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A Quantitative Study of the Limitations of the Reaction between Ammonia and Sodium Hypobromite.

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WHEN ammonia is added to an excess of an alkaline solution of sodium hypobromite at the ordinary temperature it is decomposed according to the following well-known equation:— $2\text{NH}_3 + 3\text{NaOBr} = 3\text{Na Br} + 3\text{H}_2\text{O} + \text{N}_2$.

That this reaction is not the only one that occurs has not been recognised by certain investigators, since it has been suggested as the basis of volumetric methods for the estimation of ammonia. The majority of these methods depend on the decomposition of ammonia when a solution is added to an excess of standard sodium hypobromite, the excess of the latter being determined after acidification and addition of potassium iodide, by titrating the liberated iodine with a standard solution of sodium thiosulphate. The reaction is represented by the following equation:— $\text{NaOBr} + 2\text{HI} = \text{Na Br} + \text{H}_2\text{O} + \text{I}_2$.

Krocker and Dietrich (*Zeitsch. anal. Chem.*, 1865, 3, 65), were the first investigators to apply this reaction as the basis of a quantitative titrimetric method. They employed a solution of sodium hypochlorite containing bromine. The method, however, was re-investigated by F. Mohr (*Lehrbuch Titrimethode*, 1896, 7, 406), who found that it gave uncertain results.

J. Thiele (*Annalen*, 1893, 273, 160), while studying the reaction between ammonia and hypochlorites, found that the nitrogen evolved falls below that required by the above equation and that certain secondary reactions occur leading to the formation of reducing substances, viz. hydrazine and hydroxylamine, according to the following equations:—



A. Wohl (*Ber.*, 1903, 36 1417), who employed the reaction for the gasometric estimation of ammonia, found that the total nitrogen was not given off, the deficit being about 2.5 per cent. of the theoretical amount.

A little later E. Rupp and E. Rossler (*Archiv. Pharm.*, 1905, 243, 104), published their work on the titrimetric estimation of ammonium salts with alkali

hypobromite. They pointed out that Mohr had used solutions of hypobromite of too great alkalinity, and they contended that accurate estimations by the method were possible if the alkalinity were reduced to the lowest possible limit. They recommended the preparation of hypobromite solution as follows:—Sodium hydroxide (10 grms.) is dissolved in water (500 c.c.), and to it is gradually added, with constant stirring, bromine (17 grms.). This hypobromite solution has been employed by subsequent investigators on this subject, and it was also employed by us for the purposes of the present investigation. Independently of the work of Rupp and Rossler, P. Artmann and A. Skrabal (*Zeitsch. anal. Chem.*, 1907, **46**, 5), showed that this reaction could be employed for the accurate estimation of ammonia and more recently Willard and Cake (*J. Amer. Chem. Soc.*, 1920, **42**, 2646), have used it for the estimation of ammonia produced in the Kjeldahl process. It is obvious that this application of the reaction would be in many respects a desideratum, since it would obviate distillation. It would be necessary, however, that any titrimetric method based on the reaction should be capable of measuring nitrogen to 0.01 mgrm.

Although this degree of accuracy, together with our requirement of working with quantities of nitrogen from 3 to 4 mgrms. may seem unnecessary for ordinary analytical work, we had in view the application of this reaction to a biochemical investigation wherein these two conditions were essential if the results were to be of any value. It may be said that there is hardly a volumetric method which can give anything like the accuracy of 1/100 mgrm. The fact is, however (and with this we were particularly struck at the commencement of the work), that this method seemed capable of accuracy apparently even beyond this limit. At first we were only able to judge this fact from the results of duplicate titrations while working with N/100 iodine solution which as a rule agreed within 0.2 c.c. (0.1 c.c. of N/100 iodine solution = 0.00466 mgrm. Nitrogen). Induced by this observation we have submitted the action of hypobromite on ammonia to a searching investigation, bearing in mind the observations of other workers. The results of our experiments prove that no method on this basis is sufficiently accurate for the estimation of minute quantities of ammonia.

Willard and Cake (*loc. cit.*), who worked with amounts of substances corresponding to 10 to 15 mgrms. of nitrogen, recommend the following procedure, adhering to the directions of Rupp and Rossler and of Artmann and Skrabal. The ammonia solution is decomposed with 10 c.c. of 0.6 N sodium hypobromite solution, the amount being so adjusted that not more than two thirds of it is reduced by the ammonia. The mixture is allowed to remain aside for 5 minutes for the reaction to complete. Five grms. of potassium iodide and 6 c.c. of concentrated hydrochloric acid (Sp. gr., 1, 19) are then added, and the liberated iodine titrated with 0.2N sodium thiosulphate. There are several points in this procedure to which objection may be taken. In the first place the use of solutions as strong as 0.6N sodium hypobromite and 0.2 N sodium thiosulphate, when working with quantities, of nitrogen varying between 10 to 15 mgrms., is open to criticism, since the necessary measurement of a small volume of such a strong hypobromite solution entails a

considerable error, sufficient to impair the accuracy of the results, calculated as a percentage of nitrogen in any substance beyond the permissible limits. Secondly, the necessity of using 5·grms. of potassium iodide for every titration is undesirable. In our experiments the following modified procedure was adopted: To determine the excess of sodium hypobromite the method employed was based on the well-known reaction between hypohalogenites and arsenious acid— $2\text{NaOBr} + \text{As}_2\text{O}_3 = \text{As}_2\text{O}_5 + 2\text{Na Br}$. To the solution containing an excess of sodium hypobromite is added an approximately 2 per cent. solution of pure sodium bicarbonate in excess, and subsequently a standard solution of sodium arsenite in excess. The solution is then neutralised with dilute sulphuric acid, methyl orange being used as indicator. It is then again rendered alkaline with 2 per cent. sodium bicarbonate solution, and the excess of the arsenite titrated with standard iodine solution, with the use of starch as indicator. The object of neutralisation is to remove any normal carbonate formed by the reaction of sodium bicarbonate with the free alkali in the hypobromite, before titrating with iodine.

The accuracy of this method of estimating hypobromite is beyond all doubt, and it gives results well in agreement with those obtained when acidified potassium iodide is added to the hypobromite and the liberated iodine titrated with thio-sulphate.

We have found it possible to use much more dilute solutions of sodium hypobromite than those employed by Willard and Cake, and our results show that the reaction between ammonia and sodium hypobromite is not affected by employing solutions of this salt 20 times more dilute than those recommended by the authors referred to. Consequently, the errors in measurement are insignificant. The strength of the iodine solution used to titrate the excess of the arsenite was $N/50$ or $N/100$, whichever was convenient, and perfectly sharp end-points were obtained in each case. The arsenite solution employed was $N/10$, $N/50$, or $N/100$, whichever was convenient to employ.

In table I. are given the results of titrations in which a constant quantity of sodium hypobromite and a constant amount of nitrogen were taken, but the strengths of sodium hypobromite used in each titration were different. For the standard nitrogen solution an accurately prepared $N/10$ solution of ammonium chloride was used, for quantities of nitrogen up to 7 mgrms. and for quantities below 7 mgrms. $N/50$ ammonium chloride solution was used.

TABLE I.

Strength of NaOBr used	C.c. of NaOBr used	C.c. of $N/10$ As_2O_3	C.c. of $N/50$ Iodine required	N_2 taken mgrms.	N_2 found mgrms.
0·7090 <i>N</i>	5	25	28·7	7·00	7·555
0·1772 <i>N</i>	20	„	28·8	„	7·565
0·1418 <i>N</i>	25	„	28·8	„	7·565
0·0886 <i>N</i>	40	„	28·7	„	7·555
0·0709 <i>N</i>	50	„	28·7	„	7·555
0·0354 <i>N</i>	100	„	29·0	„	7·588
0·0283 <i>N</i>	125	„	28·3	„	7·518

In table II. are given a series of results of titrations with different quantities of nitrogen. 0.028*N* sodium hypobromite was employed throughout this work. It will be apparent that these results are consistently high, and that the percentage error varies directly with the excess of sodium hypobromite used. The greater the excess used, the greater the percentage error.

TABLE II.

Amount of 0.028 <i>N</i> NaOBr used c.c.	Amount of <i>N</i> /10 As ₂ O ₃ c.c.	<i>N</i> /50 Iodine c.c.	N ₂ taken mgrms.	N ₂ found mgrms.	Percentage error
200	40	74.8	14.0	14.446	3.18
200	40	60.6	12.6	13.104	4.00
200	40	46.4	11.2	11.800	5.35
150	30	49.8	9.8	10.240	4.49
120	25	57.1	9.1	9.329	2.51
120	25	50.4	8.4	8.705	3.63
100	20	38.6	7.0	7.325	4.65
80	20	51.5	5.6	5.918	5.67
60	15	37.6	4.2	4.343	3.33
40	15	49.7	2.8	2.861	2.14

That our conclusions are well founded is confirmed by examining the results in table III. In these titrations the nitrogen taken was kept constant but the excess of hypobromite used was varied.

TABLE III.

Amount of 0.028 <i>N</i> NaOBr used c.c.	Number of c.c. in excess of theoretical	Excess per cent.	N ₂ taken mgrms.	N ₂ found mgrms.	Percentage error
110.0	56.35	105.00	7.0	7.44	6.28
100.0	46.35	86.39	"	7.37	5.29
80.0	26.35	49.11	"	7.29	4.14
70.0	16.35	30.47	"	7.21	3.00
65.0	11.35	21.15	"	7.18	2.65
60.0	6.35	11.83	"	7.13	1.85
55.0	28.175	105.00	3.5	3.704	5.82
50.0	23.175	86.39	"	3.672	4.91
40.0	13.175	49.11	"	3.644	4.11
35.0	8.175	30.47	"	3.616	3.31
32.5	5.675	21.15	"	3.590	2.57
30.0	3.175	11.83	"	3.560	1.71

Finally, to obtain an explanation of these high results, we have made a careful examination of the products of the reaction, and after having examined every reagent used for nitrites and nitrates, we find that nitrites and nitrates invariably are formed in traces as by-products of the reaction between ammonia and hypobromite.

We may point out that it is essential when titrating ammonia with alkaline hypobromite that the ammonia solution should be added to the excess of the hypobromite and not in the reverse order, the plan adopted by Willard and Cake (*loc. cit.*), since when their course is adopted the results are still more irregular, owing to the fact that excess of ammonia and low temperature bring about an under-oxidation leading to the formation of hydrazine. This particular phase of the reaction has been thoroughly studied by Raschig (*Verh. Ges. deut. Naturforsch. Aertze*, 1907, 2 [i], 120-123) whose method is now employed for the preparation of hydrazine (D.R.P., 198307).

Taking into account the whole of our experiments and the observations of other workers cited, we submit that we are justified in advocating the rejection of the hypobromite method of estimating ammonia when the greatest possible accuracy is required.

In conclusion, we have to express our thanks to Professor A. R. Ling, for his interest and advice. We have also to thank Mr. B. S. Brooks for his assistance.

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Notes on a Method for Testing the Accuracy of Babcock Cream and Milk Test Bottles.

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WHEN the Union's Dairy Industry Act, No. 16, of 1918, came into force the officers of the Division of Chemistry, Union of S. Africa, were entrusted with the task of testing dairy glassware for accuracy of measurement and accuracy of the per cent. scale, as is provided in No. 14 (1) of the regulations under the above-mentioned Act. At a later date regulations under Section 20 of the Dairy Industry Act, 1918, were published (Government Notice No. 600 of 1919), prescribing the size and the standards of graduation of dairy glassware. The standards for Babcock glassware are laid down in Nos. 6 and 7 of the last mentioned regulations, which read as follows:

6. (i) The limit of graduation for Babcock glassware shall be the true cubic centimetre.
- (ii) In the case of test bottles the capacity of each per cent. on the scale shall be two-tenths (0.20) of one cubic centimetre.
- (iii) With pipette the delivery shall be the intent of the graduation and the graduation shall read with the bottom of the meniscus in line with the mark.

BABCOCK CREAM BOTTLES.—In Section 7 the size of the cream test bottles is prescribed, and Section 7 (IV.) stipulates that the graduated scale shall extend

over at least $63\frac{1}{2}$ mm. of the neck of the bottle and correspond, when 18 grms. of cream are used for the test, to 30 per cent. milk fat, and also that "The error at any point of the scale shall not exceed one true scale division, viz. the equivalent of one-half of 1 per cent. of milk fat." Regulation 6 (11) provides that each per cent. on the scale shall be two-tenths of one c.c. Therefore the error at any point of the scale must not exceed 0.10 c.c.

BABCOCK MILK BOTTLES, SECTION (8).—Section 8 (IV.) states: "The graduated scale shall extend over at least $63\frac{1}{2}$ mm. of the neck of the bottle, and correspond, when 18 grms. of milk are used for the test, to 8 per cent. of milk fat"; and, again, "The error at any point on the scale shall not exceed one true scale division, viz. the equivalent of one-tenth of 1 per cent. of milk fat." In this case the error allowed is therefore 0.02 c.c.

VARIOUS METHODS FOR TESTING THE ACCURACY OF BABCOCK TEST BOTTLES.—The following methods have been proposed and can be found in text books—see for example, "Modern Methods of testing Milk and Milk Products," by Lucius L. van Slyke.

(i) *Testing with a Burette.*—In this method the bottle to be tested is filled with water to the zero mark. After any drops of water adhering to the sides of the neck have been removed with a piece of blotting paper, water is run in from a burette accurately graduated to 0.1 c.c. until the meniscus in the neck of the bottle reads an exact percentage. The reading on the burette and on the bottle is recorded. In the same way, by adding more water from the burette and taking the readings, several points along the neck of the bottle are found. Each per cent. graduated on the bottle should be equal to 0.2 c.c.

The objection to this method is that two errors are introduced—one in adjusting the meniscus in the neck of the bottle to read an exact percentage, and the other in reading the burette. The error in reading a 50 c.c. burette graduated to 0.10 c.c. is somewhere in the neighbourhood of 0.02 c.c., and, in addition, the error allowed by the National Physical Laboratory and the Reichanstalt, where burettes are standardised, is 0.05 c.c. for total contents of the burette and 0.025 c.c. for a volume less than 25 c.c. There are, of course, burettes of smaller capacity which are more accurate, but the error in reading the bottle itself is so great that there would be no advantage in using a very accurate burette. Milk bottles cannot be tested with a burette and water, firstly, because the burette is not accurate enough; and, secondly, because it is difficult to introduce water into a vessel with such a narrow neck without wetting the sides of the neck above the meniscus.

(2) *The Plunger Method.*—In this method a brass plunger is used. The plunger is divided into two parts, each part displacing, in the case of the milk bottle tester, exactly 0.8 c.c. or 4 per cent. on the scale. The bottle is filled with water or some other liquid to the zero mark, and the first part of the plunger is slowly immersed in the liquid until the upper surface of the liquid rises to half-way between the two parts of the plunger. The upper surface of the liquid should now be on the 4 per cent. mark. The second part of the plunger is now

immersed, and the reading on the bottle should then be on the 8 per cent. mark. There are also plungers for testing cream bottles.

This is a quick and easy method, but unfortunately it only gives two readings. If a bottle is out between the 0 and, say, 2 per cent. marks on the negative side, and if this negative error is compensated by a positive error between the 2 and the 4 per cent. mark, the plunger will only indicate that the bottle is correct for the total 4 per cent.

There are also plungers giving the 5 per cent. and 10 per cent. readings, so that by using both plungers for an 8 per cent. bottle the readings for 4, 5 and 8 per cent. can be tested. This would involve the tedious operation of adjusting the liquid in the bottle to zero for a second time and also of drying out the neck.

(3) *Testing with Mercury.*—Measure out 1.6 c.c. of mercury from a burette, run the mercury into the bottle to be tested. Now push a close-fitting cork into the neck of the bottle so that the end of the cork coincides with the top line of graduation. On turning the bottle upside down, the mercury should exactly fill the space between the 0 and the 10 per cent. graduation on the neck. The same mercury can be used for the next bottle to be tested.

Here again the objection is that only one reading can be obtained. These bottles are graduated on the assumption that the necks are of uniform calibre. The first and last graduations are determined experimentally, and then the intervening space is equally divided into the required number of divisions with a dividing machine.* It is therefore desirable that several points along the scale be tested as well as the total reading.

(4) *Weighing the Mercury.*—The bottle is filled with mercury to the zero mark or to one of the graduations above the zero, and weighed. Mercury is now run in from a fine jet, and the meniscus adjusted to any point on the scale—say, to the 3 per cent. mark. The bottle is again weighed; the difference in weight is the weight of the mercury which occupies the space between the first reading and the 3 per cent. mark. The volume is then calculated. More mercury is run in, and the weight again taken until several points along the neck have been found. This method is an accurate one, and can also be used for cream bottles.

When first we commenced testing glassware in this Division, this method was adopted for testing Babcock milk and cream bottles, but later on the cream bottles were tested with the aid of a burette and water. The methods were found to be accurate, but they were much too slow. When testing cream bottles with the burette and water the writer was unable to do more than about 45 to 50 per day. The milk bottles took even longer, and only about a dozen could be tested per day. Since we had at that time over 2400 cream bottles, 700 milk bottles, and about 900 pipettes to test, there was no prospect of finishing the work within any reasonable time; moreover bottles were continually arriving from various firms, whilst other firms were already complaining about the delay. The following method was then adopted with considerable success.

(5) *Glass Rod Method.*—The method is based on the plunger idea. Glass

* See *Milk and its Products*, by Wing.

rods of various lengths are used as plungers. The rods are made in the following way:

(a) FOR CREAM BOTTLES.—Take a piece of glass rod about 7 mm. in diameter and of a suitable length, cut this into pieces from 30 mm. to 60 mm. in length. The one end of the rod is made to taper slightly by rounding it off in a flame. This is to prevent the splash of the liquid on to the sides of the neck of the bottle when the rods are dropped into the liquid. The specific gravity of the glass is now determined by means of an ordinary specific gravity bottle, about 8 or 9 of the rods being used for one determination. The average of several determinations gave the specific gravity of the glass as 2.472 (sp. gr. of water = 1 at 4° C.). The rods are then weighed separately and the volumes calculated.

Weight of Rod	Specific Gravity	Volume in c.c.	Volume in Babcock half per cent. Divisions
2.5242	2.472	1.0212	10.2112

In this way twenty-two rods were obtained, weighing from 2 to about 6 grms., their volumes being from 1 c.c. to about 2.3 c.c. The next step was to grind the rods down until each of them represented a volume corresponding to an exact number of Babcock divisions, or, better still, to Babcock half per cent. divisions, since the error allowed is 0.5 per cent. on the bottle. Taking the example given above; the rod is equal in volume to 5.1056 Babcock divisions, or 10.2112 half per cent. divisions. The rod was ground down on the end not rounded off in the flame, until its volume was equal to 10.00 half per cent. Babcock divisions. This was done by grinding the rod on a sandstone and thus working it down to the required weight.

The required weight is naturally the required volume in c.c. multiplied by the specific gravity. One c.c. is equal to 10 Babcock half per cent. divisions, therefore the required weight = $\frac{\text{required Babcock } \frac{1}{2} \text{ per cent. Divisions}}{10} \times \text{specific gravity}$.

The following table gives the weight, volume, etc., of two of the rods prepared according to the above method, and used for testing Babcock cream bottles.

No of Rod.	Weight. Grms.	Required Weight. Grms.	Volume in Babcock $\frac{1}{2}$ per cent. Divisions.	Required Volume in Babcock $\frac{1}{2}$ per cent. Divisions.
1	2.4703	2.4720	9.993	10.000
3	2.7192	2.7192	11.000	11.000

Owing to the enormous stock of glassware in the Union which did not comply with the size laid down in the regulations, those sections dealing with actual size were waived for a time, with the result that we received Babcock cream bottles reading 30, 40 and 50 per cent. when 18 grms. of cream are used.

The bottle is filled to the zero mark on the neck with water. If, say, four readings are required, four rods are selected, which together will give a total reading of rather less than the total reading on the bottle. These rods are dropped into the bottle one after another, and the reading on the bottle as well as the volume of the rod noted.

The following tests carried out on 30 per cent. Babcock cream bottles show a comparison between the glass rod method, and testing with a burette. A standard 10 c.c. dropping pipette previously tested by weighing was used instead of a burette.

All the figures given in the table below represent Babcock $\frac{1}{2}$ per cent. Divisions:

Bottle	Burette Testing			Glass Rod Testing		
	Bottle Reading	Burette Reading	Error	Bottle Reading	Volume of Rod	Error
A.	0-10.1	0-10.0	+0.1	0-10.1	10.0	+0.1
	10.1-21.1	10.0-21.0	± 0.0	10.1-21.2	11.0	+0.1
	21.1-39.5	21.0-39.0	+0.4	21.2-39.6	18.0	+0.4
	39.5-58.8	39.0-58.0	+0.3	39.6-58.9	19.0	+0.3
		Total error	0.8		Total error	+0.9
B.	0-12.0	0-12.0	± 0.0	0-12.0	12	± 0.0
	12.0-29.0	12.0-29.0	± 0.0	12.0-29.0	17	± 0.0
	29.0-56.9	29.0-56.4	+0.5	29.0-43.0	14	± 0.0
	—	—	—	43.0-58.3	15	+0.3
		Total error	+0.5		Total error	+0.3
C.	0-13.0	0-12.7	+0.3	0-12	12	± 0.0
	13.0-31.0	12.7-30.7	± 0.0	12-31.2	19	+0.2
	31.0-46.0	30.7-45.7	± 0.0	31.2-46.3	15	+0.1
	46.0-60.0	45.7-59.5	+0.2	46.3-60.5	14	+0.2
		Total error	+0.5		Total error	+0.5
D.	0-11.1	10-21.0	+0.1	0.0-11.0	11	± 0.0
	11.1-27.5	21.0-37.0	+0.4	11.0-27.4	16	+0.4
	27.5-43.5	37.0-53.0	± 0.0	27.4-43.3	16	-0.1
	43.5-57.2	53.0-67.0	-0.3	43.3-57.2	14	-0.1
		Total error	+0.2		Total error	+0.2
E.	0-11.1	10-21	+0.1	0.0-11.1	11	+0.1
	11.1-23.1	21.0-33.0	± 0.0	11.1-23.2	12	+0.1
	23.1-40.2	33.0-50.0	+0.1	23.2-40.2	17	± 0.0
	40.2-58.4	50.0-68.0	+0.2	40.2-58.5	18	+0.3
		Total error	+0.4		Total error	+0.5

In this paper the error found when testing a bottle is shown as a + error, when the reading on the neck of the bottle is too high. It must be pointed out, though, that in such a case the neck of the bottle is actually too small.

These five bottles all pass the test by both methods, since the error allowed is one $\frac{1}{2}$ per cent. division.

The error in the specific gravity determination is about 1 in 2900. The error due to the glass rod not having been ground down to exactly the required weight can easily be kept below half the specific gravity determination error. The total error of the method is about 1 in 1900. Using four rods in testing a 40 per cent. cream bottle, each of a volume equivalent to about 20 half per cent. Babcock divisions, and assuming that each rod introduces the maximum error, and, moreover, that all go in the same way, then the total error will be $\frac{4}{1900}$ of a half per cent.

Babcock scale division, or less than 0.05 of a half per cent. Babcock scale division. This error can be considered as negligible in view of the fact that it is impossible to read a 40 per cent. Babcock bottle (18 grms.) as we received them in this office to within less than 0.4 of a half per cent. Babcock scale division. The reason for the great error in reading the bottle is that the scale on the neck is only about 63 mm. long, and this distance is divided into 40 parts. Each division is equal to 1 per cent. and the lines are only about $1\frac{1}{2}$ mm. apart. It is clear, therefore, that it would be impossible to read this to a greater accuracy than 0.2 per cent., which is 0.4 of a half per cent. Babcock scale division. The accuracy of any method is limited by the accuracy with which one can read these bottles. Even with the prescribed 30 per cent. Babcock cream bottle (18 grms.) graduated to read 0.5 per cent., it is impossible to read to a greater accuracy than 0.2 of a half per cent. Babcock scale division.

The method is by far the quickest of all methods tried. With the assistance of a laboratory boy, who emptied the bottles and dried the rods, the writer tested over 100 Babcock cream bottles in about 3 hours.

(b) FOR MILK BOTTLES.—The glass rods used for testing Babcock milk bottles were made from a piece of rod about 5 mm. in diameter. The specific gravity of the glass was again determined, and the weights of the rods were adjusted to the required calculated weights.

RODS FOR TESTING BABCOCK MILK BOTTLES.

Specific Gravity of the Glass 2.488.

No. of Rod	Weight Grms.	Required Weight Grms.	Volume in $\frac{1}{10}$ per cent. Divisions	Required Volume in $\frac{1}{10}$ per cent. Divisions
5	1.3935	1.3933	28.004	28.000
9	1.5926	1.5923	32.005	32.000
13	1.8415	1.8411	37.005	37.000

The method of making these rods and the procedure in testing the milk bottles are the same as are described for the cream bottles. In the case of the milk bottles, however, the test was not always started with the meniscus on the zero mark.

The majority of milk bottles on hand at the time when this method was first employed had a scale reading up to 10 per cent. The rods were therefore made so that sets of three gave a total volume of about 2 c.c., or 10 per cent. on the bottle. In the following tests the same rods were used in testing 8 per cent. milk bottles, so that only two readings could be obtained. These tests show the results obtained with the glass rod method compared with those obtained by weighing with mercury. Several tests were carried out on each bottle. With the mercury two methods were used.

I. The bottle was filled with mercury to the zero mark, or to a mark just above the zero, and weighed. Mercury was then added from a burette until the neck of the bottle was about half full, the reading taken, and the bottle again weighed. From the difference in weight the volume of the mercury added was calculated. The bottle was now nearly filled with mercury, and a second reading

obtained. The bottle filled with mercury weighed about 700 grms., and in order to avoid placing such a heavy load on the balance, and to weigh it more accurately, the following additional method was employed:

II. The bottle was filled to zero or one of the marks just above zero with mercury. A quantity of mercury equal to about half the volume of the graduated part of the neck of the bottle (in the case of the 8 per cent. bottle about 0.8 c.c.) was now weighed out in a glass dish, and the volume calculated. The weighed mercury was added, and the reading on the scale taken. A second reading was obtained by adding another quantity of weighed mercury.

In both these methods care had to be taken to remove air bubbles by gently tapping the bottle before the first reading was taken. Appreciable temperature changes were avoided by immersing the bottle in water at a constant temperature before each reading. In method No. II it was found that by adding the weighed mercury with a pipette the neck of the bottle could be kept free from air bubbles.

All the figures given in the table below represent $\frac{1}{10}$ per cent. Babcock divisions.

Bottle	With Rods			By Weighing added Mercury			By adding Weighed Mercury		
	Reading on Bottle	Volume of Rod	Error	Reading on Bottle	Volume of Mercury	Error	Reading on Bottle	Volume of Mercury	Error
A.	1.0-29.2	28.0	+0.2	0.0-42.7	42.2	+0.5	0.0-75.6	74.8	+0.8
	29.2-61.9	32.0	+0.7	42.7-78.0	34.9	+0.4	—	—	—
	Total error		+0.9	Total error		+0.9	Total error		+0.8
	1.0-40.8	40.0	+0.8	—	—	—	1.0-30.7	29.4	+0.3
	40.8-78.1	37.0	+0.3	—	—	—	30.7-63.9	32.6	+0.6
Total error		+1.1				Total error		+0.9	
B.	0.0-28.3	28.0	+0.3	—	—	—	1.0-29.2	28.0	+0.2
	28.3-70.9	42.0	+0.6	—	—	—	29.2-61.8	32.0	+0.6
	Total error		+0.9				Total error		+0.8
	1.0-43.9	42.0	+0.9	1.0-43.8	42.2	+0.6	1.0-77.8	75.7	+1.1
	43.9-75.3	31.0	+0.4	43.8-75.4	31.4	+0.2	—	—	—
Total error		+1.3	Total error		0.8	Total error		+1.1	
B.	1.0-43.9	42.0	+0.9	1.0-44.0	42.4	+0.6	1.0-48.0	46.3	+0.7
	43.9-76.1	32.0	+0.2	44.0-75.0	30.7	+0.3	48.0-79.9	31.5	+0.4
	Total error		+1.1	Total error		+0.9	Total error		+1.1
	0.0-39.9	39.0	+0.9	0.0-39.4	38.8	+0.6	1.0-77.9	75.7	+1.2
	39.9-76.1	36.0	+0.2	39.4-80.0	40.0	+0.6	—	—	—
Total error		+1.1	Total error		+1.2	Total error		+1.2	
			0.0-35.9	35.2	+0.7				
			35.9-77.8	41.4	+0.5				
			Total error		+1.2				

Bottle	With Rods			By Weighing added Mercury			By adding Weighed Mercury		
	Reading on Bottle	Volume of Rod	Error	Reading on Bottle	Volume of Mercury	Error	Reading on Bottle	Volume of Mercury	Error
C.	1.0-29.9	28.0	+0.9	1.0-30.0	28.6	+0.4	0.0-77.0	76.0	+1.0
	29.9-66.0	36.0	+0.1	30.0-66.0	35.5	+0.5	—	—	—
	Total error		+1.0	Total error		+0.9	Total error		+1.0
	1.0-41.0	39.0	+1.0	0.0-39.7	39.2	+0.5	1.0-30.0	28.5	+0.5
	44.0-71.1	30.0	+0.1	39.7-78.8	38.5	+0.6	30.0-66.1	35.8	+0.3
	Total error		+1.1	Total error		+1.1	Total error		+0.8
	0.0-40.8	40.0	+0.8						
	40.8-78.1	37.0	+0.3						
	Total error		1.1						
D.	0.0-40.0	39.0	+1.0	0.0-40.0	39.2	+0.8	0.0-77.2	76.3	+0.9
	40.0-70.5	30.0	+0.5	40.0-70.0	29.5	+0.5	—	—	—
	Total error		+1.5	Total error		+1.3	Total error		+0.9
	0.0-31.6	31.0	+0.6	0.0-43.0	42.3	+0.7	0.0-40.7	40.1	+0.6
	31.6-60.0	28.0	+0.4	43.0-78.7	35.2	+0.5	40.7-72.5	31.4	+0.4
	Total error		+1.0	Total error		+1.2	Total error		+1.0
	0.0-40.9	40.0	+0.9						
	40.9-77.2	36.0	+0.3						
	Total error		+1.2						
E.	2.0-30.2	28.0	+0.2	2.0-30.0	27.7	+0.3	0.0-78.7	77.6	+1.1
	30.2-71.0	40.0	+0.8	30.0-71.0	40.4	+0.6	—	—	—
	Total error		+1.0	Total error		+0.9	Total error		+1.1
	0.0-36.8	36.0	+0.8	0.0-39.2	38.8	+0.4	2.0-30.8	28.4	+0.4
	36.8-73.3	36.0	+0.5	39.2-77.7	37.9	+0.6	30.8-72.0	40.7	+0.5
	Total error		+1.3	Total error		+1.0	Total error		+0.9
	0.0-36.7	36.0	+0.7						
	36.7-77.1	40.0	+0.4						
	Total error		+1.1						
F.	1.0-37.0	36.0	+0.0				0.0-78.7	77.9	+0.8
	37.0-75.0	37.0	+1.0				—	—	—
	Total error		+1.0				Total error		+0.8
	1.0-41.1	40.0	+0.1	0.0-35.9	35.9	+0.0	1.0-38.7	37.5	+0.2
	41.1-79.1	37.0	+1.0	35.9-76.9	40.1	+0.9	38.7-76.6	37.2	+0.7
	Total error		+1.1	Total error		+0.9	Total error		+0.9
	0.0-32.0	32.0	+0.0				1.0-37.0	36.0	+0.0
	32.0-75.0	42.0	+1.0				37.0-74.9	37.0	+0.9
	Total error		+1.0				Total error		+0.9

It will be noticed that in the case of any one bottle the greatest difference between the total errors found by the three methods is equivalent to 0.06 per cent. on the bottle. The bottles used in the above tests were all on the border line.

In the case of the milk rods the error in the specific gravity determination is about 1 in 2000, whilst the error due to the grinding down of the rod is 1 in 5000. The total error of the test with each rod is 1 in 1400. With an 8 per cent. bottle the total error due to the method is therefore 80 in 1400, or 0.006 per cent. on the bottle.

The average length of the scale on the prescribed 8 per cent. milk bottle is about 70 mm., and the scale is divided into 80 parts, so that each division representing 0.1 per cent. is less than 0.9 mm. wide. The writer has found that, in many cases, the dividing lines of the scale measure 0.2 mm., representing 0.02 per cent. It is impossible to take into consideration the thickness of the line when the reading is taken with the naked eye. It would be difficult, therefore, without the aid of a cathetometer, to read these milk bottles with an accuracy within 0.03 per cent. This error is five times as great as the error due to the method. The method, therefore, besides being quick is sufficiently accurate.

After a year and four months' use, during which time over 5000 milk and cream bottles were tested, the rods were again weighed in order to ascertain whether the wear on them would make any appreciable difference. The following tables show the change in volume of some of the rods.

Cream Bottles			Milk Bottles		
Rod No.	Change in Volume after 16 months' use c.c.	Babcock $\frac{1}{2}$ per cent. Divisions	Rod No.	Change in Volume after 16 months' use c.c.	Babcock $\frac{1}{10}$ per cent. Divisions
1	0.0002	0.002	(1)	0.0003	0.015
2	0.0000	0.000	(6)	0.0002	0.010
12	0.0003	0.003	(2)	0.0001	0.005
17	0.0001	0.001	(13)	0.0002	0.010
19	0.0003	0.003			
6	0.0003	0.003			
9	0.0000	0.000			

My thanks are due to Dr. B. de C. Marchand, Chemist in the Department of Agriculture, for his interest and assistance.

DIVISION OF CHEMISTRY,
DEPARTMENT OF AGRICULTURE,
UNION OF S. AFRICA, PRETORIA.

Note.

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

THE ADDITION OF SODIUM CARBONATE BEFORE ESTIMATING FREE AMMONIA IN WATERS.

THE following experiments were conducted with a view to estimating when the addition of alkali was necessary in estimating free ammonia by distillation. The following waters were taken:—(a) Distilled water; (b) Birmingham water supply with alkalinity equivalent to 2.2 parts CaCO_3 per 100,000; (c) well-water with alkalinity equivalent to 8.4 parts CaCO_3 per 100,000.

To 500 c.c. of each of these 10 c.c. of ammonium chloride containing 0.0001 grm. of ammonia were added, and the liquid boiled, the original free ammonia having first been distilled off. In each case all the ammonia was obtained in the distillate and no further ammonia was obtained on adding 10 c.c. of 2 N sodium carbonate solution. These experiments were conducted in new flasks. As these might possibly be alkaline, the experiments were repeated in old flasks. The same results were obtained.

Another experiment was made in which ammonium acetate was added to distilled water, and again the whole of the ammonia was distilled without the addition of sodium carbonate, although the ammonium acetate was faintly acid to cochineal and to methyl orange. It was found, however, that if ammonium chloride was added to distilled water, as little as 0.5 c.c. of 0.1 N lactic acid in 500 c.c. of water was sufficient to prevent the distillation of any of the ammonia, which, however, came off completely on addition of an excess of sodium carbonate.

As the flasks used for the distillation of water are often washed out with hydrochloric acid, there is a remote possibility of a trace being left behind. Although it is exceptional to find a water that needs sodium carbonate, the simplest rule is to add it in all cases.

A. W. KNAPP.

KING'S HEATH,
BIRMINGHAM.

Notes from the Reports of Public Analysts.

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

CITY OF LONDON.

REPORT ON SAMPLES SUBMITTED TO THE PUBLIC ANALYST IN 1922.*

DURING the year 942 samples were examined, of which 830 were taken informally and 82 were found to be adulterated. Of the 112 formal samples 24 were adulterated. Ten informal samples (of 148 taken) and 2 formal samples (of 71 taken) of milk were adulterated, and there were two prosecutions.

* This forms a section of the Report of the Medical Officer of Health for the year 1922.—
EDITOR.

COFFEE EXTRACT.—Eighteen samples of coffee essence or extract contained from a trace to 7 grains of salicylic acid per lb., and one sample contained 10.5 grains of sulphur dioxide per lb. A letter was forwarded to all the manufacturers to the effect that the Sanitary Committee did not consider that coffee extract or essence required the addition of any preservative.

BORIC ACID IN CAKE AND EGG YOLK.—Out of 16 samples of slab cake submitted for analysis only 3 were free from preservative. The remainder contained quantities of boric acid ranging from 0.03 to 0.37 per cent. An egg yolk used in the preparation of cake made in the city contained 1.10 per cent. of boric acid. Until recently boracised egg yolk had been regarded as entirely an imported product, but firms are now preparing boracised liquid eggs in this country.

LEMON CURD AND LEMON CHEESE.—Nine samples were examined. Of these, 1 contained 2.1 grains of boric acid and 2 contained 1.4 and 1.75 grains of salicylic acid per lb. respectively.

CANNED SPINACH.—Of seven samples examined, two (American) were free from copper, but samples of French, Belgian and Dutch manufacture each contained copper varying from 0.022 to 0.049 per cent.

GELATIN.—Three of eight samples of gelatin prepared for domestic use contained sulphur dioxide in the proportion of 9.1, 2.8 and 4.2 grains per lb. respectively.

BOROUGH OF PORTSMOUTH.

ANNUAL REPORT OF THE BOROUGH ANALYST FOR 1922.

THE total number of samples examined was 1318, of which 1239 were food and drugs, 16 waters, 37 paints, oils, etc., and 26 miscellaneous. The samples taken under the Food and Drugs Acts represent 5.3 samples per 1000 of the population. Of the food and drugs samples 17 were inferior and 53 adulterated (=4.2 per cent.).

MILK.—The number of samples examined was 573, of which 16 were inferior and 19 adulterated. The average composition of the milk was: Fat, 3.66; solids-not-fat, 8.82 per cent.

CREAM.—Nine samples of cream and 19 of preserved cream were examined. In the case of preserved cream the label stated the sample to contain boric acid not exceeding 0.4 per cent. in every instance. The amounts of boric acid ranged from *nil* (2 samples) to 0.358. Two samples containing 0.169 and 0.182 per cent. of boric acid respectively, also contained 2 parts of formaldehyde per million.

BORIC ACID IN CAKE.—Of 25 samples of cake examined 16 contained boric acid in proportions ranging from 0.04 to 0.33 per cent. A letter was written to the local Confectioners' Association and they agreed to abandon the use of preserved liquid eggs in sponge cakes. Samples taken later in the year were free from boric acid.

DRUGS.—Of 115 samples of drugs bought from local pharmacists 4 (1 mercury ointment, 1 Gregory powder, 2 glycerin of borax) were not in accordance with the B.P. Standards.

R. P. PAGE.

Legal Notes.

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

AN AMBIGUOUS PRESCRIPTION.

ON July 25, H. C. Jenkins was summoned by the Hampstead Borough Council for selling a compounded drug containing 8 grains of potassium iodide per oz., instead of 10 grains demanded by the purchaser.

Dr. Scrase, Medical Officer for Hampstead, said that he regarded the deficiency as serious. In cross-examination, he stated that the prescription was written in Latin, and accepted the correction of Mr. Glyn-Jones, counsel for the defence, that the Latin for "grains" was "grana," and that although in this prescription he had written the contraction "grs.," in another prescription he had written it "gr."

The defendant said that he had read the prescription as "IV," for there was a downstroke with a dot above it immediately before the V of five. The abbreviation "grs." was only used in prescriptions written in English, not Latin.

Evidence was given by two other pharmacists that it was not unreasonable to say that the amount in the prescription might very well be read as either four or five.

The magistrate (Mr. W. H. Leicester) said that, personally, he should have read the prescription as a five, not as a four. But another man, noticing that the letter which was meant to be an "s" was drawn down like the figure "1," might think it was a four. There was a little mark on the paper exactly like a dot over the "1," and although that might be a mere misfortune, it might be misleading to the person who had to interpret the prescription. A prescription which might lead to the prosecution of a man ought to be absolutely unambiguous. It was not fair to him to send him a prescription as to which there was any possibility of mistake. Here there was not only a possibility, but people who were not at all interested in the case had stated as experts that in their opinion a person misreading that document would not be to blame. He should therefore acquit the defendant and dismiss the summons. Under the circumstances that the failure of the prosecution arose from the fact that the prescription was not legible he allowed the defendant five guineas costs.

COPPER IN PEAS.

ON August 1 the Civil Service Supply Association, Ltd., were summoned at Bow Street Police Court for the sale of a tin of peas containing 2·1 grains of copper sulphate, so as to render them injurious to health. On the tin was a label stating that "The colour of these peas has been preserved with sulphate of copper, the smallest quantity possible being used to ensure finest quality, and is not injurious to health."

Dr. F. J. Allan, Medical Officer for Westminster, said that the peas were rendered more indigestible to the extent of being injurious to health, by the amount of copper found in this case. The colour of peas was not retained by copper sulphate, but an artificial colour was imparted. He did not agree that the copper was entirely insoluble. Many people, to his knowledge, always required treatment after eating such peas.

Dr. P. K. Panton, clinical pathologist at the London Hospital, said that he had been unable to detect any "free" copper sulphate (*i.e.* soluble copper) in the contents of a tin of the same brand of peas. He had digested some of the peas for 8 hours with artificial gastric juice, and had then found the fluid to be quite free from copper, and from this he inferred that the copper could not have been absorbed into the system. In his opinion, the peas in the tin in question were not injurious to health.

The magistrate (Sir Charles Biron) said that it was obvious that the copper had been added to improve the appearance of the peas, and he had had clear evidence that it was likely to disagree with people. The peas in the condition in which they were sold seemed to him to be unwholesome.

A fine of £5, with £2 s. costs, was imposed.

MARGARINE CONTAINING 1 PER CENT. OF BUTTER.

On August 1st the Ballomachyle Creamery Co., Ltd., Mauchline, Ayrshire, was summoned at the Salford Police Court for the sale of margarine labelled "Ayrshire Margarine Rolls. Blended with pure butter." The sample analysed had been certified to contain 99 per cent. of margarine and 1 per cent. of butter, and the prosecution submitted that the defendants had sold an article not of the nature, quality and substance demanded. As the defendants had not complied with the law, which required the article to be described as margarine only, they were not entitled to the protection of the statute which limited them to 10 per cent. of butter. The margarine was an average product which could be purchased at 6d. to 8d. per lb., and on the date of sale of the sample butter could be purchased at 1s. 4d. per lb., so that a blend of 50 per cent. of margarine and butter could have been made for 1s. a lb., which was the retail price of the sample containing 1 per cent. of butter.

Mr. Lustgarten, for the defence, said that the only question was whether the article supplied corresponded with the description. He quoted a case heard in the Divisional Court, in 1915, when it was held that a mixture of margarine and butter containing less than 5 per cent. of butter was in accordance with the description. Referring to the evidence of Inspector Bowker, he said that the inspector's view was that nothing less than 30 per cent. of butter would justify the description. If they took the inspector's view that nothing less than 30 per cent. would give a flavour to the mixture, it did not matter whether 5 or 10 per cent. were put in. If, on the other hand, manufacturers were right in believing that the introduction of a small percentage of butter would give a flavour, the article was not misrepresented.

The stipendiary magistrate (Mr. P. W. Atkin) decided against the defence, and imposed a fine of £20. He said that he would be willing to state a case, and remarked that people paid more for this article because they thought there was butter in it. They might have a definition of an Act of Parliament, but one per cent. was not enough to make him think it fair to the purchaser.

CREAM CHEESE.

Correction.

In the report of the case in the September issue (p. 447), read "fat" for "cream" in lines 3, 5, and 9.

Department of Scientific and Industrial Research.

FOOD INVESTIGATION BOARD.

BROWN HEART—A FUNCTIONAL DISEASE OF APPLES AND PEARS.*

"BROWN heart" is an abnormal condition of the fruit resulting from the death and subsequent browning of parts of the internal fleshy tissue, while the peripheral flesh generally remains sound.

The results of the experiments, described in detail, have shown that apples which remain free from blemish when stored in air (21 per cent. oxygen; 0 per cent. carbon dioxide) develop brown heart when stored in atmospheres containing carbon dioxide exceeding a certain limit (13.6 per cent. in the experiments cited). Under the conditions which occur in practice when fruit is stored in gas-tight chambers, with little or no ventilation, the concentration of carbon dioxide rises, and that of oxygen falls correspondingly, until all the oxygen has disappeared. After this, carbon dioxide is still formed by the fruit and continues to accumulate. The formation of brown heart cannot be prevented by artificially maintaining a high oxygen concentration. The composition of the internal atmosphere which permeates the living tissue is also a matter of importance. The higher the temperature the higher the percentage of carbon dioxide and the lower the percentage of oxygen in the tissue atmosphere. It has been shown that brown heart may be produced by merely exposing the apples to high temperatures (113° F.). It is suggested that the disease known as "bitter pit," which occurs in apples in the orchard may be due to a temporary excess of carbon dioxide in the internal atmosphere of the fruit.

The disease known as "internal breakdown" occurs only in the middle or late storage life of the apple, and is not associated in any way with high carbon dioxide or low oxygen concentrations in the storage atmosphere. It is more prevalent in cold storage than in ordinary storage. Another disease described as "deep scald" occurs in apples stored under conditions characterised by the absence of oxygen, and also appears more readily in apples stored at low temperatures.

* *Special Report*, No. 12. By F. Kidd, D.Sc., and C. West, D.Sc. Pp. 54. 19 Plates. H.M. Stationery Office, Kingsway, W.C.2. Price 4s. 6d. net.

Government of Madras.

ANNUAL REPORT OF THE CHEMICAL EXAMINER.*

THE total number of analyses made during the year was 3824, including 220 in cases of human poisoning and 399 in stain cases.

MEDICO-LEGAL INVESTIGATIONS.—In the 220 cases of suspected poisoning, poison was detected in 108. For the extraction of morphine from viscera the method recommended by Lucas (*Forensic Chemistry*, pp. 190–202) for the porphyrin test has proved sensitive and trustworthy. *Madar*.—Experiments were made to discover a test for madar. An alcoholic extract of the plant, after evaporation to dryness and solution of the residue in water, gave crystals with a saturated solution of iodine in 10 per cent. potassium iodide solution, which bore some resemblance to Florence's crystals. The alcoholic extract on evaporation,

* G.O. No. 688 P.H., Apr. 21, 1923. Report for the Year 1922. By Major Clive Newcomb, M.D., A.I.C.

left a residue which gave a bluish coloration with strong hydrochloric acid, a green coloration with strong sulphuric acid, and a pink coloration with dilute acids. In two cases of suspected madar poisoning a bitter resin giving these reactions and proving fatal to a frog could be extracted from the viscera.

The quinine molybdate test (ANALYST, 1922, 47, 317) was tried and found to work satisfactorily.

HUMAN POISONING CASES.—Of the 108 cases in which poison was detected 175 persons were affected and 74 died. Mercury was the poison most commonly found (32 cases), arsenic next (23 cases), and opium third (19 cases). There were two cases of cyanide poisoning, one with iodine, one with caustic soda, and one with quinine and ergot. In one case datura was used as an aid to robbery, the victim being rendered unconscious.

ANIMAL POISONING.—There were 42 cases with 134 articles. Arsenic was the poison most commonly found (8 cases) and thevetin (the active principle of yellow oleander) the next (6 cases). Mercury was detected in 2 cases.

STAIN CASES.—The spectroscopic test was used for routine work, the hæmin test being used for confirmation. During the year 1065 specimens of blood stains were sent to the Imperial Serologist, Calcutta, and he reported 985 to be human blood. Of the remainder, 10 were ruminant blood only, 2 were non-mammalian blood, three were mixed human and non-mammalian blood, three were the blood of an equine animal, and in 62 the origin of the blood could not be discovered.

SEMINAL STAINS.—The following modification of Florence's method has been devised and used in all the cases. The suspected stain is cut out and wetted with sufficient water to give 1 drop when the cloth is squeezed. After not less than 5 minutes' contact this drop of aqueous extract is squeezed on to a microscope slide, and 1 drop of a saturated solution of iodine in 10 per cent. potassium iodide solution is put beside, but not touching it, until a cover glass is placed over the two drops. The crystals thus obtained with semen are apparently due to choline, and a pure solution of choline prepared from white of egg gave typical crystals in very great dilutions. About 150 suspected stains were examined, and in no case in which spermatozoa were found did the test give a negative result.

The test has also a positive value, however, since choline in combination does not give the test, and in practice substances which yield free choline on decomposition do not seem to occur in association with stains on garments. Only in one case (an extract of the flowers of the madar plant) were crystals resembling Florence's crystals obtained. Hence, while Florence's test does not afford absolute proof of the presence of semen, a stain which gives the characteristic crystals will probably be seminal.

HUMAN IDENTIFICATION.—In two cases there was a question of the identification of persons supposed to be murdered. Comparative experiments indicated that estimates of the height from the bones should not be given within less than 3 inches.

In another case identification of hair was required. A woman had been murdered and grasped in her hand a tuft of hair, which she had presumably torn from the murderer's head. Microscopical examination of the ends of the hair showed that it had been pulled out and not cut. It also agreed in microscopical appearance, both when mounted in caustic potash solution and in cross section, with the hair of the accused.

GENERAL ANALYSES.—The number of these was 570, including 144 from the Customs, of which 50 were analyses of gold or silver thread. These frequently showed small traces of gold or silver, probably indicating that they were plated with the genuine metals.

Analysis of Dispensed Medicines.

THE following letters have been sent by the Hon. Secretary of the Society to the Secretary of the Ministry of Health.

June 21, 1923.

Sir,

ANALYSIS OF PRESCRIBED MEDICINES.

It has been brought to the notice of my Council that difficulties have arisen in the interpretation of results of analyses of dispensed medicines, owing to variation in the accuracy of the bottles commonly employed, making it impossible to form a correct judgment as to the exactness of the dispensing.

My Council desires to submit the following suggestion to your Ministry:

That a circular be issued by the Ministry to the Local Authorities recommending that the following procedure be adopted in taking samples of prescriptions:

- (i) The inspector be instructed, prior to dividing the sample into three parts, to mark, in the presence of the vendor, the height the contents reach in the bottle in which the medicine is originally supplied to him by the vendor.

That the bottle so marked be submitted to the analyst in order to enable him to determine the total quantity of medicine supplied.

- (ii) The Analyst and the Medical Officer of Health be both consulted as to the type of prescription it is desirable to use for the purpose of checking the accuracy of dispensing, and that, in the event of any substantial inaccuracies being disclosed by analysis, both these officers be consulted as to the desirability of instituting proceedings.

I am, sir, yours faithfully,

E. R. BOLTON (Honorary Secretary).

Sir,

June 21, 1923.

The Council of the Society of Public Analysts having addressed to the Ministry a letter, dated to-day, on the question of the procedure best adapted for the efficient and just administration of the Sale of Food and Drugs Acts, in the case of dispensed medicines, desires at the same time to ask that the Ministry will use such means as they think fit to ensure that the Public Analyst shall receive adequate remuneration for the difficult work entailed by the analysis of such medicines.

It is felt that analyses, such as are required in these circumstances, were not contemplated in the Act of Parliament and that the fee, customarily paid by even the most sympathetic Local Authorities for analyses of samples taken under the Food and Drugs Acts, cannot be considered a reasonable one for the analysis of the majority of dispensed medicines.

It has often been urged upon the Ministry that year by year increased demands are made upon the Public Analyst without any corresponding increase in his fee. That the Analyst should now be required to analyse complex

mixtures, such as dispensed medicines, for a fee that is already inadequate for single substances, is putting an altogether unjustifiable burden upon the Analyst. The Council of the Society, therefore, urges that in any instructions which the Ministry issues to Local Authorities in regard to the analysis of medicines a strongly-worded opinion should be expressed that the Authority cannot fairly ask its Analyst to undertake the analysis for the fee usually paid for samples under the Food and Drugs Acts, and that an additional fee should be paid in these cases.

I am, sir, yours faithfully,

E. R. BOLTON (Honorary Secretary).

The Hon. Secretary of the Society has received a reply from the Minister of Health, dated July 25th, to the effect that these letters are receiving consideration, and that a further communication will be addressed to him in due course.

United States Department of Agriculture.

FOOD INSPECTION DECISION 192.

MUSTARD AND MUSTARD PRODUCTS.

THE following revised and amended definitions and standards for ground mustard seed, mustard cake, mustard flour, and prepared mustard were adopted by the Joint Committee on Definitions and Standards, composed of representatives of the United States Department of Agriculture, the Association of American Dairy, Food and Drug Officials, and the Association of Official Agricultural Chemists, at its meeting, March 12 to 16, 1923.

Ground Mustard Seed, Mustard Meal, is the unbolted, ground mustard seed and conforms to the standards for mustard seed.

Mustard Cake is ground mustard seed, mustard meal, from which a portion of the fixed oil has been removed.

Mustard Flour, Ground Mustard, "Mustard," is the powder made from mustard seed with the hulls largely removed and with or without the removal of a portion of the fixed oil. It contains not more than one and five-tenths per cent. (1.5 per cent.) of starch, nor more than six per cent. (6 per cent.) of total ash.

Prepared Mustard is a paste composed of a mixture of ground mustard seed and/or mustard flour and/or mustard cake, with salt, a vinegar, and with or without sugar (sucrose), spices, or other condiments. In the fat-, salt-, and sugar-free solids it contains not more than twenty-four per cent. (24 per cent.) of carbohydrates, nor more than twelve per cent. (12 per cent.) of crude fiber, nor less than five and six-tenths per cent. (5.6 per cent.) of nitrogen, the carbohydrates being calculated as starch.

The foregoing definitions and standards are adopted as a guide for the officials of this department in enforcing the Food and Drugs Act.

June 27, 1923.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Coagulation of Milk by Acid. L. Anderson. (*Trans. Faraday. Soc.*, 1923, 19, 106–111.)—It has been previously suggested that the globules of fat in milk are protected by a layer of so-called mucoid protein, but microscopic examination of the effect of the addition of dilute hydrochloric acid or sodium hydroxide solution to milk shows that casein is the protective agent for the fat corpuscles. The stability of the fat globules is due entirely to this protective layer and is independent of the fact that the fat is solid at ordinary temperatures. The addition of dilute acid causes the precipitation of the casein, the rate of which increases with the amount of acid added until a maximum is reached; the amount required to reach this maximum is inversely proportional to the dilution of the milk. The addition of a further quantity of acid redissolves the casein, with the result that the fat globules which were carried down with it rise to the surface, but they do not coalesce; still further addition of acid “salts out” casein hydrochloride. Dilute caustic soda solution also does not bring about any coalescence of the fat particles. The fat globules, after separation by dilute acid, may be freed from excess of protein and caused to coalesce by repeated washing with water, which removes the protective casein layer. Benzene or olive oil emulsions in casein solution show similar properties in respect to acid and alkali and may be made to coalesce in a similar manner.

H. E. C.

Reducing and Oxidising Properties of Milk. P. Haas and T. G. Hill. (*Pharm. J.*, 1923, 111, 94.)—When milk is heated with sodium nitrate in presence of acetaldehyde at 45° C. the nitrate is reduced, the amount of nitrite produced rising to a maximum and then falling until eventually no nitrite can be detected. This process is greatly accelerated by effective aeration. By replacing air with nitrogen the reduction of nitrate proceeds until a definite maximum amount of nitrite has been produced, after which prolonged rotation causes no further diminution of nitrate. Again, when milk is rotated in a thermostat with sodium nitrite and acetaldehyde, in the presence of air, the nitrite is rapidly destroyed, with oxidation to nitrate, up to a certain limit. From these observations the conclusion is drawn that milk contains two substances which, in presence of acetaldehyde, can exert reducing and oxidising actions, respectively. Both appear to be present only in limited amount, and since they themselves are used up as a result of their activity, it would appear that they are not enzymes, although the reducing substance is thermolabile at 75° C. It is also noteworthy that the disappearance of the oxidising substance coincides with the disappearance of the so-called peroxidase reaction—a circumstance which also casts some doubt upon the enzymic nature of the peroxidase of milk.

Notes on the Food Value of Pacific Fish and on the Constants of Whale Oil. W. M. Doherty. (*J. Royal Soc. New South Wales*, 1922, 46, 206-209.)—Analyses of various food fish of the Pacific were as follows:—

Fish	Weight in grms.	Water Loss at 100° C. per cent.	Total Organic Nitrogen	Nitrogen from Gelatin	Nitrogen other than from Gelatin	Proteins equal Flesh-forming substances	Gelatin	Fat	Non-nitrogenous extract	Ash	Phosphoric Acid
Snapper (<i>Pagrosomus auratus</i>)	1800	78.82	3.25	0.49	2.76	17.46	2.69	0.33	1.56	1.38	0.58
Sea mullet	1470	72.6	3.64	0.53	3.11	19.68	2.92	2.72	1.42	1.33	0.57
Black bream	580	74.6	3.78	0.52	3.26	20.63	2.98	2.36	1.44	1.5	0.61
Sand whiting	163	76.0	3.57	0.56	3.01	19.05	3.08	1.60	1.56	1.38	0.51
River garfish	66	75.0	3.60	0.70	2.90	18.35	3.85	1.50	1.85	1.58	0.60

Snapper muscle fat was found to give a purple coloration on dissolving in chloroform and adding sulphuric acid, possibly indicating the presence of vitamin A.

Analyses of *Whale Oils* from Pacific sources established the following figures:—

Source of Oil	Specific Gravity at 15.5° C.	Refractive Index at 15.5° C.	Saponification Value	Acid Value	Iodine Value
Blubber of Sei Whale	0.9182	1.474	195	0.75	99.6
Bones " " "	0.9186	1.471	197	1.25	86.4
Blubber of Humpback, 1.	0.9232	1.475	194.5	5.80	108
" " " 2.	0.9212	1.476	192	1.00	119
Tongue " " "	0.9212	1.473	199	2.2	105
Tongue of blue whale	0.9197	1.4715	194	0.87	95
Stearin from humpback	0.9204	—	192	35.00	92
Oil and spermaceti from head cavities of sperm whale	0.8779	1.463	145	12.7	66
Oil from blubber of sperm whale	0.8796	1.4667	127	3.0	89

D. G. H.

New Method for Estimating Proteins in Honey. O. Laxa. (*Ann. Falsificat.*, 1923, 176, 286-289.)—In cases where it is necessary to estimate the true proteins of honey but not the total nitrogen-containing matter, 8 grms. of honey are weighed into a wide-necked flask of about 150 c.c. capacity, 4 c.c. of water added and the honey dissolved by warming. One hundred c.c. of 96 per cent. alcohol are then poured in, and the flask corked and left to stand until the next day. Dextrin and albuminoids are precipitated on the bottom and sides of the flask and the clear solution is decanted. The precipitate is washed with 10 c.c. of alcohol and the flask heated in the oven at 100° C. for 2 hours. The residue is digested with cold water to eliminate dextrans and sugars, re-dried and weighed. If the alcohol precipitate should be flocculent and non-adherent to the flask, it should be filtered through a weighed paper and treated as above. Some honeys give very similar figures by Kjeldahl's method and the above procedure, but others, for example Spanish honeys, show a considerably higher Kjeldahl figure. Further, it was found that most honeys contain more non-precipitable nitrogen than protein nitrogen, since the figure from the tannin precipitate method is higher than the Kjeldahl figure. From this it is shown that Lund's method of estimation by precipitation with phosphotungstic acid is not exact. Tables of results are given for honeys of different origins.

D. G. H.

Quantitative Estimation of Inositol. J. Needham. (*Biochem. J.*, 1923, 17, 422-429.)—The methods adopted by former workers are criticised and the experimental development of the following method is given in detail. The tissue is freed from fat, finely minced and well stirred into approximately an equal weight of acetone, the mixture being left overnight. After filtration, the solution is evaporated under reduced pressure until free from acetone, and the aqueous residue is treated successively with neutral lead acetate and basic lead acetate and, after filtration, the lead remaining in solution is precipitated with hydrogen sulphide. Any inositol adsorbed is removed by extracting the precipitate twice with water at 70° C., the extracts being mixed with the aqueous solution and evaporated to a volume of about 20 c.c., to which 300 c.c. of absolute alcohol are added and the mixture is left to stand overnight. The precipitate of pure inositol is filtered off through a Gooch filter, and the alcohol is removed by continuing the current of air for 10 minutes after filtration is complete. The residue is dissolved in water, and the solution is diluted to 100 c.c., after which the amount of inositol present is estimated from the carbon content of the solution by the method described in the abstract on p. 505. Inositol is adsorbed by fuller's earth but not by kieseluhr, melts at 221° C., and does not exhibit optical activity. Results obtained show that the inositol content of freshly-killed rabbit muscle is 50 per cent. less than from muscle of an animal which has been dead two days, and that the inositol of ox muscle is identical with that obtained by the hydrolysis of phytin. Tables are given showing the action of various reagents used and also some preliminary results obtained with various rabbit tissues. T. J. W.

Halawa (Oriental Nougat). A. Azidin. (*Ann. Falsificat.*, 1923, 176, 289-292.) Halawa gozia (a sugar paste mixed with broken nuts and eaten as a sweetmeat) and Halawa tahinya, which serves as a food, are the most important of the various varieties sold in Egypt. Halawa tahinya is made from sugar and crushed sesame seed, and various samples examined, both of Egyptian and foreign manufacture, contained from 22.6 to 28 per cent. of oil with a butyro-refractive reading at 40° C. varying between 60.0 and 61.4, and giving a positive Baudouin and negative Halphen reaction, and sugar varying from in one case as little as 9 per cent., to a maximum of 54 per cent. Molasses or glucose and saccharin are liable to be present, as their introduction cheapens manufacture. D. G. H.

Constituents of the Wax Coating of Apple Skin. C. E. Sando. (*J. Biol. Chem.*, 1923, 56, 457-468.)—The mixed skins of Ben Davis and Black Ben Davis varieties of apples were rapidly dried in a current of warm air, coarsely ground and extracted in a large Soxhlet apparatus with ether. The residue obtained on evaporating the ether was shaken several times with cold 80 per cent. acetone to remove coloured impurities, etc. The acetone extracts were neglected, and the insoluble white residue was mixed into a paste with plaster of Paris and water, and, after setting, was extracted with petroleum spirit. After all material soluble in this solvent was removed the residue was further extracted with ether. From the petroleum spirit extract various fractions were obtained by fractional

crystallisation with the use of different solvents. These fractions were too small in amount for complete identification, but appeared to consist of hydrocarbons and alcohols or mixtures of these substances. In addition, triacontane, $C_{30}H_{62}$, and heptacosanol, $C_{27}H_{56}O$, were separated and identified by their melting points and analysis by combustion. From the ether extract by similar methods of purification a new crystalline alcohol, $C_{30}H_{48}O_3$, was isolated and named malol. This compound is isomeric with caryophyllin and gentiol and is the next lower homologue of oleanol and prunol. It consists of lustrous prismatic needles melting at 284 to $285^\circ C.$, is insoluble in water and petroleum spirit, slightly soluble in ether, chloroform, acetone, cold ethyl alcohol and glacial acetic acid, but dissolves readily in boiling absolute or 95 per cent. ethyl alcohol. Its alcoholic solution is dextro-rotatory and exhibits muta-rotation. The mono-acetyl, di-acetyl, mono-methyl and acetyl-methyl compounds have been prepared and their melting points and composition determined. With the Liebermann-Salkowski cholesterol reaction malol yields a pink colour which slowly changes through violet and blue to green. (Cf. Power and Chesnut, *J. Amer. Chem. Soc.*, 1920, **42**, 1509.) T. J. W.

International Standardisation of Quillaia Preparations. J. Cofman-Nicoresti and S. B. Tallantyre. (*Pharm. J.*, 1923, **111**, 103-104.)—Most of the pharmacopœias in which quillaia bark is an official drug give the species *Q. saponaria* as its source. The French Codex, however, requires the bark of *Q. smegmadermous* D.C. and the Mexican pharmacopœia mentions both species. Three other kinds of quillaia have also been described—namely, *Q. Peppigi* (Walpers), *Q. lancifolia* (Don) and *Q. braziliensis*, Martius. The chief species have the following botanical characteristics:

<i>Q. saponaria.</i>	<i>Q. smegmadermous.</i>	<i>Q. Peppigi.</i>
Strands of bast fibres placed axially.	Strands of bast fibres smaller and rarely exceeding 15 to 20 fibres. Isolated fibres of more frequent occurrence.	Strands of bast fibres placed obliquely.
Bast fibres not usually extending from one ray to another.	—	Bast fibres usually extending from one medullary ray to another.
Medullary rays, usually of 4 rows of cells.	Medullary rows, usually of 4 rows of cells.	Medullary rows, usually of 3 rows of cells.
Lignification confined to lateral cells of the medullary rays.	Large irregular sclerotic cells in the cortex; sclerotised cells almost absent in medullary rays.	Lignification continued as a rule, to the third cell of the ray.
Starch grains 4 to 6 μ in diameter.	Starch grains 15 to 20 μ in diameter.	—

Certain authorities (e.g. Reich, Gay) regard these three kinds as varieties of the same species due to variations in the conditions of growth. In analysing samples of these species or varieties the authors have found the saponogenin method the most suitable for the estimation of the saponins. The filtered saponin solution

is boiled for a short time with dilute hydrochloric acid and allowed to cool, and the precipitated saponin is separated, dried at 100° C. and weighed. No other constituent of the bark is precipitated under these conditions. Pure quillaia saponin yields on hydrolysis 31.1 per cent. of saponin, so that 1 part of saponin found by this method is equivalent to 3.22 parts of saponin, and this factor has been used in calculating the subjoined results, which were obtained with two samples of each kind of bark.

Species of Bark	Soluble in Water Per Cent.	Sapogenin Per Cent.	Saponin calculated from Sapogenin Per Cent.	Soluble in Alcohol Per Cent.	Ash Per Cent.
<i>Q. saponaria</i>	32.80	—	—	—	—
	32.40	3.35	10.78	22.25	9.05
<i>Q. smegmadermous</i>	29.40	3.00	9.66	19.75	9.58
	27.25	3.10	9.98	—	10.20
<i>Q. Peppigi</i>	26.20	3.06	9.87	22.60	9.78
	21.40	2.75	8.98	—	9.35

From this investigation the conclusion is drawn that there appears to be no reason for giving preference to any one species or variety of quillaia over the others. The proportion of bark used in the preparation of the tincture is 20 per cent., except in the B.P. (5 per cent.). For the sake of uniformity it is desirable to use 20 per cent. in England. It is suggested as a standard that an alcoholic tincture should contain a minimum of 1.8 per cent. of saponin, estimated as above described.

Activity of Pharmacopœial Preparations of Ergot. A. J. Clark and W. A. Broom. (*Pharm. J.*, 1923, 111, 89-91.)—Ergot amines and alkaloids differ in the fact that, although they all produce powerful pharmacological effects when injected, yet the amines produce little or no effect when taken by the mouth in moderate doses, whereas the alkaloids are active when taken by the mouth. A method which measures only the ergot alkaloids has been based by the authors upon the fact that adrenaline normally produces contraction of the isolated uterus of the rabbit, but after a sufficient dose of ergot alkaloids it produces either no effect or inhibition. The uterus, after excision, can be kept on ice for more than a day without losing its activity, and a strip of about 1 cm. in length is sufficient for a test. The uteri of different animals vary slightly, but can be standardised upon a solution of pure ergotamine. The results obtained by this method agree with those obtained by the cock's comb method and the cat's blood pressure method, whereas the usual methods of standardising ergot preparations upon the isolated uterus or upon the cat's uterus *in situ* measure chiefly the amine content. Comparative results, given in tabular form, show that the instructions of the British Pharmacopœia, if followed exactly, result in preparations almost devoid of ergot alkaloids, although considerable quantities of these alkaloids are yielded by the same samples of ergot when treated by the U.S.P. method. The conclusion is therefore drawn that, since the ergot alkaloids are the only known active specific principles of ergot, the methods of preparation laid down by the B.P. appear to be absurd.

Biochemical, Bacteriological, etc.

Fat-Soluble Vitamin and the Action of Light. H. Steenbock and E. M. Nelson. (*J. Biol. Chem.*, 1923, **56**, 327-373.)—Young rats raised on a stock ration and transferred to a purified ration containing yeast as its only source of vitamins will grow for a few weeks, then cease growing completely or partially, and ultimately will fail owing to the incidence of ophthalmia or infections of the respiratory tract. Aerated cod-liver oil or light from a quartz mercury-vapour lamp, both well known as antirachitic agents, will eliminate the initial failure of growth, or, when the failure is prevalent, will restore growth, without appreciably postponing the final failure due to ophthalmia or respiratory diseases. Inasmuch as aerated cod-liver oil did not cure ophthalmia, the experiments (described in detail) support the theory of McCollum that cod-liver oil contains two vitamins, viz. vitamin A and the antirachitic vitamin.

Influence of Reaction on the Oxidation of Vitamin C. S. S. Zilva. (*Biochem. J.*, 1923, **17**, 410-415.)—The author has investigated the effect of air upon decitrated lemon juice of different P_H values by its physiological action upon guinea pigs fed upon a scorbutic diet of oats, bran and milk. Lemon juice of $P_H=12.5$ exposed to the air at the ordinary temperature loses about 80 per cent. of its vitamin potency in 30 minutes and, after 3 hours, a daily dose of 5 c.c. failed to prevent or retard the onset of scurvy. A similar sample kept under the same conditions, but in the absence of air for 24 hours showed no deterioration in potency. The preparation exposed to air showed a decrease in P_H value during exposure. By aspirating air through boiling decitrated lemon juice the destruction of the vitamin was delayed by reducing the P_H value of the solution from 6.6 to 2.2.

T. J. W.

Technique of Blood Platelet Counting in Vitamin A Deficiency. W. Cramer, A. H. Drew and J. C. Mottram. (*Brit. J. Exp. Path.*, 1923, **4**, 37-44.)—The authors explain the discrepancies between their results (*Lancet*, 1921, **1**, 963; 1922, **2**, 1202; *Proc. Roy. Soc.*, 1922, B, **93**, 449) and those obtained by Bedson and Zilva (*Brit. J. Exp. Path.*, 1923, **4**, 5) by the inferiority of the optical microscope system employed by the latter workers. Owing to the minute size of the platelets a highly efficient optical system is necessary in order to distinguish between these bodies and other particles of similar dimensions occurring in blood. By the use of an ordinary Abbé sub-stage condenser Bedson and Zilva limited the numerical aperture of their optical system to 0.87, and by partially closing the iris diaphragm this was still further reduced. The authors employed a first-class apochromatic condenser yielding a numerical aperture of 1.37 with the 2 mm. objective used, thus obtaining resolution exceeding 50 per cent. higher than that of Bedson and Zilva. By comparison of the results obtained with three samples of rat's blood, with the above numerical apertures, it was shown in all cases that considerably higher results were obtained with the inferior optical system.

T. J. W.

Purification and Properties of Insulin. H. W. Dudley. (*Biochem. J.*, 1923, 17, 376-390.)—A description is given of the preparation of crude insulin from ox pancreas. This substance contains over 50 per cent. of inorganic salts, and yields positive reactions for chlorides and phosphates, biuret, tyrosin, tryptophan, the iminazole ring, carbohydrates, fructose and sulphur. The "rabbit-unit" (the weight required to reduce the blood sugar of a rabbit, kept without food about 20 hours, and weighing 2 kilos., from a normal value of 0.1 per cent. to about 0.04 per cent. and to cause typical hypoglycæmic convulsions within 4 hours) for this complex mixture is about 10 mgrms., this quantity being obtained from 15 grms. of pancreas. From a 1.5 per cent. solution of crude insulin, freed from suspended matter by centrifuging, the hormone is precipitated by the addition of half its volume of saturated aqueous picric acid solution and, after standing for 1 or 2 days, the supernatant liquid is poured off, and the residue is washed with water several times in centrifuge tubes, collected on a Buchner funnel and dried *in vacuo* over sulphuric acid. This compound is converted into the hydrochloride by grinding 1 gm. with 6 c.c. of absolute alcohol, transferring it to a centrifuge tube with a further 2 c.c. of alcohol, and adding 4.5 c.c. of an absolute alcohol solution containing 5 per cent. of dry hydrochloric acid. After stirring for 5 minutes, 30 c.c. of dry ether are run in, and the hydrochloride is precipitated as a white amorphous powder; this is washed twice with dry ether and finally dried over sulphuric acid *in vacuo*. This compound is free from phosphorus and yields positive reactions with the biuret, Pauly and organic sulphur tests, whilst the reactions for tryptophan and tyrosin are negative. The "rabbit unit" for the purified substance varies from 0.5 to 1.0 mgrm., and the total activity of the crude insulin is contained in the purer product, whilst the latter does not give rise to the physiological "depressor" effect of the former. From the aqueous solution of the hydrochloride, precipitates are obtained on the addition of either acid or alkali, both products showing a higher physiological activity than that of the original hydrochloride. Insulin is relatively stable to acids, but is readily decomposed by alkalis, pepsin and trypsin, these facts explaining its inefficiency when administered by the mouth. It is readily adsorbed from faintly acid solutions, but in solutions brought to a P_H value of 7.2 by the addition of di-sodium phosphate may be readily passed through a Berkefeld kieselguhr filter without loss. The compound appears to be a highly complex protein derivative, and its isolation as a chemically pure substance and its synthesis are probably very remote. T. J. W.

Estimation of Blood Urea. J. A. Behre. (*J. Biol. Chem.*, 1923, 56, 395-404.)—The substitution of an extract of jack bean meal for solid soya bean meal in the estimation of urea in whole blood produced lower results than those previously obtained. An investigation into the cause of this difference showed that it depended upon the quantity of soya bean used, whether in the powder form or as extract. When ox blood filtrates (obtained by heat coagulation or by precipitation with tungstic acid) were employed, practically no difference was obtained whatever the concentration of enzyme used. With human blood filtrates

a slight increase in ammonia was given by increased concentration of the extract. The substance to which this increase is due exists chiefly in the red blood corpuscles, does not diffuse through collodion, and may represent a considerable proportion of the "undetermined" nitrogen in filtrates from human blood. The results given in various tables indicate that the true urea content is more accurately given by the use of blood filtrates instead of whole blood. It is suggested that soya bean meal and crude extracts may contain a second enzyme, in addition to urease.

T. J. W.

Estimation of Urea by Means of Urease. G. M. Wishart. (*Biochem. J.*, 1923, 17, 403-405.)—When crude soya-bean meal is used for the estimation of urea the addition of sodium carbonate in sufficient amount to liberate the ammonia formed in the free state is without deleterious effect upon the activity of the urease. This observation is made use of in the following method:—Three large test tubes are constricted by drawing them out in a blowpipe flame in such a manner that the capacity below the constriction is about three times that of the liquid to be contained. These tubes are fitted with corks and connecting tubes as wash bottles, the upper end of the first inlet tube being provided with a three-way piece and stopcock. Into the first tube 20 c.c. of water, 5 c.c. of urine and 3 to 5 grms. of soya bean meal are placed, and the central narrow tube is inserted and surrounded by a few glass beads above the constriction. The remaining tubes contain known volumes of 0.1 *N* sulphuric acid for absorption of the ammonia liberated. The tube containing the mixture is immersed in a water-bath at 40° C., and a current of ammonia-free air is drawn through the apparatus at a rate exceeding 5½ litres per minute. Two c.c. of saturated sodium carbonate solution are introduced into the tube containing the mixture by means of the stopcock, and aeration is continued for 40 minutes, after which the urea is calculated from the titration of the standard acid. Results obtained with known amounts of urea show a maximum error of 0.88 per cent.

T. J. W.

Modification of the Folin-Wu Method for Obtaining Protein-free Blood Filtrates. R. L. Haden. (*J. Biol. Chem.*, 1923, 56, 469-471.)—One volume of blood is diluted with 8 volumes of 0.41 per cent. sulphuric acid and treated with 1 volume of 10 per cent. sodium tungstate solution, after which the liquid is filtered. The advantages obtained by this method over the previous one are increased rapidity of filtration, the greater volume of filtrate obtained amounting to about 15 per cent., and the more neutral reaction of the filtrate. Corresponding estimations were made of the non-protein nitrogen, uric acid, creatinine, amino-acid nitrogen, sugar and chlorides in blood by the original method and the new modification, and with twelve samples practically identical results were obtained (*J. Biol. Chem.*, 1919, 38, 81).

T. J. W.

Electrolytic Separation of Hexone Bases from Protein Hydrolysates. G. L. Foster and C. L. A. Schmidt. (*J. Biol. Chem.*, 1923, 56, 545-553.)—Electrolysis was carried out in a wooden box internally coated with asphalt paint

and divided into three compartments by membranes consisting of linen cloth impregnated with 30 per cent. gelatin solution and immersed in formalin overnight. The electrodes consisted of carbon sheets which were inserted in the end compartments and connected with a direct current lighting circuit at a pressure of 110 volts, this yielding a current of approximately 1.5 ampères through the cell. The temperature was maintained below 35° C. by means of cold water circulating through a test tube immersed in the centre compartment. Solutions of hydrolytic products of casein, gelatin, fibrin and red blood cells were submitted to electrolysis by placing them in the centre compartment and retaining the P_H value constant by frequent additions of barium hydroxide solution, whilst the anode and cathode compartments contained distilled water which in the cathode compartment was saturated with carbon dioxide throughout an experiment. After 5 hours' electrolysis with a P_H of 5.5, arginine, lysine and histidine were found in the cathode compartment, whilst with a P_H of 7.5 arginine and lysine only were transported, the histidine remaining in the central liquid. In the most favourable results approximately 90 per cent. of the hexone bases were separated. T. J. W.

Estimation of Calcium in Serum by the Kramer-Tisdall Method.

F. F. Tisdall. (*J. Biol. Chem.*, 1923, **56**, 439-441.)—The above method (*J. Biol. Chem.*, 1921, **47**, 475) has been simplified and shortened by the following modification:—Two c.c. of fresh serum are added to 2 c.c. of water contained in a 15 c.c. graduated centrifuge tube having an external diameter of 6 to 7 mm. at the 0.1 c.c. mark. One c.c. of saturated ammonium oxalate solution is added, and the liquids are thoroughly mixed by rotating and tapping the tube, this procedure being repeated after the tube has stood for 30 minutes. The tube is then centrifuged at 1500 revolutions per minute for 5 minutes, and the supernatant liquid is poured off, after which 0.6 per cent. ammonia solution is poured down the sides of the tube to give a volume of 4 c.c., and the contents are mixed by rotation until slight disturbance of the precipitate occurs, the tube being then again centrifuged for 5 minutes. The clear liquid is poured off, and the washing is repeated, after which the precipitate is dissolved in 2 c.c. of *N* sulphuric acid and, after being heated in a boiling water-bath for 1 minute, the solution is titrated with 0.01 *N* potassium permanganate solution until a definite pink colour persists for at least 1 minute. The number of c.c. of potassium permanganate solution used multiplied by 10 equals the weight in mgrms. of calcium contained in 100 c.c. of serum. Tables are given showing that similar results are obtained, whether the precipitation is allowed to proceed for 30 minutes or 18 hours, and that the results are in agreement with those obtained by the original method. T. J. W.

Toxicological and Forensic.

Diazotisation of the Benzoyl Radical in Toxicological Examinations for Alkaloids. **H. Pecker.** (*J. Pharm. Chim.*, 1923, **28**, 13-15.)—In the case of an examination of the organs of a person who had died from supposed

belladonna poisoning it was found that the residue obtained by evaporation of the ethereal extract gave a strong positive reaction with iodine solution, suggesting the presence of alkaloids. Since Vitali's reaction for atropine was negative, Guerbet's reaction for organic compounds with benzoyl radicles (*J. Pharm. Chim.*, 1920, 22, 321) was then applied, by taking up the residue two or three times with a few drops of fuming nitric acid, adding a drop of 10 per cent. stannous chloride solution, and drying. On cooling, 2 drops of a 1 per cent. sodium nitrite solution were added, and, subsequently, a few drops of 1 per cent. resorcinol in 10 per cent. ammonia solution. A bright orange-red coloration was immediately formed which turned violet on addition of 1 c.c. of concentrated sulphuric acid. This reaction could not be attributed to the presence of atropine, for in the course of carrying out Vitali's reaction a slight odour of mint was noticed; and, further, crystals of cocaine picrate were formed. Guerbet's reaction was then applied under the same conditions to atropine, cocaine, and the above ethereal extract with the addition of atropine. In the first and third cases the reaction was strongly positive, and in the second a slight rose colour was produced. It was concluded that atropine or belladonna was not responsible for the poisoning, and that the trace of cocaine present could not have produced so pronounced an orange coloration. It was further found that the presence of ptomaines resulting from putrefaction caused a positive reaction in Guerbet's test, and it appears therefore that benzoyl diazotisation cannot be used for proving the presence of benzoyl derivatives such as stovaine, cocaine and atropine.

D. G. H.

Water Analysis.

Estimation of Carbon Dioxide in Drinking Water. P. Lehmann and A. Reuss. *Zeitsch. Unters. Nahr. Genussm.*, 1923, 45, 227-236.)—Schloesing's formula (*Comptes rend.*, 1872, 74, 1552; 75, 10) has been re-calculated so that the free carbon dioxide is expressed as mgrms. of CO_2 per litre, and the dissolved calcium in its equivalent of mgrms. of combined CO_2 (1 mol. $\text{Ca}(\text{HCO}_3)_2 = 1$ mol. CO_2) per litre, and a reference table has been based on the results. When the sum of the combined and free carbon dioxide exceeds a certain limit (200 mgrms. per litre), a correction is required for the dissociation which then occurs. This correction is also embodied in the table. In practice, two to three drops of an aqueous 0.01 per cent. solution of methyl orange are added to 250 c.c. of the water, which is then titrated with 0.1 *N* hydrochloric acid. This gives the combined carbon dioxide. For the estimation of the free carbon dioxide 200 c.c. of the water are treated with 1 c.c. of Tillmann and Heublein's phenolphthalein solution (0.375 gm. in 1 litre of alcohol) and titrated with sodium carbonate solution (2.409 grms. per litre; 1 c.c. = 1 mgrm. CO_2), until a pink colour persists for at least 5 minutes at 15° to 20° C. If, for example, the first titration has given 80 mgrms., and the second 50 mgrms. of carbon dioxide, the sum (*s*) is 130, which corresponds to the value 102.6 in the table, and the difference between this value and the 80 mgrms. first found gives 22.6 mgrms. as the amount

of solvent carbon dioxide per litre. Tillmann and Heublein's curve (*Zeitsch. Unters. Nahr. Genussm.*, 1917, **33**, 289), gave 22.5 mgrms. in this instance.

Solvent Carbon Dioxide in Water in mgrms. per Litre.

s = sum of the total carbon dioxide found.

G = calculated sum of the combined and solvent carbon dioxide multiplied by the correction factor for the dissociation of the calcium hydrogen carbonate.

s	G	s	G	s	G	s	G	s	G
1	1	68	62.0	100	84.6	136	105.9	200	136.0
5	5	70	63.5	102	85.9	140	108.1	210	141.6
10	10	72	65.0	105	87.6	145	110.7	220	145.6
15	14.9	74	66.5	108	89.7	150	113.2	240	153.8
20	19.8	78	69.5	110	90.9	154	115.3	250	157.5
25	24.6	80	71.0	112	92.2	158	117.3	270	164.9
30	29.4	82	72.4	115	94.0	160	118.1	290	171.9
35	34.0	84	73.8	118	95.8	165	120.6	300	175.3
40	38.6	88	76.6	120	97.0	170	123.0	320	182.1
50	47.3	90	78.0	124	99.2	175	125.2	340	188.3
55	51.5	92	79.3	126	100.4	180	127.5	360	194.2
60	55.7	94	80.8	128	101.5	185	129.7	370	197.3
62	57.2	96	82.1	130	102.6	190	131.9	390	202.8
65	59.6	98	83.3	134	104.8	195	134.0	400	205.7

Organic Analysis.

New Volumetric Method of Elementary Analysis. L. Hackspill and G. de Heeckeren. (*Comptes rend.*, 1923, **177**, 59-60.)—In presence of cupric oxide, but in absence of free oxygen, the combustion of an organic compound may be rendered complete if carried out in a vacuum and at 800° to 900° C., carbon dioxide, nitrogen and water being obtained. At 80° C. the water may be completely condensed free from the least trace of carbon dioxide, so that the latter, together with the nitrogen, may be collected by means of a mercury pump and the mixture analysed. Passage of the water vapour over calcium hydride at the ordinary temperature results in quantitative liberation of the hydrogen, $\text{CaH}_2 + 2\text{H}_2\text{O} = \text{Ca}(\text{OH})_2 + 2\text{H}_2$; the volume of this hydrogen may be measured.

In applying this method to the elementary analysis of an organic compound, use is made of a silica tube of 350 mm. length and 12 mm. bore, the rest of the apparatus including a bulb in which the water is condensed, a calcium hydride tube with terminal cocks, and a Sprengel pump, all these being fixed together; a side tube is arranged so that carbon dioxide and nitrogen may be drawn off without traversing the hydride. The weighed substance is introduced into the closed end of the silica tube, which is then charged with calcined cupric oxide to the extent of two-thirds and with a plug of very fine copper wire; the tube is then arranged in a vertical position and connected with a mastic joint to the remainder of the apparatus. The substance is then cooled with solid carbon dioxide and acetone, so that its vapour pressure becomes practically zero, and the pressure in the apparatus reduced to 0.01 mm. The silica tube is now heated gradually from the top downwards by means of an electric furnace 200 mm. in length, the water bulb being cooled meanwhile with the carbon dioxide and acetone. The whole of the carbon dioxide and nitrogen is collected in about 2 hours, the lateral tube being then

closed, the calcium hydride tube opened, the water bulb allowed to assume the ordinary temperature, and the liberated hydrogen collected; this reaction occupies at least three hours, or more if the hydride is coated with lime from use.

The method gives good results except for the nitrogen, for which rather more than the true percentage is found. If measuring tubes reading to 0.01 c.c. are employed, it requires only 0.02 to 0.03 grm. of substance. T. H. P.

Estimation of the Carbon Content of Solutions. J. Needham. (*Biochem. J.*, 1923, 17, 431-434.)—The following method is applicable to solutions containing less than 20 mgrms. of carbon-containing substance per 100 c.c. and may be carried out in about 30 minutes. A source of oxygen is connected with a series of wash-bottles and towers containing sulphuric acid, soda-lime, potassium hydroxide and baryta solution, followed by an ordinary silica combustion tube containing a silica boat into which the solution under examination is measured, copper gauze and copper oxide. To the other end of the combustion tube is attached a bulb for collecting condensed moisture, followed by a test tube containing saturated baryta solution and, suspended above it, a small vertical tube containing saturated tartaric acid solution. On completing the combustion the carbon dioxide formed is in combination with the baryta and, after the exit tube has been connected with a gas-analysis apparatus, the tartaric acid solution is tilted into the baryta solution, thus liberating the carbon dioxide, which is then transferred to the measuring apparatus by means of a stream of oxygen. From time to time a blank estimation should be carried out, the value of which becomes more constant as the apparatus is used. An estimation of 5 c.c. of a solution containing 0.184 per cent. of inositol showed an error of approximately 0.1 per cent. on the solid present. T. J. W.

Detection of Methyl Alcohol in Ethyl Alcohol. R. Meurice. (*Ann. Chim. anal.*, 1923, 5, 204-205.)—If 10 c.c. of 95 per cent. ethyl alcohol are shaken with 10 c.c. of 22 per cent. aqueous solution of ammonium sulphate and allowed to stand for about 2 minutes the mixture separates into two layers, the lower one being saturated ammonium sulphate solution and the upper layer aqueous alcohol. After a longer time interval the lower layer becomes cloudy and deposits crystals. If the alcohol contains 10 per cent. of methyl alcohol the liquid no longer separates into two layers, but produces a voluminous crystalline precipitate. In the presence of 3.5 per cent. of wood spirit the same precipitation takes place, but with 1.7 per cent. there is separation into two layers, and the methyl alcohol only produces a more intense turbidity in the lower layer. The limit of sensitiveness of the test is therefore 3 per cent. The method is available for the detection of wood spirit in alcoholic liquids by first rectifying them to 95 per cent. and then applying the above test, which should be carried out at about 18° C. H. E. C.

Estimation of the easily hydrated Alcohols of Essential Oils. L. S. Glichitch. (*Comptes rend.*, 1923, 4, 268-270.)—The author finds that a more exact method than that of Boulez (*Bull. Soc. Chim.*, 1907, 117) and modified by

Schimmel et Cie (*Bull. Sem. Schimmel*, 1907, 123) for estimating tertiary alcohols partly dehydrated by boiling, such as linalol, is to esterify them in the cold with the mixed anhydrides of acetic and formic acids, and he proceeds as follows:—One part of formic acid (sp. gr., 1.22) is poured into 2 parts of acetic anhydride (100 per cent.) free from chlorine, and the temperature kept below 15° C. The temperature is subsequently raised to 50° C., and the mixture then quickly cooled. Fifteen c.c. of this mixture are placed in a 30 c.c. flask, and 10 c.c. of the essence to be analysed added. The flask is shaken, corked and kept in ice water. The temperature is gradually allowed to come to normal and the flask is left for 72 to 96 hours at 20° C. The excess of anhydride is then hydrated by digestion with 50 c.c. of water for 2 hours in the cold, the oil washed with water, with 5 per cent sodium bicarbonate solution and again with water, dried over anhydrous sodium sulphate and saponified, the boiling being continued for 1½ hours in order to saponify the small quantities of terpene esters formed by isomerisation. The proportion of alcohol is then calculated from the formula of Schimmel:—

$$\text{Alcohol per cent.} = \frac{n.M.}{10(p - 0.028n)}$$

where n is the number of c.c. of normal potassium hydroxide solution required to saponify p grms. of the formylated compound, and M the molecular weight of the alcohol to be estimated. In order to calculate the proportion of free and total alcohols, taking account of the esters existing in the essence before esterification, the following formulæ may be used:—

$$\text{Free Alcohol, per cent.} = \frac{M(n' - n)}{10(p - bn')}$$

$$\text{Total Alcohol} = \frac{p - bn}{p - bn'} \times \frac{n'M}{10p}$$

where M is the molecular weight of the alcohol to be estimated, n the number of c.c. of normal potassium hydroxide necessary to saponify p grms. of the original essence, n' the number required by the esterified essence, and b the factor corresponding to the esterifying acid, which is $\frac{R.COOH - 18}{1000}$ (*i.e.* 0.0042 for acetic, and 0.028 for formic acid). The method has so far been limited to the estimation of linalol, although it should prove useful in doubtful cases, and where the alcohol is readily destroyed on warming in acid medium. D. G. H.

Variation of Refractive Index of Chinese Wood (Tung) Oil with Temperature. F. H. Rhodes and H. E. Goldsmith. (*J. Ind. Eng. Chem.*, 1923, 15, 786.) —Results of determinations of the refractive index of two specimens of genuine Chinese wood oil showed that, between 10° and 40° C., the value decreases 0.000395 for each rise of 1° C. in the temperature. W. P. S.

Titration of Sodium Formaldehydesulphoxalate. B. Salkin. (*J. Ind. Eng. Chem.*, 1923, **15**, 848-849.)—A combination of the copper sulphate and iodimetric methods is recommended for the estimation of sodium formaldehydesulphoxalate; the titrations also give the quantity of sodium bisulphite formaldehyde present. The ammoniacal copper sulphate solution used is prepared by dissolving 500 grms. of crystallised copper sulphate in 2 litres of water, adding 1600 c.c. of 28 per cent. ammonia, and diluting the solution to 4 litres; the exact copper content of this solution must be estimated. Only recently boiled distilled water is used, and the sulphoxalate solution must always be kept under an atmosphere of nitrogen. Fifteen grms. of the samples are dissolved in 100 c.c. of water (Solution A); 10 c.c. of this solution are then diluted to 250 c.c. (Solution B). *First Titration.* Fifty c.c. of the ammoniacal copper solution and 40 c.c. of 28 per cent. ammonia solution are placed in a flask, the mixture is heated at 55° C., a current of nitrogen is passed into the flask, and the copper is titrated with solution A until the mixture is colourless. Each c.c. of copper solution (containing 0.07982 gm. of copper sulphate) is equivalent to 0.0295 gm. of sodium formaldehydesulphoxalate. *Second Titration.* A flask is filled with nitrogen, 25 c.c. of solution B are introduced and titrated with 0.1N iodine solution; 2 c.c. of the iodine solution are added in excess and, after five minutes, this excess is titrated with 0.1 N thiosulphate solution. Each c.c. of 0.1N iodine solution is equivalent to 0.00295 gm. of sodium formaldehydesulphoxalate. *Third Titration.* Into a flask filled with nitrogen are introduced 25 c.c. of solution B and a quantity of 0.1N iodine solution equal to 50 c.c. more than was used in the second titration. Ten per cent. sodium hydroxide solution is then added until the colour of the mixture is slightly yellow and, after ten minutes, the mixture is acidified with dilute hydrochloric acid and the iodine is titrated with 0.1N thiosulphate solution. From the number of c.c. of iodine solution reduced in the titration subtract 1.5 times the number of c.c. reduced in the second titration; the difference is multiplied by 0.00335 to obtain the quantity of sodium bisulphite formaldehyde present. A difference in the amounts of sodium formaldehydesulphoxalate found in the first and second titrations indicates the presence of sodium hyposulphite in the sample. W. P. S.

Estimation of Benzene in Coal Gas by Means of Active Charcoal. R. Kattwinkel. (*Chem. Zeit.*, 1923, **47**, 682-684.)—In reply to Krieger's criticism (*ANALYST*, 1923, **48**, 347), the author states that charcoal loses its efficiency only after hundreds of estimations. The apparatus and method used are described in detail, and figures are adduced to prove that under the conditions given, Krieger's objection as to the moisture retained by the charcoal reducing its absorbing power, cannot be upheld; the charcoal, after treatment with superheated steam, is air-dry (average moisture content 7.0 per cent.) and again ready for immediate use. For drawings, etc., the original must be consulted. W. R. S.

Estimation of Phenols in Coal-Tar Oils and Crude Carboic Acid. J. B. Hill. (*J. Ind. Eng. Chem.*, 1923, **15**, 799-800).—Results of an investigation of various methods are recorded, particular attention being given to the contraction

method described by Weiss (ANALYST, 1919, 44, 58), in which the contraction in volume of an oil on extraction with sodium hydroxide solution is taken as a measure of the phenols present. Statements that this method yields high results are without foundation. High results are given, however, by the methods in which the tar acids are extracted from the oil with sodium hydroxide solution, liberated from the phenolate solution by the addition of acid, and then measured. W. P. S.

Modified Test for Phthalates, with Particular Reference to the Detection of Diethyl Phthalate. R. E. Andrew. (*J. Ind. Eng. Chem.*, 1923, 15, 838.)—The following test is recommended for the detection of phthalates and especially of diethylphthalate, as this substance is now used for denaturing alcohol intended for the preparation of toilet products. Ten c.c. of the solution to be tested are evaporated to dryness, with the addition of 5 drops of 10 per cent. sodium hydroxide solution, and the residue is treated with 0.5 c.c. of 5 per cent. resorcinol solution, again evaporated, and treated with 6 drops of concentrated sulphuric acid. The cold acid solution is diluted with 20 c.c. of water, transferred to a test-tube, and treated with 5 c.c. of 10 per cent. sodium hydroxide solution. A green fluorescence appears if the original solution contained a phthalate. The test will detect as little as 0.0002 grm. of diethyl phthalate in 10 c.c. of alcohol. In testing solutions containing large quantities of extractives it is best to extract the phthalate with petroleum spirit, evaporate the solvent at ordinary temperature, dissolve the residue in alcohol, and use this solution for the test. W. P. S.

The Gelatin-Tannin Reaction. A. W. Thomas and A. Frieden. (*J. Ind. Eng. Chem.*, 1923, 15, 839–841).—Gelatin is precipitated completely by gallotannin when the ratio of tannin to gelatin is not less than 2:1, and the gelatin-tannin precipitate is not soluble in excess of tannin. There is a definite hydrogen ion concentration (at or near $C_{H^+} = 10^{-4.4}$) at which each different vegetable tannin gives the best precipitation; if the hydrogen ion concentration is not adjusted near this point the precipitate may fail to form. The presence of a neutral electrolyte broadens the range for precipitation. At the optimum hydrogen ion concentration as little as 1 part of tannin in 200,000 parts of water can be detected, but this depends to some extent on the source of the tannin, that contained in wattle bark extract being the most sensitive. Provided that there has been no bacterial decomposition, the age of the gelatin solution is without effect on the sensitiveness of the test. W. P. S.

Estimation of Tryptophan. G. E. Holm and G. R. Greenbank. (*J. Amer. Chem. Soc.*, 1923, 45, 1788–1792).—The errors inherent in the estimation of tryptophan by means of aldehydes are considered, and Herzfeld's *p*-dimethylamino-benzaldehyde method (*Biochem. Zeitsch.*, 1915, 56, 256) is found satisfactory if the reaction time allowed is sufficiently long for the attainment of the maximum intensity of colour. At 25° C. the reaction is slow, but at 37° C. it is much more rapid, and the colour shows its maximum development in 6 to 8 days. At higher temperatures secondary changes set in and the colour is not permanent. About

3 mgrms. of the tryptophan are added to 100 c.c. of 20 per cent. hydrochloric acid, to which has been added an excess of the aldehyde, and allowed to stand at 37° C. for 8 days; the colour is then compared in a colorimeter with standards similarly prepared from known tryptophan. Although the tryptophan content of proteins may be estimated as above without previous hydrolysis, it is preferable to submit it to enzymic hydrolysis before the estimation; otherwise the colour attains a maximum intensity which is not permanent. It is found that Gortner and Holm's method of estimating tryptophan by "humins" formation (*J. Amer. Chem. Soc.*, 1917, **39**, 2485) gives results which agree well with those of the colorimetric method.

H. E. C.

Inorganic Analysis.

Analysis of Hydrogen for Traces of Nitrogen. R. L. Dodge. (*J. Amer. Chem. Soc.*, 1923, **45**, 1688-1691.)—The method consists in the combustion of a volume of about 15 litres of the hydrogen by passing it over heated copper oxide in an evacuated system. The residual gases are circulated over the copper oxide to ensure complete removal of the hydrogen; and the unburned gas, which is designated as nitrogen, is measured by the usual gas volumetric method. Errors due to incomplete evacuation are eliminated by carrying the initial and final evacuation to the same point (0.01 mm.). The volume of the hydrogen burned is calculated from the volume of water formed by the combustion. A special apparatus is described and figured in which the residual gas is pumped off by a Topley pump and measured; the results are capable of an accuracy of 0.005 per cent. Ordinary commercial hydrogen shows about 0.47 per cent. of nitrogen, and special electrolytic hydrogen contains 0.04 per cent., which is probably a minimum quantity.

H. E. C.

New Indicator for Acidimetry. A. Reisenleitner. (*Chem. Zeit.*, 1923, **47**, 689.)—The colouring matter of the red petals of the flower of the wild carrot (*Daucus carota*) is readily soluble in hot alcohol. The solution is wine-red with acids and dark green with alkalis, and when very dilute, is colourless at the point of neutrality. The indicator is not sensitive to carbonic acid, and may be used in the titration of organic acids.

Hexamethylene Tetramine as a Microchemical Reagent. H. E. Cole. (*Philippine J. Sci.*, 1923, **22**, 631-640.)—Solutions of salts of antimony, bismuth, cadmium, gold, iridium, mercury, palladium, platinum, silver and tin form characteristic crystalline precipitates with hexamethylenetetramine in the presence of hydrochloric acid (silver in nitric acid solution). Salts of calcium, ferrous iron, magnesium, manganese and titanium also form crystalline compounds, but these are not sufficiently characteristic to enable them to be distinguished. Other metallic salts yield very soluble crystalline compounds. The test is best applied by adding a fragment of the solid reagent to a drop of a solution of the metallic salt.

Antimony forms colourless octahedra and dodecahedra. (Sensitiveness, 1:1500).

If no crystals appear, a fragment of potassium iodide is added. Yellow octahedra are formed in presence of antimony (1:15,000). Bismuth and tin also yield yellow octahedra, but the crystals are smaller.

Bismuth forms large colourless highly refractive octahedra and dodecahedra (sensitiveness, 1:8000). The crystals never polarise light, and may thus be distinguished from antimony. On adding potassium iodide to the solution an amorphous yellow precipitate forms, gradually changing into minute yellow octahedra. This is an exceedingly delicate test for bismuth (sensitiveness, 1 in 100,000). Bismuth may be distinguished from tin by adding to the test drop a fragment of caesium chloride and then a fragment of potassium iodide. Bismuth yields orange-red hexagonal plates, whilst tin yields yellow octahedra.

Tin forms colourless octahedra with the reagent alone (sensitiveness, 1:1000), and minute yellow octahedra on the addition of potassium iodide (1:1000).

Cadmium forms thin colourless plates belonging to the hexagonal system (1:200), and, in presence of potassium iodide, it yields square-ended colourless prisms and hexagonal plates (1:2200).

Gold yields characteristic pale yellow needles or thin plates, showing strong polarisation with parallel extinction. Mercury and silver interfere with the test (sensitiveness, 1:900).

Iridium salts yield very characteristic red-brown octahedra or crosses belonging to the isometric system (sensitiveness, 1:2000).

Platinum salts yield highly refractive pale yellow octahedra (1:5000). In the case of mixtures of platinum and palladium, the platinum crystallises first.

Palladium. Palladous salts give very thin colourless plates with strong polarising power (sensitiveness, 1:8000).

Mercury salts yield colourless square-ended slender prisms (1:1000).

Silver salts in nitric acid solution form colourless needles or plates belonging to the monoclinic system (1:200).

Free sulphuric, hydrochloric or nitric acids give crystals with the reagent and potassium iodide, but these are readily distinguished from those formed with the above-mentioned metals.

Microchemical Separation of Antimony and Tin. A. P. Ortodogsu and M. Ressay. (*Bull. Soc. Chim.*, 1923, 33, 991-994.)—When a solution of tin and antimony in dilute hydrochloric acid is evaporated to dryness with sodium chloride solution, characteristic crystals having the composition respectively 2SnCl_2 , 3NaCl and SbCl_5 , NaCl are obtained. The antimony salt is in the form of a cross and tin in the form of six-branched stars. These are easily distinguishable under the microscope and by simply mixing a drop of sodium chloride solution with a drop of the solution to be tested, slowly evaporating to dryness and examining the crystals under the microscope the presence of either or both of these metals is easily detected. The limit of sensitiveness using magnifying power of 100 is 0.06 mgm. for antimony and 0.08 mgm. for tin. The solution should not be more concentrated than 20 to 30 per cent., as in such cases it is difficult to distinguish the crystals. No other known metals give crystals of these kinds. H. E. C.

Micro-Chemical Estimation of Copper by means of Molybdenum.

Fontès and Thivolle. (*Bull. Soc. Chim.*, 1923, **33**, 835–844.)—Phospho-molybdic acid dissolves finely divided metallic copper, with formation of a blue solution (MoO_2), and the colour of the solution can be discharged by careful addition of a weak solution of potassium permanganate (with formation of MoO_3). The phospho-molybdic acid solution is prepared by dissolving 40 grms. of ammonium molybdate in 100 c.c. water, adding 60 c.c. of sodium hydroxide solution (sp. gr. 1.36), boiling off the ammonia and cooling, after which 200 c.c. water are added, then 200 c.c. of phosphoric acid (sp. gr. 1.38), and the whole boiled for 15 minutes, cooled and diluted to 1 litre. The potassium permanganate solution used contains 0.08 gm. per litre. If the copper to be estimated is in solution, it is reduced to the metallic state either (A) by electrolysis or (B) by precipitation with α -nitroso- β -naphthol, but only in absence of iron and cobalt. In the case of method A the solution to be tested is acidified with 3 drops each of strong sulphuric and nitric acids. It should not exceed 5 c.c., and should contain between 0.1 mgrm. and 5 mgrms. of copper. It is contained in a small tube 2 cm. in diameter by 8 cm. high, provided with a cock at the lower end to facilitate washing without interrupting the current. This is most important so as to avoid re-solution of the copper in the acid solution. The cathode is a cylinder of platinum gauze 3 cm. by 1 cm. The time required for electrolysis is 10 minutes with a current of 0.3 to 0.5 ampère. In the case of method (B) a measured volume of the copper solution is placed in a 30 c.c. decantation flask and rendered neutral or very slightly acid. An excess of nitroso- β -naphthol solution containing 4 per cent. of acetic acid is added, and the whole stirred and allowed to stand some minutes, after which it is filtered, the precipitate washed, dried, and ignited in a platinum or porcelain boat, in a combustion tube, first in air and then in hydrogen. The metallic copper obtained by either (A) or (B) is dissolved in a few c.c. of the phospho-molybdic acid solution, which process may be accelerated by moderate warming, and the blue coloured solution is titrated with the permanganate solution (1 c.c. = 0.0783 mgrm. Cu). The permanganate solution can be standardised by means of pure ferrous ammonium sulphate in phosphoric acid solution. R. F. I.

Micro-chemical Estimation of Iron by means of Molybdenum. **Fontès and Thivolle.** (*Bull. Soc. Chim.*, 1923, **33**, 844–849.)—This process is based on the following facts:—1. Ferrous salts in phosphoric acid medium are practically unoxidisable in the cold. 2. Metallic copper in acid medium at the boiling point reduces ferric salts to the ferrous condition, the quantity of copper dissolved being molecularly proportional to the quantity of iron reduced. To a solution of ferric sulphate containing 1 to 5 mgrms. of iron is added a slight excess of phosphoric acid (1 to 2 c.c. at 40° Bé), then one or two fragments of copper turnings previously cleaned in nitric acid. The liquid is boiled for 10 minutes, cooled rapidly, and filtered on to a small Buchner funnel. The vacuum-flask for receiving the filtrate already contains 5 c.c. of the phosphomolybdic acid solution. (See preceding abstract.) The precipitate is washed with 0.1 per cent. phosphoric acid

solution, and the filtrate and washings titrated with permanganate solution as above. If large amounts of sulphates are present, as in Kjeldahl estimations, it is best to precipitate the iron by means of sodium hydroxide and a few drops of magnesium sulphate (to render the precipitate granular), dissolve the ferric hydroxide in a few c.c. of phosphoric acid and proceed as above. A more accurate method for quantities of 0.1 mgrm. to 1 mgrm. of iron is to precipitate the iron with nitroso- β -naphthol as in the case of copper (preceding abstract). The finely divided metallic iron obtained readily dissolves in the phospho-molybdic solution.

R. F. I.

Microscopical Appearance of Potassium and Sodium Picrate and Tartrate Crystals. E. Justin-Mueller. (*J. Pharm. Chim.*, 1923, 28, 15-17.)—

Microscopical examination of sodium and potassium picrate crystals shows the former to consist of long yellow prisms and the latter of fine yellow needles. Potassium tartrate crystals are only visibly formed in a medium of certain concentration, whilst sodium tartrate crystals are invisible microscopically. A microscopical examination of the deposits shows, however, that the potassium tartrate is for the most part in the form of fairly abundant diamond-shaped crystals, whilst the sodium tartrate is present as thinly distributed octagonal plates and monoclinic prisms.

D. G. H.

Physical Methods, Apparatus, etc.

Quantitative Estimation of Radium by the Emanation Method. C. E. Baumgarten and H. H. Barker. (*J. Ind. Eng. Chem.*, 1923, 15, 597-599.)—

When sulphuric acid is used as the solvent for the estimation of radium in an ore it is necessary to decompose the ore with a small quantity of nitric acid followed by boiling with concentrated sulphuric acid to reduce the activity to zero; for the subsequent collection of the emanation the mixture must be boiled again and a current of air aspirated through it. Phosphoric acid may be used in place of sulphuric acid, 30 c.c. of 85 per cent. phosphoric acid being sufficient for 1 to 3 gm. quantities of ore; the mixture is heated until it boils gently (five to seven minutes being required), and the boiling is continued for two minutes to ensure that the temperature reaches 300° C. The boiling tube is then sealed and set aside for a definite period of growth of emanation previous to the collection of the latter in the usual way.

W. P. S.

Tungsten Wire for Hydrogen Ion Determinations. J. R. Baylis. (*J. Ind. Eng. Chem.*, 1923, 15, 852-853.)—For the determination of the change in hydrogen ion concentration produced by the addition of purifying chemicals to a water supply, the author has used the tungsten wire in an ordinary 40-watt electric lamp as an electrode. A hole was ground into the lamp globe large enough for the introduction of two small tubes, one for the potassium chloride solution and the other for the water. The maximum range of the hydrogen ion concentration

of the water in question was from 6.5 to 8.6, and for each unit there was a voltage difference of 0.09. The use of hydrogen is eliminated, and it is possible that the electrode may be employed over a wider range than that mentioned. W. P. S.

Reviews.

CHEMISTRY: INORGANIC AND ORGANIC. By C. L. BLOXAM. Eleventh Edition. Revised by A. G. BLOXAM, F.I.C. and S. JUDD LEWIS, D.Sc., F.I.C. Pp. ix.+832. 310 Illustrations. London: J. & A. Churchill. 1923. Price 36s. net.

Bloxam's Chemistry has served as a chemical bible for many generations of students, and it well deserves the reputation which it has built up. So far as the reviewer is aware, there is no other elementary textbook on chemistry which covers so wide a field, and in which the answers to even out-of-the-way questions can so readily be found.

The first edition was published as far back as 1867, and so the demand for an eleventh edition is in itself a proof that the book continues to supply the want for a concise hand-book dealing with both inorganic and organic chemistry in a single volume.

Various additions have been made to this edition to bring it up to date. For example, brief descriptions are given of the quantum theory, the phase rule, the structure of crystals, etc., and the section on spectroscopy has been thoroughly revised.

Notwithstanding the large amount of additional matter, the size of the volume has been kept within its acceptable limits by the use of a somewhat smaller type. In the 7th edition of 1890 there were 799 pages, whilst this new edition contains 832 pages of a rather larger size.

One feature for which the book has long been noted is the way in which it interests a student in the chemical reactions involved in manufacturing processes, and this feature is still to be found in the new edition, in which short sections are given to dyeing, the manufacture of various explosives, the refining of sugars, and so on. In this connection it might perhaps be pointed out that the description of the manufacture of vinegar is not altogether accurate, and that the use of the term *Mycoderma aceti* for acetic bacteria is quite obsolete.

The section on oils and fats also requires bringing up-to-date in certain places. For instance, cottonseed oil (as well as sesame oil) contains linolin; "jecolein" is not now accepted as a glyceride of cod liver oil; and there is no reference to clupanodonic acid. The statement that stearic acid exists in fats in the form of stearin mixed with palmitin and a little olein requires some modification. It is probable that free tristearin is only present to a very small extent in fats.

The editors rather disarm criticism of their method of grouping aliphatic with

aromatic compounds by adopting the parliamentary plan of ranging critics who approve of their method against those who object to it. In the present reviewer's opinion, the chief objection to this classification is the mechanical one that it makes a sequence of references to other works on chemistry somewhat less easy.

Although, as already mentioned, the type is smaller than in previous editions, the clearness of the printing and the good spacing of the paragraphs make the book easy to read, and, regarded as a whole, the new edition can be warmly recommended, not only as a textbook for students, but also as a general work for preliminary reference in the laboratory.

EDITOR.

PRACTICAL BACTERIOLOGY FOR CHEMICAL STUDENTS. By DAVID ELIIS, Ph.D., D.Sc., F.R.S.E. Illustrated. Pp. viii. + 136. London: Longmans, Green & Co. 1923. Price 4s. 6d. net.

This book contains a series of 108 exercises in the elements of bacteriology, and is designed to give trained chemists and chemical students sufficient guidance to enable them to master the general principles of the subject. The author has succeeded in producing what might well become a useful companion for the beginner in bacteriology. The hints to students are very good, although they might usefully be expanded, as, for instance, in an instruction to workers not to partake of food in a bacteriological laboratory where pathogenic organisms may be under investigation; and a warning not to put labels, pens or pencils near the mouth. Many things that may often be done with impunity in a chemical laboratory are unwise in a bacteriological laboratory, and students are often thoughtless on such points. Further, it is desirable that the worker should be taught to wash his hands thoroughly after the bench work is done, and before touching the microscope.

Some of the methods recommended by the author will scarcely meet with the general approval of teachers who may put this book into the hands of their students. For example, in the examination of water (page 96) the worker is instructed to draw up repeatedly the mixture of water and medium into the pipette and blow it out again, to get a good mixture before plating out. It is surely better to pipette the water direct into the petri dish, and pour the melted gelatin or agar over it; and this method of working obviates the doubtful expedient of adding five per cent. to the result! Again, the incubation of gelatin plates at room temperature for a variable period may be satisfactory for obtaining cultures in general examinations, but in water bacteriology it leaves much to be desired, as determinations of the number of organisms growing on gelatin should always be done by a strictly uniform procedure, and a definite incubation temperature of 20°—22° C. for three days is preferable on all grounds. It is surprising that the volume contains no mention of the standardisation of reaction of media by means of the colorimetric estimation of the H-ion concentration and its adjustment, as the chemical titration method described is out-of-date. The bacteriology of foods and the simpler methods of examination are not touched upon, and might usefully be added in any future edition.

Although good in intention, unfortunately the real value of this little book is marred by various small defects which should not appear in a volume to be largely used by students with no knowledge of the subject. The reading of the proofs leaves something to be desired, whilst unusual contractions of words, and lack of proper punctuation in many places, are irritating in a scientific textbook. Some of the illustrations are unnecessary (Figs. 9d, 16, 21), and others referred to in the text do not appear in the volume sent for review (Figs. 36 and 37, pages 85 and 101). The "needle" and "loop" are used indiscriminately and wrongly in the descriptions of Exercises 6 (p. 23) and 14 (pp. 29, 30). The descriptions of Figures 9f and 40, referred to on pages 6, 8 and 71, do not agree with the illustrations so numbered. The author would be well advised to shorten considerably the space taken up with details of calculations of dilutions and the use of standard solutions, since, after the first example, even the student will find them unnecessary. On page 44 it is stated that 3 per cent. alcoholic solution of hydrochloric acid is prepared by mixing 3 c.c. of alcohol and 97 c.c. of acid, but these figures should obviously be reversed.

When the defects pointed out have been removed in a later edition, this book will be found to offer the beginner in bacteriological technique a valuable series of exercises which cannot fail to be helpful, stimulating the worker's powers of observation, and introducing him to a science which will frequently be of service in investigating many problems where a knowledge of chemistry and bacteriology are alike necessary.

ARNOLD R. TANKARD.

AN INTRODUCTION TO THEORETICAL AND APPLIED COLLOID CHEMISTRY. By Prof. WOLFGANG OSTWALD. Authorised Translation from the Eighth German Edition by Prof. MARTIN H. FISCHER (Cincinnati). Second and Revised American Edition. Pp. xiii.+266. New York: John Wiley & Sons, Inc.; London: Chapman & Hall, Ltd. 1922. Price 12s. 6d. net.

In the winter of 1913-4, Professor Ostwald gave fifty-six lectures on colloid chemistry at various Universities in the United States and Canada. The five lectures given most often are presented in this volume, having first appeared in German under the title *Die Welt der Vernachlassigten Dimensionen* ("The World of Neglected Dimensions.").

Prof. Ostwald states that this selection of his popular lectures is an "attempt to give a general survey of modern colloid chemistry as a pure and as an applied science, and in a form readily intelligible to the general reader." He also designates the volume as "a propaganda sheet for colloid chemistry."

The five lectures (now expanded and revised) are entitled:

- (1) Fundamental properties of the colloid state. Colloids as examples of dispersed systems. Methods of preparing colloid solutions.
- (2) Classification of the colloids. The physico-chemical properties of the colloids and their dependence upon the degree of dispersion.
- (3) The change in state of colloids.

- (4) Some scientific applications of colloid chemistry.
- (5) Some technical applications of colloid chemistry.

An enthusiast wrote this book, himself a pioneer of modern colloid chemistry. Prof. Ostwald believes "that colloids constitute the most universal and the commonest of all the things we know" (p. 194), and that "colloid chemistry is the promised land of the biological scientist" (p. 165).

The treatment throughout is elementary and, perhaps, somewhat cursory, but it is exceptionally well presented and exceedingly clear. The translation, too, is admirable. Written in the first person, the style is unusual but arresting. The reviewer recommends the book as an excellent stimulus to all colloid chemists, and a pleasing introduction for students.

Prof. Ostwald lays stress on the principle of continuity, the gradual transition from coarse suspensions to molecularly dispersed solutions. "The colloids merely represent a realm differentiated for practical purposes from a continuous series of systems" (p. 22).

There is a more detailed account of the Tyndall cone than is usually presented in text-books on colloids. The subject of Liesegang rings is briefly touched upon, but no attempt is made to give any explanation of this interesting but complex phenomenon.

The last two lectures are particularly fascinating, and most readers will be surprised to find how extraordinarily wide and important are the ramifications of colloid phenomena in both pure science and industrial processes. As Prof. Ostwald himself concludes: "Things not only begin in colloid chemistry, but in colloid chemistry they end."

WILLIAM CLAYTON.

THE MANUFACTURE OF HYDROCHLORIC ACID AND SALTCAKE. By A. C. CUMMING, O.B.E., D.Sc., F.I.C. London: Gurney & Jackson. 1923. Price 31s. 6d

This is a volume of the revised edition of Lunge, in which the editor, Dr Cumming, is arranging the volumes so that technical data relating to specific industries are collected together, and much that is now obsolete in former editions is curtailed or cut out altogether. In this case the editor has acted in the capacity of author and has dealt with that part of the heavy chemical industry producing saltcake, hydrochloric acid and Glauber's salts.

Chapters I. and II. deal with raw materials and products, their analysis and purity; Chapters III. and IV. deal with the manufacture of saltcake and hydrochloric acid; Chapter V. is mainly historical, giving in a condensed form a summary of the process of Hargreaves and Robinson; Chapter VI., which is very short, deals with pure sulphate of soda and the manufacture of Glauber's salts; Chapter VII. with the condensation of hydrochloric acid; Chapter VIII. with the manufacture of hydrochloric acid from chlorine; and Chapter IX. with the manufacture of the acid by other than the usual methods; whilst Chapter X. deals with control, yields, costs and purification in the industry.

The requirements of Glauber's salts in the textile industry appear to have been overlooked in dealing with this product in Chapters II. and VI.; with the

substitution of soda-ash for saltcake in the glass industry on the increase, this outlet for saltcake is becoming of greater importance. On page 58 in the titration of chlorides by silver nitrate it would have been well to have recommended a blank titration for colour comparison and the detection of any error likely to creep in from chlorides contained in the water, &c. On page 79 *et seq.* the use of secondary air in the flues to the pot should have been given more prominence, as by this means the heat of the pot is mainly regulated when both furnace and pot are heated by the one furnace. It would have been valuable if some account could have been given of the new Tahn proposals for making saltcake, in which the bisulphate is made in one operation, and from this strong and fairly pure hydrochloric acid recovered, the bisulphate either being crushed and mixed with further salt for treatment in a furnace of the mechanical type such as the Mannheim furnace, or run, whilst molten, with fresh salt into a mechanical furnace of the Mactear type. The yields and costs are mainly historical and refer to pre-war conditions and must be re-cast to give any reference to altered conditions of to-day.

In dealing with processes for making sulphate of soda the author has omitted the application of the phase rule to mixed solutions, and a reference to the large quantities of this product which were produced during the war years as a by-product in the manufacture of ammonium nitrate; this should have been included, as further applications of these methods may have important industrial effects in the future. More details would also have been welcome of the various processes for the manufacture of hydrochloric acid from chlorine, as it appears that such processes will become of increasing importance in the near future. In the summary of the uses of hydrochloric acid, that of carbonising of the cotton in rags should have been mentioned, fairly large quantities being used in Yorkshire when trade is brisk.

The book is well written and deals very completely with the subjects treated; no doubt a future volume will deal with the manufacture of electrolytic chlorine and bleach, as modern developments on these lines are making much that is included in this volume of mainly historical interest, although questions of considerable technical importance, such as the comparative merits of open and blind roasters in saltcake manufacture, are very fully discussed.

It is somewhat difficult when dealing with an industry which has been of such capital importance, and yet to-day is being largely superseded by different methods of manufacture, such as the ammonia-soda and electrolytic methods, to give a concise account of details which yet may be of practical value, and the author is to be congratulated on the result of his task in this direction. The book will take its place in every technical library and will be appreciated by both students and technical men.

H. J. BAILEY.

BLEACHING POWDER AND ITS ACTION IN BLEACHING. By R. L. TAYLOR, F.I.C.
Pp. 78. Manchester: John Heywood, Ltd. 1922. Price 4s. 6d.

This little book consists of collected reprints of eight papers by the author on bleaching powder, originally published in the Transactions of the Chemical Society

and in the *Journal of the Society of Dyers and Colourists*, from 1910 to 1922. In the first paper is given a method of distinguishing analytically between chlorine and hypochlorous acid in mixtures of the two, depending on the different amounts of hydrogen chloride produced when the two gases are passed through a known volume of sodium arsenite. The author applies this method to the investigation of the action of air and of carbon dioxide upon bleaching powder under different conditions. A remarkable difference is found in the action on bleaching powder of ordinary air and of air free from carbon dioxide. In the former case the action proceeds fairly rapidly with evolution of a high proportion of chlorine, whilst in the latter it is much slower, and the product consists almost wholly of hypochlorous acid. The author points out that the action of chlorine ($2\text{Ca}(\text{OH})_2 + 2\text{Cl}_2 \rightleftharpoons \text{CaCl}_2 + \text{Ca}(\text{OCl})_2 + 2\text{H}_2\text{O}$) is a reversible one, and that the action of the carbon dioxide present in ordinary air is to remove free lime, thus allowing the reaction to proceed from right to left with production of more chlorine. Air free from carbon dioxide is practically without action on bleaching powder and merely sweeps out the small amount of hypochlorous acid originally present.

When pure carbon dioxide is passed through a mixture of bleaching powder and water a different reaction occurs. At the ordinary temperature chlorine is the main product, but at high temperatures hypochlorous acid predominates. In the latter case carbon dioxide acts, therefore, like boric acid, which, when boiled with bleaching powder, gives almost pure hypochlorous acid. The author discusses at length the mechanism of these reactions and presents evidence for the view that at ordinary temperatures carbon dioxide acts in the same way as the strong mineral acids, decomposing both calcium chloride and calcium hypochlorite, the resulting hydrochloric and hypochlorous acids reacting to give chlorine. At high temperatures very little true carbonic acid (H_2CO_3) can exist in solution, and consequently, carbon dioxide will decompose only the calcium hypochlorite. The author holds that the active bleaching agent in bleaching powder is chlorine, and that hypochlorous acid plays only a minor part. Observations on the action of bleaching powder on the colouring matters of linen and other fibres and textiles form the subject of Chapters IV. and V., and subsequent chapters deal with the estimation of chloric acid and chlorates and with the action of light on bleaching powder. In the last chapter the comparative bleaching action of chlorine and hypochlorous acid is discussed, and it is shewn that the various phenomena observed can be explained on the assumption that the reaction $\text{Cl}_2 + \text{H}_2\text{O} \rightleftharpoons \text{HCl} + \text{HClO}$ is reversible.

The book presents in a handy and convenient form the results of a valuable series of investigations, and should be of great interest and assistance to all chemists who are concerned with bleaching. Taylor's work, moreover, has an intimate bearing on the question of disinfection by hypochlorite solutions, so many of which are now on the market under proprietary names. G. W. MONIER-WILLIAMS.