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### The Estimation of Boric Acid in "Liquid Eggs" and other Foodstuffs.

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*(Read at the Meeting, April 4, 1923.)*

AMONG the various methods which have been proposed for the estimation of boric acid in foodstuffs, that of R. T. Thomson (Glasgow City Anal. Soc. Reports, 1895, 3) has always been regarded as one of the simplest and most universally applicable. In Thomson's method advantage is taken of the fact that calcium borate is fairly soluble in a weakly alkaline solution, while calcium phosphate is insoluble. Unless Thomson's directions be closely followed, and the addition of alkali carried out with the greatest care, one is liable to obtain low results, owing apparently to the solution becoming too strongly alkaline, with the result that some calcium borate is precipitated together with the phosphate. The margin of safety in the addition of alkali is rather narrow, and with some foodstuffs, such as liquid egg yolks, containing a large amount of phosphoric acid, it is not at all easy to avoid loss of boric acid by precipitation.

With a view to avoiding the occurrence of excessive alkalinity in the liquid, several experiments were carried out in which the neutralisation was effected by ammonia in place of sodium hydroxide, but the results obtained were frequently too low, indicating that calcium borate had been precipitated.

A more certain method, and one which appears to be universally applicable to foodstuffs, depends upon the removal of most of the lime from the solution in the form of calcium sulphate before neutralisation, and upon subsequent precipitation of the phosphoric acid by magnesia mixture (*cf.* L. de Koningh, *J. Amer. Chem.*

*Soc.*, 1897, XIX. 385). A convenient weight of the foodstuff is mixed with concentrated sodium hydroxide solution in a porcelain dish and the mixture thoroughly charred. In deciding on the quantity of sodium hydroxide to be used so as to secure retention of all the boric acid present, it is best to assume that the whole of the phosphorus and sulphur in the foodstuff will form phosphoric and sulphuric acids on ignition, an assumption which leaves an ample margin of safety. In the case of samples of liquid egg yolk containing approximately 1.20 per cent. of phosphoric acid and 2 to 4 per cent. of boric acid, sodium hydroxide is added at the rate of one gramme for every 10 grms. of egg yolk.

The char is extracted with a small quantity of hot water and the solution filtered direct into a 100 c.c. or 200 c.c. flask. The filtrate is acidified with sulphuric acid. If the original material contained a high proportion of silica, a slight precipitate may be formed on acidification, which need not be filtered off. In the absence of silica the solution, on acidifying, remains clear. The residual carbon, together with the filter, is dried and completely ashed, preferably in a muffle. The ash is taken up with a small quantity of water and acidified with sulphuric acid, the lime present being thus converted into calcium sulphate. The contents of the dish are filtered through a small filter into the measuring flask, and the residue washed free from acid, using at first a small quantity of cold water and completing the washing with boiling water. The liquid in the flask now contains the whole of the phosphoric acid and boric acid of the original char, together with a small quantity of calcium sulphate in solution. If the foodstuff contained much lime, most of this is left behind on the filter as calcium sulphate, since the solubility of calcium sulphate in dilute sulphuric acid is of the order of 0.2 per cent. In such cases it is advisable to use a 200 c.c. flask instead of 100 c.c. owing to the extra quantity of water required for washing the calcium sulphate.

To the solution in the flask, which should not amount to more than 70–80 c.c., or a correspondingly larger volume if a 200 c.c. flask be used, one drop of methyl red solution is added, and then sodium hydroxide solution until the liquid is neutral or only slightly alkaline to methyl red. Owing to the absence of a heavy precipitate of calcium phosphate, etc., it is easy to recognise the colour change of the indicator, and moreover even if the liquid be made slightly too alkaline there is not sufficient lime present to cause precipitation of calcium borate.

Magnesia mixture\* is now added in quantity sufficient to precipitate the whole of the phosphoric acid as magnesium ammonium phosphate, avoiding any great excess, and the mixture made up to the mark with distilled water.

Under these conditions there is no danger, with the quantities of phosphoric and boric acid usually present in foodstuffs, of boric acid being precipitated either as calcium or magnesium borate.

After being allowed to stand for 12 hours, or, alternatively, after vigorous shaking for five minutes in a stoppered bottle, the liquid is filtered. Fifty c.c. of

\* The magnesia mixture used is made by dissolving 68 grms.  $MgCl_2 \cdot 6H_2O$  and 165 grms.  $NH_4Cl$  in 300 c.c. of water, adding 75 c.c. of 0.880 ammonia and making up with water to one litre.

the filtrate are transferred to an Erlenmeyer flask, a small quantity of phenolphthalein added, and the liquid heated to boiling. Concentrated sodium hydroxide solution is then added drop by drop, until the liquid is strongly alkaline, the boiling being continued until all the ammonia has been driven off. At this stage the liquid should be strongly pink, and the excess of magnesia present should have separated out as a precipitate. This need not be removed, as it does not interfere with the final titration. The flask is cooled, one drop of methyl-red solution added, and then a strong solution of sulphuric acid drop by drop until the liquid is just acid to methyl-red, and the whole of the magnesia has been re-dissolved. From this point the procedure is the same as in Thomson's method. Carbon dioxide is removed by boiling for a few minutes, and the liquid, after cooling, made exactly neutral to methyl-red with 0.1 N sodium hydroxide. A fifty per cent. solution of glycerine, previously neutralised to phenolphthalein, is added in quantity sufficient to give about 30 per cent. of glycerine in the liquid, and the resulting solution is titrated with 0.1 N sodium hydroxide to the point of neutrality to phenolphthalein. It is important to add plenty of phenolphthalein to secure a sharp end-point.

The following test analyses were carried out by this method:—

Egg Yolk Grms.	Boric Acid added Grms.	NaOH added Grms.	Boric Acid found Grms.
5	0.195	0.75	0.200
10	0.205	1.0	0.200
10	0.099	1.0	0.099
20	0.106	2.0	0.104

Dried Milk Powder Grms.	Boric Acid added Grms.	NaOH added Grms.	Boric Acid found Grms.
20	0.200	2.0	0.200*
20	0.100	2.0	0.097

\* 200 c.c. flask used.

Mixture of:

1.338 gm.  $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$  (=0.25 gm.  $\text{P}_2\text{O}_5$ ).

0.2 gm.  $\text{H}_3\text{BO}_3$ .

0.7 gm.  $\text{CaCO}_3$ .

10 gm. dextrose.

Five c.c. of a 40 per cent. solution of NaOH.

A 200 c.c. flask was used and the amount of boric acid found was 0.209 gm.

## Estimation of Boric Acid in Cream, Milk and other Foods.

(Process used in the Government Laboratory).\*

THE method is a modification of the process described by R. T. Thomson (ANALYST, 1896, 21, 64).

The original process was found to be deficient in two aspects.

In the first place, it was found that boric acid, even when present in very small quantities, was precipitated along with the phosphates if the solution was made more than faintly alkaline to phenolphthalein indicator. The addition of lime water to the neutral solution at this stage was discontinued because it sometimes precipitated nearly all the boric acid. For the same reason, dilute sodium hydroxide solution should be added drop by drop, with constant shaking, during the whole period of phosphate precipitation, thereby avoiding regional alkalinity and the precipitation of boric acid which would not re-dissolve in the nearly neutral solution.

It was also found that boiling in beakers, covered with clock glasses to avoid loss by spirting, for a period of less than 15 minutes was not sufficient to drive off the whole of the carbonic acid from the heavily charged liquid, and the carbonic acid remaining in solution caused the titration for boric acid to be high. The greater part of the carbonic acid can be got rid of with safety from the slightly acid solution as indicated in the method, but the final boiling to get rid of carbonic acid should last for not less than 10 minutes.

Weigh out a suitable quantity of the sample in a platinum basin, and add three drops of phenolphthalein solution. Neutralise with normal solution of sodium hydroxide, and add 2 c.c. in excess. Evaporate on steam bath, stirring with a glass rod, until nearly free from moisture. With samples of liquid egg warm until the albumin coagulates before neutralising.

Extract fat twice with ether, and filter the ether extracts through paper into a small separating funnel. Wash the platinum dish and the filter paper with ether until free from fat. Wipe adhering solid from the glass rod with the filter paper, place the paper in the platinum dish, and wash the rod with water into the dish. Wash the ethereal extract first with 5 c.c. of *N* sodium hydroxide solution and then with 5 to 6 c.c. water, adding the washings to the platinum dish. Add sodium hydroxide solution, if necessary, until not less than 2 c.c. of *N* sodium hydroxide solution have been added, including that used for original neutralisation, for each grm. of dry solid in the sample. Heat the platinum basin gradually to drive off the ether without loss by spirting, evaporate to dryness, and ignite over a low Argand burner. Cover dish with platinum cover and continue heating until all organic matter is thoroughly charred. Cool, and wash the charred residue with 10 c.c. of water, filtering the solution into a 100 c.c. flask. Wash the dish and paper three times with 5 c.c. of water. Return paper to dish, dry and ignite carefully over Argand burner until all carbon is burned.

Dissolve the ash in dilute hydrochloric acid, pour the solution through the filter funnel into the 100 c.c. flask, and wash the funnel. Add hydrochloric acid carefully until no precipitate remains. Place the flask on a steam bath and shake until there is no more effervescence of carbonic acid. Cool. Add 5 c.c. of *N*

\* The Society is indebted to Sir Robert Robertson for kindly giving these details of the process used in the Government Laboratory.—EDITOR.

calcium chloride solution, five drops of phenolphthalein solution, and *N* sodium hydroxide solution until a slight permanent precipitate is formed. Then add 0.1 *N* sodium hydroxide solution, drop by drop, with constant shaking, until there is a distinct, but faint, permanent pink colour. Make up to 100 c.c. Shake thoroughly, filter off 75 c.c. and catch any filtrate in excess of the 75 c.c. in the 100 c.c. flask (*vide infra*).

Transfer the filtered solution to a 400 c.c. hard glass beaker. Add 1 drop of methyl orange indicator and 0.1 *N* sulphuric acid until the liquid is just acid to methyl orange. Cover with a clock glass and boil for 10 minutes to expel carbonic acid; cool quickly; wash cover into beaker with water; add 10 drops of phenolphthalein, and bring the liquid to neutral point to methyl orange. (One drop of 0.1 *N* acid or alkali is usually sufficient.) Add 0.5 gm. of mannitol, and titrate with 0.1 *N* sodium hydroxide solution to a pink colour. Add another portion of mannitol and, if the pink colour is not discharged, note the number of c.c. added. If the colour is discharged, add more sodium hydroxide until further addition of mannitol fails to discharge the colour. A blank obtained by using the same quantities of sodium hydroxide solution and other reagents as in the test experiment is subtracted from the number of c.c. used. Calculate the number of c.c. required for the 100 c.c. of liquid, representing the boric acid in the quantity of sample weighed out. Each c.c. of 0.1 *N* sodium hydroxide solution is equivalent to 0.0062 gm. of boric acid ( $H_3BO_3$ ).

The residue on the filter and in the 100 c.c. flask is dissolved in hydrochloric acid and collected in the 100 c.c. flask. Phenolphthalein and 1 c.c. of *N* calcium chloride are added, and the process of precipitation of phosphate, filtration and titration is repeated as described above. The difference between this titration figure and one-fourth of the original titration figure is negligible if the phosphate has been carefully precipitated. If, however, the difference is appreciable, it should be added to the original titration.

## An Investigation into the Chemistry of the Reinsch Test for Arsenic and Antimony, and its Extension to Bismuth.

By B. S. EVANS, M.C., M.B.E., PH.D., F.I.C.

PART II. (*Concluded from p. 367.*)

MECHANISM OF THE REACTION.—The large influence which chlorides exercise on the velocity of the Reinsch test raises the question of the mechanism of the reaction. Three different solutions of the question suggested themselves, and an attempt was made to discriminate among them experimentally:—

(a) The reaction may be a specific one between copper and the halide of the element in question.

(b) The reaction may be electrolytic in character, similar, for example, to the deposition of copper on iron. In this case the action of the chlorides would be explained by their great influence in activating passive copper.

(c) The third hypothesis was suggested by the notable difference of behaviour of copper towards hydrochloric and, say, dilute sulphuric acid. In the former case the copper dissolves slowly (presumably with liberation of hydrogen), and quite large amounts may be converted into cuprous chloride, even in the absence of air or any oxidising agent; in the latter case there appears to be no action at all. In view of this fact, it appeared probable that the Reinsch reaction might be due to reduction of the arsenic, antimony, or bismuth, salt by the film of hydrogen on the surface of the copper; the hydrides formed in the first two instances, as shown by the author (*ANALYST*, 1920, 45, 8), would immediately combine with the copper to form arsenides or antimonides, respectively.

COMBINATION AS HALIDE.—A few experiments were carried out with a view to finding out whether arsenic and antimony chlorides will react directly with copper to form arsenides or antimonides.

1. As a preliminary rough test, a crystal of antimony chloride was dissolved in glacial acetic acid, a slip of copper was cleaned in 1.2 sp. gr. nitric acid, washed, dried, rinsed once or twice with glacial acetic acid, dropped into the antimony chloride solution, and the whole heated on the plate. The antimonides deposited fairly promptly.

Arsenious oxide heated with sodium chloride and a slip of dry copper under glacial acetic acid similarly gave a deposit of arsenide. Of course in these experiments there is a strong probability that there was a trace of water in the glacial acetic acid.

2. Three dry flasks were taken; two or three grms. of tartar emetic were added to one, a gm. or two of arsenious oxide to a second, and the third was left blank; about 50 c.c. of concentrated sulphuric acid were run into each, and then about 2 grms. of sodium chloride added; a slip of clean bright dry copper was suspended in each flask, out of contact with the liquid, and the flasks tightly corked. Almost instantaneously the slips in the antimony and arsenic flasks darkened, the blank copper remaining bright; after standing overnight the antimony and arsenic slips were each coated with a black velvety deposit, the blank slip being only slightly tarnished. The deposits were dissolved off the slips with sodium peroxide following the procedure given by the author ("Estimation of Antimony in Copper," *ANALYST*, 1922, 47), and the solutions tested, after purification with zinc sulphide, with hydrogen sulphide; the antimony and arsenic deposits gave precipitates of the orange and yellow sulphides respectively, the blank gave no precipitate.

3. A strong solution of antimony chloride in benzene was prepared and

allowed to stand over phosphorus pentoxide for a considerable time. A copper slip was cleaned with nitric acid (sp. gr. 1.2), washed with water, rinsed three times with alcohol and twice with ether, and finally allowed to stand in ether over phosphorus pentoxide for some time. It was then removed from the ether with forceps and dropped straight into the antimony chloride solution, which was then heated. There was an immediate blackening of the copper and fumes were given off which reddened litmus paper and caused a white precipitate to form in a drop of silver nitrate solution suspended on a watch glass. The deposit, on being dissolved and tested as before, gave an orange precipitate of antimony sulphide.

In view of the association of phosphorus with arsenic, antimony, and bismuth, in the periodic table, it was thought worth while to repeat the last experiment with phosphorus pentachloride in place of antimony chloride; a purplish-black deposit was formed in patches, and with some difficulty, and acid fumes were evolved. The copper was removed, thoroughly washed with ether, dilute hydrochloric acid, and water; on being tested with ammonium molybdate it gave a slight but distinct precipitate of phosphomolybdic acid.

These experiments seemed to give ground for the assumption that a reaction could take place directly between metallic copper and the chlorides of arsenic and antimony, but did not prove that the reaction was the basis of the Reinsch test. If the Reinsch reaction depends upon the formation of arsenic trichloride, the velocity of a test carried out in fairly strong hydrochloric acid should be increased by the addition of solid sodium chloride. Two solutions were prepared, each containing 0.001 gm. of arsenic in 20 c.c. of 25 per cent. hydrochloric acid (*i.e.* 25 c.c. of the strong acid made up to 100 c.c.); to one of the solutions were added 2 grms. of solid sodium chloride; both were heated to boiling on the plate and slips of cleaned copper were dropped into both simultaneously. The slip in the sodium chloride solution became coated much more rapidly than the other. It should be noted, however, that the sodium chloride would raise the boiling point of the solution, and that, therefore, the temperatures would not be the same; the experiment, therefore, was repeated, both the beakers being immersed in a third containing water, which was kept boiling; under these conditions the order of deposition was reversed. It would seem, therefore, that the addition of sodium chloride hindered the deposition of the arsenide, presumably by reducing the ionisation of the hydrochloric acid, and that, therefore, a direct interaction between arsenic trichloride and copper does not play the main part in the Reinsch reaction; whether it plays any part at all, under ordinary conditions, could not be ascertained.

**ELECTROLYTIC HYPOTHESIS.**—In order to test the electrolytic hypothesis, as a preliminary, a few measurements of electric potential of antimony deposits in various solutions were obtained. These were kindly carried out by Mr. Macnaughtan, of the Research Department, Woolwich.

The results were as follows:—

Temperature °C.	Solution	Electrode	Potential millivolts	Sign
(a) 20	Faintly alkaline solution of sodium chloride	White film obtained from tartar emetic in dilute $H_2SO_4$ + NaCl	185 m.v.	Negative to copper
(b) 20	Ditto	Purple film obtained from tartar emetic in trichloroacetic acid + NaCl	200 m.v.	Negative to copper
(c) 20	Ditto	Pure antimony	130 m.v.	Negative to copper
(d) 20	Dilute sulphuric acid	Same film as (a)	170 m.v.	Negative to copper
(e) 22	35 c.c. strong HCl made up to 100 c.c.	White film as for (a)	100 m.v.	Negative to copper
(f) 21	Ditto	Purple film as for (b)	115 m.v.	Negative to copper
(g) 97	Ditto	White film as for (a)	185 m.v.	Negative to copper
(h) 97	35 c.c. strong HCl made up to 100 c.c.	Purple film as for (b)	180 m.v.	Negative to copper
(j) 97	Ditto	Pure antimony	230 m.v.	Negative to copper
(k) 20	$CCl_3CO_2H$ + NaCl	White film as for (a)	25 m.v.	Negative to copper
(l) 20	Ditto	Purple film as for (b)	100 m.v.	Negative to copper
(m) 97	Ditto	White film as for (a)	165 m.v.	Negative to copper
(n) 97	Ditto	Purple film as for (b)	150 m.v.	Negative to copper

(In all cases the sign convention used is that zinc is electropositive to copper.)

These measurements, as far as they go, seem to lend support to the electrolytic hypothesis; it appeared doubtful, however, whether that hypothesis fully explained all the peculiarities of the Reinsch reaction. The following experiments were devised with a view to finding out whether the reaction could be made to take place under conditions which negated electrolytic action:—

(a) A solution of antimony in dilute sulphuric acid was placed in a clean platinum dish and a few fragments of zinc were added; the dish was allowed to stand until a dark deposit of antimony had formed on it. The zinc was then removed, the solution poured out of the dish and the latter thoroughly washed; the dish was then filled with dilute sulphuric acid and about 2 grms. of sodium chloride added, a clean slip of copper placed in it in contact with the platinum, and the solution in the dish was heated to boiling. The antimony left the dish and deposited on the copper as the purple antimonide, the dish being left perfectly clean except for the very slight greyish stain at one point, which stain was subsequently found to be very difficult of removal and appeared to be an antimonide of platinum. The same operation was carried through, by way of a blank, with silver in place of antimony; in this case the silver stayed on the dish, and the copper remained bright.

(b) An electric couple was made of a strip of copper foil and a strip of platinum foil joined by a wire, the foils were suspended in a beaker containing the solution to be tested, the wire connection being outside the liquid.



The method of testing was to place the cold solution in the beaker, set the foils in place, and then heat to boiling. Results were as follows:—

Solution	Result
0.5 per cent. tartar emetic, 5 per cent. NaCl, 25 per cent. 1 : 3 H <sub>2</sub> SO <sub>4</sub> , a little tartaric acid	Purple deposit of antimonide on copper, quite as prompt as if no platinum were present. Very faint grey deposit of Sb on platinum
0.1 per cent. arsenic (as arsenide), 5 per cent. NaCl, 25 per cent. 1 : 3 H <sub>2</sub> SO <sub>4</sub>	Prompt grey silvery deposit of arsenide on copper; nothing on platinum
0.1 per cent. bismuth (as sulphate), 5 per cent. NaCl, 25 per cent. 1 : 3 H <sub>2</sub> SO <sub>4</sub>	Grey deposit of bismuth on copper; faint deposit of bismuth on platinum
A little silver (as AgNO <sub>3</sub> ), 25 per cent. 1 : 3 H <sub>2</sub> SO <sub>4</sub>	Immediate deposit of silver on both copper and platinum in cold; when boiling deposit on Cu showed tendency to thin, and that on Pt to increase

(c) A piece of copper foil was wrapped tightly round a piece of platinum and boiled in a solution of tartar emetic in hydrochloric acid.

The antimony deposited first on the copper as purple antimonide; this then began to turn white in patches in the usual manner, and simultaneously with this antimony began to deposit on the platinum; ultimately both platinum and copper became of a uniform grey-white colour.

(d) Attempts were made to get deposits on bismuth and antimony crystals boiled in copper solutions; these, in the main, were unsuccessful, though once or twice slight films were obtained which, on testing, showed the presence of copper.

The results of this group of experiments appear rather more favourable to the hydrogen film hypothesis than to that of electrolytic action.

It may be mentioned, in passing, that sulphur in the form of sulphite appears to behave exactly in the same way as arsenic in the Reinsch test, *i.e.* no copper sulphide is formed merely by boiling a slip of copper in dilute sulphuric acid containing sulphur dioxide, but, if sodium chloride is added, the sulphide promptly deposits. No curves of this reaction were attempted.

The deposition of sulphide mentioned above takes place quite readily in the cold; a measurement of electric potential between a film so obtained and copper, in the solution in which the film had been deposited, gave: Copper sulphide 30 millivolts electronegative to copper.

To test the question of the activation of the copper by hydrochloric acid two solutions were prepared, each containing 0.10 gm. of arsenic and 10 c.c. of 1 : 3 sulphuric acid in 100 c.c., and were heated to boiling in two flasks. Two slabs were made from electrolytic copper which had been melted and annealed, and these were thoroughly cleaned by warming with nitric acid of sp. gr. 1.2; one of the slabs was then placed in distilled water, the other one in strong hydrochloric acid, and allowed to stand for some minutes, after which they were thoroughly rinsed with distilled water, taken up with forceps, and simultaneously dropped into the

boiling solutions of arsenic, one into each flask. There was no immediate deposition of arsenide on either slab, but after several minutes' boiling a faint darkening was noticeable on the one which had been treated with hydrochloric acid, the other retaining its original colour. At the end of 20 minutes' boiling the slab which had been treated with the acid was of a faint purplish colour, which showed no tendency to darken further, the untreated one remained unchanged; this experiment was confirmed. This result might be taken as lending some support to the activation theory, but, on the whole, it is more in accord with the idea of the formation of a film of hydrogen. If the copper is activated, to the extent seen in the curves, by the comparatively small amount of hydrochloric acid present, the copper which had been immersed in strong hydrochloric acid should have been in a highly active condition. This, however, was not the case, and the apparent cessation in the deposition of the arsenide after some minutes' boiling agrees much better with the presence of a film of hydrogen on the surface which caused a certain amount of deposition before it was used up. In passing, it may be noted that the copper which had been treated with hydrochloric acid was of a different shade of colour from the untreated, being distinctly pinker.

It may be mentioned that several instances of what looked like passivity of the copper were noticed in the course of the work done in the preparation of the curves; now and again a point would be obtained which lay right off the curve in the direction of a considerable increase of time of reaction; in one or two cases it became necessary even to reject a certain slab of copper in order to get concordant results at all; the reason of this effect was never cleared up, and, in any case, it was very exceptional.

On one occasion, in the course of an experiment on the influence of the physical condition of the copper, it was necessary to grip the piece of cleaned copper strongly between the fingers before dropping it into a solution containing sulphuric acid and arsenic, but no chlorides; the result was that a clear finger print developed on the copper in deposited arsenide, the areas of contact being the areas where the arsenide deposited. This observation is reminiscent of a statement of W. R. Dunstan's (*J. Chem. Soc.*, 1911, 1856) that handling passive iron transforms it into the active modification. It is conceivable that in this instance the traces of chlorides in the perspiration caused the action, but it is difficult to explain the magnitude of the effect. Attempts at repetition were mainly, but not invariably, unsuccessful, and in some instances the reverse effect took place.

EFFECT OF HYDROGEN IN THE COPPER SURFACE.—The following experiments were carried out to ascertain if the presence of hydrogen in the copper surface enhanced the velocity of the reaction.

(a) A piece of electrolytic copper was unchanged after 15 minutes', but blackened after 40 minutes' boiling in a solution containing 0.0625 gm. of arsenic and 10 c.c. of conc. sulphuric acid in 60 c.c.; it was then taken out, immersed in warm nitric acid (sp. gr. 1.2), washed, dried and ignited in a current of hydrogen for 15 minutes; at the end of this time it was cooled, removed from the tube, and

dropped into the same solution of arsenic; it darkened in streaks almost immediately, and was completely black at the end of 15 minutes.

(b) Some electrolytic copper was melted, poured, and annealed by slow cooling from 700°C.; it was then cut into slabs, and one of these was heated to redness (900°C.) in a current of hydrogen, allowed to cool in the hydrogen, and then removed.

Time curves were then determined for both the slab exposed to hydrogen and one of the untreated slabs, the two being treated in the usual manner, and exactly alike in the matter of cleaning, polishing, etc. The results were as follows:—

Solution used 0.08 As. 25 c.c. of 1 : 3 H<sub>2</sub>SO<sub>4</sub> in 100 c.c.

NaCl	Annealed slab Seconds	Hydrogen treated slab Seconds
1.5 grms.	80	65
1.0 "	130	110
0.6 "	200	200

(c) Copper was deposited on two platinum dishes (1) from cupric nitrate and (2) from cupric sulphate, and a solution of arsenic in dilute sulphuric acid was subsequently boiled in each dish, with the following results:—

- (1) Slight darkening after 15 minutes, pink colour still showing after 30 minutes.
- (2) Copper (which was very red, showing absorbed H) darkened almost at once in patches, which gradually spread all over.

HYDROGEN FILM HYPOTHESIS.—These experiments are all in agreement with the hydrogen film hypothesis in that they show in hydrogen-treated copper a certain activation which gives that copper a certain definite start over untreated copper, but though the reaction starts sooner, it does not proceed faster. Examination of the curves showing the influence of varying acid concentration indicates that beyond a certain concentration of acid no further effect is produced, the curve then becoming a horizontal straight line; this concentration of acid appears to be independent of the amount either of sodium chloride or of arsenic (or of antimony or bismuth), and therefore is presumably dependent on the third reagent present, *i.e.* the copper. The action of copper on hydrochloric acid is to form cuprous chloride, thereby necessarily liberating hydrogen; the fact that this action will proceed to the entire solution of the copper if the cuprous chloride is allowed to get oxidised: that the same thing happens, in presence of an arsenic compound, without oxidation of the cuprous chloride: and the fact that hydrogen arsenide generated in presence of copper reacts to form copper arsenide (*cf.* Author's paper on Marsh test, *ANALYST*, 1920, **45**, 8) all combine to make this view reasonable.

Looked at in this way, the Reinsch test and the Marsh test depend on the same reaction, *viz.*, the reduction of arsenic salts by nascent hydrogen to form hydrogen

arsenide; in the Marsh test the hydrogen arsenide is set free unless copper happens to be present; in which case it combines to form copper arsenide. In the Reinsch test the hydrogen is liberated at the surface of the copper, and the latter accordingly becomes coated with a layer of the arsenide. As, however, copper does not dissolve in most acids, and therefore no hydrogen is produced, and as an oxidising acid such as nitric acid precludes the formation of hydrogen, the presence of a halogen acid and the absence of oxidising agents is necessary to the reaction or, at any rate, to its rapid progress.

It was not found possible to boil a strong arsenic solution with copper, even in the absence of chlorides, for any length of time without a deposit forming. The following curve was obtained with a solution containing 0.0125 gm. of arsenic and 25 c.c. of 1:3 sulphuric acid in 60 c.c. The standard used was not the ordinary alloy, but a slip of copper coated with a slight purplish film of arsenide.

Sodium chloride, grms.	4.0	2.0	1.0	0.7	0.6	0.5	0.25	0.20	0.15	0.10	0.05	0.01	blank
Time, seconds ..	30	30	60	60	75	85	130	180	260	450	960	1800*	1800*

\* Approximate only; end point very difficult to distinguish.

The results shown in this curve were obtained with the use of copper foil.

0.0625 gm. of arsenic in the same volume completely blackened a slab of electrolytic copper in 40 minutes.

0.38 gm. of arsenic in the same volume completely blackened a slab of electrolytic copper in 15 minutes.

By the use of copper deposited electrolytically on platinum from cupric nitrate the following results were obtained:—

A 0.10 per cent. solution of arsenic in dilute sulphuric acid gave no deposit after an hour's boiling.

A 0.59 per cent. solution of arsenic in dilute sulphuric acid gave a slight darkening after 15 minutes; the pink colour of the copper still showed after 30 minutes.

With solutions of arsenic of this strength it is impossible to get concordant quantitative results as, apparently, the formation of a very slight trace of hydrogen brings down the arsenic. In any case results, in the absence of halide salts, are far less concordant than those obtained in their presence.

EFFECT OF DIFFERENT KINDS OF COPPER.—Variations in the behaviour of different samples of copper towards arsenic in the presence of sodium chloride have occasionally been observed. The initial work for this research was done on copper foil; on the exhaustion of one stock of foil it was found that, with the new stock, the entire set of curves had been displaced, the reaction time having become shorter.

The following are comparison curves carried out on the new foil and on electrolytic copper, respectively, with two different amounts of arsenic. The

volume of solution in each case was 60 c.c., containing 25 c.c. 1 : 3 of sulphuric acid, and the standard used was a purple film of arsenide deposited on copper.

Sodium chloride Grms.	Arsenic, 0.02 gm.		Arsenic, 0.04 gm.	
	Copper foil Seconds	Electrolytic copper Seconds	Copper foil Seconds	Electrolytic copper Seconds
0.10	—	—	—	840
0.15	210	—	180	—
0.20	—	600	—	270
0.30	—	—	80	—
0.40	100	225	—	—
0.50	—	—	50	—
0.60	65	160	—	120
0.75	—	—	35	—
0.80	45	120	—	85
1.00	40	—	32	60
1.50	—	—	30	—
2.00	30	35	—	20
5.00	—	20	—	10

With antimony the results were entirely different. Using the same two samples of copper alternately, the points for the two samples are found to lie along one curve.

The volume of the solutions used was 100 c.c. containing 25 c.c. 1 : 3  $H_2SO_4$ .

Sodium chloride Grms.	Antimony, 0.01 gm.		Antimony, 0.10 gm.	
	Electrolytic copper Seconds	Copper foil Seconds	Electrolytic copper Seconds	Copper foil Seconds
0.20	320	—	220	—
0.25	—	—	—	160
0.30	—	170	—	—
0.33	—	—	—	90
0.40	100	—	62	—
0.50	—	80	—	45
0.60	70	—	38	—
0.70	—	65	—	35
0.80	60	—	30	—
0.90	—	55	—	—
1.00	50	45	26	—
1.17	—	—	—	22
1.50	—	42	20	—
2.50	42	—	—	—

An analysis of this sample of copper foil gave the following results:—Tin, slight trace; lead, nil; iron, 0.017; nickel, zinc and arsenic, nil; and antimony, 0.009 per cent.

The difficulty had to be left unsolved. The analysis did not show anything which was likely to accelerate the reaction for arsenic, but not for antimony, and the amounts of impurity were very slight.

Experiments to ascertain whether passivity of the old batch of copper foil had anything to do with the higher readings gave negative results.

Solution used: 0.2 gram. sodium chloride, 0.02 gram. arsenic, 25 c.c. of 1 : 3 sulphuric acid, made up to 100 c.c.

	Seconds		Seconds
New copper treated in ordinary way	40	New copper immersed in $\text{CrO}_3$ solution after cleaning with $\text{HNO}_3$	35
New copper immersed in $\text{CrO}_3$ solution before cleaning with $\text{HNO}_3$	40	New copper boiled with sodium hypophosphite after cleaning with $\text{HNO}_3$	35
		Old copper	65

The new copper results are constant within the limits of experimental error, and are totally different from the old copper figure. That the effect is due to absorbed hydrogen is rendered unlikely by: (a) The fact seen above that immersion in  $\text{CrO}_3$  does not retard the reaction; and (b) the fact that the point of maximum effect of acid, in the curves showing the influence of varying acid, occurs at a lower concentration of acid for antimony than for arsenic; hence, if the effect is due to absorbed hydrogen, one would expect the antimony curve to be more affected than the arsenic curve.

A few experiments were carried out to see if the physical state of the copper influenced the rate of deposition.

No difference from the normal rate was observed in the following cases: (a) Copper melted and annealed by slow cooling from  $700^\circ\text{C}$ .; (b) copper under stress caused by bending by holding in a vice and striking with a hammer.

In getting the curves for this research, polishing of the copper was adopted. This was on account of the greater evenness of the deposits and steadiness of the results obtained, not because of any change in the rate of deposition. This point was tested by the following experiment.

An annealed electrolytic copper block was highly polished on one side; one half of the polished surface was then roughened with coarse emery paper, and the block was heated up in a solution containing 2.0 grms. of sodium chloride, 0.1 gram. of arsenic, and 20 c.c. of 1 : 3 sulphuric acid per 100 c.c. Arsenide deposited on both the polished and roughened surfaces simultaneously.

EXPERIMENTS ON COMPLETION OF THE REACTION.—Some estimations were made with a view to finding out whether the Reinsch reaction could be pushed to completion. In the first estimations some arsenic or antimony was always found in the solution at the end; it was thought that the oxidation of the ever-increasing amount of cuprous chloride might account for this; and, in consequence, arrangements were made for deposition to take place in an atmosphere of carbon dioxide. The solution and the copper were contained in a large flask, the neck of which was closed by a cork with two holes, one carrying a tube connected with a Kipp's apparatus generating carbon dioxide, and the other a straight vertical tube about three feet long, closed at the top with a Bunsen valve, to act as a condenser; the inlet tube was carried below the surface of the liquid, and was drawn out to a jet.

A slow stream of carbon dioxide was passed all the time the deposition was taking place. The results were as follows:—

*Arsenic.*—Solution: 0.025 gm. of arsenic, 10 grms. of sodium chloride, 50 c.c. of 1 : 3 sulphuric acid, in 200 c.c. Time of boiling, 5 hours.

The copper was stripped by treatment with sodium peroxide, followed by hydrogen peroxide and dilute sulphuric acid. Copper was removed from the solution as thiocyanate, and the arsenic was ultimately estimated by titration with iodine.

Arsenic added	0.0250 gm.	=	6.70 c.c.
„ recovered	0.0242	„	= 6.50 c.c.
„ left in solution	Nil		

This result is probably a little low, as no arsenic could be detected in the solution and no indication of hydrogen arsenide could be detected in the steam issuing from the flask. The error was possibly due to undissolved arsenide left adhering to the copper.

*Antimony.*—Solution: 0.025 gm. of antimony (as tartar emetic), 70 c.c. of conc. hydrochloric acid, made up to 200 c.c. Time of boiling: 5 hours first copper, 2 hours each second and third.

In the case of antimony, in relatively large amounts, the white secondary film is so dense that, unless it can be induced to strip itself automatically, as is sometimes the case, it practically stops the reaction; this is the reason for the treatment with a second and third roll of copper; a fourth roll remained bright. A little sodium hypophosphite was added with each roll after the first. The antimony was stripped with sodium peroxide, the dissolved copper separated as sulphide in alkaline tartrate solution, the antimony separated as sulphide in slightly acid solution, and ultimately after dissolving estimated by Rowell's potassium bromate method.

Antimony added	.. .. .	0.0250 gm.
„ recovered (1st copper)	.. .. .	0.0225 „
„ „ (2nd and 3rd copper together)	.. .. .	0.0025 „
		<hr/>
		0.0250 „
		<hr/>
Antimony left in solution	.. .. .	Nil

*Bismuth.*—Solution: 0.025 gm. bismuth, 10 grms. of sodium chloride, 50 c.c. of 1 : 3 sulphuric acid in 200 c.c. Time of boiling: 5 hours.

The film was stripped off with sodium peroxide, followed by hydrogen peroxide and dilute sulphuric acid. Bismuth was separated as basic carbonate, and weighed as  $\text{Bi}_2\text{O}_3$ .

Bismuth added	.. .. .	0.0250 gm.
„ recovered	.. .. .	0.0252 „ (0.0282 $\text{Bi}_2\text{O}_3$ )
„ left in solution	.. .. .	Nil

It is evident that, in all three cases, in an inert atmosphere the reaction is complete; its value, from an analytical point of view, is limited, at present, in the

cases of arsenic and antimony, by the difficulty of redissolving the film without dissolving at the same time excessive amounts of copper.

A number of estimations were made of copper left in solution as cuprous chloride after the deposition was complete. These are not worth recording, as they were very variable and always in excess of that theoretically required (less so in an inert atmosphere). This result is to be expected in view of the distinct solubility of copper in hydrochloric acid.

APPLICATION TO SMALL AMOUNTS OF BISMUTH.—A few estimations were made to ascertain whether the reaction was applicable to minute amounts of bismuth.

Solution used: 30 c.c. of acetic acid, 5 grms. of sodium chloride, varying amounts of bismuth. Time of boiling: 2 hours.

Bismuth added Grm.	Bismuth recovered Grm.	Bismuth added Grm.	Bismuth recovered Grm.
0.00100	0.00100	0.00400	0.00415
0.00200	0.00200	0.00500	0.00500
0.00300	0.00290		

It was found that, with these small amounts of bismuth, better results were obtained with acetic than with a stronger acid. The films (which were often invisible) were stripped with sodium peroxide, and the bismuth estimated in the solution, after acidifying and boiling, by the ordinary colorimetric iodide process.

SENSITIVENESS OF THE REACTION.—As a qualitative test the sensitiveness of the Reinsch reaction has been somewhat underrated. By using the copper in the form of a thin wire fixed to a cork in the neck of the flask and dipping about  $\frac{1}{4}$  inch into the liquid the following results were obtained with pure solutions:—

Element	Amount Mgram.	Solution	Volume	Time of boiling	Result
Arsenic	0.01	20 per cent. 1 : 3 $H_2SO_4$ , 2 per cent. NaCl	100 c.c.	1 hour	Very distinct purple-grey film
Antimony	0.01	20 per cent. 1 : 3 $H_2SO_4$ , 2 per cent. NaCl	100 c.c.	1 hour	Distinct purple film
Bismuth	0.01	20 per cent. acetic acid, 2 per cent. NaCl	100 c.c.	1 hour	Rather indistinct red-grey film. In this case the colour of the film is not sufficiently distinct from that of copper

Time did not permit of this aspect of the subject being pursued further.

CONCLUSIONS.—In conclusion, it seems probable that this reaction might be made a basis for a variety of analytical processes in certain cases where existing methods have the drawbacks of uncertainty, difficulty, or tediousness, as, for example, the estimation, or the removal from solution, of arsenic, antimony, or bismuth.

The principal difficulties remaining are, on the one hand, the solution of the



film without at the same time dissolving large amounts of copper, and, on the other, the inevitable passage of considerable quantities of copper into solution.

The principal precaution to be observed is thorough removal of any substance likely to act as an oxidising agent.

It is suggested that, in cases of doubt, two or three determinations of the reaction velocity, carried out as described in this paper, will quickly decide the question as to whether a given solution will, or will not, interfere with the Reinsch test, thus affording a criterion which is lacking in the Marsh and Gutzeit tests.

## Notes on the Examination of Preserved Meats, etc.

By OSMAN JONES, F.I.C.

*(Read at the Meeting, February 7th, 1923.)*

IN a laboratory attached to a factory producing much of its own raw material and handling fairly large quantities of articles preserved in tins and glasses, one must perforce conduct a good many routine examinations. In the case of meats, etc., packed in tins and glasses, it is necessary to take into consideration the points discussed under the following heads:—

(1) THE CAN THAT IS TO CONTAIN THE ARTICLE OF FOOD.—The first essential is that it must be made of tin plate of uniform coating, for if the iron upon which the tin is plated comes in contact with the food, discoloration is almost certain to occur. A few sheets of tin plate are therefore selected at random from each consignment and tested as follows:—

The sheet of tin plate is flooded with an almost cold 3 per cent. solution of potassium ferricyanide containing approximately about 10 per cent. of gelatin. If blue spots of ferrous ferricyanide develop in the gelatin the consignment of tin plate is rejected as unsuitable. It should be noted that the size of the spots gives no true indication of the dimensions of the surface of the iron which has been improperly coated with tin.

Within reasonable limits the actual thickness of the coating of tin is not of undue importance; a suitable specification for food preserving containers is 0.75 gm. per sq. dcm.

The finished, but still empty, can is next examined for zinc and lead. Some manufacturers still use the soldered can in place of the more modern "sanitary can," but even with the latter there is some danger of the soldering flux contaminating the inside of the tin; moreover, zinc chloride is still frequently used as a flux; and my experience has been, that the presence of even a very small quantity of zinc chloride has a marked effect upon the amount of tin taken up by the food contents of a can.

Meat packed in an unsoldered or "sanitary can" and in a soldered can, respectively, on the same day and treated in precisely the same manner, yielded the following amounts of tin (calculated as tin dioxide):—

					In sanitary can Per Cent.	In soldered can Per Cent.
At end of	7 days	...	...	...	0·001	0·006
" "	" 3 months	...	...	...	0·0015	0·0093
" "	" 12 months	...	...	...	0·0015	0·0103

A similar result is brought about by introducing a trace of zinc chloride into an unsoldered can before it is closed; whereas the introduction of a small piece of solder does not increase the quantity of tin taken up by the food. It would appear, therefore, that it is the presence of zinc chloride that accelerates the reaction.

SEALING MATERIAL.—When a sanitary can is used the joints between the body and the lid and the body and the bottom are rendered air-tight by double seaming, and the seam is treated, before closing, with a solution of rubber in a light solvent (b. pt., 70 to 90° C.) such as benzene. I mention this point because it has happened on occasions that the solvent used has not been sufficiently volatile to evaporate completely during the course of the manufacture of the tin, and, as a result, the food contents have become contaminated with the solvent, a trace of which is quite sufficient to impart a most disagreeable flavour. The unpleasant effects sometimes noticed, flatulence, etc., after eating canned food, as well as the so-called "tinny" flavour, can often be traced to the same source.

The following simple tests may be applied to determine whether the sealing fluid material complies with the above specification:—

The bottom seam of the can is carefully cut open from the outside, and a portion of the sealing material removed and placed in a small test tube, which is then heated in a glycerin or oil bath to a temperature of about 120° C.; should any solvent be present in the seam, a distinct petrol-like odour is noticed. A further portion of the seaming material should be placed upon the tongue, and should be entirely tasteless.

(2) THE ARTICLE OF FOOD.—I am assuming that the materials used by the manufacturer are in a fresh and clean condition at the time of filling the container. It is a matter of common knowledge that a tin properly filled should have its ends dished inwards, *i.e.* concave; a raised end sometimes indicates decomposition of the contents, with the formation of gas; it may also point to faulty manufacture, the air space above the contents not having been sufficiently exhausted before the tin was closed. It may be noted, in passing, that the absence of a blown appearance does not necessarily indicate sterility of the contents, or that no decomposition has occurred, because certain bacteria do not produce sufficient gas to cause the tin to bulge, or the tin may possess a minute perforation which will allow the escape of any gas generated.

If the tin is blown it may be desirable to examine the gaseous contents, in

which case the method of Doremus (*J. Amer. Chem. Soc.*, 1897, 733) may be adopted.

After the removal of the gas, a bacteriological examination of the contents is made. In the case of a blown tin the easiest method is to remove the top carefully, the usual precautions with regard to the sterility of the instruments being taken, and to transfer a loop full of the liquid, if present, to a tube containing sterile nutrient broth or other suitable medium. A portion of the solid matter, if present, is transferred to a larger tube containing a sterile medium. The tubes are then incubated for 48 hours at 37° C., after which plate cultivations of the contents of the first tube are made in the usual manner, whilst the sub-culture is made from the contents of the second tube, and this sub-culture is incubated and used for a plate cultivation.

In the case of a tin which is not blown, it is desirable to incubate it before making an examination as to the sterility of its contents. Various authorities differ in their opinion as to the most suitable length of time of incubation; of course the longer the better, but my own experience is that at 37° C., about 8 days is usually sufficient to allow any spores which may be present to germinate, and in a works laboratory one has to bear in mind the commercial aspect. No hard and fast rule can be laid down, however; for example, it has recently been demonstrated that the spores of *B. botulinus* can remain dormant for 400 days.

After incubation the tin or glass is treated in the usual manner, the top is sterilised either by burning upon it some methylated spirit, or by heating it with a Bunsen flame. In either case the sterile top is pierced with a sterile awl, without removing the flame, which is still allowed to play upon the top of the tin during the time that the awl is withdrawn from the perforation it has made, thus preventing a rush of unsterile air into the tin. A sterile platinum needle is then inserted into the contents of the can, and a loopful of the material removed and placed in a tube of sterile medium. The tube is now incubated for 48 hours at 37° C., and its appearance noted, the familiar turbidity indicating non-sterility. If such be the case, it is sometimes desirable to make a bacteriological examination, with a view to tracing the cause of the trouble. Should it be necessary to examine the condition of any solid matter which the tin may contain, the method previously mentioned under the heading of "solid matter in blown tins" can be adopted.

In some cases an examination for the presence of anaerobes may be necessary. I have found that the method of Novy, in which a dilute solution of alkaline pyrogallol is used, is satisfactory.

METALLIC CONTAMINATION.—In addition to lead and zinc, already mentioned, it is frequently necessary to make an examination of the contents of a tin or glass for tin. I have always found some tin to be present in every sample I have examined; occasionally only a trace; sometimes (in the case of fruits and vegetables) as much as 0.027 per cent. The tin is usually present as an insoluble salt, and the amount found is higher in the drained solids than in the liquor, but I have experienced cases where practically all the tin was present in solution. Discoloration

of the inside of the tin does not always indicate that a large quantity of tin has been absorbed by the contents.

The reaction between the food material and the tin lining appears to take place most rapidly during the time of processing or cooking, that is, when the highest temperatures are reached, and in the case of meat products it seems almost to cease after about four months. The following figures (obtained with a 1 lb. can of brawn [sanitary can]) will make this clear:

Time						Tin (as SnO <sub>2</sub> ) per cent.
After 48 hours	...	...	...	...	...	0·001
„ 7 days	...	...	...	...	...	0·001
„ 28	...	...	...	...	...	0·0012
„ 56	...	...	...	...	...	0·0017
„ 84	...	...	...	...	...	0·0017
„ 168	...	...	...	...	...	0·0019
„ 335	...	...	...	...	...	0·0020
„ 2 years	...	...	...	...	...	0·0021

The above remarks do not necessarily apply to fruits or vegetables in cans, as I have not made a similar set of tests with them.

Absorption of tin takes place more rapidly when the air space in the container has not been as completely exhausted as possible.

It has been stated that zinc salts have been used by some manufacturers to improve the colour of peas and beans, but the process is supposed to be a secret, and I have never examined a sample in which it appeared probable that zinc salts had been used for this purpose.

*Copper*.—The presence of salts of copper is usually attributed to their addition as colouring agents for vegetables, but I have known its presence to arise through the use of brass or copper vessels in the course of manufacture.

ARTIFICIAL COLOURING MATTERS.—New dyes are continually being introduced which are stated to be suitable for food colouring, and their identification is a matter of great difficulty, owing to the very small quantities employed. The number of colouring matters that can be used in the preserving of meats, etc., in tins and glasses is restricted to those which will remain fast at a temperature of 121° C., the temperature at which most manufacturers process these articles. The colours most commonly employed, especially in potted meats, are the Ponceau dyes and naphthol yellows. Artificial colours may be detected by the methods usually adopted in the case of sausages. I have found the method of Arata the best.

PRESERVATIVES.—The presence of preservatives in meats, etc., preserved in cans and glasses is uncommon, and their use should not be necessary. In some cases their presence may be attributed to the use of preserved material in the manufacture of the article, *e.g.* (1) certain brands of imported hams and bacon which have been cured with a pickle containing borax, and (2) gelatin, which frequently gives a reaction for sulphurous acid.

Manufacturers sometimes use a mixture of agar-agar and gelatin in the preparation of such meat products as brawn, tongues, etc. Gelatin readily hydrolyses above the boiling point of water, and thus loses, to a large extent, its setting properties. This difficulty can, to some extent, be overcome by the use of a mixture of agar and gelatin. Agar alone is unsuitable, as the jelly it produces is too brittle.

A useful test for agar in the presence of gelatin depends upon its behaviour with a dilute potassium iodide solution of iodine. This reagent, when added to a solution of agar, gives a deep crimson coloration, whilst with gelatin it produces an orange-coloured precipitate. By using a 1 per cent. solution of gelatin as a standard for colour comparison, it is possible to detect and estimate approximately the presence of 10 per cent. of agar in gelatin. This test cannot, of course, be applied in the presence of starch. (*Cf.* Parkes, *ANALYST*, 1921, **46**, 231.)

The above notes also apply to the examination of meats, etc., in glass vessels, except in so far as metallic contamination is concerned. This need not be looked for unless the metal lid of the container is corroded. Such corrosion occurs when the lid has been improperly lacquered, an action being set up between the metallic lid, the vulcanised rubber sealing band, and the contents of the glass. Such corrosion rapidly causes discoloration of the contents of the glass, the action being considerably accelerated by exposure to light.

Fruits containing a considerable quantity of tartaric acid and certain sugars will, even when perfectly sterile, set up, when exposed to direct sunlight, an action which is apparently similar to fermentation by yeast.

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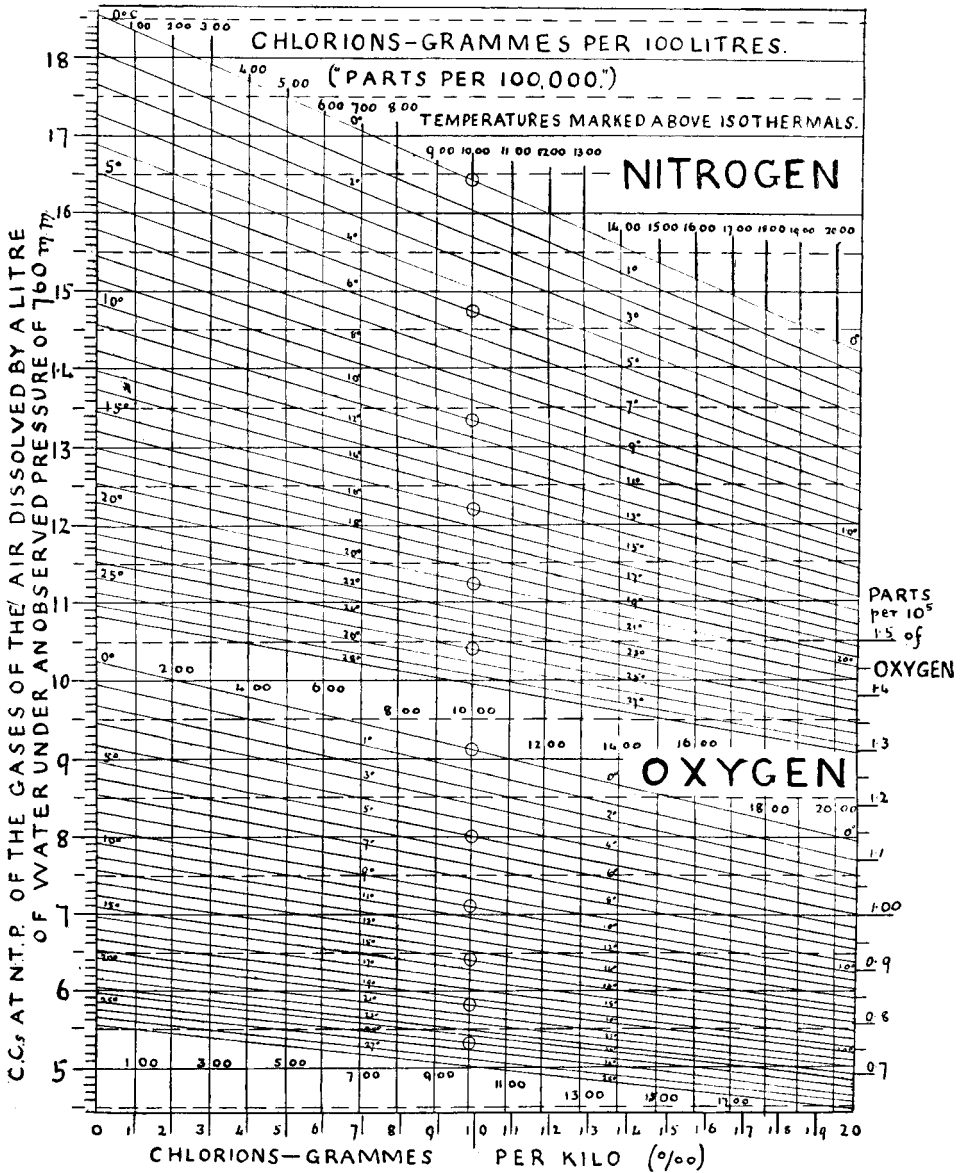
## The Absorption of Atmospheric Gases by Water.

By J. H. COSTE, F.I.C.

IN the examination of waters, *e.g.* of tidal rivers, which contain a relatively large proportion of dissolved salts and yet can hardly be considered definitely as sea water, some doubt may be felt as to what value should be taken to represent full saturation with dissolved gases, more particularly oxygen. For ordinary river or lake waters, the values given by various workers for distilled water may be used without correction. The values to be used for sea water have been less definite, as "sea water" is, after all, only a relative term, but since Fox's very exact work on various dilutions of sea water uncertainty has been removed. Nevertheless, the need for a means of quickly ascertaining the saturation value of mixtures of fresh and sea water of any salinity, or at any temperature, must have been felt by many workers.

My colleagues and I have used for some years a graph for dissolved oxygen, calculated from Fox's values, consisting of a series of isothermal curves showing as ordinates the volume of oxygen at N.T.P. dissolved in one litre of water of any

salinity from 0 to 20 grms. of chlorions per kilo. of water ( $\text{‰}$  of oceanographers), or alternately per 100 litres ("parts per 100,000" of water analysts).



The accompanying graph is of a similar kind, but it includes atmospheric "nitrogen" ( $\text{N}_2 + \text{A}$ ) as well as oxygen, a value which is sometimes required, and without which the results of estimations of dissolved oxygen may be difficult to interpret. The upper series of isothermals refers to "nitrogen," and the lower

to oxygen; for use with the latter a scale showing "parts of dissolved oxygen per 100,000 parts of water," *i.e.* grms. per 100 litres, has been given on the right hand.

Examples of the method of using the graphs may help to make the matter perfectly clear. Water containing 1750 parts of chlorine per 100,000, temperature 8.3° C.  $N_2 + A = 12.57$  c.c. p.l.  $O_2 = 6.74$  c.c. p.l. (=0.96 per 100,000). Water containing 8.30 ‰ Cl at 17° C.  $N_2 + A = 12.00$  c.c. pl.  $O_2 = 6.27$  c.c. pl. (=0.90 parts per 100,000 nearly).

It should be noted that this graph has been calculated for a total pressure of 760 m.m., that is, actual barometric reading (pressure of dry air + tension of aqueous vapour). The correction to any other observed pressure is approximately linear.

The ratio between oxygen and nitrogen is not constant. It is affected much more by variations of temperature than of salinity, and in a more regular manner. The following table shows the saturation ratios calculated from Fox's results, and also the ratios calculated from Winkler's formula—percentage of oxygen in mixed gases =  $35.47 - 0.0338t$  where  $t = °C$ .

Volume ratio  $\frac{N_2 + A}{O_2}$  in water of varying salinity at temperatures from 0° to 28° C. (Fox):

Cl ‰	0	4	8	12	16	20	24	28
0	1.82	1.84	1.86	1.88	1.90	1.92	1.93	1.96
4	1.81	1.84	1.86	1.89	1.90	1.92	1.94	1.97
8	1.81	1.83	1.87	1.89	1.90	1.93	1.94	1.98
12	1.80	1.84	1.86	1.89	1.91	1.93	1.96	2.00
16	1.80	1.83	1.86	1.89	(1.97)	1.94	1.97	2.02
20	1.80	1.83	1.86	1.89	1.92	1.95	2.00	2.04

Winkler's ratio:

1.82	1.83	1.84	1.85	1.86	1.87	1.88	1.89
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It will be seen that Winkler's formula does not give as high a ratio as Fox's results would indicate as appropriate to the higher temperatures. Fox's value for nitrogen as printed in the table in *Trans. Farad. Soc.*, at 16 per thousand of chlorine, and 16° C. is certainly wrong.

## Tables for Sugar Analysis.\*

By G. D. ELSDON, B.Sc., F.I.C.

ONE of the standard methods of sugar analysis used in this country is that due to Brown, Morris and Miller (*J.C.S.*, 1897, 71, 281). The tables which they give are, however, not very complete, and some time ago the writer, to save repeated calculations, interpolated from these tables the quantities of various sugars corresponding to weights of copper oxide for every milligram from 100 to 460;

\* Reprints of these tables, printed on one side of the paper only to facilitate mounting if required, may be obtained at 1/- each (post free) from the writer, at 143, Regent Road, Salford.

they are now published in the hope that they may be of some service to others using this process. The figures for dextrose, lævulose and invert sugar are based on the results obtained by Brown, Morris and Miller (*vide supra*), while those for maltose are taken from another paper by the same authors (*J.C.S.*, 1897, 71, 100). The figures for starch have been obtained by multiplying those for dextrose by 0.900; those for cane sugar by multiplying the figures for invert sugar by 0.950. The table for hydrated lactose is due to the Government Laboratory, and was published in *Food Reports*, No. 24, p. 179, issued by the Ministry of Health in 1918; that for anhydrous lactose was calculated from this. The actual process is conducted in the following manner:—Place 50 c.c. of the mixed Fehling solution ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , 59.2 grms. per litre; Rochelle salt, 346 grms.; NaOH, 130 grms. per litre) into a 400 c.c. squat form beaker, add such a quantity of water that, with the sugar solution to be added subsequently, the total volume will be 100 c.c., cover the beaker with a clock glass and heat for a few minutes in a boiling water bath. Then add the accurately measured quantity of the sugar solution, and continue the heating for exactly twelve minutes. The precipitate of cuprous oxide is then filtered off and weighed as  $\text{Cu}_2\text{O}$ , CuO or Cu. The most convenient plan is to filter through a Gooch crucible and ignite to CuO.

QUANTITIES OF COPPER AND COPPER OXIDE PRODUCED UNDER STANDARD CONDITIONS BY VARIOUS CARBOHYDRATES.

QUANTITIES EXPRESSED IN MILLIGRAMS IN ALL CASES.

Cupric Oxide.	Cuprous Oxide.	Copper.	Dextrose.	Starch.	Lævulose.	Hydrated Lactose. $\text{C}_{12}\text{H}_{22}\text{O}_{11} \cdot \text{H}_2\text{O}$ .	Anhydrous Lactose.	Maltose.	Invert Sugar.	Cane Sugar.
100	89.9	79.9	—	—	—	59.2	56.2	72.5	—	—
101	90.8	80.7	—	—	—	59.8	56.8	73.2	—	—
102	91.7	81.5	—	—	—	60.4	57.3	74.0	—	—
103	92.6	82.3	—	—	—	61.0	57.9	74.7	—	—
104	93.5	83.1	—	—	—	61.6	58.5	75.4	—	—
105	94.4	83.9	—	—	—	62.2	59.1	76.2	—	—
106	95.3	84.7	—	—	—	62.8	59.6	76.9	—	—
107	96.2	85.5	—	—	—	63.4	60.2	77.6	—	—
108	97.1	86.3	—	—	—	64.0	60.8	78.3	—	—
109	98.0	87.1	—	—	—	64.6	61.3	79.0	44.9	42.6
110	98.9	87.9	—	—	—	65.2	61.9	79.8	45.3	43.0
111	99.8	88.7	—	—	—	65.8	62.5	80.5	45.7	43.4
112	100.7	89.5	—	—	—	66.4	63.0	81.3	46.1	43.7
113	101.6	90.3	—	—	—	67.0	63.6	82.0	46.5	44.1
114	102.5	91.1	—	—	—	67.6	64.2	82.7	46.9	44.5
115	103.4	91.9	—	—	49.8	68.2	64.8	83.5	47.3	44.9
116	104.3	92.7	—	—	50.2	68.8	65.3	84.2	47.7	45.2
117	105.2	93.5	—	—	50.6	69.4	65.9	84.9	48.0	45.6
118	106.1	94.3	—	—	50.9	70.0	66.5	85.7	48.4	46.0
119	107.0	95.1	—	—	51.3	70.6	67.0	86.4	48.8	46.3
120	107.9	95.9	46.5	41.8	51.7	71.2	67.6	87.2	49.2	46.7
121	108.8	96.7	46.9	42.2	52.1	71.8	68.2	87.9	49.6	47.1
122	109.7	97.5	47.3	42.5	52.5	72.4	68.7	88.7	50.0	47.4



	Cupric Oxide.	Cuprous Oxide.	Copper.	Dextrose.	Starch.	Lævulose.	Hydrated Lactose, $C_{12}H_{22}O_{11} \cdot H_2O$ .	Anhydrous Lactose.	Maltose.	Invert Sugar.	Cane Sugar.
123	110.6	98.3		47.7	42.9	52.8	73.0	69.3	89.4	50.4	47.8
124	111.5	99.1		48.1	43.2	53.2	73.6	69.9	90.2	50.8	48.2
125	112.4	99.9		48.5	43.6	53.6	74.2	70.5	91.1	51.2	48.6
126	113.3	100.7		48.8	44.0	54.0	74.8	71.0	91.9	51.5	49.0
127	114.2	101.5		49.2	44.3	54.3	75.4	71.6	92.8	51.9	49.3
128	115.1	102.3		49.6	44.7	54.7	76.0	72.2	93.6	52.3	49.7
129	116.0	103.1		50.0	45.0	55.1	76.6	72.7	94.5	52.7	50.0
130	116.9	103.9		50.4	45.4	55.5	77.2	73.3	95.3	53.1	50.4
131	117.8	104.7		50.8	45.7	55.9	77.8	73.9	95.9	53.5	50.8
132	118.7	105.5		51.2	46.1	56.3	78.4	74.4	96.6	53.9	51.2
133	119.6	106.3		51.5	46.4	56.7	79.0	75.0	97.2	54.2	51.5
134	120.5	107.1		51.9	46.8	57.1	79.6	75.6	97.9	54.6	51.9
135	121.4	107.9		52.3	47.1	57.5	80.2	76.2	98.5	55.0	52.2
136	122.3	108.7		52.7	47.4	57.9	80.8	76.7	99.2	55.4	52.6
137	123.2	109.5		53.1	47.8	58.3	81.4	77.3	99.8	55.8	53.0
138	124.1	110.3		53.5	48.1	58.7	82.0	77.9	100.5	56.2	53.3
139	125.0	111.1		53.8	48.5	59.1	82.6	78.4	101.3	56.6	53.7
140	125.9	111.9		54.2	48.8	59.5	83.2	79.0	102.0	57.0	54.2
141	126.8	112.6		54.6	49.1	59.9	83.8	79.6	102.7	57.4	54.6
142	127.7	113.4		54.9	49.5	60.3	84.4	80.2	103.4	57.8	55.0
143	128.6	114.2		55.3	49.8	60.8	85.0	80.7	104.2	58.2	55.3
144	129.5	115.0		55.7	50.2	61.2	85.6	81.3	104.9	58.6	55.7
145	130.4	115.8		56.0	50.5	61.7	86.2	81.9	105.6	59.0	56.1
146	131.3	116.6		56.4	50.8	62.1	86.9	82.4	106.3	59.4	56.5
147	132.2	117.4		56.8	51.2	62.6	87.5	83.0	107.0	59.8	56.9
148	133.1	118.2		57.2	51.5	63.0	88.1	83.6	107.8	60.2	57.2
149	134.0	119.0		57.6	51.9	63.5	88.7	84.2	108.5	60.6	57.6
150	134.9	119.8		58.0	52.2	63.9	89.3	84.8	109.2	61.0	58.0
151	135.8	120.6		58.3	52.5	64.4	90.0	85.4	109.9	61.4	58.4
152	136.7	121.4		58.7	52.9	64.8	90.6	86.0	110.7	61.8	58.8
153	137.6	122.2		59.1	53.2	65.2	91.2	86.5	111.4	62.2	59.1
154	138.5	123.0		59.5	53.6	65.6	91.8	87.1	112.2	62.7	59.5
155	139.4	123.8		59.9	53.9	66.0	92.4	87.7	113.0	63.1	59.9
156	140.3	124.6		60.3	54.2	66.5	93.0	88.3	113.8	63.5	60.3
157	141.2	125.4		60.7	54.6	66.9	93.6	88.8	114.5	63.9	60.7
158	142.1	126.2		61.1	54.9	67.3	94.2	89.4	115.3	64.3	61.0
159	143.0	127.0		61.5	55.3	67.7	94.8	90.0	116.0	64.7	61.4
160	143.9	127.8		61.8	55.6	68.1	95.4	90.6	116.8	65.1	61.8
161	144.8	128.6		62.2	56.0	68.5	96.0	91.2	117.5	65.5	62.2
162	145.7	129.4		62.6	56.3	68.9	96.6	91.7	118.2	65.9	62.6
163	146.6	130.2		63.0	56.7	69.4	97.2	92.3	119.0	66.3	63.0
164	147.5	131.0		63.4	57.0	69.8	97.8	92.9	119.7	66.7	63.4
165	148.4	131.8		63.8	57.4	70.2	98.4	93.5	120.4	67.1	63.8
166	149.3	132.6		64.2	57.7	70.6	99.0	94.0	121.2	67.6	64.1
167	150.2	133.4		64.5	58.1	71.1	99.6	94.6	121.9	68.0	64.5
168	151.1	134.2		64.9	58.4	71.5	100.2	95.2	122.7	68.4	64.9
169	152.0	135.0		65.3	58.7	71.9	100.8	95.8	123.4	68.8	65.3
170	152.9	135.8		65.7	59.1	72.4	101.4	96.3	124.2	69.2	65.7
171	153.8	136.6		66.1	59.5	72.8	102.0	96.9	124.9	69.6	66.1
172	154.7	137.4		66.5	59.8	73.3	102.6	97.4	125.6	70.0	66.5
173	155.6	138.2		66.9	60.2	73.7	103.2	98.0	126.4	70.4	66.9
174	156.5	139.0		67.3	60.5	74.1	103.8	98.6	127.1	70.8	67.3
175	157.4	139.8		67.6	60.9	74.6	104.4	99.2	127.9	71.2	67.7

Cupric Oxide.	Cuprous Oxide.	Copper.	Dextrose.	Starch.	Lavulose.	Hydrated Lactose. $C_{12}H_{22}O_{11} \cdot H_2O$ .	Anhydrous Lactose.	Maltose.	Invert Sugar.	Cane Sugar.
176	158.3	140.6	68.0	61.2	75.0	105.0	99.7	128.6	71.7	68.1
177	159.2	141.4	68.4	61.6	75.4	105.6	100.3	129.4	72.1	68.5
178	160.1	142.2	68.8	61.9	75.8	106.2	100.9	130.1	72.5	68.9
179	161.0	143.0	69.2	62.3	76.3	106.8	101.5	130.8	72.9	69.3
180	161.9	143.8	69.6	62.6	76.7	107.4	102.0	131.5	73.4	69.7
181	162.8	144.6	70.0	63.0	77.1	108.0	102.6	132.2	73.8	70.1
182	163.7	145.4	70.4	63.3	77.5	108.6	103.2	133.0	74.2	70.5
183	164.6	146.2	70.8	63.7	77.9	109.2	103.8	133.7	74.6	70.9
184	165.5	147.0	71.2	64.0	78.3	109.8	104.3	134.4	75.0	71.3
185	166.4	147.8	71.6	64.4	78.8	110.4	104.9	135.1	75.4	71.6
186	167.3	148.6	72.0	64.8	79.2	111.0	105.5	135.8	75.8	72.0
187	168.2	149.4	72.4	65.1	79.5	111.6	106.1	136.6	76.2	72.4
188	169.1	150.2	72.8	65.5	80.0	112.2	106.7	137.3	76.6	72.8
189	170.0	151.0	73.2	65.8	80.4	112.8	107.3	138.1	77.0	73.2
190	170.9	151.8	73.6	66.2	80.9	113.5	107.9	138.8	77.5	73.6
191	171.8	152.6	74.0	66.6	81.3	114.1	108.5	139.6	77.9	74.0
192	172.7	153.4	74.4	66.9	81.8	114.7	109.1	140.3	78.3	74.4
193	173.6	154.2	74.8	67.3	82.2	115.4	109.6	141.1	78.7	74.7
194	174.5	155.0	75.2	67.6	82.7	116.0	110.2	141.8	79.1	75.1
195	175.4	155.8	75.6	68.0	83.1	116.6	110.8	142.6	79.5	75.5
196	176.3	156.6	76.0	68.4	83.6	117.2	111.3	143.3	79.9	75.9
197	177.2	157.4	76.4	68.7	84.0	117.8	111.9	144.1	80.3	76.2
198	178.1	158.2	76.8	69.1	84.5	118.4	112.5	144.8	80.7	76.6
199	179.0	159.0	77.2	69.4	84.9	119.1	113.1	145.5	81.1	77.0
200	179.9	159.8	77.6	69.8	85.3	119.7	113.7	146.3	81.5	77.4
201	180.8	160.6	78.0	70.2	85.7	120.3	114.3	147.0	81.9	77.8
202	181.7	161.4	78.4	70.5	86.1	121.0	114.9	147.7	82.4	78.2
203	182.6	162.2	78.8	70.8	86.6	121.6	115.5	148.4	82.8	78.6
204	183.5	163.0	79.2	71.2	87.0	122.2	116.1	149.2	83.2	79.0
205	184.4	163.8	79.6	71.6	87.4	122.8	116.7	149.9	83.6	79.4
206	185.3	164.6	80.0	71.9	87.8	123.4	117.3	150.6	84.0	79.8
207	186.2	165.4	80.4	72.3	88.2	124.1	117.9	151.4	84.4	80.2
208	187.1	166.2	80.8	72.7	88.7	124.7	118.5	152.1	84.8	80.6
209	188.0	167.0	81.2	73.0	89.1	125.4	119.1	152.9	85.2	81.0
210	188.9	167.8	81.6	73.4	89.5	126.0	119.7	153.6	85.7	81.4
211	189.8	168.6	82.0	73.8	89.9	126.6	120.3	154.4	86.1	81.8
212	190.7	169.4	82.4	74.1	90.4	127.2	120.9	155.1	86.5	82.2
213	191.6	170.2	82.7	74.5	90.8	127.8	121.5	155.8	87.0	82.6
214	192.5	171.0	83.1	74.8	91.3	128.5	122.1	156.6	87.4	83.0
215	193.4	171.8	83.5	75.2	91.7	129.1	122.6	157.3	87.8	83.5
216	194.3	172.6	83.9	75.6	92.2	129.7	123.2	158.1	88.3	83.9
217	195.2	173.4	84.3	75.9	92.6	130.3	123.8	158.8	88.7	84.3
218	196.1	174.2	84.7	76.3	93.1	130.9	124.4	159.6	89.2	84.7
219	197.0	175.0	85.1	76.6	93.5	131.6	125.0	160.3	89.6	85.1
220	197.9	175.8	85.5	77.0	94.0	132.2	125.6	161.0	90.0	85.5
221	198.8	176.6	86.0	77.4	94.4	132.8	126.2	161.8	90.4	85.9
222	199.7	177.4	86.4	77.8	94.9	133.4	126.8	162.5	90.9	86.3
223	200.6	178.2	86.9	78.2	95.4	134.0	127.4	163.2	91.3	86.8
224	201.5	179.0	87.3	78.6	95.8	134.7	127.9	163.9	91.8	87.2
225	202.4	179.8	87.8	79.0	96.3	135.3	128.5	164.7	92.2	87.6
226	203.3	180.6	88.2	79.4	96.8	135.9	129.1	165.4	92.6	88.0
227	204.2	181.4	88.7	79.8	97.2	136.5	129.7	166.2	93.1	88.5
228	205.1	182.2	89.1	80.2	97.7	137.2	130.3	166.9	93.5	88.9

Cupric Oxide.	Cuprous Oxide.	Copper.	Dextrose	Starch.	Laevulose.	Hydrated Lactose $C_{12}H_{22}O_{11} \cdot H_2O$ .	Anhydrous Lactose.	Maltose.	Invert Sugar.	Cane Sugar.
229	206.0	183.0	89.6	80.6	98.1	137.8	130.9	167.6	93.9	89.3
230	206.9	183.8	90.0	81.0	98.6	138.4	131.5	168.3	94.4	89.7
231	207.8	184.6	90.4	81.4	99.1	139.0	132.1	169.1	94.8	90.1
232	208.7	185.4	90.8	81.8	99.5	139.6	132.7	169.8	95.2	90.5
233	209.6	186.2	91.2	82.1	100.0	140.2	133.3	170.5	95.6	90.9
234	210.5	187.0	91.7	82.5	100.4	140.9	133.9	171.3	96.1	91.4
235	211.4	187.7	92.1	82.9	100.9	141.5	134.4	172.0	96.5	91.8
236	212.3	188.5	92.5	83.3	101.3	142.1	135.0	172.8	97.0	92.2
237	213.2	189.3	92.9	83.6	101.7	142.7	135.6	173.5	97.4	92.6
238	214.1	190.1	93.4	84.0	102.1	143.3	136.2	174.3	97.8	93.0
239	215.0	190.9	93.8	84.4	102.6	144.0	136.8	175.0	98.3	93.4
240	215.9	191.7	94.2	84.8	103.0	144.6	137.4	175.7	98.7	93.8
241	216.8	192.5	94.6	85.2	103.4	145.2	138.0	176.5	99.2	94.2
242	217.7	193.3	95.0	85.5	103.8	145.8	138.6	177.2	99.6	94.6
243	218.6	194.1	95.4	85.9	104.3	146.4	139.1	177.9	100.0	95.0
244	219.5	194.9	95.8	86.3	104.7	147.0	139.7	178.6	100.4	95.4
245	220.4	195.7	96.2	86.6	105.1	147.6	140.3	179.3	100.8	95.8
246	221.3	196.5	96.6	87.0	105.5	148.2	140.8	180.1	101.2	96.2
247	222.2	197.3	97.0	87.3	106.0	148.9	141.4	180.8	101.7	96.6
248	223.1	198.1	97.5	87.7	106.4	149.5	142.0	181.6	102.1	97.0
249	224.0	198.9	97.9	88.1	106.9	150.1	142.6	182.3	102.5	97.4
250	224.9	199.7	98.3	88.5	107.3	150.7	143.2	183.1	102.9	97.8
251	225.8	200.5	98.7	88.9	107.8	151.4	143.8	183.8	103.3	98.2
252	226.7	201.3	99.1	89.3	108.2	152.0	144.4	184.5	103.8	98.6
253	227.6	202.1	99.5	89.6	108.7	152.6	145.0	185.3	104.2	99.0
254	228.5	202.9	99.9	90.0	109.1	153.2	145.6	186.1	104.6	99.4
255	229.4	203.7	100.3	90.4	109.6	153.8	146.2	186.8	105.0	99.7
256	230.3	204.5	100.8	90.7	110.0	154.4	146.8	187.6	105.4	100.1
257	231.2	205.3	101.2	91.1	110.5	155.0	147.4	188.3	105.8	100.5
258	232.1	206.1	101.6	91.5	110.9	155.7	148.0	189.1	106.3	100.9
259	233.0	206.9	102.1	91.9	111.4	156.4	148.6	189.8	106.7	101.3
260	233.9	207.7	102.5	92.3	111.8	157.0	149.2	190.5	107.1	101.7
261	234.8	208.5	102.9	92.7	112.3	157.6	149.8	191.3	107.5	102.1
262	235.7	209.3	103.4	93.0	112.7	158.2	150.3	192.0	108.0	102.5
263	236.6	210.1	103.8	93.4	113.2	158.8	150.9	192.7	108.4	102.9
264	237.5	210.9	104.2	93.8	113.6	159.4	151.5	193.4	108.8	103.3
265	238.4	211.7	104.7	94.1	114.1	160.0	152.1	194.2	109.2	103.8
266	239.3	212.5	105.1	94.5	114.5	160.6	152.6	194.9	109.7	104.2
267	240.2	213.3	105.5	94.8	115.0	161.2	153.2	195.6	110.1	104.6
268	241.1	214.1	105.9	95.2	115.5	161.8	153.8	196.4	110.5	105.0
269	242.0	214.9	106.3	95.6	115.9	162.4	154.4	197.1	111.0	105.4
270	242.9	215.7	106.7	96.0	116.4	163.0	154.9	197.9	111.4	105.8
271	243.8	216.5	107.1	96.4	116.9	163.7	155.5	198.6	111.8	106.2
272	244.7	217.3	107.6	96.7	117.3	164.4	156.1	199.4	112.3	106.6
273	245.6	218.1	108.0	97.1	117.8	165.0	156.6	200.1	112.7	107.0
274	246.5	218.9	108.4	97.4	118.2	165.6	157.2	200.8	113.1	107.4
275	247.4	219.7	108.8	97.8	118.7	166.2	157.8	201.6	113.6	107.9
276	248.3	220.5	109.2	98.2	119.2	166.8	158.4	202.3	114.0	108.3
277	249.2	221.3	109.6	98.5	119.6	167.4	158.9	203.0	114.4	108.7
278	250.1	222.1	110.0	98.9	120.1	168.0	159.5	203.7	114.8	109.1
279	251.0	222.9	110.4	99.3	120.6	168.6	160.1	204.5	115.2	109.5
280	251.9	223.7	110.8	99.7	121.0	169.2	160.7	205.2	115.7	109.9
281	252.8	224.5	111.2	100.1	121.5	169.9	161.3	205.9	116.1	110.3

Cupric Oxide.	Cuprous Oxide.	Copper.	Dextrose.	Starch.	Lavulose.	Hydrated Lactose. $C_{12}H_{22}O_{11} \cdot H_2O$ .	Anhydrous Lactose.	Maltose.	Invert Sugar.	Cane Sugar
282	253.7	225.3	111.7	100.5	121.9	170.5	161.9	206.7	116.5	110.7
283	254.6	226.1	112.1	100.8	122.4	171.1	162.5	207.4	117.0	111.2
284	255.5	226.9	112.5	101.2	122.8	171.7	163.1	208.2	117.5	111.6
285	256.3	227.7	112.9	101.6	123.2	172.3	163.7	208.9	117.9	112.0
286	257.2	228.5	113.4	101.9	123.7	173.0	164.3	209.7	118.3	112.5
287	258.1	229.3	113.8	102.3	124.1	173.6	164.9	210.4	118.8	112.9
288	259.0	230.1	114.2	102.7	124.6	174.2	165.5	211.1	119.2	113.3
289	259.9	230.9	114.6	103.1	125.1	174.8	166.1	211.9	119.6	113.7
290	260.8	231.7	115.0	103.5	125.6	175.5	166.7	212.6	120.1	114.1
291	261.7	232.5	115.4	103.9	126.0	176.1	167.4	213.3	120.5	114.5
292	262.6	233.3	115.9	104.3	126.5	176.7	168.0	214.1	121.0	115.0
293	263.5	234.1	116.3	104.7	126.9	177.6	168.7	214.8	121.4	115.4
294	264.4	234.9	116.7	105.1	127.4	178.4	169.3	215.5	121.9	115.8
295	265.3	235.7	117.2	105.5	127.8	179.0	170.0	216.3	122.3	116.3
296	266.2	236.5	117.6	105.9	128.3	179.8	170.6	217.0	122.8	116.7
297	267.1	237.3	118.1	106.3	128.7	180.4	171.3	217.8	123.2	117.1
298	268.0	238.1	118.5	106.7	129.2	181.0	171.9	218.5	123.7	117.6
299	268.9	238.9	118.9	107.1	129.6	181.7	172.6	219.3	124.1	118.0
300	269.8	239.7	119.4	107.5	130.1	182.3	173.2	220.0	124.6	118.4
301	270.7	240.5	119.8	107.9	130.6	183.0	173.8	220.7	125.0	118.8
302	271.6	241.3	120.2	108.3	131.0	183.6	174.5	221.5	125.5	119.2
303	272.5	242.1	120.6	108.6	131.5	184.3	175.1	222.2	125.9	119.7
304	273.4	242.9	121.1	109.0	132.0	185.0	175.7	222.9	126.4	120.1
305	274.3	243.7	121.5	109.4	132.4	185.6	176.4	223.6	126.8	120.5
306	275.2	244.5	121.9	109.7	132.9	186.2	177.0	224.4	127.3	121.0
307	276.1	245.3	122.2	110.1	133.3	186.9	177.6	225.1	127.7	121.4
308	277.0	246.1	122.8	110.5	133.8	187.6	178.3	225.8	128.2	121.8
309	277.9	246.9	123.2	110.9	134.3	188.2	178.9	226.6	128.6	122.2
310	278.8	247.7	123.7	111.3	134.7	188.9	179.5	227.3	129.1	122.6
311	279.7	248.5	124.1	111.7	135.2	189.6	180.1	228.1	129.6	123.0
312	280.6	249.3	124.5	112.1	135.7	190.2	180.7	228.8	130.0	123.5
313	281.5	250.1	125.0	112.5	136.2	190.8	181.3	229.6	130.5	123.9
314	282.4	250.9	125.5	112.9	136.7	191.4	181.9	230.3	130.9	124.4
315	283.3	251.7	125.9	113.4	137.2	192.2	182.6	231.0	131.4	124.8
316	284.2	252.5	126.4	113.8	137.6	192.8	183.2	231.8	131.9	125.2
317	285.1	253.3	126.8	114.2	138.1	193.4	183.8	232.5	132.3	125.7
318	286.0	254.1	127.3	114.6	138.6	194.1	184.4	233.2	132.8	126.1
319	286.9	254.9	127.7	115.0	139.0	194.8	185.0	234.0	133.2	126.6
320	287.8	255.7	128.2	115.4	139.5	195.4	185.6	234.7	133.7	127.0
321	288.7	256.5	128.6	115.8	140.0	196.0	186.2	235.4	134.2	127.4
322	289.6	257.3	129.1	116.2	140.5	196.7	186.9	236.2	134.6	127.9
323	290.5	258.1	129.5	116.6	141.0	197.4	187.5	236.9	135.1	128.3
324	291.4	258.9	130.0	117.0	141.5	198.0	188.1	237.7	135.5	128.7
325	292.3	259.6	130.4	117.3	142.0	198.7	188.7	238.4	136.0	129.2
326	293.2	260.4	130.9	117.7	142.5	199.3	189.4	239.2	136.4	129.6
327	294.1	261.2	131.3	118.1	143.0	200.0	190.0	239.9	136.9	130.0
328	295.0	262.0	131.8	118.5	143.5	200.7	190.7	240.6	137.3	130.4
329	295.9	262.8	132.2	118.9	144.0	201.4	191.3	241.4	137.8	130.9
330	296.8	263.6	132.6	119.3	144.5	202.0	191.9	242.1	138.2	131.3
331	297.7	264.4	133.1	119.7	145.0	202.7	192.6	242.8	138.7	131.7
332	298.6	265.2	133.5	120.1	145.5	203.4	193.2	243.5	139.1	132.1
333	299.5	266.0	133.9	120.5	146.0	204.0	193.9	244.3	139.6	132.5
334	300.4	266.8	134.4	120.9	146.5	204.7	194.5	245.0	140.0	133.0

Cupric Oxide.	Cuprous Oxide.	Copper.	Dextrose.	Starch.	Lactulose.	Hydrated Lactose, C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> ·H <sub>2</sub> O.	Anhydrous Lactose.	Maltose.	Invert Sugar.	Cane Sugar.
335	301.3	267.6	134.8	121.4	147.0	205.4	195.1	245.7	140.4	133.5
336	302.2	268.4	135.3	121.8	147.5	206.0	195.8	246.5	140.9	133.9
337	303.1	269.2	135.7	122.2	148.0	206.7	196.4	247.2	141.4	134.4
338	304.0	270.0	136.2	122.6	148.5	207.4	197.1	248.0	141.9	134.8
339	304.9	270.8	136.7	123.0	149.0	208.0	197.7	248.7	142.3	135.3
340	305.8	271.6	137.1	123.4	149.5	208.7	198.3	249.5	142.8	135.7
341	306.7	272.4	137.6	123.8	150.0	209.4	198.9	250.2	143.2	136.2
342	307.6	273.2	138.0	124.2	150.4	210.0	199.5	250.9	143.7	136.6
343	308.5	274.0	138.5	124.6	150.9	210.7	200.2	251.7	144.2	137.1
344	309.4	274.8	139.0	125.0	151.4	211.3	200.8	252.4	144.7	137.5
345	310.3	275.6	139.4	125.5	151.9	212.0	201.4	253.2	145.1	138.0
346	311.2	276.4	139.9	125.9	152.3	212.6	202.0	253.9	145.6	138.5
347	312.1	277.2	140.4	126.3	152.8	213.3	202.7	254.7	146.1	138.9
348	313.0	278.0	140.8	126.7	153.2	214.0	203.3	255.4	146.6	139.4
349	313.9	278.8	141.3	127.1	153.7	214.6	203.9	256.1	147.2	139.8
350	314.8	279.6	141.7	127.5	154.2	215.3	204.5	256.9	147.7	140.3
351	315.7	280.4	142.2	127.9	154.6	216.0	205.1	257.6	148.2	140.8
352	316.6	281.2	142.6	128.3	155.1	216.6	205.8	258.3	148.7	141.3
353	317.5	282.0	143.1	128.8	155.6	217.3	206.4	259.1	149.2	141.7
354	318.4	282.8	143.5	129.2	156.1	218.0	207.1	259.8	149.7	142.2
355	319.3	283.6	144.0	129.7	156.5	218.7	207.7	260.5	150.2	142.7
356	320.2	284.4	144.5	130.1	157.0	219.4	208.3	261.3	150.7	143.2
357	321.1	285.2	144.9	130.5	157.5	220.0	209.0	262.0	151.2	143.6
358	322.0	286.0	145.4	131.0	158.0	220.7	209.6	262.8	151.7	144.1
359	322.9	286.8	145.9	131.4	158.5	221.3	210.3	263.5	152.2	144.6
360	323.8	287.6	146.4	131.8	158.9	222.0	210.9	264.3	152.7	145.1
361	324.7	288.4	146.9	132.2	159.4	222.6	211.5	265.0	153.2	145.5
362	325.6	289.2	147.4	132.7	159.9	223.4	212.1	265.7	153.7	146.0
363	326.5	290.0	147.9	133.1	160.4	224.0	212.8	266.5	154.2	146.4
364	327.4	290.8	148.4	133.5	160.9	224.6	213.4	267.2	154.7	146.8
365	328.3	291.6	148.9	134.0	161.3	225.3	214.0	267.9	155.2	147.3
366	329.2	292.4	149.4	134.4	161.8	226.0	214.7	268.6	155.7	147.7
367	330.1	293.2	149.9	134.9	162.3	226.6	215.3	269.4	156.1	148.2
368	331.0	294.0	150.3	135.3	162.8	227.3	216.0	270.1	156.6	148.6
369	331.9	294.8	150.8	135.7	163.3	228.0	216.6	270.8	157.0	149.1
370	332.8	295.6	151.2	136.1	163.8	228.6	217.2	271.6	157.5	149.6
371	333.7	296.4	151.6	136.5	164.2	229.3	217.8	272.3	157.9	150.0
372	334.6	297.2	152.1	136.9	164.7	230.0	218.4	273.0	158.4	150.4
373	335.5	298.0	152.5	137.3	165.2	230.6	219.1	273.7	158.8	150.9
374	336.4	298.8	152.9	137.7	165.7	231.3	219.7	274.5	159.3	151.3
375	337.3	299.6	153.4	138.1	166.1	232.0	220.3	275.2	159.8	151.7
376	338.2	300.4	153.8	138.5	166.6	232.6	220.9	275.9	160.2	152.1
377	339.1	301.2	154.3	138.9	167.1	233.2	221.6	276.7	160.6	152.6
378	340.0	302.0	154.7	139.3	167.6	234.0	222.2	277.5	161.1	153.0
379	340.9	302.8	155.1	139.7	168.0	234.6	222.8	278.2	161.5	153.4
380	341.8	303.6	155.6	140.1	168.5	235.2	223.4	279.0	161.9	153.8
381	342.7	304.4	156.1	140.5	169.0	235.9	224.0	279.7	162.4	154.2
382	343.6	305.2	156.6	141.0	169.4	236.6	224.7	280.4	162.8	154.7
383	344.5	306.0	157.1	141.4	169.9	237.2	225.3	281.2	163.3	155.1
384	345.4	306.8	157.5	141.9	170.4	237.9	225.9	281.9	163.7	155.5
385	346.3	307.6	158.0	142.3	170.9	238.6	226.6	282.7	164.1	156.0
386	347.2	308.4	158.5	142.7	171.4	239.2	227.2	283.4	164.6	156.4
387	348.1	309.2	159.0	143.2	171.9	239.9	227.9	284.2	165.0	156.9

	Cupric Oxide.	Cuprous Oxide.	Copper.	Dextrose.	Starch.	Laevulose.	Hydrated Lactose. $C_6H_{12}O_{11} \cdot H_2O$ .	Anhydrous Lactose.	Maltose.	Invert Sugar.	Cane Sugar.
388	349.0	310.0	159.5	143.6	172.5	240.6	228.5	284.9	165.5	157.3	
389	349.9	310.8	160.0	144.0	173.0	241.2	229.2	285.6	166.0	157.8	
390	350.8	311.6	160.5	144.5	173.5	241.9	229.8	286.4	166.5	158.2	
391	351.7	312.4	161.0	144.9	174.0	242.6	230.4	287.1	167.0	158.7	
392	352.6	313.2	161.4	145.3	174.5	243.2	231.1	287.8	167.5	159.1	
393	353.5	314.0	161.9	145.8	175.0	243.9	231.7	288.5	168.0	159.6	
394	354.4	314.8	162.3	146.2	175.5	244.6	232.4	289.3	168.5	160.0	
395	355.3	315.6	162.8	146.6	176.0	245.2	233.0	290.0	169.0	160.5	
396	356.2	316.4	163.3	147.1	176.5	245.9	233.6	290.7	169.5	160.9	
397	357.1	317.2	163.7	147.5	177.0	246.6	234.3	291.5	170.0	161.4	
398	358.0	318.0	164.2	147.9	177.4	247.2	234.9	292.2	170.5	161.8	
399	358.9	318.8	164.7	148.3	177.9	247.8	235.6	293.0	170.9	162.3	
400	359.8	319.6	165.2	148.7	178.4	248.6	236.2	293.7	171.4	162.8	
401	360.7	320.4	165.7	149.1	178.9	249.2	236.8	294.5	171.9	163.2	
402	361.6	321.2	166.2	149.6	179.4	249.8	237.4	295.2	172.4	163.7	
403	362.5	322.0	166.7	150.0	179.9	250.5	237.9	295.9	172.8	164.1	
404	363.4	322.8	167.2	150.5	180.4	251.1	238.5	296.7	173.3	164.5	
405	364.3	323.6	167.6	150.9	180.9	251.7	239.1	297.4	173.8	165.0	
406	365.2	324.4	168.1	151.3	181.4	252.3	239.6	298.2	174.3	165.4	
407	366.1	325.2	168.6	151.8	181.9	252.9	240.2	298.9	174.7	165.9	
408	367.0	326.0	169.1	152.2	182.4	253.5	240.8	299.7	175.2	166.3	
409	367.9	326.8	169.6	152.7	182.8	254.1	241.4	300.4	175.7	166.8	
410	368.8	327.6	170.1	153.1	183.3	254.7	242.0	301.1	176.2	167.2	
411	369.7	328.4	170.6	153.5	183.8	255.4	242.6	301.8	176.7	167.7	
412	370.6	329.2	171.1	154.0	184.3	256.0	243.2	302.6	177.2	168.2	
413	371.5	330.0	171.6	154.4	184.8	256.6	243.8	303.3	177.7	168.7	
414	372.4	330.8	172.1	154.9	185.3	257.2	244.4	304.0	178.2	169.2	
415	373.3	331.5	172.5	155.3	185.8	257.8	244.9	304.7	178.7	169.6	
416	374.2	332.3	173.0	155.7	186.3	258.4	245.5	—	179.2	170.1	
417	375.1	333.1	173.5	156.2	186.8	259.0	246.1	—	179.7	170.6	
418	376.0	333.9	174.0	156.6	187.3	259.6	246.7	—	180.2	171.1	
419	376.9	334.7	174.5	157.1	187.8	260.2	247.3	—	180.7	171.6	
420	377.8	335.5	175.0	157.5	188.3	260.9	247.9	—	181.2	172.1	
421	378.7	336.3	175.5	157.9	188.8	261.5	248.5	—	181.7	172.6	
422	379.6	337.1	176.0	158.4	189.3	262.1	249.1	—	182.2	173.1	
423	380.5	337.9	176.5	158.8	189.8	262.8	249.7	—	182.7	173.5	
424	381.4	338.7	177.0	159.3	190.3	263.4	250.2	—	183.2	174.0	
425	382.3	339.5	177.4	159.7	190.8	264.0	250.8	—	183.7	174.5	
426	383.2	340.3	177.9	160.1	191.3	264.6	251.4	—	184.2	175.0	
427	384.1	341.1	178.4	160.6	191.8	265.4	252.0	—	184.7	175.4	
428	385.0	341.9	178.9	161.0	192.3	266.0	252.6	—	185.2	175.9	
429	385.9	342.7	179.4	161.5	192.7	266.6	253.2	—	185.7	176.4	
430	386.8	343.5	179.9	161.9	193.2	267.2	253.8	—	186.2	176.9	
431	387.7	344.3	180.4	162.4	193.7	267.9	254.4	—	186.7	177.4	
432	388.6	345.1	180.9	162.8	194.2	268.6	255.1	—	187.2	177.9	
433	389.5	345.9	181.4	163.3	194.7	269.2	255.7	—	187.7	178.3	
434	390.4	346.7	181.9	163.7	195.2	269.9	256.3	—	188.2	178.8	
435	391.3	347.5	182.5	164.2	195.7	270.5	257.0	—	188.7	179.3	
436	392.2	348.3	183.0	164.7	196.3	271.2	257.6	—	189.2	179.8	
437	393.1	349.1	183.5	165.1	196.8	271.8	258.2	—	189.7	180.2	
438	394.0	349.9	184.0	165.6	197.3	272.4	258.9	—	190.2	180.7	
439	394.9	350.7	184.5	166.0	197.8	273.1	259.5	—	190.7	181.2	
440	395.8	351.5	185.0	166.5	198.4	273.8	260.1	—	191.2	181.7	

Cupric Oxide.	Cuprous Oxide.	Copper.	Dextrose.	Starch.	Lævulose.	Hydrated Lactose. $C_{12}H_{22}O_{11} \cdot H_2O$ .	Anhydrous Lactose.	Maltose.	Invert Sugar.	Cane Sugar.
441	396.7	352.3	185.5	167.0	198.9	274.4	260.7	—	191.7	182.2
442	397.6	353.1	186.0	167.4	199.5	275.1	261.4	—	192.2	182.7
443	398.5	353.9	186.5	167.9	200.0	275.8	262.0	—	192.7	183.2
444	399.4	354.7	187.0	168.3	200.5	276.4	262.6	—	193.2	183.6
445	400.3	355.5	187.5	168.8	201.1	277.0	263.3	—	193.7	184.1
446	401.2	356.3	188.0	169.2	201.6	277.7	263.9	—	194.2	184.6
447	402.1	357.1	188.5	169.7	202.1	278.4	264.5	—	194.7	185.0
448	403.0	357.9	189.0	170.1	202.6	279.0	265.2	—	195.2	185.5
449	403.9	358.7	189.5	170.6	203.1	279.7	265.8	—	195.7	186.0
450	404.8	359.5	190.0	171.0	203.6	280.4	266.4	—	196.3	186.5
451	405.7	360.3	190.5	171.5	204.2	281.0	267.0	—	196.8	187.0
452	406.6	361.1	191.0	171.9	204.7	281.7	267.7	—	197.3	187.5
453	407.5	361.9	191.5	172.4	205.2	282.4	268.3	—	197.9	188.0
454	408.4	362.7	192.0	172.8	205.7	283.0	269.0	—	198.4	188.5
455	409.3	363.5	192.5	173.3	206.2	283.7	269.6	—	198.9	189.0
456	410.2	364.3	193.0	173.7	206.8	284.4	270.2	—	199.5	189.5
457	411.1	365.1	193.5	174.2	207.3	285.1	270.9	—	200.0	190.0
458	412.0	365.9	194.0	174.6	207.8	285.8	271.5	—	200.5	190.5
459	412.9	366.7	194.5	175.1	208.3	286.5	272.2	—	201.1	191.0
460	413.8	367.5	195.0	175.5	208.8	287.2	272.8	—	201.6	191.5

## Notes.

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

### AN ABNORMAL SAMPLE OF MILK.

THE highest percentage of fat recorded in any sample of milk examined in the laboratory of the Aylesbury Dairy Company is given by Richmond (*Dairy Chemistry*, p. 150) as 12.52 per cent. Recently a sample of milk was examined by me, which gave the following extraordinary analysis:—Fat, 19.50; lactose, 4.78; protein, 3.77; and ash, 0.82 per cent. The genuineness of the milk is beyond all question; it was received exactly as it came from the cow, and had not been tampered with in any way; I have known the farm from which it came, and also the farmer, for the last 35 years; this particular farmer has always been noted for the richness of the milk sold by him.

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## THE ESTIMATION OF DIRTY SEDIMENT IN MILK.

In an article published in the *British Food Journal* (1919, 21, 61), the author discussed the general principles underlying the work done, in connection with the production of a clean milk-supply, by local authorities through their Public Analysts, and showed that by the estimation of the dirty sediment in milk reliable data could be obtained which would enable such authorities to set up a reasonable standard of cleanliness. These methods have in several areas resulted in a decided improvement in the apparent cleanliness and bacterial contamination of milk. In a second article in the same *Journal* (1922, 24, 51), the author described the apparatus and method employed by him in the determination of dirty sediment, and gave figures showing the reasonableness of the limit of 2 parts by volume of moist extraneous sediment in 100,000 parts of the milk, which he—in common with other workers—has adopted as a standard.

The procedure followed up to a year ago in the treatment of the dirty sediment, prior to its final measurement, has now been superseded by a method which gives much better results. The treatment of the separated sediment in the small centrifuge tube with dilute sodium carbonate solution is fairly satisfactory in most cases; but frequently one examines samples of milk which have a high cell-content, and occasionally others which give a small quantity of curdy deposit, and these deposits are sometimes difficult to separate completely from the true extraneous sediment. The new method to be described appears to be uniformly successful in all cases in giving a complete separation of the white cellular deposit from the extraneous matter, and allows of the further examination of the sediment, after measurement.

When the centrifuge tube has been removed from the centrifugal machine, the milk in the upper part of the tube is poured away, leaving all the sediment, including any cells, &c., in the constricted lower part of the tube. By means of a clean brass rod of such a thickness that it will readily pass to the bottom of the narrow end of the tube, the remaining portion of the milk in the constricted part of the tube immediately above the sediment can be drawn away without disturbing the deposit. About 10 c.c. of strong sodium chloride solution (a nearly saturated brine of specific gravity 1.18 to 1.20 at 15.5° C.) are now poured into the centrifuge tube, and the contents stirred with the brass rod until an intimate mixture has been obtained. The centrifuge tube is closed with a rubber stopper and allowed to stand upright for one to two hours, when it will be found that the cells, etc., in the milk are entirely at or near the surface of the liquid. If the whole of the extraneous sediment has not fallen completely into the lower constricted part of the tube, a short period (1 minute) in the centrifugal machine at a low rate of speed will suffice to bring this about, when by a rapid jerking movement the contents of the upper part of the tube, including all the cellular matter, can be readily ejected. In many cases the simple pouring-off of the brine in the top portion of the tube will carry the cells with it, and this part of the tube can be washed with distilled water, which is then poured off. A final whirling in the centrifugal machine for a few minutes at about 2000 revolutions will now bring the sediment, free from all cellular matter, into the bottom graduated portion of the tube in a compact form, when it can readily be measured. A washing with distilled water, before or after removal of the sediment from the tube, should be given prior to carrying out the microscopical examination and the test for bile-acids.

ARNOLD ROWSBY TANKARD.

40, LOWGATE, HULL.

28th June, 1923.



## 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.*

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### COUNTY OF LANCASTER.

#### ANNUAL REPORT OF THE COUNTY ANALYST FOR THE YEAR 1922.

THE number of samples examined under the Food and Drugs Acts was 4977, of which 4631 were from the County Police, 343 from inspectors of local authorities, and 3 from other persons. Of the samples submitted by the police, 374 (8 per cent.) were condemned, and of those from the inspectors, 18 (5·2 per cent.). The percentage of adulterated samples was the lowest on record in the County.

MILK.—Of 2814 samples taken 267 (9·48 per cent.) were adulterated, deficient or dirty, but only 89 (3·16 per cent.) were serious cases. Of the condemned samples 0·5 per cent. contained more than 5 parts per 100,000 of cow dung, and 1·7 per cent. more than 2 parts, but not more than 5 parts. Two samples contained 10 parts and one 20 parts per 100,000.

The average composition of the milk samples during the year was:—Fat, 3·69; solids-not-fat, 9·03 per cent.

The highest percentage of fat in a supervised milking was 6·95 per cent., and the average 3·51 per cent., the corresponding figures for solids-not-fat being 9·43 and 9·02 per cent.

MARGARINE.—Four samples, labelled “blended with butter,” contained, in three cases, 10 per cent., and in one 4 per cent., of butter respectively. It is submitted that from the report of the Royal Commission on Food Adulteration of 1896, and the debates in Parliament on Clause 8 of the Act of 1899, together with the wording of the clause itself, the intention of the law is that there shall be *no* admixture of butter (as such) with margarine for sale. Ten per cent. of butter *fat* was allowed as a limit to meet contingencies arising from the use of milk in the manufacture of margarine.

MARMITE (YEAST EXTRACT).—A sample contained 1·4 grains of copper per lb., apparently derived from the use of copper vessels in the manufacture.

WHISKEY.—Of 18 samples 17 were below 35 u.p., and 12 from 40 to 48 u.p. In every case but one a notice was exhibited on the premises that “to meet the requirements of the Food and Drugs Act” the spirit was diluted, and that no alcoholic strength was guaranteed. In former years some licensing benches have refused to renew licences unless such notices were removed.

BORAX.—Four samples of borax sold by grocers contained 1·7, 3·4, 4·2, and 8·4 grains of arsenic per lb., respectively. It is suggested that such borax, if sold at all, should be sold under the restrictions of the Poisons or Dangerous Drugs Acts.

INFORMAL SAMPLES.—The number taken was 2691, and of these 48 were adulterated, and 29 corresponding formal samples were taken. Of these 19 were similarly adulterated and 10 were genuine.

W. COLLINGWOOD WILLIAMS.

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## 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.*

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### COPPER IN PEAS.

THE International Tea Co.'s Stores, Ltd., were summoned at Dunstable on June 19, for selling tinned peas containing 0·01 per cent. of copper sulphate (=2·765 grains per lb.), so as to render the food injurious to health.

Professor H. R. Kenwood, Medical Officer of Health for Bedfordshire, said that the amount of copper sulphate required to give a bright colour to peas had been worked out, and was less than half the amount found in these particular peas. Individuals varied in their susceptibility to drugs, but he was convinced that there were some members of the community whose health would be temporarily disturbed as the result of eating about one-third of a pound of these peas. From 11 to 33 per cent. of copper taken into the body was stored in the liver, and must prove injurious to the function of the liver. He could cite two cases within his own experience in which he was satisfied that persons were upset as a result of eating peas. In one case the peas contained a little over  $2\frac{1}{2}$  grains of copper sulphate, and in the other case somewhat more than 3 grains per lb. Very small doses of copper would check the action of the digestive ferments, and the astringent effect was also undesirable during the period of digestion. In cross-examination, he said that he was not aware that 3 grains of copper sulphate per lb. were allowed in New York. He believed that in Italy the amount allowed was practically the same as found in this tin; but he also knew that in other more enlightened countries the use of copper in peas was absolutely prohibited.

Mr. Whiteley, K.C., for the defence, submitted that the amount of copper found in this case was not injurious to health, and said that, although varying decisions had been given by local benches of magistrates, the only case taken to the High Court was in 1904. In that case the percentage of copper was 2·55 grains, and the High Court judges declined to interfere with the decision of the local magistrates, who had dismissed the case.

Dr. P. A. Panton, Director of Clinical Laboratories, London Hospital, described experiments he had made with tinned peas, from which he concluded that about 60 per cent. of the copper remained insoluble when the peas were eaten (*cf. ANALYST, 1923, 267*). In his opinion the amount of copper sulphate per lb. in these peas was not injurious to health, even in the case of a person suffering from kidney disease.

Dr. O. Layton, London Hospital, said that copper sulphate had been given to children suffering from croup, up to six grains per day, and medical literature did not record that any harm had resulted. He did not believe that three grains of copper sulphate would have any effect, if taken in peas during a meal.

The magistrates said they considered the case proved, and imposed a fine of £10 with £9 1s. 6d. costs.

Mr. Whiteley gave notice of appeal on the facts.

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## COPPER IN PEAS: DISCLOSURE ON TIN.

ON June 19th, E. C. Rosevear, trading as Mouser and Co., was summoned at Kensington for selling peas containing 0·005 per cent. of copper, equivalent to 1·380 grains of copper sulphate per lb.

The Town Clerk (Mr. H. Rapson), stated that the summons was taken out under Sec. 3 of the Food and Drugs Acts.

The Inspector said that there was a disclosure on the tin as follows:—"In accordance with the method followed for many years past, and approved by the highest authorities, the contents of this package has been prepared with a small quantity of copper."

Dr. J. Fenton, Medical Officer of Health for Kensington, said that he considered copper in food to be injurious to health. It was a cumulative poison, and copper in the quantity found in these peas would be injurious.

In cross-examination, he stated that he was not aware that the Ministry of Health had issued a circular on the subject of copper in peas, or that negotiations had been proceeding between representatives of the trade and the Ministry of Health.

No evidence was called for the defence, and the defendant was fined £5 and 10s 6d. costs.

## CREAM CHEESE.

ON July 5, A. Mager, provision dealer, was summoned by the Southwark Borough Council for selling as "cream cheese" a "Bondon" cheese containing only 1·2 per cent. of cream.

Mr. C. Dickinson, Public Analyst for Southwark, said he expected to find at least 20 per cent. of cream. In his opinion cream cheese should be made from whole milk or milk to which cream had been added. He admitted that there was no legal standard for this kind of cheese, and was aware that the public called it "cream" cheese.

For the prosecution it was stated that the standard of 20 per cent. of cream had been suggested by the Board of Agriculture. There was to be a standard fixed owing to a recent decision by Mr. Graham Campbell at Bow Street Police Court (ANALYST, 1923, 383).

The Magistrate (Mr. Waddy) said that, although there was no statutory authority as to the quantity of fat, it was not a good defence, since anyone buying cream cheese expected to find he had a cheese which was made, to some extent, of cream; that was only commonsense. In his opinion it was reasonable to expect, as had been suggested, a minimum of 20 per cent. He did not think 1·2 per cent. sufficient, and must decide against the defendant.

The second point raised for the defence was more ingenious—that the public had come to regard "cream cheese" in quite another way, and that they were prepared to accept something without cream when they asked for cream cheese. In his opinion, when a person asked for cream cheese he expected to get something of the "nature, substance and quality" of cream cheese. If this point was to be carried further, they could go on Mr. Graham Campbell's decision.

Defendant was fined 40s., with 42s. costs.

**COMPOUNDED DRUG CONTAINING EXCESS OF POTASSIUM IODIDE.**

ON July 10, C. A. Noble appeared at the Marylebone Police Court to answer a summons by the Hampstead Borough Council for selling a compounded drug containing 11·62 grains of potassium iodide, instead of the prescribed amount, 10 grains per oz.

Dr. Scrase, Medical Officer of Health for Hampstead, stated that, in his opinion, the excess of potassium iodide (16·2 per cent.) was serious, the error being approximately 1 grain per dose.

Dr. Macdonald Brown, consulting physician, said that he should not consider the excess serious; but, in cross-examination, he admitted that an error of 10 per cent. was avoidable.

Mr. Glyn-Jones, referring to the fact that the Government analyst had certified that the excess of potassium iodide was not less than 13·8 per cent., contended that if two analysts could vary to the extent of 3 per cent., surely the pharmacist could. Unless the magistrate had distinct evidence that the patient received appreciably less than his dose, and that there was carelessness on the part of the dispenser, he would not be justified in convicting.

The Magistrate (Mr. Symmons) said that the evidence of Dr. Brown was conclusive. An error of 10 per cent. was avoidable, but it was not a very serious error, and, under the circumstances, the summons would be dismissed, under the Probation of Offenders Act, on payment of four guineas costs.

**Parliamentary Notes.****HOUSE OF COMMONS.**

JULY 11.

**DIRTY MILK VANS.**—Replying to Sir Arthur Churchman, the Minister of Health said that his attention had been directed to the lack of clean and well-ventilated milk vans on certain railway lines, and that he proposed to ask the Minister of Transport to bring the recommendations of the Departmental Committee on the distribution and prices of agricultural produce to the notice of the railway companies.

**CHEESE, MILK, AND CREAM STANDARDS.**—In reply to Mr. Hurd, the Minister of Health said that he understood that the Minister of Agriculture was considering the practicability of giving effect to the recommendation of the Departmental Committee for the imposition of a legal minimum standard of quality for whole-milk cheese, cream, and milk powder.

**Department of Scientific and Industrial Research.****FOOD INVESTIGATION BOARD.****STUDIES IN SWEETENED AND UNSWEETENED CONDENSED MILK.\***

**PART I.—SWEETENED CONDENSED MILK.—INTRODUCTION.**—Statistics are given of the importations of condensed milk for the years 1910 to 1920.

**METHODS OF MANUFACTURE.**—An outline is given of the methods of manufacturing condensed milk, based on the study of the processes used in nine factories

\* Special Report, No. 13. By W. G. Savage, M.D., and R. F. Hunwicke, B.Sc., pp. 103. H.M. Stationery Office. 1923. Price 4/- net.

in England and U.S.A. The actual temperature for the preliminary heating of the milk varies, but is always below boiling point. In the few cases tested it was between 80° and 90° C. The temperature at which the mixture of sugar and milk is heated in the vacuum pans ranges from about 53° to 63° C., and the evaporation takes about 3 hours. Non-sporing bacteria frequently survive the preliminary heating, and the method of cooling in vessels with open tops circulating in water near the ground leads to aerial contamination.

**METHODS OF INSPECTION AT THE PORTS.**—The methods of testing the tins are very similar to those used for the examination of other canned foods (*cf.* ANALYST, 1922, 47, 350). In comparative tests applied to 45 tins the judgment of the inspectors was found to be correct in 87 per cent., or, excluding blown samples, in 50 per cent.

**SUMMARY OF PREVIOUS WORK.**—An outline is given of the work of 16 previous investigators from 1892 to 1920.

**THE BACTERIOLOGY OF CONDENSED MILK.**—The types of organisms isolated comprised yeasts, moulds (to which certain abnormalities have been attributed), micrococci (which are invariably present), true anaerobes, gas-producing aerobic bacilli, sporing aerobic bacilli, thermophilic bacilli, and other aerobic bacilli. *True anaerobes* were isolated from only two tins, and do not appear to be an important cause of decomposition. *Gas-forming aerobes* were found in 9.3 per cent. of the tins. Experiments indicated that *B. coli* strains are not inhibited by the percentage of sucrose present, but that they will not grow, and ultimately die out in sweetened condensed milk. Five types of *sporing aerobic bacilli* were isolated, but it was found that while they can multiply and produce acid, they do not peptonise or even clot the milk. *Thermophilic bacteria* were isolated from 47 of the 76 samples examined. It is obvious that they are very widely distributed in condensed milk, both good and bad. They have less proteolytic capacity than the sporing aerobes. Probably they represent survivals from the milk, and, apart from the possibility of any relationship between their number and the cleanliness of the milk being established, their presence has little significance in the condensed product.

**Examination of Stained Centrifugalised Deposit.**—A ready means of forming an idea of the number of yeasts and bacteria (living and dead) in condensed milk is to mix a little of the sample with water, to centrifuge it, and to fix and stain the deposit on a cover glass. Micrococci of various kinds were detected in almost all the films, and the yeast findings agreed with the cultivation tests.

**YEASTS IN RELATION TO UNSOUND TINS.**—The methods of isolation included the use of glucose agar plates, of a mineral medium containing sucrose, and of a sterilised yeast-water medium. Another method consisted in adding a sterile solution of tartaric acid to the milk so as to bring the final tartaric acidity to 2 per cent. This permits yeasts to grow while inhibiting the growth of other organisms.

**Distribution and Types of Yeasts.**—Yeasts were found in 65 per cent. of the tins, pipe deposits, etc., examined, the most striking result being the high proportion of freshly canned sound factory samples containing them. Nine distinct types of yeast were isolated, four of which had fermentation properties. Rejected blown tins almost invariably contain yeasts, and in 70 per cent. of those examined these yeasts were of the fermenting types. The experiments (described in detail) show conclusively that, in addition to the presence of fermenting yeasts, another factor is of primary importance in the production of fermentation, *i.e.* the ability to multiply sufficiently in sweetened condensed milk under the conditions of anaerobiosis which prevail. Only the yeasts actually isolated from blown tins were capable of fermenting condensed milk, with the production of a blown condition.

*Factors Influencing the Growth of Yeasts.*—Yeasts do not readily multiply in condensed milk. This is not due to the high percentage of sugar, but to the fact that condensed milk is an unsuitable medium, and especially to the restricted supply of oxygen. If the yeasts are present in sufficient numbers, or if they can multiply sufficiently with the little oxygen available, they will be able to produce sufficient enzyme to hydrolyse the sucrose and produce gas, and this process may then continue, even though the yeasts do not increase materially in number. When only a few yeasts gain access they are unlikely to increase sufficiently before anaerobic conditions become established, with the result that they probably gradually die out. This is the probable explanation of the fact that so few of the shop samples examined contained living yeasts.

*Sources of the Yeasts.*—Occasionally, living yeasts derived from the fresh milk may gain access to the vacuum pans. The sugar is usually suspected as the cause of trouble, but the work here described does not support this view. The air in the factory and deposits in the pipes must be regarded as not infrequent sources of infection. The condensed milk is frequently not put into the tins until next day, or even later, so that any yeasts admitted have time for multiplication.

*CHANGES ON KEEPING.*—The  $P_H$  values were determined by colorimetric tests, bromthymol blue being a suitable indicator for the range required. With the batches showing low bacterial counts this value varied very little, being about 6.2. Although various micrococci produce a little acid, great increase in the number of bacteria was not associated with pronounced acidity, and this was never sufficient to alter the composition of the milk, with the production of clots, etc.

*Colorimetric Changes.*—The colour of freshly condensed milk, as measured with Lovibond's tintometer, was: Red, 0.76; yellow, 2.2; blue, 0.16. The gradual colour change which takes place with time, through light yellow to buff, and finally to brown, is probably due to caramelisation of some of the sugar. It occurs much less readily in unsweetened milk. Colour changes are only of importance from the commercial point of view.

*Changes in Consistency.*—These are due to the same physical changes as affect colloids generally. If the preliminary heating is sufficiently high to remove the albumins, which act as protective colloids, subsequent precipitation of the compound of casein with calcium phosphate will be favoured and the viscosity increased.

*Bacterial Content.*—The usual numbers are low (below 5000 per grm.), but in one batch counts of over 40,000 were obtained. In general, the effect of keeping unopened tins at 21° C. and 37° C. was to reduce the number of bacteria. With the admission of a small amount of air there was a fairly rapid increase, mainly due to the multiplication of micrococci. Only air-tight containers should be used; certain types of cans with close-fitting lids are not air-tight. As a practical point, sweetened condensed milk, if used for infant feeding at all, should be put up in tins small enough to contain only one day's supply.

**II. UNSWEETENED CONDENSED MILK.—METHODS OF MANUFACTURE.**  
—An outline is given of the processes used. After condensation in a vacuum pan, the milk is homogenised, cooled and delivered into the cans, which are then usually closed by crimping, and heated in retorts for 30 to 40 minutes at 110° to 113° C., or for 15 to 20 minutes at 110° C.

**BACTERIOLOGY.**—Yeasts are of small importance in relation to unsweetened condensed milk, but micrococci must be regarded as a possible cause of decomposition. Unsoundness in 33 tins was found to be due to the following organisms:—Spring aerobes, 3; micrococci, 18; coccoidal bacillus type, 5; yeast, 1; and gas-producing aerobic bacilli, 6. It is unlikely that tins of evaporated milk ever become blown from chemical means alone.

**TITRATABLE ACIDITY.**—Twenty-four sound shop samples showed a lactic acidity of only 0·3 to 0·41 per cent.; average, 0·36 per cent. In 26 samples with clotted contents the average was 0·90 per cent. (0·45 to 1·70 per cent.). An acidity of 1·49 per cent. was recorded (associated with a diplococcus), although the milk showed no clots. If the acidity is raised to 1·0 per cent. by the addition of lactic acid, and the tin incubated, coagulation will always result, but if the acidity is slowly developed coagulation does not necessarily occur. Given a tin free from organisms, prolonged incubation at 21° C. or 37° C. has very little effect on the acidity or the appearance of the milk.

**GENERAL CONSIDERATIONS.**—The prevalence of sporing aerobic bacilli in a considerable proportion of samples renders it questionable whether the temperatures of processing which can be used without damaging the product will invariably give a sterile product. It appears, however, that it is the micrococci and other non-sporing forms which are the chief danger, and it seems probable, therefore, that a longer processing at a lower temperature would give results as good as those obtained by a shorter time at a higher temperature, and that there would be less risk of damaging the milk.

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## Ministry of Health.

### DRIED MILK.

THE Minister of Health is about to make Regulations as to the labelling and composition of dried milk on the lines of the Regulations recently made with regard to condensed milk. The Regulations will come into operation on the 1st January, 1924, and will apply to all dried milk intended for sale for human consumption in England and Wales.

Copies of the draft Regulations which have been prepared for this purpose can be purchased under the description "Draft of the Public Health (Dried Milk) Regulations, 1923," from H.M. Stationery Office, Imperial House, Kingsway, W.C.2., either directly or through any bookseller (price 2d.). Any representations on the subject should be addressed to the Secretary to the Ministry at an early date.

MINISTRY OF HEALTH, WHITEHALL, S.W.1.  
24th July, 1923:

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## ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

### Food and Drugs Analysis.

**Behaviour of Potassium Nitrate in Minced Meat.** E. Baier and K. Pfizenmaier. (*Zeitsch. Unters. Nahr. Genussm.*, 1923, 45, 192-199.)—The formation of nitrite from the nitrate used for salting meat is due to bacterial action, but the whole of the nitrate disappearing is not converted into nitrite or into products of more profound reduction. The formation of nitrite is favoured by warmth and by the putrefaction of the meat. Prescher's statement that sulphite in presence of nitrite gives no blue colour with starch and potassium

iodate paper is not confirmed; either acid alone gives the blue coloration which, under some conditions, disappears rapidly when the two are present together.

In estimating nitrate in presence of nitrite, the nitrite must first be destroyed by boiling the solution with urea in presence of sulphuric acid. The nitrate is then estimated by means of a sulphuric acid solution of brucine, a solution containing 0.1 mgrm. of potassium nitrate per c.c. being used for purposes of comparison. The nitrite may be estimated by the German official method.

T. H. P.

**Estimation of Starch Content in the Presence of Interfering Polysaccharides.** G. P. Walton and M. R. Coe. (*J. Agric. Research*, 1923, 23, 995-1006.)—The method is devised primarily for the estimation of starch in linseed meal, which is sometimes adulterated with non-flax material. The usually accepted limit for such material is 6 per cent., and, as its starch content is generally 50 per cent., the presence of more than 3 per cent. of starch in linseed indicates adulteration. The presence of polysaccharides, plant mucilages and pectins renders the ordinary hydrolysis methods useless for this purpose. The method worked out is in the following stages: (a) Extraction of the material with five portions of ether, then with 35 per cent. (by volume) alcohol, then with 95 per cent. alcohol, and finally again with ether to eliminate interfering substances. (b) The starch is gelatinised and converted by digestion with infusion of malt. The malt infusion is prepared by digesting 5 grms. with 100 c.c. of water for two hours, then filtering it until clear. The charge of material, after removal of the alcohol and ether by drying, is mixed into a smooth paste with water, 100 c.c. of boiling water are added, and after boiling, the mixture is cooled to 50° or lower temperature. Twenty c.c. of malt infusion are now added, and the temperature is gradually raised during an hour to 80°. After the further addition of 20 c.c. of malt infusion, the mixture is kept at 55° C. for an hour, and the temperature is then raised above 80°. (c) The interfering polysaccharides are precipitated with 60 per cent. alcohol by diluting the mixture to 200 c.c. with water, then adding 316 c.c. of 95 per cent. alcohol and sufficient water to make the total volume up to 500 c.c., plus 3 c.c. for every four grms. of material taken. The mixture is filtered, and 200 c.c. of filtrate are evaporated to 20 c.c. to remove the alcohol. (d) The resulting dextrose solution is defecated with phosphotungstic acid; the 20 c.c. residue is diluted to 200 c.c., 20 c.c. of hydrochloric acid are added, and the mixture heated on the water bath under a reflux condenser for 2½ hours, after which it is cooled, and 1 c.c. of 10 per cent. solution of phosphotungstic acid is added. After being allowed to stand the mixture is diluted to 250 c.c. and filtered, and 200 c.c. of the filtrate neutralised, first with sodium hydroxide, and towards the end with sodium carbonate. (f) The dextrose is estimated by gravimetric copper reduction in the usual way. From the weight of dextrose obtained is deducted the weight of dextrose given by a blank test on the same volume of the malt infusion, and the resultant quantity of dextrose multiplied by 0.90 represents the yield of starch, though the actual proportion is more accurately represented by the empirical factor 0.93.

H. E. C.



**The Highly Unsaturated Fatty Acids of Fish Oils.** J. B. Brown and G. D. Beal. (*J. Amer. Chem. Soc.*, 1923, **45**, 1289-1303.)—The authors were able to prepare and analyse the methyl, ethyl and normal butyl esters of the acids of menhaden oil and the bromination products, and obtained indications of the presence of more highly unsaturated acids than clupanodonic acid. A comparative analysis of commercial fish oils showed that these oils, as a class, contain acids whose esters distil at too high a temperature for the C<sub>18</sub> series, and among them were again found acids more highly unsaturated than clupanodonic acid. The fatty acids of menhaden oil were then roughly separated according to molecular weight by fractionating the esters from 3 kilos. of oil six times under diminished pressure. The presence of acids with 16, 18, 20 and 22 carbon atoms was indicated. The lead soap and ether method of separation was not found very satisfactory, but the products obtained by this method and the barium soap and benzene method were esterified, fractionated, and their bromine addition products analysed. Further, the ether-insoluble bromine addition products were analysed. Details of methods used, together with tables of constants for many of the compounds prepared, are given in the paper, and the authors claim to have demonstrated the presence in fish oils of the following acids:—Myristic, palmitic, palmitolic (hexadecatrienoic), clupanodonic (octodecatetrenoic), arachidonic, eicosapentenoic, docosapentenoic and docosahexenoic.  
D. G. H.

**Inactive Malic Acid as a Food Acidifier.** J. M. Weiss, C. R. Downs and H. P. Corson. (*J. Ind. Eng. Chem.*, 1923, **15**, 628-630.)—Synthetic malic acid is now produced on a large scale, and is stated to have a flavour superior to that of citric acid. Experiments as to its physiological action show that it may be used safely in place of citric and tartaric acids in such food products as jellies, confectionery, etc., and in beverages.  
W. P. S.

**Methyl Anthranilate in Grape Beverages and Flavours.** R. D. Scott. (*J. Ind. Eng. Chem.*, 1923, **15**, 732-733.)—A method described by Erdmann (*ANALYST*, 1902, **27**, 125) for the estimation of methyl anthranilate is, briefly, as follows:—The diazotised solution of the methyl anthranilate is added slowly from a burette to a standard alkaline solution of  $\beta$ -naphthol; portions of the liquid are filtered from time to time, and the filtrate is tested with more of the diazotised solution until no further precipitation occurs. This volumetric method is unsatisfactory, owing to the action of the alkali on the compound, the difficulty of regulating the diazotisation, and to the fact that not less than 0.5 gm. of the ester can be titrated with accuracy. A gravimetric modification of the method, however, yields trustworthy results. About 0.03 gm. of methyl anthranilate dissolved in 5 c.c. of ethyl alcohol is diluted to 100 c.c. with water, and 2 c.c. of *N* hydrochloric acid and 1 c.c. of 5 per cent. sodium nitrite solution are added. After one minute the mixture is poured into a beaker containing 20 c.c. of 0.2 per cent.  $\alpha$ -naphthol solution (alcoholic) and 3 c.c. of *N* sodium bicarbonate solution. The mixture is placed aside for fifteen hours, the precipitate then collected on a filter, washed with 50 c.c. of water, dried at 100° C. for two hours, and weighed.

The weight of the precipitate is multiplied by 0.4935 to obtain the equivalent amount of methyl anthranilate. In the case of grape juices, 500 c.c. of the sample are steam-distilled, and 500 c.c. of distillate are collected in a receiver cooled with ice and provided with a mercury seal; 100 c.c. of the distillate are used for the estimation. Twenty-five c.c. of the sample are sufficient for the analysis of artificial grape concentrated extracts; this quantity is steam-distilled until 500 c.c. of distillate have been collected. Seven samples of Concord grape juice examined contained from 0.80 to 1.49 parts per million of the ester. For very small quantities of methyl anthranilate (less than 0.00025 gm.) a colorimetric method may be used. One hundred c.c. of the ester solution is treated for thirty seconds with 1 c.c. of *N* hydrochloric acid and 0.5 c.c. of 5 per cent. sodium nitrite solution, and the mixture is then poured into a Nessler cylinder containing 0.5 c.c. of 2 per cent.  $\alpha$ -naphthol solution (alcoholic) and 3 c.c. of *N* sodium hydroxide solution. The maximum coloration develops within fifteen minutes; it is then compared with the colorations shown by standards prepared under the same conditions. In both the gravimetric and colorimetric methods better results are obtained with  $\alpha$ -naphthol than with  $\beta$ -naphthol.

W. P. S.

**Hesperidin Deposits in Apricots.** C. Griebel (*Zeitsch. Unters. Nahr. Genussm.*, 1923, 45, 238-240).—During last year apricots were frequently submitted for examination on account of their bitter taste. White dendritic deposits were observed about the endocarp of these fruits, and under the microscope these were seen to be composed of aggregates of spherical masses made up of fine acicular crystals. These crystals were insoluble in hot water, alcohol, ether or dilute acids, but dissolved readily in dilute alkali solutions to form a yellow solution. When treated with concentrated sulphuric acid they became deep yellow to orange and reddish brown on warming. The yellow alkaline solution gave a bright red coloration with blue tinge on the addition of sulphuric acid, and the crystals were also identified by other tests as the glucoside hesperidin.

**Injurious Potatoes rich in Solanin.** C. Griebel. (*Zeitsch. Unters. Nahr. Genussm.*, 1923, 45, 175-183.)—In two different cases, burning and itching in the throat, nausea, and retching following the eating of potatoes, were found to be due to the presence of solanin in excessive amounts. The percentages of solanin were: (1) 0.0495, of which 0.0177 occurred in the peel, and (2) 0.079, with 0.0235 in the peel. These cases are the first to be recorded of such trouble arising in the winter. It appears possible that the formation of solanin in such large proportions is due, to some extent, to the action of light. (*Cf. ANALYST*, 1918, 43, 133.)

T. H. P.

**High Solanin Content of Potatoes.** A. Bömer and H. Mattis. (*Zeitsch. Unters. Nahr. Genussm.*, 1923, 45, 288-291.)—Six separate cases in 1922 are recorded in which fresh potatoes of normal appearance had a bitter taste, or had caused vomiting and other symptoms, suggestive of solanin poisoning. The amounts of solanin in these samples were accordingly estimated, a special method

being devised for the purpose, since the methods of Schmiedeberg and Meyer (*Arch. exper. Path. u. Pharmacol.*, 1895, **36**, 361), and of Morgenstern (*Landw. Versuchsstat.*, 1907, **65**, 301) were found to give unsatisfactory results:—About 200 grms. of the ground-up potatoes were mixed with 250 c.c. of water, and the mass expressed after some time. The residue was then digested for 30 minutes with three successive portions of 250 c.c. of water acidified with 5 drops of acetic acid, and expressed after each digestion. The united pressings were rendered slightly alkaline with ammonia and evaporated to dryness with 10 grms. of kieselguhr, and the dry residue powdered and extracted for 5 hours with 95 per cent. alcohol in a Soxhlet extractor, the residue being then ground up and again extracted for 5 hours. The total extract was evaporated, the residue dissolved in water acidified with a few drops of acetic acid, the solution made slightly alkaline with ammonia and heated on the water bath, and the flocculent precipitate of solanin filtered off and washed with warm 2.5 per cent. ammonia solution. Finally, the solanin was purified by dissolving it in alcohol, filtering the solution, evaporating the filtrate and treating the residue as described above, and the final colourless residue was collected on a weighed filter. Three normal varieties of potatoes of different kinds contained, 2.0, 2.1 and 7.5 mgrms. of solanin in 100 grms. of potatoes, whilst the six suspected samples gave the following results:—(1) 57.8 and 58.8; (2) 50.5; (3) 37.6; (4) 43.4 and 41.2; (5) 26.1 and 25.3; and (6) 32.9 mgrms. per 100 grms.

**Seeds of *Chydenanthus excelsus*. M. Duyster.** (*Pharm. Weekblad*, 1923, **60**, 777–799.)—The seeds of *Chydenanthus excelsus*, Miers (*Lecythidaceæ*), a tree growing in the Dutch Indies, are used in the preparation of medicinal remedies for flatulence and diarrhoea. A sample of the powdered seed gave the following results on analysis:—Water, 10.01; ash, 2.47; nitrogen, 1.37; crude cellulose, 2.48; glucose, 68.7; soluble in petroleum spirit, 0.50; soluble in ether, 0.12; and soluble in 70 per cent. alcohol, 14.87 per cent. The fatty oil, extracted with petroleum spirit, had the following characteristics:—Sp. gr. at 15° C., 0.9495;  $n_D^{25}$ , 1.4877; saponification value, 140.6; and iodine value, 112.25. The ethereal extract contained gallic acid, and the alcoholic extract contained a crystalline glucoside, chydenanthin,  $C_{21}H_{34}O_{10}$ , which, on hydrolysis, yielded a mixture of sapogenins, arabinose and galactose. Chydenanthin has a strong hæmolytic action and is a cardiac poison.

## Bacteriological, Biochemical, etc.

**The Whittles Vibration Method of Obtaining a Suspension of the Bacteria in a Soil. H. G. Thornton.** (*J. Agric. Sci.*, 1923, **13**, 352–353.)—It is pointed out that the results obtained by the vibration method (ANALYST, 1923, 280) are much too high. Thus one billion bacterial cells taken at one cubic micron each would mean a volume of 1 c.c., whereas Whittles records finding 27 billions in a single grm. of soil. The discrepancy may be due to the difficulty of adequately sterilising the vibration apparatus described by Whittles. It is also pointed out that physical factors, such as moisture content, affect the ease with which soil

particles can be dispersed by shaking. With regard to the direct counting of films prepared by the same author's method, there remains the difficulty of distinguishing between live bacteria, dead bacterial cells and soil particles, and a number of parallel counts are required to show the experimental error. H. E. C.

**Estimation of Sodium in Biochemical Work. A. Blanchetière.** (*Bull. Soc. Chim.*, 1923, **33**, 807-818.)—The qualitative micro-chemical method of Streng (*Zeitsch. anal. Chem.*, 1884, **23**, 115) has been adapted to the estimation of sodium in small quantities (1 c.c. or less) of blood. This method depends upon the formation of a triple acetate of uranium, magnesium and sodium ( $3\text{UO}_2(\text{C}_2\text{H}_3\text{O}_2)_2 + \text{Mg}(\text{C}_2\text{H}_3\text{O}_2)_2 + \text{Na}(\text{C}_2\text{H}_3\text{O}_2) + 9\text{H}_2\text{O}$ ), which is extremely insoluble in excess of the uranium and magnesium reagent. This reagent is prepared by mixing equal volumes of the two solutions A and B:—

A. Uranium acetate, 100 grms.; acetic acid, 60 grms.; water to 1000 c.c.

B. Magnesium acetate (dry), 333 grms; acetic acid, 60 grms.; and water to 1000 c.c. The mixed solutions are allowed to stand and filtered before use.

Ammonium, potassium, magnesium and calcium in normal solution do not affect the estimation, but barium and zinc in more or less concentrated solutions form precipitates. Phosphoric acid must be removed either by the uranium or the magnesia-mixture method.

*Method of estimation in presence of phosphate.*—To remove the phosphate, a known excess of solution A only is added to a known volume of the solution to be analysed, the liquid left until the precipitate has settled, and then centrifuged, and an aliquot part of the clear solution is taken for the sodium estimation and added to 10 times its volume of the mixed reagents A and B. If the phosphate is to be removed by the magnesia method, a known volume of the test solution is treated with a known volume of 10 per cent. ammonium chloride solution, 1 c.c. of strong ammonia solution and a small amount of magnesia. After several hours' standing the liquid is centrifuged, and an aliquot part of the clear solution taken for the sodium estimation after evaporation of the excess ammonia. In either case, complete precipitation is induced by rubbing the walls of the beaker with a glass rod, and the whole allowed to stand for at least half an hour, with occasional stirring, after which the precipitate is washed by decantation three times with the mixed reagent, and then three times with 95 per cent. alcohol, and is finally collected in a weighed Gooch crucible, dried at 110° C. for at least half an hour, cooled and weighed. The molecular weight of this dried product is 1390, thus being 60.43 times the weight of the sodium present. R. F. I.

**Estimation of Urea alone and in the Presence of Cyanamide by means of Urease. E. J. Fox and W. J. Geldard.** (*J. Ind. Eng. Chem.*, 1923, **15**, 743-745.)—The method proposed is based on the conversion of urea into ammonium carbonate by the action of the enzyme urease, and subsequent titration of the ammonia thus formed. The urease solution used is prepared by mixing soya bean flour with twenty times its weight of water, neutralising the mixture, after fifteen minutes, with 0.1 N hydrochloric acid, and filtering the solution. For the estima-

tion of urea alone, about 0.5 gm. of the sample is dissolved in 250 c.c. of water, 25 c.c. of this solution are placed in a stoppered bottle and neutralised, methyl-red being used as indicator, and 10 c.c. or more of the urease solution are added. After one hour a definite excess of 0.1 N hydrochloric acid is added, air is aspirated through the mixture for ten minutes to remove all carbon dioxide, and the excess of acid is then titrated with 0.1 N sodium hydroxide solution. To estimate urea in cyanamide, 2 grms. of the sample are extracted with 400 c.c. of water for two hours, 2 grms. of sodium carbonate are added to precipitate the calcium, the mixture is shaken for thirty minutes, filtered, and the urea is estimated in 25 c.c. of the filtrate as described, after the solution has been neutralised and the carbon dioxide expelled. In the case of phosphate mixtures, the aqueous extract is treated with barium hydroxide to precipitate phosphates, and the excess of barium, together with calcium salts, is removed by treatment with sodium carbonate and filtration.

W. P. S.

## Toxicological and Forensic.

**Formation of Phenol by Intestinal Bacteria.** L. R. Bouwman. (*Pharm. Weekblad.*, 1923, 60, 845-847.)—Tests for the presence of phenol-producing bacteria in faeces were made by means of Rhein's method (*Biochem. Zeitsch.*, 1917, 246). Cultivations are made in a nutrient medium consisting of 0.3 gm. of tyrosin (which yields phenol on decomposition) 5 grms. of asparagin, 5 grms. of ammonium lactate, 2 grms. of dipotassium hydrogen phosphate, and 0.2 gm. of magnesium sulphate in 1 litre of water. The liquid is sterilised in flasks of about 15 c.c. capacity, inoculated with the faeces, and kept for 3 days at 37° C., after which it is tested for phenol by adding a few drops of 10 per cent. formaldehyde solution, followed by strong sulphuric acid, poured down the side of the vessel. A reddish violet ring is obtained in the presence of 1 part of phenol in 40,000. By the use of this method phenol-producing bacteria were detected in 7 out of 32 specimens of faeces examined. Sub-cultivations showed that these bacteria belonged to the *Coli* group. (Cf. Maclaurin, *ANALYST*, 1922, 47, 294.)

## Organic Analysis.

**Estimation of Formaldehyde in Paraformaldehyde.** P. Borgstrom and W. G. Horsch. (*J. Amer. Chem. Soc.*, 1923, 45, 1493-1497.)—Examination of the published methods for the estimation of formaldehyde in paraformaldehyde shows that the undermentioned four methods are accurate. Romijn's iodimetric method (*ANALYST*, 1897, 22, 221), the hydrogen peroxide method of Blank and Finkenberg (*ibid.*, 1899, 24, 22), the neutral sulphite method (cf. Lemme, *ibid.*, 1903, 28, 363), and Messenger's method of oxidation by dichromate (*Ber.*, 1888, 21, 2914). The fixed alkali method, which consists in heating with excess of alkali and subsequently titrating the excess, is not satisfactory, as the solution always becomes coloured. (Cf. Fresenius and Grünhut, *Zeitsch. anal. Chem.*, 1905, 44, 15.)

H. E. C.

**Analysis of Gas Mixtures Containing Methyl Chloride.** R. H. McKee and S. P. Burke. (*J. Ind. Eng. Chem.*, 1923, **15**, 578-579.)—In the case of a gaseous mixture containing methyl chloride, methyl ether, methane and other components, the methyl chloride may be estimated by burning the gas over concentrated potassium hydroxide solution, in a combustion pipette, and then titrating the chloride in the alkaline solution. The gas burette and the combustion pipette are connected by a three-way tap, and mercury is used as the containing fluid; a layer of concentrated potassium hydroxide solution is introduced into both parts of the apparatus, a volume of oxygen more than sufficient for the combustion is admitted to the combustion pipette, and the platinum spiral in the pipette is heated to bright redness by passing an electric current through it. The gas to be analysed is measured in the burette and then passed slowly into the pipette. Combustion usually proceeds smoothly and readily. At the end of the operation, the alkali solution is washed out of the burette and pipette, neutralised, and the chloride is titrated. In conjunction with the acetic acid absorption method, the method described above furnishes a means of analysing a mixture containing other components soluble in organic solvents. If ethylene is present in the gas mixture, it may be estimated as follows:—300 c.c. of a mixture of 10 per cent. of ethylene and 90 per cent. of air are shaken with bromine-water of known concentration, and the maximum volume of bromine-water which it will decolorise is ascertained. An equal volume of the gas mixture under examination is treated in the same way, the maximum volume of bromine-water decolorised being noted, and the percentage volume of ethylene is then found by calculation. W. P. S.

**Estimation of Mucic Acid.** E. O. Whittier. (*J. Amer. Chem. Soc.*, 1923, **45**, 1391-1397.)—A small excess of potassium permanganate solution is added to the solution of mucic acid acidified with sulphuric acid, the mixture boiled and allowed to stand for five minutes, after which a known quantity of standard oxalic acid solution is added, and the titration completed with permanganate. As the oxidation of mucic acid by permanganate does not strictly follow the stoichiometric equation it is necessary to standardise the solution against a known quantity of mucic acid. Tartaric and racemic acids may be estimated in the same manner. Mucic acid, but not tartaric or racemic acids, may be estimated in the same solution with oxalic acid by first titrating the oxalic acid with permanganate at 50° C. to an end-point persistent for ten seconds, then adding excess of permanganate, boiling, and continuing the estimation as already described. There is a small constant error, which the operator should determine for himself, due to slight oxidation of mucic acid during the titration of the oxalic acid. When 0.05 *N* potassium permanganate is used the error amounts to 1.5 c.c., which is deducted from the volume of permanganate solution required for the oxalic acid titration and added to that required by the mucic acid. Other oxidising or reducing substances must not be present; purification of the oxalic and mucic acids may be effected by their precipitation as calcium salts, but in the case of mucic acid four days are necessary for the complete precipitation of calcium mucate. H. E. C.

**Heat Treatment of Chinese Wood (Tung) Oil and Linseed Oil.** C. E. Mabery. (*J. Ind. Eng. Chem.*, 1923, **15**, 365–367.)—Chinese wood oil polymerises slowly when heated at temperatures below 150° C. and out of contact with air, but at 200° C. it is transformed rapidly into the solid, brittle condition. The change proceeds more quickly when the oil is heated in a current of air, and a considerable quantity of oxygen is absorbed. It appears that the most coherent polymerised form of the oil is obtained by slow heating in a current of air at 125° to 150° C., or at a higher temperature in a current of carbon dioxide. Linseed oil is not affected, or only slightly, when heated below 150° C. in the absence of air; in a current of air the polymerisation proceeds slowly at 150° C., and more rapidly at 200° C.

W. P. S.

**Changes in Powdered Rosin stored in Closed Containers.** F. P. Veitch and W. F. Sterling. (*J. Ind. Eng. Chem.*, 1923, **15**, 576–577.)—When powdered rosin is kept in a closed bottle its composition, as shown by the usual analytical constants, changes to a considerable extent. The change is appreciable within one week's storage, and is not complete within six weeks. In the case of powdered samples kept for six weeks there was an increase of 5.1 to 9.3 in the acid values, an increase of 5.1 to 8.9 in the saponification values, a decrease of 47.3 to 52.2 in the iodine values, and an increase of 8.9 to 11.3° C. in the melting points. Samples should therefore be prepared for analysis immediately before the analysis is commenced.

W. P. S.

**Analysis of Chrome-Tanned Leather.** E. Little and E. Sargent. (*J. Ind. Eng. Chem.*, 1923, **15**, 633.)—The peroxide fusion method may be modified by using ammonium fluoride in acid solution to remove ferric iron, in order to eliminate any error due to the presence of iron and barium (action of ferric salts on potassium iodide and the precipitation of barium chromate). About 1.5 gm. of the leather is ignited in a porcelain crucible and the ash is fused for 15 minutes in an iron crucible with 5 grms. of sodium peroxide. After cooling, the fused mass is dissolved in 150 c.c. of water, 0.1 gm. of sodium peroxide is added to ensure complete oxidation, and the solution is boiled for twenty minutes to decompose the excess of peroxide. The cold solution is then treated with hydrochloric acid until all the ferric hydroxide is dissolved, and an excess of 5 c.c. of concentrated hydrochloric acid is added for each 100 c.c. of the solution. Ammonium fluoride is added until a drop of the solution ceases to give a reaction for ferric salt when tested with potassium ferrocyanide, and an excess of 1 gm. of the fluoride is introduced. Three grms. of potassium iodide are then added and, after three minutes, the liberated iodine is titrated with 0.1 N thiosulphate solution.

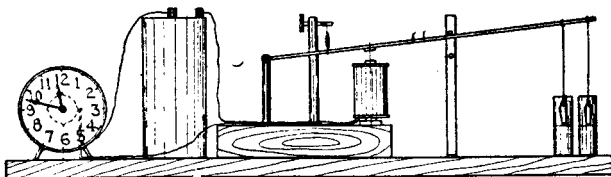
W. P. S.

**Examination of Writing Inks.** F. F. Rupert. (*J. Ind. Eng. Chem.*, 1923, **15**, 489–493.)—Practical methods are described for testing and comparing ink intended for definite purposes, a reference being given to the methods of the Bureau of Standards (*ANALYST*, 1921, **46**, 61), for the analytical examination. *Streak tests*:—A sheet of bond paper of good quality, 11 inches long, is stretched

over a sheet of glass, 8 inches wide and secured by means of rubber bands. The glass is fixed at an angle of  $45^\circ$ , and about 0.6 c.c. of the ink is delivered on to the top of the paper from a special pipette, held vertically with its outlet almost touching the paper. *Penetration and fluidity*:—The ink should penetrate into the fibres of the paper, but not pass through it. A normal ink forms a streak with an oval head, and the remainder should be nearly uniform in width; a very fluid ink gives a wide head, and the streak rapidly narrows. *Temporary Colour*:—The streaks are compared soon after making and graded in the order of the colours. Dye which is not in solution produces a speckled or irregular streak. *Permanent Colour*:—The streaks are exposed for 7 days to diffused light in an atmosphere free from excessive dust, and the colours again compared, a streak made with the standard ink being made the basis of comparison. For exposure tests, the paper is cut horizontally into strips of uniform width, preferably 1 inch, and the top and bottom strips are kept as blank specimens. *Exposure to light*:—One strip is exposed for at least 2 weeks in a position where it will obtain as much light as possible. Or the strips may be exposed for 48 hours or longer at a short distance from a source of ultra-violet light. A nitrogen-filled 100 watt tungsten light may be used in the same way, as the source of light. The nearest approximation to sunlight is obtained by exposure for part of the time to each of these sources of artificial light. A short exposure enables differences to be noted in the colour of different inks, but a complete light test for permanence requires several months' exposure. *Exposure to weather*:—One of the strips is exposed in a position where it will be as free as possible from dust and smoke and the progress of the test is noted at weekly intervals. *Exposure to water and reagents*:—Narrow strips are immersed in water, 10 per cent. ammonia solution, 2 per cent. hydrochloric acid, and bleaching powder (or nearly neutralised sodium hypochlorite) solution containing 0.005 *N* available chlorine. The solutions should be stirred frequently and the strips observed from time to time. For inks which are easily removed a good measure of permanence is the time required to remove all the ink except a pale yellow stain. For others, the amount of ink left after a given time is the best indication. The resistance of the dye, as indicated by the rate of disappearance of the blue colour, should also be noted. In some cases it is advantageous to vary the strength of the hypochlorite solution. *Stability*:—Any sediment in the original bottle should be noted. The ink is then filtered, and about 15 c.c. put into each of two small bottles of the same size and shape, one of which is tightly corked and the other covered with filter paper. After 7 days the condition of each is noted, and each sample is then filtered again and the relative amount of sediment noted. Any sediment beyond fine loosely-scattered flakes on the paper is to be regarded as excessive. *Corrosion (Complete Immersion Test)*:—Pens of unplated steel, of the same size and make, are used, and it is advisable to take the average of 5 separate tests with each ink. The pens are washed successively with alcohol and ether and weighed, and one put into 10 or 20 c.c. of each of the samples of ink, which are then left for 7 days in loosely covered bottles. The pens are then removed, wiped free from deposit, washed, dried and weighed. *Corrosion (Mechanical Tester)*:—Inks may be tested under nearly working



conditions by the use of a device which dips the pens at intervals into the ink and allows it to dry between the immersions. An apparatus of this kind is shown in the diagram, in which the clock has a circular plate with four equidistant projections attached to the minute-hand shaft, and there is a spring which makes contact with each projection for a few seconds as it is carried round. The motive force is supplied by a dry cell, and a 2-spool electro-magnet of suitable strength and



MECHANICAL INK TESTER

resistance; there is also an aluminium bar 10 inches long, pivoted at one end so as to move vertically, an armature at the middle of the bar just over the electro-magnet, a cross-bar at the other end to support the pens, and a brass spring (or rubber band) to raise the bar when released. Four pen nibs may be supported from the bar and from the cross-bar by means of iron wires passing through holes in the bars and through the slots in the pens. The ink is contained in four small jars each holding about 20 c.c.; when contact is made the nibs are practically completely immersed, and they are just out of the ink when the bar is released. A test of 5 to 7 days is sufficient for the purpose. The results obtained with this tester are usually in the same order as those given by the immersion test, but not in the same ratio, especially in cases where "negative catalysers" have been added to reduce corrosiveness. One of these substances reduced the corrosion to a very low amount when the pen was immersed all the time, but showed little improvement when tested by the mechanical tester. The appearance of the pen after it has been used for a day or so longer, in the tester, enables an estimate of the amount of deposition on the pen to be made.

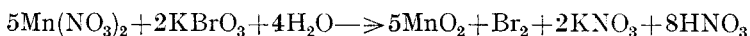
*Comparison and grading.*—It is suggested that corrosiveness, stability, permanence, and colour should be rated at 25 each on a scale of 100. *Colour.*—The initial and final colours are each rated at 12.5, a satisfactory ink of known composition being selected as the standard, and the same ink diluted to a point where it is quite unsatisfactory taken as zero. The intermediate steps are found by mixing the extremes. *Permanence.*—The results of the weather test or of the hypochlorite test are used, and the ink under examination compared with a series of inks of varying concentration prepared from the standard ink. *Corrosion.*—High and low limits are chosen arbitrarily. *Stability.*—No sediment is rated at 25 and a chosen maximum as 0.

## Inorganic Analysis.

**Influence of Alkali Metals on the Ferrocyanide Titration of certain Metals.** W. D. Treadwell and D. Chervet. (*Helv. Chim. Acta*, 1923, 6, 550-559).—The behaviour of zinc, lead, and cadmium has already been investigated

electrometrically (ANALYST, 1922, 47, 535). *Nickel* yields with lithium ferrocyanide  $\text{Ni}_2\text{Fe}(\text{CN})_6$ , with the sodium salt  $\text{Ni}_3\text{Na}_2(\text{Fe}(\text{CN})_6)_2$ ; potassium ferrocyanide behaves at first like the sodium salt, but at a later stage of the titration the precipitate becomes richer in potassium and poorer in nickel. The department of *cobalt* is similar to that of nickel. *Manganese*, in neutral or weak acetic acid solution, furnishes  $\text{Mn}_2\text{Fe}(\text{CN})_6$  with lithium or sodium ferrocyanide, whereas the potassium salt causes formation of almost pure  $\text{MnK}_2\text{Fe}(\text{CN})_6$ . In the case of *silver*, the compound  $\text{Ag}_4\text{Fe}(\text{CN})_6$  is always obtained. W. R. S.

**Use of Bromate in Volumetric Analysis. I. Stability of Bromic Acid in Boiling Solutions. G. F. Smith.** (*J. Amer. Chem. Soc.*, 1923, 45, 1115.)—Hitherto the use of bromic acid in quantitative analysis has, in general, been limited to those estimations where an excess of the reagent was not required, but in the course of estimating manganese in its ores the reaction—



—was found to proceed better in presence of an excess of standard potassium bromate in acid solution, and a study of the stability of bromates in boiling acid solutions was therefore undertaken. This showed that (1) 0.025 *N* solutions of pure potassium bromate and 2 *N* solutions with perchloric, nitric and acetic acids are not decomposed after boiling for 5 minutes, but with sulphuric and phosphoric acids the solutions are much less stable. (2) The presence of free bromine does not affect the stability of hot acid solutions except in the case of acetic acid. (3) The bromide in bromate may be determined by acidifying the solution and distilling the pure bromine liberated. (4) Barium bromate gives results similar to the potassium salt. D. G. H.

**Use of Bromate in Volumetric Analysis. II. Influence of Mercuric Mercury upon Bromic Acid Reactions. G. F. Smith.** (*J. Amer. Chem. Soc.*, 1923, 45, 1417–1422.)—The stability of bromic acid in boiling solutions containing mineral acid has been shown (see preceding abstract). It is now proved that the mercury in the form of mercuric perchlorate enables bromate reactions to be carried either to the formation of bromide or free bromine as desired. The addition of mercuric perchlorate prevents the oxidation of chloride, bromide, or iodide by bromate in solutions containing either nitric or perchloric acids; so that the following reactions of bromate (*inter alia*) take place in the presence of sufficient mercuric perchlorate to form undissociated mercuric bromide.

- (1)  $3\text{Mn}(\text{NO}_3)_2 + \text{KBrO}_3 + 3\text{H}_2\text{O} \longrightarrow 3\text{MnO}_2 + \text{KBr} + 6\text{HNO}_3$ .
- (2)  $2\text{Cr}(\text{NO}_3)_3 + \text{KBrO}_3 + 5\text{H}_2\text{O} \longrightarrow 2\text{H}_2\text{CrO}_4 + \text{KBr} + 6\text{HNO}_3$ .
- (3)  $3\text{HNO}_2 + \text{KBrO}_3 \longrightarrow 3\text{HNO}_3 + \text{KBr}$ .
- (4)  $3\text{H}_2\text{C}_2\text{O}_4 + \text{KBrO}_3 \longrightarrow 6\text{CO}_2 + 6\text{H}_2\text{O} + \text{KBr}$ .

Also, bromides in the presence of mercuric perchlorate are not decomposed by potassium permanganate in hot acid solutions; hydrogen peroxide does not liberate iodine from potassium iodide in the presence of sufficient mercuric mercury; and

the oxidation of oxalic acid in dilute sulphuric acid solution by potassium iodate is entirely prevented. These results are due to the formation of slightly dissociated mercuric halides.

H. E. C.

**Separation of Copper from Cadmium.** P. Wenger and N. Dürst. (*Helv. Chim. Acta*, 1923, 6, 642-647.)—The authors recommend Dallimore's method as being the quickest and most accurate. The solution is heated with excess of a 25 per cent. solution of hypophosphorous acid and boiled until the hydrogen evolution ceases. The precipitate of metallic copper is filtered off; the cadmium is recovered from the filtrate as sulphide and weighed as sulphate.

W. R. S.

**Separation of Copper from Selenium.** A. Angeletti. (*Gazz. Chim. Ital.*, 1923, 53, I., 339-341.)—In presence of selenium, copper may be precipitated by addition of "cupferron" to the solution rendered slightly acid with hydrochloric acid. The precipitate is filtered off, with the aid of gentle suction, and is washed first with cold water and then with 1 per cent. sodium carbonate solution. The precipitate and filter together are dried in a capacious platinum or quartz crucible at 100-105° C., the crucible being then heated over a gradually increasing flame, and finally over a Teclu burner, and the resulting cupric oxide weighed. The selenium is precipitated by passing a current of sulphur dioxide through the filtrate.

T. H. P.

**Estimation of Cobalt in the Presence of Nickel and other Metals of the Iron Group by Dinitroso-resorcinol.** W. R. Orndorff and M. L. Nichols. (*J. Amer. Chem. Soc.*, 1923, 45, 1439-1444.)—Cobalt is quantitatively precipitated by dinitroso-resorcinol when applied in the following manner. To 50 c.c. of a warm solution containing about 0.2 gm. of cobalt and 2 per cent. of hydrochloric acid are added 300 c.c. of a hot 0.1 per cent. aqueous solution of the reagent. The mixture is well stirred, and 10 c.c. of a 10 per cent. solution of sodium acetate are added; then the mixture is heated gently for about 15 minutes, stirred, and the precipitate allowed to settle, filtered off on a Gooch crucible, washed with 1 per cent. hydrochloric acid, dried at 125° C. and weighed. The composition of the dried precipitate is  $(C_6H_3N_2O_4)_2CO$ . The method is applicable without modification in the presence of manganese or zinc, but iron or copper, if present, must be removed. Large quantities of nickel interfere with the estimation, but in the presence of small amounts of this element the method is accurate if the supernatant liquor from the precipitate is filtered off and the precipitate digested for half an hour with warm 0.1 N hydrochloric acid, then filtered off and dried as before. The method is applicable to the estimation of cobalt in steel, but the iron must be first removed.

H. E. C.

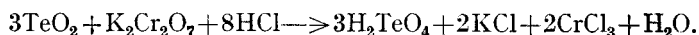
**Gravimetric Estimation of Iron and its Separation from Manganese.** B. Solaja. (*Chem. Zeit.*, 1923, 47, 557.)—Iron is precipitated quantitatively from ferric chloride solution by aminomercuric chloride ("infusible white precipitate"); the precipitate is dense, and easily filtered off and washed. The reaction quantitatively separates iron from manganese. The paper, which is a preliminary notice, contains no directions as to procedure.

W. R. S.

**Estimation of Praseodymium.** P. H. Brinton and H. A. Paget. (*J. Amer. Chem. Soc.*, 1923, 45, 1460–1465.)—Praseodymium may be estimated by ignition of the precipitated oxalate, but, contrary to statements in previous literature, the oxide obtained on ignition in air is  $\text{Pr}_6\text{O}_{11}$ , not  $\text{Pr}_4\text{O}_7$ . On reduction in a current of hydrogen  $\text{Pr}_2\text{O}_3$  is obtained. The ignition of the oxide may be carried out with the filter paper without any special precautions, but the oxide should be cooled in a desiccator containing potassium hydroxide, as it readily absorbs carbon dioxide. A volumetric method is satisfactory in the presence of only small quantities of cerium; the presence of lanthanum tends to high results. The sample is added to concentrated nitric acid (1 c.c. for each 0.1 gm. of oxide), and solution effected by heating, after which it is diluted to 100 c.c. and warmed to 60° to 70° C. Excess of 0.1 *N* solution of oxalic acid is now slowly added, and the mixture is stirred and kept warm for an hour. The excess of nitric acid is very slowly neutralised with ammonium hydroxide until the liquid is only faintly acid. The oxalate precipitate is filtered off and washed, and the excess of oxalic acid in the filtrate is titrated at 80° C. in the presence of dilute sulphuric acid as usual.

H. E. C.

**Volumetric Estimation of Tellurium by the Dichromate Method.** V. Lenher and H. F. Wakefield. (*J. Amer. Chem. Soc.*, 1923, 45, 1423–1425.)—The method depends upon the reaction—



As hydrochloric and telluric acids react, it is necessary to control the conditions carefully. A quantity of telluric oxide between 0.1 and 0.3 gm. is dissolved in 10 c.c. of hydrochloric acid, and the solution is diluted to 200 c.c. Standard solution of potassium dichromate (0.1 *N*) is added until there is an excess of 1 to 5 c.c., after which the solution is allowed to stand for half an hour. A measured volume of standard solution of ferrous sulphate is added, and the excess of ferrous salt is titrated with the dichromate as usual, with the use of an outside indicator. It is essential that the concentration of hydrochloric acid should be 2 per cent., so that, if *aqua regia* is used for the solution of the tellurium oxide, the nitric acid must be driven off by evaporation on the water bath with hydrochloric acid, and the concentration of the latter adjusted to 2 per cent.

H. E. C.

**Characteristic Reaction of Hydroxylamine.** W. N. Hirschel and J. A. Verhoeff. (*Chem. Weekblad*, 1923; 20, 319–320.)—Diacetylmonoxime reacts with hydroxylamine in dilute alkaline solution to form dimethylglyoxime, which forms with nickel the well-known red precipitate of nickel glyoxime. The reaction is capable of detecting 0.01 mgrm. of hydroxylamine sulphate in solution. With quantities over 1 mgrm. of hydroxylamine per c.c. the red precipitate is formed in the cold, but for the detection of smaller quantities it is necessary to heat the solution to the boiling point, then to cool it, and, if necessary, neutralise it with dilute nitric acid.

**Gravimetric Estimation of Nitric Acid.** H. Rupe and F. Becherer. (*Helv. Chim. Acta*, 1923, 6, 674–676.)—Catalytic reduction of  $\alpha$ -naphthonitril gives  $\alpha$ -dinaphthodimethylamine  $(C_{10}H_7.CH_2)_2NH$ . The nitrate of this base is quite insoluble in water (100 c.c. dissolved 0.00004 grm. at 24° C.). For the estimation of nitric acid or nitrate, the dilute, boiling solution containing a little free sulphuric acid is treated with a 10 per cent. solution of the base in 50 per cent. acetic acid. The precipitant is used after standing on the water bath till clear, because the acetate crystallises out in the cold at the above concentration. The nitrate solution remains clear at first, but, on cooling, the nitrate of the base deposits quantitatively in flaky crystals. The precipitate is collected on a Gooch crucible, washed with cold water, dried, and weighed. Sulphate and phosphate do not interfere, but the chloride is less soluble. The limit of sensitiveness for the detection of nitric acid by precipitation with the new reagent is given at about 1:1,000,000. The qualitative test is carried out in the cold; very dilute nitrate solutions give an opalescence, which on standing develops into a precipitate.

W. R. S.

**Analysis of Alkali Phosphotungstates.** R. Mellet. (*Helv. Chim. Acta*, 1923, 6, 656–661.)—The salt is carefully heated to constant weight in a platinum crucible; the loss represents water, as well as ammonia in the case of ammonium salts. The residue is fused with 4 to 5 parts of sodium potassium carbonate, the fused mass dissolved in water, and the solution treated with 8 to 10 parts of solid ammonium chloride, magnesia mixture, and excess of ammonia. The precipitate is dissolved on the filter in dilute hydrochloric acid and the filtrate re-precipitated as usual. The results for phosphoric acid ( $P_2O_5$ ) show a negative error of 1 to 1.5 per cent. Tungstic acid is determined by precipitation with a solution containing 20 grms. of quinoline and 50 grms. of glacial acetic acid per litre. The solution is treated gradually at 60° C. with a large excess of the above solution. The liquid is kept hot and stirred frequently until the precipitate settles well. The precipitate is filtered off, washed with diluted quinoline acetate solution, dried, and calcined very cautiously in a platinum crucible to constant weight; it should be moistened repeatedly with nitric acid for the purpose of re-oxidising any blue oxide formed in the ignition. The weighed residue is fused with sodium carbonate and the phosphoric acid estimated as before; tungstic acid is found by difference. A small quantity of reduced tungsten adheres to the crucible, which is treated with hot hydrochloric acid, dried, and weighed; the increase in weight is deducted from that of the mixed oxides, converted into tungsten trioxide, and added to the bulk of the tungsten trioxide found by difference. With ammonium phosphotungstates, cautious and moderate ignition gives a residue of phosphotungstic anhydride in which phosphoric acid ( $P_2O_5$ ) is estimated by fusion as above. The filtrate from the quinoline phosphotungstate contains the alkali metal; it is evaporated to dryness in a platinum basin, the residue dissolved in dilute hydrochloric acid, the solution filtered if necessary, evaporated, and the residue, after gentle ignition, weighed as alkali chloride. In cases where quinoline does not precipitate the

whole of the phosphoric with the tungstic acid, the direct estimation of the alkali is very difficult, and this is best taken by difference. The ammonia of ammonium phosphotungstate may be estimated by the usual distillation method.

W. R. S.

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## Apparatus.

**Hydrogenated Oil for Oil Baths.** G. R. Robertson. (*J. Ind. Eng. Chem.*, 1923, 15, 701.)—Hydrogenated sesame oil having an iodine value of 10 and a melting point of about 60° C., is much superior to mineral oils for use in oil-baths; it does not burn below 320° C., and, when cooled, solidifies to a brittle mass which does not adhere to either iron or glass. The hydrogenated oil has a somewhat unpleasant odour resembling that of refined cottonseed oil, but the odour becomes less after repeated heatings.

W. P. S.

**Estimation of Solids or Moisture by Means of a Gauze Dish.** A. Seidenberg. (*J. Ind. Eng. Chem.*, 1923, 15, 737-741.)—For the estimation of total solids in such liquids as milk, syrups, sugar solutions, etc., the use of a gauze dish is recommended. The dishes are made of fine mesh platinum gauze, 200 cm. square, which is corrugated into 31 to 33 lateral ridges and compressed in this way into an area of 8.5 by 5.5 cm. The dish rests on a platinum-gold stand. The liquid under examination is distributed, drop by drop, over the surface of the gauze, and a dish of the dimensions given will readily hold 5 c.c. of liquid without any falling through the meshes. Each drop spreads and forms a thin film on the gauze. For drying, the highest possible temperature should be used which does not decompose the solids left on the gauze. The dehydration is very rapid, and the results are more concordant than are those obtained when liquids are dried on powdered pumice stone, sand, etc.

W. P. S.

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## Reviews.

IDENTIFICATION OF PURE ORGANIC COMPOUNDS. Vol. IV. S. P. MULLIKIN. Pp. 238. New York: J. Wiley & Sons, Inc.; London: Chapman & Hall. Price \$6.00 net.

The reviewer has a difficult task in dealing with a book of this nature; his remarks must of necessity be either of a very general or of an ultra-specific character. It is impossible to do more than select a very few compounds at random for comment and hope that they will prove fairly representative of the whole.

Vol. IV. deals with the identification of organic compounds containing halogens, sulphur and nitrogen alone or together. The use of physical constants—melting point, boiling point and, sometimes, refractive index as a method of

preliminary classification is a highly commendable practice in this, as in the preceding volumes, and should be more widely adopted in chemical laboratories. In this connexion, however, it should be emphasised that rotatory power may be of quite restricted value; for instance, it is doubtful whether another preparation of *act*-amyl iodide (p. 99) would have  $[\alpha]_D 4.28^\circ$ —so much depends on the previous history of the specimen.

The most disadvantageous feature of the book is the comparative lack of particulars of easily prepared derivatives which would be useful as confirmatory evidence of identification.

It is always possible, of course, to think of compounds which are not sufficiently well known to be included in this book, but in view of their importance in gas warfare (which is naturally suggested by the number of irritant and lachrymatory compounds included in this volume), the omission of benzal bromide and phenylbromacetonitrile is surprising. Cyanogen bromide is wrongly indexed on p. 220; **51, 647** should read **51, 149**. The fact that chloropicrin explodes violently when heated slightly above its boiling point is possibly of sufficient interest to deserve inclusion in a future edition. The lachrymatory nature of allyl bromide is not recorded on p. 87, and, in addition, it is incorrectly indexed on p. 211; **4, 3020**, should read **4, 4535**. The method of indexing adopted does not appear the most convenient one available—the listing of the page as well as the index number of the compound would greatly facilitate search. We are all familiar with the vagaries of American spelling, but it is not obvious that the variation “alleaceous” for “alliaceous” on p. 18 has anything to recommend it, whilst it entirely obscures the derivation of the word (L. *allium* = garlic). It is pleasant to record, however, that we have been spared in this book the monstrosities “sulfur” and “quantitativ.” The publishers have played their part well—the appearance of the book and the accuracy of the script are quite up to Wiley’s usual high standard. JOSEPH KENYON.

**BIBLIOGRAPHY OF COLLOID CHEMISTRY.** By Prof. H. N. HOLMES, Chairman, Committee on the Chemistry of Colloids, Division of Chemistry and Chemical Technology (U.S.A.). Pp. 135. Preliminary Edition, issued in mimeographed form by the National Research Council, Washington, D.C. 1923. Price \$1.00.

Very great interest is being shown in America regarding colloid chemistry. Some time ago, Prof. Bancroft, at the request of the National Research Council, issued a paper giving two hundred problems for research in colloid chemistry. An international book on colloid chemistry is being edited in New York by Jerome Alexander. Now, appears the preliminary edition of an ambitious work which shall contain references to all the important papers ever published on colloid phenomena.

The introduction says: “This bibliography of about 1800 references is only moderately comprehensive. But the only way an effective and complete bibliography in this field can be built up is by first issuing in mimeographed form a tentative list of references. With this as a basis for criticism, other colloid workers

can suggest improvements and offer additional important references in the subjects on which they specialise. After a time, the author will edit these suggestions and arrange for formal publication."

One hundred and nine tentative headings are given, under which papers may be classified. Naturally, much overlapping occurs. Whilst there is a heading: "Industrial Applications" (p. 71), there are also headings: "Baking" (p. 11), "Flotation" (p. 56), "Colloidal Fuel" (p. 581), "Margarine" (p. 76), "Varnishes and Paints" (p. 130), etc.

It would seem a better plan to divide the bibliography into two main divisions: (I.) Theoretical Colloid Chemistry, (II.) Industrial Applications. The second division is easily sub-divided into the various industries and technical processes. Under Division I. will come, of course, many theoretical papers arising out of, or intimately reflected in, industrial subjects. Since, as Prof. Holmes says, "the ideal reference should have a sentence or two of comment indicating the scope or general nature of the paper mentioned," cross-references to sub-headings in Division II. could be made.

The choice of suitable and comprehensive sub-divisions for the theoretical papers is more difficult. As example: At present, there are the headings "Cataphoresis" (p. 22), "Electrical Endosmose" (p. 48), and "Electrical Properties" (p. 42). A better arrangement would seem to be the heading "Electrical Phenomena," with sub-headings dealing with the electric charge on colloids, electric migration (cataphoresis), electric endosmose, etc.

When it comes to examining the degree of completion of references to given subjects, there are naturally many omissions. Thus, under "Fogs, Mists and Smokes," we find four references only, whereas, in the Fourth Colloid Report of the British Association, Gibbs gives hundreds of references. On "Emulsions" there are ninety-three references. The reviewer has prepared a bibliography of over two hundred references in this field (*vide* ANALYST, March, 1923, p. 147). Under "Surface Films," p. 112, are only three references, and no mention of Ramsden's several papers in this connection.

Much more criticism could be made if space allowed, but it must be admitted that Prof. Holmes has done well in compiling, single-handed, the present preliminary volume. His task is not an easy one, and even to edit the final book, based on the criticisms, suggestions, and additions given by other workers, will not be simple. All colloid investigators will wish him every success in his scheme, which, when completed, should prove of inestimable value for research workers and students of a science so wide-embracing in its theory and industrial applications.

WILLIAM CLAYTON.

SULPHURIC ACID AND SULPHUR DIOXIDE RAW MATERIALS. By W. WYLD. (Lunge and Cummings), Vol. I. Pp. xiii.+558. London: Gurney & Jackson. Price 36s. net.

This is the first volume of a revised edition of Dr. Lunge's treatises on Acids and Alkalis, which is being edited by Dr. A. C. Cumming. As explained



in the editor's preface, it has been found desirable to recast the whole of Dr. Lunge's work and to deal with the various subjects in more direct relationship to the industries concerned, and judging from this first volume, this will greatly assist the reader, as matter related to one section of the industry is dealt with under one definite heading, thus a subject such as "calcination" of various materials is dealt with in one section of the book, and thus much time is saved when it is desired to refer to the known facts on a particular manufacturing operation.

There are a very few misprints which will, no doubt, be corrected in later editions; on page 60, line 9, "sulphuric" acid should be "nitric" acid; page 442, line 28, "nitre-oven" in place of "metre-oven"; and on page 499, in the table of production of pyrites cinders in U.K. for 1917, the figure of 609,000 tons is for both cuprous and non-cuprous.

In the section dealing with Flowers of Sulphur, it would have been useful if some information could have been given of precipitated sulphur prepared from polysulphides, such as is used in sheep-dips, etc. In the section dealing with pyrites statistics, pp. 78-87, discrepancies occur, but the writer found how difficult it was to obtain definite figures from the Board of Trade, as some copper pyrites would be classed as sulphur pyrites, and *vice versa*.

The book deals very thoroughly with the works tests both of the main constituents required and also of impurities; in dealing with arsenic in pyrites, it would have been useful if a quick distillation test as arsenic trichloride, such as is commonly used in copper works, had been included.

The section on Zinc Blende is good, and due notice is given to the difference between sulphate and sulphide in the calcined blende, but the author appears to be somewhat optimistic about the Ridge muffle calciner; such optimism has hardly been borne out by experience up to the present. In referring to spent oxide at the gas works, the author mentions revivification 30 or 40 times, whereas with modern practice some 6 to 8 times is more nearly correct. The ammonia oxidation section is a valuable contribution, but in any future edition due note should be taken of Mr. Imison's paper in 1922. Tests for cadmium and zinc in sulphuric acid would be useful in cases where zinc blende is the source of sulphur. In regard to electrical precipitation of dust and fume there is some experience in this country, and now that the Lodge-Cottrell interests are combined more rapid development may be expected; during the war precipitators were erected to deal with acid fume at Government factories, and to-day precipitators are working on both dust and fume in the United Kingdom.

The above criticisms are only of minor points; as a whole the book is very well arranged, and well written, and the subject matter is very thoroughly dealt with. Much that was obsolete in former editions of Lunge has been omitted, much of minor importance abbreviated, and in some cases only references given. The wheat of former editions has swelled to finer grain, and the chaff been winnowed out, while new matter has brought the volume right up to date. The book should be welcomed in every works dealing with the industries concerned, should be added to every technical chemist's library, and, in addition, should be welcomed.

by our training establishments as giving a real insight into practical works chemistry. The editor is to be congratulated on his selection of an author, and the author is to be thanked for having put his practical works' experience into the arrangement and writing of what will no doubt be soon looked upon as a technical classic.

H. J. BAILEY.

MONOGRAPHS ON INDUSTRIAL CHEMISTRY. Edited by SIR EDWARD THORPE.  
SYNTHETIC COLOURING MATTERS. VAT COLOURS. J. F. THORPE and C. K.  
INGOLD. Pp. xv.+491. London: Longmans, Green & Co. 1923.  
Price 23s. net.

The addition of a work on Vat Colours to the series of Monographs on Industrial Chemistry is very welcome. The production of fast shades in, rather than on, the fibre, by the use of a vat is one of the earliest examples of the dyeing art, but until recent years the method was applied only to a few natural dyestuffs. Of these, indigo has always been one of the most important, whilst its 6:6-dibromo-derivative forms the Tyrian Purple extracted by the ancients from shell-fish (*Murex* and *Purpura*).

The authors naturally start with these ancient dyes, and give an interesting and entertaining account of their history and use. A tale relating to the derivation of the name "Tyrian Purple," raises a serious question for youthful chemists. Julius Pollux states that a nymph named Tyros walked on the sea shore with her lover, and a dog followed them. The dog cracked a shell fish, with the result that his mouth was stained purple. Tyros admired the colour and desired a dress of the same shade, the result being that her lover discovered the method of obtaining Tyrian purple. The moral seems to be to take the dog with you when courting, and that a quiet stroll may be more conducive to scientific discovery than a motor bicycle provided with a pillion seat.

It is remarkable that, although indigo has been known since early times, and its composition ascertained early in the 19th century, its molecular weight was not definitely established until 1879, when the vapour density was determined by von Sommaruga. Von Baeyer's first syntheses were effected in the early eighties, and processes for dyeing with artificial indigo soon followed. The earlier syntheses were too expensive for the economic preparation of the dye, but Heumann's work on the production of indoxyl (1890) and indoxylcarboxylic acid (1893) from phenylglycine and its *o*-carboxylic acid respectively promised success, and by 1900 the Badische Anilin-und Soda Fabrik were manufacturing indigo on commercial lines.

The authors give an excellent account of the history of these discoveries, and of the developments in the synthesis of indigo which followed at a rapid rate, whilst substitution derivatives, some of which have attained technical importance, are also described. Then follow vat dyes, the molecules of which contain different heterocyclic rings from indigo itself, though the carbonyl groups and double

linkage which confer the capability of giving a vat dye on reduction are preserved. This chapter, in which dyes such as Thioindigo, Ciba Scarlet G and Alizarin Indigo are described, will prove of great interest to the synthetic organic chemist.

Passing on from indigo, its derivatives and analogues, we find that nearly half the book is occupied with an account of the anthracene vat dyes. It is but little over 20 years since René Bohn discovered Indanthrene, and the subsequent progress in utilising derivatives of anthraquinone as vat dyes is one of the striking developments of colour chemistry during this century.

Part II. begins with an account of anthraquinone and its substitution derivatives. The reviewer doubts whether 40 per cent. anthracene is often directly oxidised to anthraquinone on the commercial scale (p. 174), and believes that homologues, rather than pyridine itself, are generally used for the removal of impurities from anthracene.

The vat colours derived from anthraquinone fall into two classes, viz. chain and fused ring derivatives, and, regarding the former, it is a matter of interesting speculation as to how many chemists at the end of the 19th century could have predicted that substances having the constitutions of Algol Yellow R (1:5-dibenzoyl-diamino-anthraquinone) and its analogues would prove to be useful as vat dyes.

Amongst the fused ring derivatives of anthraquinone, indanthrene is naturally first mentioned, and a capital account is given of this compound, flavanthrene, benzanthrene and their derivatives. It is interesting to note that the important indanthrene is now produced by four firms in this country.

Part III. is very short, and deals with the compounds discovered by Kardos, indophenol and vat dyes of unknown constitution.

In Part IV. directions are given for the preparation of numerous intermediates and a certain number of typical vat dyes; the reviewer supposes this is for the benefit of students who intend to take up the chemistry of dyestuffs as a career.

The work concludes with indexes of authors and subjects; that of the latter appears to be very complete. With regard to the former, the desirability of giving the initials of Christian names of authors as well as their surnames might be considered. This has been done in the case of K. Meyer, but not in that of (A. G.) Perkin. It is not suggested that the practice should be extended to meet the cases of Aaron, Caesar, Moses, or Queen Elizabeth.

There are but few misprints in the text; some of the formulæ have suffered, however, as on pages 145, 153, 200, 201, 207, 226, 282.

The clear and logical arrangement of the subject matter and mode of expression are altogether commendable, and the reviewer would like to see books of this type in the hands of advanced organic students. Occasionally there is a tendency to despise a knowledge of a good many facts, and turning oneself into an animated dictionary is admittedly not the highest form of mental exercise. At the same time, facts are quite useful things on which to hang theories, and their study may, in some cases, be recommended.

J. T. HEWITT.

COLLECTED PAPERS FROM THE MEDICAL RESEARCH LABORATORIES OF PARK DAVIS & Co., DETROIT, MICH. Reprints Vol. 8. Nos. 194 to 266. Pp. 619. 1923.

This publication comprises reprints of 73 papers which have appeared in various American Scientific Journals during the years 1919 and 1920. The subjects dealt with are very varied; the large majority may be conveniently grouped under the following headings:—Bacteriology (and Serum Therapy), Parasitology, Pharmacology, Botany, and Nutrition.

The papers on bacteriological subjects include studies on the haemo-culture of the meningococcus; on the influenza bacillus, its toxin and antitoxin; the requirements of the diphtheria bacillus for luxuriant growth and toxin production; on the preparation of polyvalent anti-pneumococcic serum; several interesting papers by Hoskins on veterinary bacteriological subjects; contagious abortion of cattle, haemorrhagic septicaemia of cattle and sheep, on the bacillus bronchisepticus and its infections of the white rat and rabbit, and on anaplylaxis in veterinary practice.

Ten interesting studies in parasitology are contributed by Maurice Hall, entitled "Studies in Anthelmintics," dealing with the relative efficacy of oil of chenopodium, santonin, carbon disulphide and chloroform with different infections of different hosts.

The papers on pharmacological subjects include the pharmacology of digitalis, ergot, Cannabis indica and various gland extracts, and discussions of the value of pharmacological assaying. Several papers are contributed on the interesting drug trichlor-tertiary-butyl-alcohol (chloretone) and its uses as a general and local anaesthetic and as a preservative for biological specimens. The study of its distribution in the organs of the animal body after absorption is perhaps particularly interesting in actually demonstrating its selective affinity for the cells of the nervous system.

Several papers on botanical subjects are contributed, and several papers on nutrition with special reference to the vitamins.

The publication is well compiled and well printed, and should afford interesting reading to those who are engaged in any of the subjects studied and discussed.

D. R. WOOD.

## Publications Received.

- LABORATORY MANUAL OF PHYSICAL CHEMISTRY. By A. W. DAVISON and H. S. VAN KLOOSTER. Pp. 182. London: Chapman & Hall. Price 10s. net.
- THE SEPARATION AND USES OF CACAO SHELL. By A. W. KNAPP. Pp. 8. Publication Dept., Bournville Works. Price 6d.
- THE APPLICATION OF SCIENCE TO CACAO PRODUCTION. By A. W. KNAPP. Pp. 8. Publication Dept., Bournville Works. Price 6d.
- ANALES DE LA DIRECCION DE SANIDAD NACIONAL for 1922. Caracas, Venezuela.