is distilled in a flask with a side-tubulure at the rate of 2 drops per second, and measured fractions are collected at 85° C., 90° C., 95° C., 100° C., and 108° C. Reference to Table I. will then give the percentage of toluene. The distillation is continued until the total percentage of distillate falls within the range of the corresponding column in the table. No. II. fraction is similarly distilled, fractions being collected at 115° C., 120° C., 125° C., 130° C., and 137° C., and the total percentage distilling at each temperature noted as before. Reference is then made to Table II. In the case of naphthas containing less than 30 per cent. of toluene the error should not reach 1 per cent. When only a very small amount of toluene is present it is advisable to add a known quantity of toluene, and to make the necessary correction in the results. A correction is made for paraffinoid hydrocarbons, when present. The specific gravity of fraction II. should lie between 0·865 and 0·870 at 15·5° C., and it may be assumed that the "pure" toluene separated from a naphtha would contain 1 per cent. of paraffinoid hydrocarbons for every 0·002 by which the sp. gr. falls short of 0·867.

Coal Tar.—The crude naphtha is separated by fractional distillation, washed with sulphuric acid, and 250 c.c. distilled as described above. The fractions separated from the tar are (*a*) light oil and water up to 220° C.; (*b*) creosote oil 220° to 260° C. The distillate obtained from the light oil (up to 190° C.) is mixed with the similar distillate from *b*, and the mixture shaken. Any water separating is withdrawn and measured. The residue constitutes the crude naphtha of the tar. It is purified by shaking with 10 per cent. of its volume of strong sulphuric acid, the flask being kept cool in water. After thirty minutes the brown "acid tar" is drawn off and the residue washed, without agitation, with successive portions of 50 c.c. of water, until the washings cease to be opalescent. The naphtha is then gently shaken with more portions of water, then with 10 c.c. of caustic soda solution (sp. gr. 1·075), and finally twice more with 50 c.c. of water. C. A. M.

INORGANIC ANALYSIS.

Behaviour of Antimony Stains or Deposits towards Hypochlorite Solution. W. Vaubel and A. Knocke. (*Chem. Zeit.*, 1916, **40**, 209-210.)—The ordinary hypochlorite test for distinguishing between antimony and arsenic stains, deposits, or spots, such as are obtained in the Marsh apparatus, gives reliable results only when freshly-prepared hypochlorite solution is employed. Old hypochlorite solution contains chlorite, and the latter dissolves the antimony mirror. Antimony stains on a porcelain surface have a varying composition; the velvet black stain is soluble in chlorite solution, whilst the brown-black or grey stains are insoluble. After twenty-four hours, the velvet-black stain becomes more or less insoluble. W. P. S.

Detection of Cadmium. R. Salvadori. (*Annali Chim. Applic.*, 1916, **5**, 25-26.)—An ammoniacal solution of ammonium perchlorate (20 per cent. in ammonia solution of sp. gr. 0·90) affords a sensitive test for cadmium, which it precipitates as a white crystalline double perchlorate [Cd.$(ClO_4)_2 . 4NH_3$]. In applying the test to the mixed sulphides of copper, cadmium and bismuth as precipitated in the second group, the precipitate is dissolved in nitric acid, and the solution treated with ammonia in excess. The bismuth hydroxide is filtered off, and the blue filtrate treated with the reagent, which precipitates the cadmium, even in the presence of five times its amount of copper. C. A. M.

Solubility of Mineral Phosphates in Citric Acid Solution. G. S. Robertson. (*J. Soc. Chem. Ind.*, 1916, **35**, 217-220.)—Experiments described in detail show that 2 per cent. citric acid will completely dissolve mineral phosphates if a sufficient number of extractions be made. In the case of most of the phosphates examined from 90 to 100 per cent. of the phosphoric acid was dissolved. The presence of a small amount of free lime or calcium carbonate caused a pronounced reduction in the solubility, and when much calcium carbonate is present the citric acid test, as ordinarily applied, is a test of the calcium. On the other hand, the greater the proportion of lime in actual combination with the phosphoric acid the greater the solubility. The fineness to which the slag is ground affects the solubility to the extent of about 10 per cent. decrease for each of the grades "Passes 100," "Refuses 100," "Refuses 60," and "Refuses 30." The solubility is progressively decreased by calcining. The general conclusion drawn is that mineral phosphates are as valuable sources of phosphoric acid as basic slag for plants, and that the citric acid test is valueless as a criterion of the relative suitability of phosphatic manures for plant life. C. A. M.

Influence of Fluorspar on the Solubility of Basic Slag in Citric Acid Solutions. G. S. Robertson. (*J. Soc. Chem. Ind.*, 1916, **35**, 216-217.)—The solubility of basic slag produced by the use of fluorspar ranges from 20 to 50 per cent. instead of from 70 to 90 per cent. in the citric acid test. Two samples (5 grms. each) of "fluorspar slags," when extracted five consecutive times with 500 c.c. of 2 per cent. citric acid solution, showed fairly constant ratios between the amounts of phosphoric acid and lime dissolved, whereas the ratios between the amounts of silica and phosphoric acid dissolved (*e.g.*, 1 : 0·53 and 1 : 1·209) were so divergent as to indicate that silica was not in combination with the phosphate. In the case of highly citric-soluble slag, the ratio between the dissolved phosphoric acid and silica is fairly constant in successive extraction (*e.g.*, 1 : 1·63 to 1 : 1·76). Hence the conclusion is drawn that the citric acid test is no true criterion of the solubility of phosphates in "fluorspar slags" or of their value as manures. These slags are completely soluble if sufficient time be given. Moreover, the phosphates in these slags show a close resemblance in composition to those in mineral phosphates, such as are used in the United States, in preference to highly citric-soluble slags. C. A. M.

Detection and Estimation of Halogens in Organic Compounds. I. Drogin and M. A. Rosanoff. (*J. Amer. Chem. Soc.*, 1916, **38**, 711-716.)—Stepanoff (ANALYST, 1907, **32**, 52) described a simple method for the estimation of halogens in organic compounds, depending on solution of the compound in absolute alcohol, reduction with sodium, and subsequent estimation of the halogen acid by Volhard's method. Bacon (ANALYST, 1909, **34**, 111) failed to get satisfactory results when following Stepanoff's directions, but described a modification which he found satisfactory. Maryott also (ANALYST, 1911, **36**, 287) devised a modification of the method which he found satisfactory. On the other hand, Walker and McRae (*J. Amer. Chem. Soc.*, 1911, **33**, 598) obtained poor results when following Bacon's directions. In view of the convenience of the method, as compared with that of Carius, the authors have reinvestigated it, have failed to get satisfactory results by either of the modifications above referred to, but have found conditions under which every organic substance tested by them yields the whole of its halogen as halogen acid, which can be readily estimated by Volhard's method. Since the history of the method shows that apparently trifling details may affect the results, the authors describe their modification at some length.

The reagents required are absolute alcohol, sodium, nitric acid, $\frac{N}{15}$ silver nitrate, $\frac{N}{15}$ ammonium thiocyanate, and a solution of ferric ammonium alum. The alcohol is redistilled from sodium. The sodium must be free from chlorine, or its chlorine content known. The ferric indicator is made up by adding concentrated nitric acid to a saturated solution of iron alum until the red colour changes to straw yellow.

The following quantities of alcohol and sodium are necessary and sufficient for the complete extraction of the halogen from apparently any organic combination. If W denotes the weight of substance taken (between 0·2 and 0·3 grm.), and if the halogen present is chlorine, then the number of c.c. of alcohol required is 156 W, and the number of grms. of sodium 21·5 W. If the halogen is bromine, then 68 W c.c. of alcohol and 9·4 W grm. of sodium are required. If the halogen is iodine, 44 W c.c. alcohol and 6·1 W grm. sodium are required.

The substance is dissolved in the requisite quantity of alcohol in a 300 c.c. Kjeldahl flask fitted with a reflux condenser, using a free flame to warm the alcohol if necessary. After cooling, small pieces of sodium are introduced through the condenser—a few at a time—so that the addition occupies about thirty minutes, and during at least the second half of this time the dissolution of the sodium is aided by a small flame under the flask. The mixture is next boiled for an hour with occasional shaking, cooled and diluted with water, the first portions being added 1 c.c. at a time so long as vigorous action ensues. When finally diluted to about 250 c.c., the mixture is acidified by addition of dilute (1 : 3) nitric acid, transferred to a beaker, mixed with a measured and more than sufficient volume of silver nitrate solution, and filtered. The filtrate, mixed with 10 c.c. (no less) of the ferric indicator, is then titrated with thiocyanate.

As a qualitative test, a few mgrms. of the substance are dissolved in 1 c.c. of alcohol, small fragments of sodium are added one at a time, and the mixture warmed. When cool the solution is acidified with 1 c.c. of dilute nitric acid, filtered if necessary, and tested with 10 drops of approximately $\frac{N}{15}$ silver nitrate. G. C. J.

Use of Hydrofluoric Acid in Separation of some Heavy Metals from Tin, Antimony, Tungsten, and Molybdenum by Means of the Electric Current. L. W. McCay and N. H. Furman. (*J. Amer. Chem. Soc.*, 1916, **38**, 640-652.)—It has already been shown that from a nitrohydrofluoric acid solution of copper, lead, tin, and antimony, the last two metals being in the higher state of oxidation, the electric current precipitates the copper and lead only, the former at the cathode, the latter as peroxide at the anode (McCay, *J. Amer. Chem. Soc.*, 1914, **36**, 2375.) It is now shown that silver and mercury can be separated from tin and antimony in a similar manner, and further that neither tungsten nor molybdenum interferes. The silver deposits contain no weighable amounts of platinum, but the mercury deposits usually do. The amount of platinum present varies, but seems to increase with the quantities of tin and hydrofluoric acid, and with the time during which the current acts. When the current strength is as high as 3 ampères, and a Classen rotating anode is employed, the amount of platinum found in the mercury deposits is about 0·5 mgrm. On treating the deposits with nitric acid, the platinum remains in the form of a black powder, or dark and very thin film, and can be rapidly and accurately determined, and the necessary correction made. G. C. J.

Estimation of Sulphur in Spent Oxide. E. V. Espenhahn. (*J. Soc. Chem. Ind.*, 1916, **35**, 292-293.)—The method depends on the fact that sulphur, when heated with iron, forms ferrous sulphide, which gives off its sulphur as hydrogen sulphide on treating with a strong acid. About 0·5 grm. of the oxide, finely powdered, is washed by decantation, and finally on a filter with hot water, to remove sulphates and thiocyanates. After drying, the oxide is scraped into a hard-glass crucible about 30 mm. long and 10 mm. in diameter, mixed with 4 grms. reduced iron and covered with a layer of the same. The crucible is fitted into a hole in an asbestos board, and the air displaced by carbon dioxide led in through a tube, the end of which is just above the layer of iron. A small flame is first applied, and the temperature raised until the crucible reaches dull redness, which is maintained for ten minutes. After cooling in an atmosphere of carbon dioxide, the contents of the crucible are allowed to fall into a small Erlenmeyer flask which is connected to three wash bottles, each containing 150 c.c. of $\frac{N}{10}$ iodine acidified with hydrochloric acid, and a fourth with a few c.c. of $\frac{N}{10}$ thiosulphate. The mass is covered with water and, after passing carbon dioxide for a few minutes, 50 c.c. of concentrated hydrochloric acid are run in through a funnel. As the reaction slackens, the solution is heated to boiling, and a steady stream of carbon dioxide passed through the flask for ten minutes. The excess of iodine is titrated back with thiosulphate, and the free sulphur calculated.

For the estimation of total sulphur, 0·5 grm. of the unwashed sample is reduced with iron and treated as described.

For the estimation of tar, a weighed portion of the sample is extracted with carbon bisulphide in a Soxhlet extractor. The difference between the percentage of extract so obtained and the percentage of free sulphur gives the percentage of tar.

G. C. J.

Decomposition of Tetrathionates in Alkaline Solution as a Source of Error in Certain Iodine Titrations. R. M. Chapin. (*J. Amer. Chem. Soc.*, 1916, **38**, 625-626.)—If acid solutions containing tetrathionates are to be titrated with iodine, or subjected to any treatment involving assumption that the tetrathionate present has remained unaffected, they should never be neutralised by any substance of distinctly alkaline properties. It has long been known that tetrathionates react with hot, concentrated caustic alkali to form thiosulphate and sulphite, but that the reaction might progress with significant rapidity in cold and dilute solutions does not appear to have been suspected. On investigation it was found that in $\frac{N}{10}$ cold, caustic alkaline solution the reaction is almost complete in fifteen minutes, whilst even in $\frac{N}{100}$ solution it proceeds to the extent of about 40 per cent. in the same time. $\frac{N}{10}$ alkali carbonate is about as active as $\frac{N}{100}$ caustic alkali, whilst even $\frac{N}{100}$ sodium carbonate or $\frac{N}{10}$ ammonia are active enough to bring about very serious changes in a solution in fifteen minutes. If it is necessary temporarily to make alkaline a solution containing tetrathionate (*e.g.*, a solution to which iodine and excess of thiosulphate have been successively added), sodium bicarbonate seems to be the only tolerable alkali, and even when this reagent is used it is necessary to keep the temperature low and the cencentration of alkali down to $\frac{N}{2}$, whilst it is a good precaution to saturate the solution with carbon dioxide. G. C. J.

Water Analysis. L. W. Winkler. (*Zeitsch. angew. Chem.*, 1916, **29**, 44-46). —Methods are described for the estimation of dissolved oxygen in waters containing much nitrite and organic matter, and for the estimation of alkalis and of zinc.

Dissolved Oxygen.—The nitrites and organic matter are first destroyed by treating the water with an excess of chlorine, and the oxygen estimation is then carried out as usual; the result is a measure of the dissolved oxygen plus the excess of chlorine. The second portion of the water is now treated with chlorine, and the excess of the latter titrated; the difference between the results of the two estimations gives the quantity of dissolved oxygen. The details of the method are as follows: A bottle of 250 c.c. capacity is filled with water, 10 drops of 50 per cent. sulphuric acid are added, and 1 c.c. of hypochlorite solution (a filtered solution containing 0·5 grm. of calcium hypochlorite and 25 grms. of crystallised sodium sulphate per 100 c.c.) is run in by means of a pipette reaching to the bottom of the bottle. The latter is closed with its stopper, the contents mixed, and, after ten minutes, 1 c.c. of manganous chloride solution and 2 c.c. sodium hydroxide-potassium iodide solution are added, and the mixture is titrated in the usual way with thiosulphate solution. The bottle is then filled a second time with the water, the same quantities as before of sulphuric acid and hypochlorite solution are added, and, after ten minutes, 3 c.c. of the contents of the bottle are withdrawn (to compensate for the 3 c.c. of manganous chloride, etc., added in the first estimation), a crystal of potassium iodide is added, and the mixture titrated with thiosulphate solution. Experiments with distilled water showed that the same result was obtained whether the oxygen was estimated directly or after treatment with chlorine and allowing for the excess of the latter.

Alkalis.—The method described depends, essentially, on the relative insolubility of sodium and potassium chlorides in isobutyl alcohol and on the solubility of magnesium chloride in this solvent (*cf.* ANALYST, 1913, **38**, 531); the potassium is then separated from the mixed chlorides as potassium hydrogen tartrate and estimated by titration. From 200 to 2,000 c.c. of the water, according to the quantity of chlorides and nitrates present, are evaporated to dryness with the addition of 1 grm. of barium carbonate, the dry residue is extracted with five successive quantities of 10 c.c. of hot water, the extracts are filtered, the filtrate is treated with a few c.c. of hydrochloric acid and 1 c.c. of nitric acid, and evaporated in a small glass basin; a few c.c. of hydrochloric acid are then added to the residue and the evaporation is repeated. The residue, while still warm, is rubbed down with 1 c.c. of absolute alcohol and, after some minutes, when the greater part of the alcohol has evaporated, a further 1 c.c. of the same is added, followed by 2 c.c. of freshly-distilled isobutyl alcohol. The alcoholic liquid is decanted through a very small filter, and the residue extracted several times with isobutyl alcohol, using about 10 c.c. of this solvent altogether. The residue in the basin and on the filter is now dissolved in water, the solution treated with a drop of hydrochloric acid, evaporated to dryness, dried at 150° C., and weighed. The residue still contains traces of sulphate and of magnesia, but this error is counterbalanced by the very small quantities of the alkali chlorides which are dissolved by the isobutyl alcohol. To estimate the potassium, the residue of mixed chlorides thus obtained is dissolved in lithium hydrogen tartrate solution (0·5 grm. of lithium carbonate and 2 grms. of tartaric acid are dissolved in 100 c.c. of water, 50 c.c. of 95 per cent. alcohol are added, the solution then shaken with 1 grm. of potassium hydrogen tartrate, and allowed to settle), 10 c.c. of this solution being used for each 0·1 grm. of mixed alkali chlorides; the mixture is stirred and placed aside for two hours, the vessel containing it being covered to prevent evaporation of the alcohol. The precipitated potassium hydrogen tartrate is then collected on a small filter which has been moistened with 50 per cent. alcohol (by vol.) saturated previously with potassium hydrogen tartrate, and this alcoholic solution is used for washing the precipitate; the latter is now dissolved in hot water and the solution titrated with $\frac{N}{10}$ sodium hydroxide solution.

Zinc.—Water which has been passed through galvanised iron pipes frequently contains a small quantity of zinc. The presence of this metal may be detected by the following test, which also affords some indication of the amount of the metal contained in the water: Two quantities of 100 c.c. of the water are each treated with 1 c.c. of 10 per cent. hydrochloric acid and 1 c.c. of hydrogen sulphide solution; to one portion 5 c.c. of a 15 per cent. ammonium acetate solution are then added. If the water contains 10 mgrms., or more, of zinc per litre, a turbidity appears almost at once after the addition of the ammonium acetate; with 5 mgrms. per litre the turbidity is seen after the lapse of thirty seconds, whilst 2 mgrms. of the metal per litre produce a very faint turbidity after one minute. The other portion of the water, which serves for comparison, remains clear for at least five minutes even when the water contains 10 mgrms. of ferrous iron per litre. Ferric salts vitiate the test, since a turbidity, due to the separation of sulphur, forms in each portion of the water.

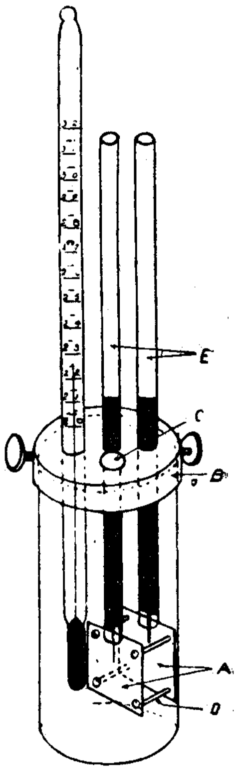
W. P. S.

APPARATUS, ETC.

Collodion Membranes for Ultrafiltration and Pressure Dialysis. G. S. Walpole. (*Biochem. J.*, 1915, 9, 284-297.)—Cylindrical collodion bags, prepared by coating the inside of a test-tube with collodion solution, allowing the solvent to evaporate partially, then adding water and removing the film, have been used for simple ultrafiltration and dialysis experiments; but they lack uniformity, are difficult

of accurate reproduction, and do not withstand any excessive pressure. Much more satisfactory membranes may be made by pouring alcohol-ether collodion solution on to levelled plate glass and then plunging the film, from which the alcohol and ether have more or less evaporated, into water. By keeping as far as possible to constant conditions, these membranes may be described, for purposes of reproduction and as a guide to their properties, by stating the kind of nitrocotton used, the weight of it they contain per sq. cm., and the "wetness," *i.e.*, the ratio of the weight of a piece to its weight when dry. Films containing 8 mgrms. of nitrocotton per sq. cm., and weighing, with contained water, 40 mgrms. per sq. cm., have been found to retain all antigens. They may be used in a filter-press. It is suggested that the channels through the gel structure are of uniform size, and that they are small and numerous.
W. P. S.

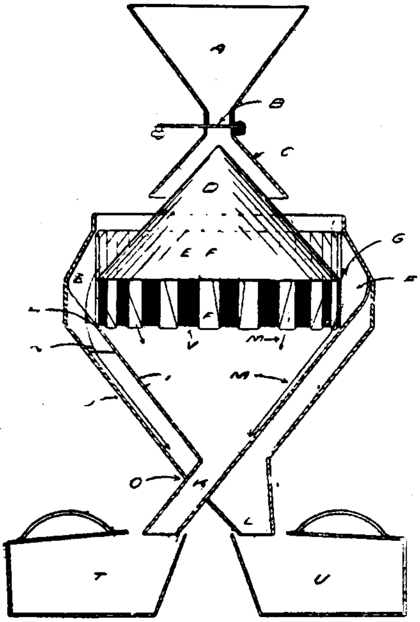
New Dip Electrode. G. J. Van Zoeren. (*J. Amer. Chem. Soc.*, 1916, **38**, 652-653.)—For determining the electrical conductivity of viscous solutions very rigid electrodes are required, so that the distance between them may not be altered by the somewhat vigorous stirring which is usually necessary. The electrode illustrated is sufficiently rigid for determining the conductivity of diluted maple syrup, and also serves well for the determination of the "volumetric lead number" of such syrups, for which purpose horizontal electrodes are unsuitable, inasmuch as the precipitate settles on them. The electrode is placed in a hard glass cylinder 6·5 cms. high by 3·7 cms. in diameter, open at both ends. One end is fitted with a vulcanite cap, *B*, fastened to the cylinder by means of set screws. A thermometer and two glass tubes pass through the cap. The tube *C* allows the air to escape when the electrode is immersed in the solution. The platinum connecting wires, which are welded to the electrodes, are protected by being sealed in the tubes, E, in which mercury is placed to connect the electrodes with the bridge leads. The electrodes, *A*, are of No. 30 B. and S. gauge platinum foil, 2 × 2 cm. squares, set 1 cm. apart. They are firmly fastened together by means of four glass pins as shown, *D*. The pins have small enlargements 2 mms. from each end, which serve as collars, and prevent the electrodes from being crowded together. The ends of the pins pass through the electrodes and are clinched. In this way they are firmly held in place. An ordinary 100 c.c. beaker may be used to complete the cell. G. C. J.



Possible Source of Error in Colorimetric Observations. J. H. Long. (*J. Amer. Chem. Soc.*, 1916, **38**, 716-718.)—The prisms of the Dubosc colorimeter seem to be fastened in their brass sockets by means of a wax which is too soft for the purpose, and which softens enough in summer in Illinois (33° C.) to allow the prisms to drop down a little. Considerable errors may arise if this happens and is

not detected. Such instruments should, therefore, be kept away from heat radiators and sunny positions, and the zero should be controlled whenever the instrument is used.
G. C. J.

Device for Sampling Grain, Seeds, and other Material. E. G. Boerner. (*U.S. Dept. of Agric. Bull.*, No. 287, 1915; through *J. of Agric. Intell. and Plant Diseases*, 1915, **6**, 1688.)—The material is placed in the hopper *A*, communicating through the valve *B* and the shield *C* with the cone *D*, which is provided at its base with a series of separated ducts, *F*, having uniform spaces, *E*, between them. Adjusted to the bottom of the ducts at *H* is a funnel *I*, having a spout *K* at its bottom part. When the valve *B* is opened the material spreads on the cone *D*; then, entering the ducts, passes through them and the inner funnel, and issues through the spout at the bottom into the receptacle *T*, while all the material which enters the spaces between the ducts falls into the outer funnel and thence into the other container *U*. As the widths of the ducts are equal to the spaces between the ducts, two equal portions of the original material are obtained. A very small sample of the original material can be obtained by adjusting the proportion between the widths of the ducts and the spaces between them; or the material in either *T* or *U* may be again put through the apparatus as often as may be required.



H. F. E. H.

REPORT.

Report on the Work of Inspectors of Foods for the Year 1914-15. A. W. J. MacFadden. (Extract from the Annual Report of the Medical Officer of the Local Government Board.)—Since the commencement of the war the inspectors have been continuously engaged in supervising the preparation of Army food supplies. Whilst the quality of the materials used in the preparation of the foods was generally found to be satisfactory, exception had, in many cases, to be taken regarding the conditions under which the food was being prepared. Steps were taken, where needed, to insure perfect cleanliness in the factories; in some cases the personal cleanliness of the workers left much to be desired. Although great improvement has been effected, it is to be feared that the standards of cleanliness which have been secured will not be maintained by many of the firms when they are no longer engaged on War Department work, and this emphasises the desirability, to which reference was made in last year's report (*cf.* ANALYST, 1915, **40**, 178), of conferring on sanitary authorities more stringent powers for securing that food for sale for human consumption should be prepared under hygienic conditions.

The following reports were published in the year under review: I. On the Use of Proprietary Foods for Infant Feeding, by F. J. H. Coutts; II. On the Analysis and Composition of Some Proprietary Foods for Infants, by J. L. Baker; III. On the Effects of Certain Condensing and Drying Processes used in the Preservation of Milk, upon its Bacterial Contents, by S. Delépine. Baker's report (*cf.* ANALYST, 1914, **39**, 464) showed that a large proportion of the foods contained considerable quantities of starch, that this existed in a practically unchanged condition, and that the majority of the foods examined contained a very low percentage of fats. In many cases the statements in printed matter accompanying the packets of the foods were inaccurate and misleading, and the exaggerated claims made as to the value of the foods were not justified. Coutts pointed out (*cf.* ANALYST, 1914, **39**, 464) that many of the foods, even when prepared with milk and water according to the directions given on the labels, showed a composition differing markedly from that of human milk or cow's milk. He came to the conclusion that foods containing unchanged starch or starch altered only by heating should not be given to infants under seven months of age, except on the advice and under the supervision of a medical man. The report also dealt with the inaccurate statements made in labels or advertisements as to proprietary foods for infants, and discussed the possible methods of exercising control over these articles of food. The results of experiments recorded in Delépine's report (*cf.* ANALYST, 1915, **40**, 176) indicate that the usual process of preparing sweetened condensed milk is effective in destroying the virulence of the tubercle bacillus, and that this process and the other methods of drying milk which were investigated resulted in a considerable destruction of the bacteria present in the original milk. Attention was drawn to the necessity of preventing recontamination of the condensed or dried milk during the final stages of the processes.

The Local Government Board has received several representations that hardships have been caused by the administration of the Milk and Cream Regulations, 1912, as far as cream is concerned, and numerous conferences have been had with the trade. The President has stated in the House of Commons (May 21, 1914) that, in view of the somewhat contradictory evidence available as to the effect of boric acid on health, he had decided to nominate a small expert committee to investigate the question afresh; owing, however, to the war and to the absence on military service of several of the proposed members, it has not so far been possible to proceed with the appointment of the committee. The President also expressed the opinion that, pending the completion of this investigation, it would be inadvisable for local authorities to institute proceedings in respect of boric acid in cream except in cases where the amount of preservative exceeded the amount declared on the label or where the amount so declared was clearly excessive. Inquiry was made by a public analyst whether "cream" as defined in the Regulations included the article known as "clotted" or "Devonshire" cream. The Board replied that such cream did come within the definition of "cream."

Several outbreaks of bacterial food poisoning were brought to the Board's notice during the year. The two most serious of these had their origin in milk; they occurred in July at Withnell and Chorley and in October and November at Newcastle-under-Lyme and Stoke-on-Trent. At the former places 329 persons were affected and at the latter 375; two deaths occurred in each case. The source of the outbreak in both instances was traced to a cow which had been ailing for several days antecedent to the outbreak. A food poisoning organism allied to the *Bacillus enteritidis* of Gaertner was isolated from the organs of the fatal cases available for examination, as well as from material derived from the affected cows. An outbreak involving eighty-five persons occurred in May at Cambridge, the alleged cause being fresh cooked salmon. A few fragments of the salmon, all that remained over, were examined bacteriologically, but no organism such as is commonly associated with outbreaks of food poisoning was discovered. Three fish were served at the dinner, and, as many persons other than those affected consumed some of the fish, it is possible that not all of the latter were infected; this hypothesis is confirmed by the result of the bacteriological analysis. Boiled pressed pork was supposed to be the cause of food poisoning at Oswestry; eighty-six persons were affected, and one died. An organism of the *enteritidis* group was isolated from the organs of the fatal case as well as from the fæces of two of the sufferers. Owing to the delay in reporting the outbreak, none of the original material which was suspected of having caused illness could be obtained for examination. W. P. S.

LAW REPORT.

Boric Acid in Cream. Haigh *v.* Aerated Bread Company, Limited.

(*Before* Mr. Justice Ridley, Mr. Justice Bray *and* Mr. Justice Avory.) (*Times*, April 6, 1916.)—The Court heard an appeal on a case stated by Mr. Mead, the magistrate sitting at Marlborough Street Police Court. On August 13, 1915, and November 19, 1915, an information was heard by the magistrate which was preferred by the appellant, an inspector under the Sale of Food and Drugs Acts, 1875 to 1907, against the Aerated Bread Company. The company was charged with having sold to him on June 28, 1915, cream mixed with 0·34 per cent. by weight of boric acid (H_3BO_3), equivalent to 23·8 grains of boric acid per pound of cream, so as to render this article of food injurious to health. The magistrate dismissed the information, and this was an appeal from his decision.

The appellant asked for, and was served with, a shillingsworth of preserved cream. On analysis the cream was found to contain boric acid in the percentage stated above. The magistrate found that cream containing boric acid in any quantity was injurious to health, particularly to the health of children and adults who were invalids, and was harmful to persons suffering from kidney disease. He also found that preserved cream—that is, a mixture of cream and boric acid preservative—was sold as a well-known commodity.

The appellant contended that the respondents had sold an article of food mixed with an ingredient rendering it injurious to health, and had thereby committed an offence under Section 3 of the Sale of Food and Drugs Act, 1875, notwithstanding that the respondents had sold the cream as preserved cream.

The respondents contended that the appellant had asked for preserved cream and had received it, and that as preserved cream was a mixed article of food there had been no mixing within the meaning of Section 3 of the Act of 1875. They referred to the case of Cullen *v.* McNair (24 *The Times* Law Reports, 692).

The magistrate held that no offence had been committed, as the cream had been bought and sold as preserved cream, and that he was bound by Cullen *v.* McNair, and he dismissed the information.

Mr. McCall, K.C., and Mr. D. C. Bartley appeared for the appellant; and Mr. W. E. Bousfield for the respondents.

Mr. JUSTICE RIDLEY, in giving judgment, said that the magistrate had come to an erroneous decision in consequence of following what was a mere *obiter dictum* of Mr. Justice Darling in the case of Cullen *v.* McNair (*supra*). Under Section 3 of the Act, if it were shown that an article of food such as the cream had been mixed with an ingredient, so as to render it injurious to health, there must be a conviction on such a charge, unless the defence had established under Section 5 that the person liable to be convicted did not know of its being so mixed, and could not with reasonable diligence have obtained that knowledge. The magistrate had not distinguished Section 3 from Section 6. He ought to have convicted the respondents, and the case must go back to the magistrate with a direction to convict.

Mr. JUSTICE BRAY and Mr. JUSTICE AVORY delivered judgments to the same effect.

Solicitors, Messrs. Allen and Son for the appellant; Messrs. Bristows, Cooke, and Carpmael for the respondents.

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REVIEWS.

VOLUMETRIC ANALYSIS. By A. J. BERRY, M.A. 1915. Cambridge University Press. Price 6s. 6d. net.

THIS little book of about 140 pages is essentially a classbook for students, and not a technical work for the industrial laboratory. The methods described are chosen to illustrate the uses of the most generally employed volumetric solutions—potassium permanganate and bichromate, iodine, sodium thiosulphate, silver nitrate, ammonium thiocyanate, acid and alkali—each of these forming the subject of a separate chapter. A few special methods, not readily classified, are dealt with in a further chapter; and there are others in which the application of volumetric methods to the analysis of mixtures is illustrated, and concrete examples are worked out. Thus, although a fair number of methods in common use are described, and in more theoretical detail than is usual in technical works, only a selection of processes is given, and by no means a complete list of methods in general use.

The descriptions, as a rule, are clear and to the point. In some cases much detail is given of processes of little practical value, and, on the other hand, many well-known and widely used methods are not mentioned. A case in point occurs on p. 16, where it is stated that nitric acid is inadmissible for volumetric work with permanganate, although in the analysis of steel one of the most accurate methods of estimating manganese (not described in this book) depends upon its oxidation by sodium bismuthate in nitric acid solution, and after excess of ferrous sulphate has been added the surplus is titrated by means of standard potassium permanganate. Again, on p. 21, the reduction of iron by means of H_2S or SO_2, previous to titration, is described, though such methods are of no practical value. The use of palladium hydrogen as a reducing agent is not mentioned. The standardisation of acids by means of metallic sodium, as described on p. 70, will not appeal to the practical analyst when so many methods of greater simplicity and accuracy are available.

Calibration of apparatus and the proper method of using measuring vessels are only touched upon very briefly. A short chapter is devoted to the theory of indicators. An abridged table of atomic weights, a short table of formula values, a hardness table, tables showing the relationship between specific gravity and percentage composition of hydrochloric acid and caustic soda solutions, and short tables of logarithms and anti-logarithms complete the work. A student who works conscientiously through this book should obtain a good knowledge of the principles of the subject.

L. ARCHBUTT.

WATER PURIFICATION PLANTS AND THEIR OPERATION. By MILTON F. STEIN, Assistant Engineer, Cleveland Filtration Plant. New York: John Wiley and Sons (Inc.). London: Chapman and Hall, Ltd., 1915. Pp. viii + 258. Price 10s. 6d. net.

This book gives a very comprehensive account of modern methods of water purification as practised in America. The preface states that the book has been written for the non-technical operator and for the qualified chemist on first taking charge. This being so, the inclusion of chapters on chemical and bacteriological tests, with detailed directions for preparing standard solutions and culture media, seems unnecessary. Such important work should not be put into the hands of a non-technical man, as any works not possessing its own chemist should have a consultant to whom the work can be sent. On the other hand, for the trained chemist and bacteriologist such information is superfluous, as it will be already in his possession. Moreover, some of the directions given seem rather peculiar, as in preparing a standard solution of sodium carbonate, when dissolving the weighed amount in a small quantity of water in a beaker, "*no* stirring rod can be introduced to aid solution," and presumably also no rod can be used in transferring the liquid from the beaker to the graduated flask.

The other chapters in the book are on the impurities present in water (in which the statement occurs that fungi "are parasitic and can only live on organic matter"), types of purification plants, interpretation of tests, coagulation and sterilisation, softening, sedimentation basins, and general management. It is interesting to note that in slow sand filtration the tendency is towards increased rates of filtration, and the most recent plant has been designed for double the usual rate. Sterilisation, too, of the slow sand filter effluent is becoming customary. It appears that the process of coagulation and sedimentation, without filtration, which was never much in vogue, is gradually dying out, and mechanical filters are being added to the plant in most cases.

The foregoing chapters are packed with information on all points, although the size of the book has necessitated some of the explanations being extremely concise, as the author recognises. The chapters, however, are admirably suited for the young chemist on first taking up duty at a water-works, and also are of interest to the general reader. Especially good and full is the account of coagulation, and in the explanations of this phenomenon and of filtration it is pleasing to note that the importance of colloids and of adsorption has been fully recognised.

No expense has been spared in publication, many plans, sketches, photographs, and charts being included in the pages. The index is rather amateurish.

J. H. JOHNSTON.