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A MANUAL
OF
Practical Chemistry
FOR
PUBLIC HEALTH STUDENTS
Especially arranged for those Studying for the D.P.H.

BY

A. W. STEWART, D.Sc.
Assistant Demonstrator of Chemistry at The Royal Institute of Public Health

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FOREWORD.

This book has been designed to supply in a concise manner the necessary practical chemical exercises for D.P.H. students. It has been the author's aim to produce a cheap book which shall also be as complete as is required by students for the D.P.H., for whom it has been especially written. The student should bear in mind that it is only by continual practice that familiarity with the methods and accuracy are obtained. The author's thanks are due to E. Garratt, M.Sc., A.I.C., for valuable help and suggestions. I have also to thank Messrs. Baird and Tatlock, and Messrs. A. Gallenkamp for the loan of blocks.

London, A. W. S.
October, 1913.

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PREFACE.

SOME time ago I had occasion to draw up a report and discussion on the methods in use in different countries for the analysis of certain foods. A book such as you have written necessarily requires that select methods should be given which are rapid, practical and exact. My experience enables me to say that you have succeeded in your object. The theoretical data given in the first part of your book on acidimetry and alkalimetry will recall to students their previous knowledge, and remind them that theoretical chemistry is an indispensable basis of applied chemistry. The list of Local Government Reports you give will prove useful to those desirous of acquiring a deeper knowledge of certain subjects, and the list of examination questions will enable the student to form a good idea of the knowledge required. Chemistry is a fundamental science; more than any other branch, hygiene borrows largely and profitably from chemistry. The publication of your manual constitutes a further proof. Both medical men and others interested in public health may derive instruction and profit from its perusal. On account of the excellent way in which your manual is written and arranged, this book should find its place in the laboratory.

DR. A. J. J. VANDEVELDE.

Director of the Chemical and Bacteriological Laboratories;
Professor at the Institute of Fermentation.

Ghent, September 25, 1913.

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ATOMIC WEIGHTS.

Element.	Symbol.	Atomic wt.
Arsenic	As	75
Barium	Ba	136·4
Boron	B	11
Bromine	Br	80
Calcium	Ca	40
Carbon	C	12
Chlorine	Cl	35·5
Copper	Cu	63
Hydrogen	H	1
Iodine	I	127
Iron	Fe	56
Lead	Pb	207
Manganese	Mn	55
Magnesium	Mg	24
Nitrogen	N	14
Oxygen	O	16
Phosphorus	P	31
Potassium	K	39
Silver	Ag	108
Sodium	Na	23
Strontium	Sr	87·3
Sulphur	S	32
Zinc	Zn	65

ERRATA.

Page	9	line	3,	*for*	.9953	*read*	·0053
,,	9	,,	3,	,,	N_2CO_3	,,	Na_2CO_3
,,	9	,,	10,	,,	$_2MnSO_4$	,,	$2MnSO_4$
,,	10	,,	1,	,,	KMnO	,,	$KMnO_4$
,,	14	,,	10,	,,	5·6642	,,	5·6442
,,	15	,,	18,	,,	water	,,	air
,,	26	,,	6,	,,	2,714	,,	2·714
,,	39	,,	29,	,,	0·074	,,	0·084
,,	50	,,	15,	,,	4 per cent.	,,	·4 per cent.

A Manual of Practical Chemistry for Public Health Students.

PART I.—ACIDIMETRY AND ALKALIMETRY.

In public health laboratory work two kinds of solutions are usually employed, viz.:—

(1) **A standard solution,** one which contains a known weight of substance dissolved in a known volume of water. A very convenient standard is to make the solutions so that 1 c.c. contains 1 milligramme (·001 grm.) of the active substance.

(2) **A normal solution,** one which contains the equivalent weight in grammes of the active substance dissolved in 1,000 c.c. water.[1] A normal solution of caustic soda is usually written as N. NaOH, and a decinormal solution as $\frac{N}{10}$ NaOH, the latter containing one-tenth of the former. In food analysis decinormal solutions are usually employed. The equivalent or combining weight is the weight equivalent to, or which will combine with, 1 grm. H_2. The equivalent weight $= \frac{\text{atomic wt.}}{\text{valency}}$; thus equivalent weight of oxygen $= \frac{16}{2} = 8$.

Note.—All normal, decinormal, centinormal, &c., solutions are equivalent, *i.e.*, 20 c.c. of $\frac{N}{10}$ H_2SO_4 would exactly neutralize 20 c.c. of $\frac{N}{10}$ KOH or $\frac{N}{10}$ Na_2CO_3, &c. Similarly, 20 c.c. of $\frac{N}{100}$ NaOH would exactly be neutralized by 20 c.c. $\frac{N}{100}$ acetic or any other acid; for the same reason 1 c.c. of normal H_2SO_4 would exactly neutralize 10 c.c. of a decinormal solution of NaOH.

In order to find out the equivalent weight we may consider the substance to be prepared as being:—

(1) **A base,** *e.g.*, KOH, NaOH, $Ba(OH)_2$, $Sr(OH)_2$. If there is only one hydroxyl group – (OH), then the formula

---

[1] It is to be understood that "water" means "distilled water."

weight, *i.e.*, the sum of the atomic weights, represents the equivalent weight. Thus $NaOH = 23 + 16 + 1 = 40$; hence 40 grm. of caustic soda dissolved in 1,000 c.c. water would constitute a normal solution, and 4 grm. dissolved in a similar volume of water would be decinormal. If the substance contains two hydroxyl groups then the formula weight is divided by 2; thus a normal solution of baryta $Ba(OH)_2$ would contain $136{\cdot}4 + 32 + 2 = \frac{170{\cdot}4}{2} = 80{\cdot}2$ grm. per litre. If there are three hydroxyl groups then the total weights would be divided by 3, and so on.

(2) **An acid.** *(A) Organic.—e.g.*, acetic acid $CH_3 - COOH$, oxalic acid $COOH - COOH + 2H_2O$, citric acid $C_3H_4OH - (COOH)_3 + H_2O$. If there is only one carboxyl group $- (COOH)$, then the formula weight gives the equivalent weight. Thus, acetic acid $CH_3 - COOH = 12 + 3 + 12 + 16 + 16 + 1 = 60$ grm. dissolved in 1,000 c.c. of water would constitute a normal solution, and 6 grm. a decinormal one. If there are two or three carboxyl groups then the formula is divided by 2 or 3 respectively, thus:—

Oxalic acid $COOH - COOH + 2H_2O = \frac{126}{2} = 63$ grm. per litre = N. solution.

Citric acid $C_3H_4OH\ (COOH)_3 + H_2O = \frac{210}{3} = 70$ grm. per litre = N. solution.

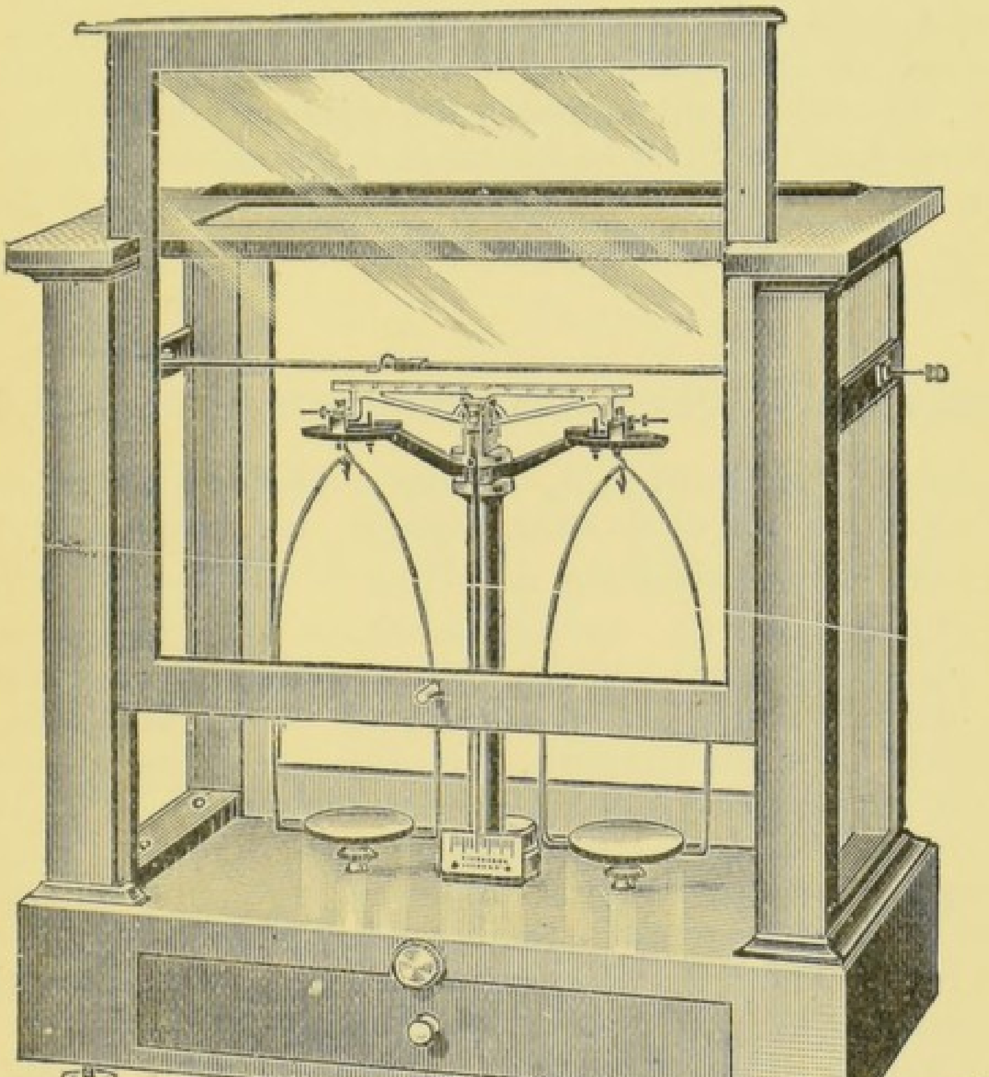
(B) Inorganic, e.g., HCl, H_2SO_4. Here the basicity of the acid must be considered, *i.e.*, the number of H atoms that can be replaced. If the acid is monobasic, such as HCl, HNO_3, then the formula weight would constitute the equivalent weight. Thus $HCl = 1 + 35{\cdot}5 = 36{\cdot}5$ grm. in 1,000 c.c. would be a normal solution. If the acid is dibasic, then the sum of the atomic weights would be divided by 2, as in $H_2SO_4 = 2 + 32 + 64 = \frac{98}{2} = 49$ grm. per litre for a normal solution.

(3) **A salt,** *e.g.*, $NaCl$, Na_2CO_3. In the case of these we consider the basicity of the acid from which they are derived. Thus Na replaces H of HCl, which is monobasic and the total weights of $NaCl = 23 + 35{\cdot}5 = 58{\cdot}5$ grm. would constitute a normal solution. Similarly, Na_2 replaces 2H of the theoretical carbonic acid H_2CO_3 and a normal solution of sodium carbonate would contain $\frac{46 + 12 + 48}{2} = 53$ grm. of $NaCO_3$ in 1 litre.

Other substances, such as permanganate of potash, arsenious acid, &c., are described under their exercises (see pp. 9 and 12).

Preparation of Decinormal Sodium Carbonate Solution, $\frac{N}{10}$ Na_2CO_3.

From the formula Na_2CO_3 it will be seen that $46+12+48 = \frac{106}{2} = 53$ grm. per litre would constitute a normal solution, the total weights being divided by 2 as the salt in question is derived from a dibasic acid. A decinormal solution would require 5·3 grm. dissolved in 1,000 c.c. water. As the sodium carbonate employed in laboratories usually contains water, ammonium salts and



THE BALANCE.

When using the balance the scales must be brought to rest upon their supports when putting on or taking off weights or substances. The front window of the balance should be closed when making the final weighing. No chemicals should ever be placed on the pans. Substances should be weighed on a tared watch-glass.

other impurities, it is preferable, in order to obtain it in a pure state, to heat the bicarbonate. For this purpose about 15–20 grm. of the bicarbonate are heated in a porcelain dish and occasionally stirred with a glass rod. Sodium carbonate is produced according to the equation: $2NaHCO_3 = Na_2CO_3 + CO_2 + H_2O$, whilst water and ammonium salts would be volatilized. The residue is allowed to cool in a desiccator and when cold 5·3 grm. are weighed out accurately and dissolved in 1,000 c.c. distilled water. In making up solutions the substance is first placed in about $\frac{3}{4}$ of the bulk to be made up, and when dissolved more water is added up to the graduation on the flask: 1 c.c. of $\frac{N}{10}$ Na_2CO_3 therefore contains ·0053 grm. Na_2CO_3.

Preparation of Decinormal Sulphuric Acid, $\frac{N}{10}$ H_2SO_4.

Sulphuric acid—H_2SO_4—being a dibasic acid, in order to determine its equivalent weight, its formula weight will have to be divided by 2. Thus $\frac{2 + 32 + 64}{2} = 49$ grm. of H_2SO_4 in 1,000 c.c. would constitute a normal solution and 4·9 a decinormal solution. H_2SO_4 cannot be accurately weighed out owing to its affinity for water and it is not always of a definite strength. In cases where the substance cannot be weighed out accurately, such as H_2SO_4, HCl, NaOH, KOH, &c., the solutions are made up slightly stronger than the amount required, and the amount of water to be added to make them exactly N. or $\frac{N}{10}$, as the case may be, is calculated by titration. The specific gravity of concentrated H_2SO_4 is approximately 1·84, *i.e.*, 1 c.c. weighs 1·84 grm. Hence to obtain 4·9 grm. we should require approximately 2·6 c.c. In practice take 3 c.c. of concentrated H_2SO_4, add it to a graduated litre flask about half full with water and make it up to the mark. Shake well and pipette 20 c.c. into a small flask, add about 20 c.c. water, a few drops of methyl orange, and then run in from a burette $\frac{N}{10}$ Na_2CO_3 until the reddish colour changes to straw yellow.

Indicators.—*Methyl Orange* gives a reddish colour with acids and a straw yellow with alkalies. It is unreliable with organic acids but quite reliable with carbonic acid. *Phenolphthalein* is colourless with acids and red with alkalies. It is affected by CO_2 and solutions of ammonia.

All pipettes and burettes before being used should be rinsed out with a little of the solution that is going to be placed in them. A little vaseline put on the burette taps will enable them to turn easily.

Two titrations should be made and the mean taken. Supposing it was 24·6 c.c.: this means that to every 20 c.c. of the acid liquid 4·6 c.c. of water must be added to make it decinormal. Measure in a cylinder the volume of the remaining acid and calculate how much water to add. Example: Volume of acid 900 c.c. Amount of H_2O to be added will therefore be $\frac{4·6 \times 900}{20} = 207$ c.c. Measure out this quantity, pour in about 180 c.c., keeping the remainder in reserve. If all the water is added at once the new solution on titration will probably be found too weak. The diluted acid liquid is now titrated as described above in the first operation. This time 20 c.c. will probably require 20·3 c.c. $\frac{N}{10}$ Na_2CO_3. The reserve water is then added by 5 or 10 c.c. portions until 20 c.c. exactly require 20 c.c. of $\frac{N}{10}$ Na_2CO_3. Such a solution would then be decinormal.

Preparation of Decinormal Caustic Soda, $\frac{N}{10}$ NaOH.

From the formula NaOH we see that there is only one hydroxyl group (OH), consequently 23 + 16 + 1 = 40 grm. of caustic soda dissolved in 1,000 c.c. water would constitute a normal solution, and 4 grm. would be $\frac{N}{10}$.

As caustic soda usually contains traces of impurities, such as carbonate and cyanide, and as it cannot be accurately weighed out on account of its being hydroscopic, we have to make the solution slightly stronger than decinormal, and by titration determine the amount of water to be added in order to make it exact. For this purpose approximately 4·5 grm. of NaOH are weighed out, dissolved in water, and finally made up to 1,000 c.c. Pipette out 20 c.c., add a little water and a few drops of phenolphthalein. Run in from a burette $\frac{N}{10}$ H_2SO_4 until the pink colour is just discharged. The process is then continued precisely in the same manner as described under the preparation of $\frac{N}{10}$ H_2SO_4.

Estimation of Acetic Acid in Vinegar.

Pipette into a 100 c.c. graduated flask 10 c.c. of vinegar. Make up to the mark with water and shake well.

Pipette out 10 c.c. into a flask, add about 50 c.c. water, a few drops of phenolphthalein and run in from a burette $\frac{N}{10}$ soda till a permanent pink colour appears.

Example : Required 5·7 c.c.

Now 1 c.c. $\frac{N}{10}$ NaOH = 1 c.c. $\frac{N}{10}$ acetic acid and a normal solution of acetic acid ($CH_3 - COOH$) would contain $12 + 3 + 12 + 32 + 1 = 60$ grm. per litre ; $\frac{N}{10}$ acetic acid = 6 grm. per litre, *i.e.*, 1 c.c. acetic acid = ·006 grm.

Amount of acetic acid in 10 c.c. diluted vinegar = ·006 × 5·7 grm.

Amount of acetic acid in 100 c.c. diluted vinegar = ·006 × 5·7 × 10.

100 c.c. diluted vinegar = 10 c.c. original vinegar.

100 c.c. original vinegar = ·006 × 5·7 × 10 × 10 = 3·42 per cent.

Test for Acetic Acid.—Boil some vinegar with sodium carbonate solution ; filter. To a portion of the filtrate add a few drops of perchloride of iron. A red colour is obtained.

A malt vinegar generally contains about 5 per cent. acetic acid.

Wood vinegar and other artificial samples contain about 3 per cent.

Vinegar should be the liquid derived wholly from alcoholic and acetous fermentations. It should contain not less than 4 grm. $CH_3 - COOH$ in 100 c.c. No sulphuric acid or other mineral acid, neither lead, copper or any foreign substance or colouring matter except caramel, should be present.

Artificial Vinegar is any vinegar or substitute for vinegar containing, or derived from, any preparation containing any added acetic acid, which is not wholly the product of alcoholic and subsequent acetous fermentations.

Tests for Mineral Acids.—To 5 c.c. of vinegar in a test tube add a few c.c. of aqueous solution of methyl violet. If mineral acids are present a green colour is produced. Free sulphuric acid may be detected also by moistening a piece of filter paper in the sample. Dry in

the water oven. If free sulphuric acid is present the paper is more or less charred.

Estimation of Citric Acid in Lime-juice.

This is done in the same manner as the acetic acid in vinegar.

Example: Required 9·6 c.c.

1 c.c. $\frac{N}{10}$ NaOH = 1 c.c. $\frac{N}{10}$ citric acid.

A normal solution of citric acid $C_3H_4OH(COOH)_3H_2O$ contains $\frac{210}{3} = 70$ grm. per litre.

We divide by 3 because there are 3 carboxyl groups. Hence 1 c.c. $\frac{N}{10}$ citric acid = ·007 grm.

Amount of citric acid in 10 c.c. diluted lime-juice = ·007 × 9·6. Amount of citric acid in 100 c.c. diluted lime-juice = ·007 × 9·6 × 10.

100 c.c. diluted lime-juice = 10 original; 100 original lime-juice = ·007 × 9·6 × 10 × 10 = 6·72 per cent.

Test for Citric Acid.—Boil some lime-juice with solution of sodium carbonate. Filter. To the filtrate add ammonium chloride, ammonia, and then calcium chloride. Boil. A white precipitate of calcium citrate is obtained.

Tartaric Acid gives a precipitate with the above reagents in the cold, whereas citric acid is only precipitated on boiling. Potassium chloride added to tartaric acid precipitates acid potassium tartrate (cream of tartar), but citric acid is not precipitated. A good lime-juice should contain about 7 per cent. citric acid.

Determination of the Strength of Lime-water, $Ca(OH)_2$.

Pipette into a small flask 25 c.c. lime-water, add a few drops of phenolphthalein and run in from a burette $\frac{N}{10}$ H_2SO_4 till the pink colour is just discharged.

Example: 12 c.c. acid.

$$1 \text{ c.c. } \frac{N}{10} H_2SO_4 = 1 \text{ c.c. } \frac{N}{10} Ca(OH)_2$$

A normal solution of $Ca(OH)_2$ would contain $\frac{40 + 34}{2} = 37$ grm. per litre.

We divide by 2 because there are 2 hydroxyl groups.

An $\frac{N}{10}$ solution therefore contains 3·7 grm. in 1,000 c.c., and 1 c.c. $\frac{N}{10}$ $Ca(OH)_2$ = ·0037 grm. Hence ·0037 × 12

= ·0444 is amount $Ca(OH)_2$ in 25 c.c. lime-water, and $\frac{\cdot 0444 \times 100}{25} = \cdot 177$ in 100 c.c.

Liquor calcis B.P. contains ·153 per cent. $Ca(OH)_2$.

Estimation of Free and Combined Alkali in Soda Ash.

The free alkali is NaOH and the combined Na_2CO_3.

Weigh out 1 grm. of soda ash, dissolve in water and make up to 100 c.c. in a graduated flask. Shake well and pipette 10 c.c. into a small flask, add a few drops of phenolphthalein and run in from a burette $\frac{N}{10}$ H_2SO_4 till the pink colour is just discharged. Read the burette. Into the same liquid add a few drops of methyl orange and continue the addition of the acid till the yellow colour has passed to red. Read the burette.

In the first titration we obtain all the free alkali and one half of the combined alkali, as will be seen from the equations :—

$$2\,NaOH + H_2SO_4 = Na_2SO_4 + 2H_2O$$
$$Na_2CO_3 + H_2SO_4 = NaHCO_3 + NaHSO_4$$

The second titration gives the remaining half of the combined alkali, viz. :—

$$NaHCO_3 + H_2SO_4 = NaHSO_4 + H_2O + CO_2$$

Methyl orange is used here as the carbonic acid liberated acts on phenolphthalein.

Example : 1st titration, 12 c.c. using phenolphthalein.
2nd ,, 7 ,, ,, methyl orange.

Total 19 ,, $\frac{N}{10}$ H_2SO_4

Double 2nd titration = 14 c.c. = whole of combined alkali (Na_2CO_3)
Difference = 5 c.c. = whole of free alkali (NaOH)

1 c.c. $\frac{N}{10}$ H_2SO_4 = ·004 grm. NaOH
1 ,, ,, = ·0053 ,, Na_2CO_3

Hence ·004 × 5 = amount of NaOH in 10 c.c. of solution and ·004 × 5 × 10 = amount of NaOH in 100 c.c. of solution; but 100 c.c. solution contains 1 grm. soda ash. ∴ 100 soda ash contains ·004 × 5 × 10 × 100 = 20 per cent. NaOH.

Similarly: ·0053 × 14 = amount of Na_2CO_3 in 10 c.c.

of solution and ·0053 × 14 × 10 = amount of Na_2CO_3 in 100 c.c. of solution, whence 100 grm. soda ash contains ·9953 × 14 × 10 × 100 = 74·2 per cent. N_2CO_3.

Preparation of Decinormal Permanganate of Potash, $\frac{N}{10}$ $KMnO_4$.

Here we require to find out how much permanganate corresponds to the equivalent weight of oxygen (= 8), which is the active principle in the substance.

This is got from the following equation :—

$$
\begin{aligned}
& 2KMnO_4 + 3H_2SO_4 = 5O + 3H_2O + K_2SO_4 + {}_2MnSO_4 \\
\textit{i.e.}, & \ 2 \times 158 = 5 \times 16 \text{ (oxygen)} \\
& 316 = 80 \\
& 31{\cdot}6 = 8 = \text{equivalent weight of oxygen} \\
& \qquad = \text{N. solution} \\
& 3{\cdot}16 = {\cdot}8 = \tfrac{N}{10} \text{ solution}
\end{aligned}
$$

Weigh out accurately 3·16 grm. of $KMnO_4$, dissolve in water and make up to 1,000 c.c. in a graduated flask.

Estimation of Ferrous Iron in Ferrous Sulphate (Sulphate of Iron).

This is effected by means of the permanganate solution.

Weigh out 5 grm. sulphate of iron ; dissolve in water and make up to 250 c.c. in a graduated flask. Pipette 25 c.c. into a flask, add 20 c.c. H_2SO_4 (1 in 4) and then run in $\frac{N}{10}$ $KMnO_4$ until a permanent pink colour is obtained. The oxygen from the $KMnO_4$ oxidizes the ferrous salt to the ferric state. The following is the equation :—

$$
\overset{\textit{[Ferrous sulphate]}}{10FeSO_4} + 2\,KMnO_4 + 8\,H_2SO_4 = 5\,\overset{\textit{[Ferric sulphate]}}{Fe_2(SO_4)_3} + 2\,MnSO_4 + 8\,H_2O + K_2SO_4
$$

The oxygen is more in evidence in the following equation :—

$$
\underset{112}{\overset{\textit{[Ferrous oxide]}}{2\,FeO}} + \underset{16}{O} = \overset{\textit{[Ferric oxide]}}{Fe_2O_3}
$$

From this we see that 2 × 56 = 112 Fe correspond to 16 oxygen.

56 Fe correspond to 8 oxygen (equivalent weight).

Hence an $\frac{N}{10}$ Fe = 5·6 grm. per litre, *i.e.*, 1 c.c. = ·0056 grm. Fe.

Example : 17·8 c.c. $\frac{N}{10}$ KMnO.

17·8 × ·0056 = amount of Fe in 25 c.c. of the solution.

17·8 × ·0056 × 10 = amount of Fe in 250 c.c. of the solution, but 250 c.c. solution contain 5 grm. iron sulphate.

∴ 100 grm. contain 17·8 × ·0056 × 10 × $\frac{100}{5}$ = 19·93 per cent. Fe.

Theoretically ferrous sulphate contains 20·1 per cent. Fe since $FeSO_4 + 7\,H_2O$ contains 1 Fe, *i.e.*—

278 contains 56 Fe

$$100 \text{ contains } \frac{56 \times 100}{278} = 20{\cdot}1 \text{ per cent. Fe.}$$

Estimation of Available Oxygen in an Unknown Solution of Permanganate.

This is conveniently done by using decinormal oxalic acid. Oxalic acid crystallizes with two molecules of water and has the formula : $(COOH)_2 . 2H_2O$. A normal solution would contain $\frac{126}{2}$ = 63 grm. per litre.

Pipette into a small flask 20 c.c. of $\frac{N}{10}$ oxalic acid, add 10 c.c. H_2SO_4 (1 in 4), warm to about 60—70° C., and from a burette run in the solution of $KMnO_4$ until a permanent pink colour is obtained. The colour ought not to be discharged on warming to 70°C.

The equations are :—

$$5\,C_2H_2O_4 + 2\,KMnO_4 + 3\,H_2SO_4$$
$$= 2\,MnSO_4 + K_2SO_4 + 8\,H_2O + 10\,CO_2.$$

Example : 25 c.c. $KMnO_4$ solution.

Under the preparation of $\frac{N}{10}$ $KMnO_4$ we saw that 1 litre of this solution was equivalent to ·8 grm. of oxygen, *i.e.*, 1 c.c. = ·0008.

As all decinormal solutions are equivalent it follows that 1 c.c. $\frac{N}{10}$ oxalic acid = ·0008 grm. oxygen, 20 c.c. oxalic acid = ·0008 × 20 = ·016, whence 25 c.c. $KMnO_4$ solution = ·016 oxygen and 100 c.c. $KMnO_4$ solution = ·016 × 4 = ·064 per cent. available oxygen.

Bleaching Powder.

Bleaching powder, bleach, chlorinated lime, or chloride of lime, is a complex mixture, composed chiefly of calcium chlorohypochlorite, slaked lime and moisture. It is best represented by the formula :—

$$CaOCl_2 .\quad Ca(OH)_2 .\quad H_2O$$

When treated with water it breaks down according to the equation :—

$$2\ \overset{\textit{[Chlorohypochlorite]}}{CaOCl_2} + Ca(OH)_2 + H_2O$$

$$= \overset{\textit{[Hypochlorite]}}{Ca(OCl)_2} + CaCl_2 + Ca(OH)_2 + H_2O$$

The hypochlorite so formed splits up in the presence of organic substances into nascent oxygen and calcium chloride :—

$$Ca(OCl)_2 = CaCl_2 + O_2$$

It must be borne in mind that bleaching powder in its industrial application of bleaching, deodorizing or disinfecting does not act by its chlorine but by its oxygen. The action is one of oxidation, not "chlorination."

$$O_2 \text{ is equivalent to } 2Cl_2$$

Bleaching powder slowly decomposes in contact with air and moisture, with formation of hypochlorous acid, which is very unstable.

$$Ca(OCl)_2 + CO_2 + H_2O = CaCO_3 + 2\ \overset{\textit{[Hypochlorous acid]}}{HOCl}$$

In water sterilization the latter is liberated from solutions of hypochlorites due to the CO_2 in the water or in the air. The HOCl is the active oxidizing agent of chloride of lime. It gives up nascent oxygen with readiness according to the equation :—

$$2HOCl = 2HCl + O_2$$

The total amount of available chlorine has been found to be diminished in very hot seasons by 1 per cent. per month and in winter by 1 to 3 per cent. per month.

When treated with a mineral acid bleaching powder gives off chlorine, a reaction which is made use of for its detection :—

$$Ca(OCl)_2 + 4\ HCl = 2\ Cl_2 + CaCl_2 + 2\ H_2O$$

Estimation of Available Chlorine in Bleaching Powder.

Weigh out 5 grm. of the powder. Place in a mortar and triturate with water. Decant off supernatant fluid into a 500 c.c. graduated flask. Add more water and continue until all the powder has thus been transferred to the flask. Make up to the mark with water. This turbid liquid is then well shaken and 25 c.c. pipetted out into a small flask. Decinormal arsenious acid is now run into the solution from a burette until a drop of the liquid placed on a starch and potassium iodide paper ceases to give a blue spot.

The equations representing the reactions with the arsenious acid are:—

(1) $As_2O_3 + Ca(OCl)_2 = As_2O_5 + CaCl_2$
(2) Or $As_2O_3 + O_2 = As_2O_5$
(3) With the starch and KI paper :—

$$Ca(OCl)_2 + 4KI + 2H_2O = 2I_2 + 4KOH + CaCl_2$$

Preparation of Decinormal Arsenious Acid, $\frac{N}{10}$ As_2O_3.

From the equation $As_2O_3 + O_2 = As_2O_5$ we see that 198 As_2O_3 are equivalent to 32 of oxygen, or 49·5 As_2O_3 are equivalent to 8 of oxygen (equiv. wt.). Hence 4·95 grm. As_2O_3 would make an $\frac{N}{10}$ solution.

As arsenious acid is only slightly soluble in water, weigh out about 20 grm. Na_2CO_3, add it to the 4·95 grm. As_2O_3 and boil in a beaker with 300 c.c. water. When cold transfer to a graduated 1,000 c.c. flask, wash out the beaker with a little water, and finally make up to bulk. The sodium carbonate forms with the As_2O_3 a soluble salt (arsenite of soda).

Example : Required 20 c.c. $\frac{N}{10}$ As_2O_3
1 c.c. $\frac{N}{10}$ As_2O_3 = 1 c.c. $\frac{N}{10}$ Cl
,, = ·00355 grm. Cl
20 ,, ,, = ·00355 × 20 Cl

In 25 c.c. bleaching solution there are ·00355 × 20 grm. Cl ; in 500 c.c. bleaching solution there are ·00355 × 20 × 20 grm. Cl, but this is in 5 grm. of the powder ; therefore in 100 grm. of the powder there is ·00355 × 20 × 20 × 20 = 28·4 per cent. available chlorine.

A good sample of bleach should contain 35 per cent. available Cl.

PART II.—AIR ANALYSIS.

The average composition of air is :—

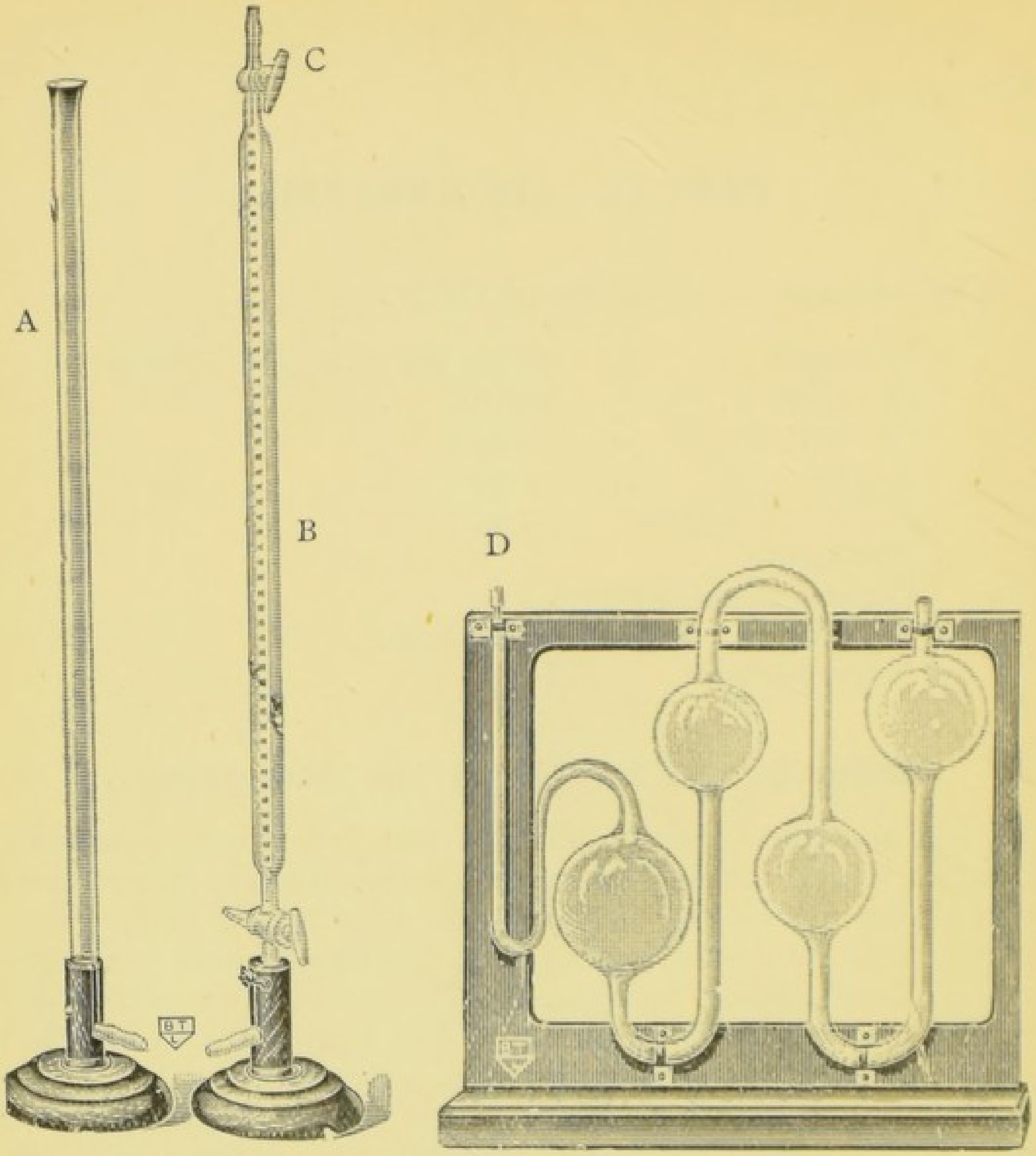
Nitrogen... ...	76·95	per cent.	by volume.
Oxygen	20·66	,,	,,
Carbonic acid ...	0·034	,,	,,
Argon	0·94	,,	,,
Aqueous vapour	1·40	,,	,,
Ammonia			
Ozone, nitric acid	Traces.		
Hydrogen, methane			
Rare gases			

Estimation of Oxygen in the Atmosphere.

This is conveniently carried out by using Hempel's burette, which consists of several bulbs (usually two or four) containing a solution of pyrogallic acid and KOH. This is prepared by dissolving 15 grm. pyrogallic acid in a little water, then adding 50 grm. KOH dissolved in water and finally making up to 1,000 c.c. The bulbs are then filled with this solution. (See figure, p. 14.)

The tubes A and B are filled about three-quarters full with water. 50 c.c. of air are taken into the tube B by lowering A and opening the tap C. Before shutting the tap the tubes must be put under the same pressure. When C is shut connect up with the apparatus by means of a small piece of rubber tubing. On a piece of paper placed at D mark where the brown liquid is. Open C and force the 50 c.c. of air into the apparatus by raising A. Allow to stand for fifteen minutes. The oxygen is absorbed. After this period open the tap C and manipulate A so as to bring the brown fluid back to the mark previously made on D. Close the tap C, place the two tubes under the same pressure and note the burette reading on B.

Example : 39·6 c.c. Hence $50 - 39{\cdot}6 = 10{\cdot}4$ c.c. oxygen are in 50 c.c. air, *i.e.*, 20·8 per cent.



Hempel's Gas Burette and Absorption Apparatus.

Estimation of Carbonic Acid in Air. Pettenkofer's Method.

Measure the volume of a Pettenkofer bottle: they have hollow stoppers and a capacity of about 3 litres. Add 50 c.c. of baryta water (approximately 4·5 grm. per litre). Allow the bottle to lie on its side for $\frac{1}{2}$ to $\frac{3}{4}$ of an hour, shaking from time to time. In the meantime titrate rapidly 50 c.c. $Ba(OH)_2$ with standard oxalic solution, 1 c.c. = 1 c.c. CO_2 at N.T.P. (5·6642 grm. per litre).

[This amount is weighed, since 1,000 c.c. CO_2 weigh 1·965 grm. Now 44 grm. CO_2 are equivalent to 126 grm.

of oxalic acid $(COOH)_2 . 2H_2O$; 1·965 grm. CO_2 are equivalent to $\frac{126 \times 1·965}{44} = 5·6442$ grm. oxalic acid.] Use phenolphthalein as indicator.

As there is no CO_2 present we can use this indicator. Barium carbonate, being heavy, settles in the hollow stopper when the bottle has been inverted long enough.

The baryta water in the bottle absorbs the CO_2 and forms $BaCO_3$. After sufficient time invert the bottle and allow the precipitate to settle for ten minutes. From the hollow stopper pipette out 25 c.c. of clear liquid, place in a flask, add phenolphthalein and from a burette run in standard oxalic acid solution till the pink colour is discharged. Double the titration of the latter.

Example :—

50 c.c. $Ba(OH)_2$ blank experiment = 34·7 c.c. oxalic acid.
50 ,, ,, experiment = 32·4 ,, ,,
Difference = 2·3 c.c. oxalic acid = 2·3 c.c. CO_2.
Volume of water = 2910 – 50 c.c. for $Ba(OH)_2$ = 2860.
Pressure, 757 mm. Temp., 15·5° C.

According to Boyle's law the volume of gas varies inversely as the pressure, and according to Charles' law a gas increases by $\frac{1}{273}$ of its bulk at 0° C. for every degree increase in temperature.

Expressing the result at N.T.P. (760 mm. and at 0° C.), as 1 c.c. of the oxalic acid solution = 1 c.c. of CO_2 at *N.T.P.*

$$\frac{V_1P_1}{\theta_1} = \frac{V_2P_2}{\theta_2}$$

$V_1 = 2910–50 = 2860$
$P_1 = 757$ mm.
$\theta_1 = t + T$, *i.e.*, $15·5° + 273° = 288·5$
$\theta_2 = T$, *i.e.*, 273
$P_2 = 760$

$$\text{Whence } V_2 = \frac{273 \times 2860 \times 757}{760 \times 288·5} = 2695·6 \text{ c.c.}$$

In 2695·6 c.c. air there are 2·3 c.c. CO_2
100 c.c. ,, ,, $\frac{2·3 \times 100}{2695·6} = ·085$ % at N.T.P.

Expressing the result at the temperature and pressure of experiment.—Volume of CO_2 observed at 15·5° C. (and at 757 mm. in volume of air 2860) $= 2·3 \times \frac{760}{757} \times (1 + \frac{15·5}{273}) = 2·4$ c.c. In 2,860 c.c. air there are 2·4 c.c. CO_2; in 100 c.c. air there are $\frac{2·4 \times 100}{2860}$ = ·084 per cent. at temperature and pressure of experiment.

Conclusions to be drawn from the amount estimated.—Kenwood states that CO_2 is so inert itself that it may exist to the extent of 2 to 3 per cent. without serious consequences, and fatal results would not accrue with less than 5 to 10 per cent. The lowest estimation of CO_2 made in any atmosphere appears to have been ·02 per cent. with a sample of air taken at a high altitude. The air of cities often reaches ·07 to ·09 per cent.; in a sitting-room well lighted with gas ·2 per cent. may frequently be found. The normal amount of CO_2 is known as ·033 per cent. by volume.

The Detection of Poisonous Gases.

Poisonous gases often exist in notable quantities in the atmosphere of chemical works and in industries which necessitate the use of chemicals having or producing obnoxious odours. It is, therefore, necessary to test samples of air for the presence of poisonous gases.

The samples may be taken in air-jars.

In dealing with gases it is a good plan to eliminate the gases which cannot be present and then to apply special tests for those which can reasonably be present. Thus, if the gas is alkaline it is useless testing for acid gases. Often the odour of the gas gives a clue to its detection.

The following procedure will be found useful:—

I.—Open the stopper partially and cautiously smell.

Sulphuretted hydrogen —smells of rotten eggs.
Ammonium sulphide —fœtid, but when pure has a sweetish ethereal odour.
Chlorine —straw-coloured gas with a suffocating odour.
Nitrous fumes —reddish-brown colour, irritating smell.
Sulphurous acid —pungent and suffocating odour.
Carbonic acid —faint pungent smell, feebly acid taste.
Carbon monoxide —colourless, odourless gas
Ammonia —powerfully pungent smell, strong caustic taste.
Hydrochloric acid —choking pungent odour.

II.—Take the reaction of the gas by inserting at the neck of the jar a moist red litmus paper. If the paper goes blue an alkaline gas is present, and it may be (A) Ammonia; (B) Ammonium sulphide.

Ammonia (NH_3).

Add about 10-15 c.c. of water to the jar and shake well. Pour a little into a test tube and add Nessler's reagent. An orange-yellow coloration is obtained, or if much ammonia is present a reddish precipitate is formed.

Ammonia and ammonium sulphide may be present as an atmospherical impurity owing to organic decomposition, trade processes, &c.

Ammonium Sulphide $(NH_4)_2S$.

(1) A solution of sodium nitroprusside added to a few c.c. of the gas solution gives a violet coloration.

(2) Another drop of the solution placed on a lead acetate paper blackens it.

(3) A few c.c. of the solution added to a copper salt gives a black precipitate.

Reactions (2) and (3) are given by sulphuretted hydrogen, but the latter gives no colour with sodium nitroprusside.

III.—If the red litmus paper remained unchanged, insert a moist blue one. An acid gas will be denoted by the paper turning red, and the gas may be—(A) Sulphurous acid; (B) Carbonic acid; (C) Nitrous acid; (D) Nitric acid; (E) Hydrochloric acid; (F) Chlorine.

(*A*) *Sulphurous Acid* (SO_2).

(1) Moisten a starch and potassium iodate paper, and insert it in the jar. A blue colour is obtained.

(2) A piece of filter paper moistened with potassium bichromate is turned green.

(3) Shake up the gas with 10-15 c.c. of water. A few c.c. of this solution will decolorize a solution of permanganate of potash.

(4) Add a drop of ferric chloride to a few c.c. of the solution, then potassium ferricyanide. A blue precipitate is produced.

Sulphurous acid gains access to air from combustion, pyrites in fuel, bleaching works and copper smelting.

(*B*) *Carbonic Acid* (CO_2).

(1) The acidity is not very pronounced.

(2) Add clear baryta water to a few c.c. of the gas solution and shake. A turbidity appears due to the formation of $BaCO_3$.

(3) Silver nitrate added to another portion only gives a faint precipitate, soluble in nitric acid.

Carbonic acid gains access to the air from combustion, respiration, organic decomposition, chemical works, soda-water factories, brewing.

(*C*) *Nitrous Acid* (HNO_2).

(1) The acidity is marked.

(2) To a few c.c. of the gas solution add a drop of potassium iodide, a drop of dilute H_2SO_4, and then about 3 c.c. of fresh starch solution. A blue colour is obtained.

(3) A few c.c. are well diluted in a Nessler glass, acidify with dilute H_2SO_4 and add 3 c.c. of metaphenylene-diamine solution. A yellowish orange colour is obtained.

(4) A few drops are diluted in another Nessler glass, acidify with dilute HCl, add 6 drops of naphthylamine hydrochloride and 6 drops of a saturated solution of sulphanilic acid. A pink colour develops. It is important to do this test in a well diluted solution. Nitrous acid and nitric acid occur in the air from organic decomposition, trade processes, &c.

(*D*) *Nitric Acid* (HNO_3).

(1) Add Brucine and H_2SO_4 to a few c.c. of gas solution. A red colour is obtained which changes to yellowish brown.

(2) To a few c.c. add a solution of ferrous sulphate and pour cautiously Conc. H_2SO_4 down the side of the tube. A black ring is formed at the separation zone of the liquids.

(3) Warm a few c.c. with a few drops of phenol-sulphonic acid. Dilute and add ammonia. A yellow colour is obtained.

(*E*) *Hydrochloric Acid* (HCl).

(1) To a few c.c. of the gas solution add silver nitrate. A white curdy precipitate is formed. Divide it into two portions and notice that one part is insoluble in nitric acid and the other part is soluble in ammonia. HCl may occur in the atmosphere from alkali works.

(*F*) *A moistened blue litmus paper is first reddened*, and then bleached, which indicates *Chlorine*.

(1) Insert a moistened starch and potassium iodide paper. A blue colour is produced.

(2) Shake with 15 c.c. of water and test a portion with a ferrous salt and then add ammonium sulphocyanide. The chlorine oxidizes the ferrous salt to the ferric state and so gives a blood red colour with the sulphocyanide. Cl. occurs in the atmosphere from bleaching works.

IV.—If a red or blue litmus paper is not affected the gas is apparently neutral and may be (A) Sulphuretted hydrogen; (B) Carbon bisulphide.

(*A*) *Sulphuretted Hydrogen* (H_2S).

(1) A moist lead acetate paper is blackened.

(2) A few c.c. of the gas solution gives a black colour with a copper salt.

(3) Characteristic odour.

(4) No nitroprusside reaction. (Difference from ammonium sulphide.)

H_2S occurs in the atmosphere from combustion of coal, putrefaction, chemical works, sewers.

(*B*) *Carbon Bisulphide* (CS_2).

(1) Burns with a blue flame and deposits sulphur.

(2) A few c.c. of gas solution, dissolved in alcohol, ther add ammonia, boil, acidify with HCl, and add a drop of ferric chloride.

A blood-red colour is obtained, due to the formation ammonium sulphocyanide.

$$3CS_2+6NH_3+3H_2O=2NH_4SCN+(NH_4)_2CO_3+4H_2S$$

CS_2 may occur in the air from vulcanization in india-rubber works.

(*C*) *Carbon Monoxide* (CO) may be detected by shaking up with blood. The oxyhæmoglobin is changed to carboxyhæmoglobin, and when seen through the spectroscope the spectrum yields two bands in the yellow and green parts of the spectrum, both between the D and E lines, and resembling those of oxyhæmoglobin. The addition of ammonium sulphide does not change them, whereas ordinary blood forms a single band. Blood treated with carbon monoxide assumes a cherry-red colour.

Welzel's Test.—To 10 c.c. of the blood solution add 15 c.c. of 20 per cent. potassium ferrocyanide solution and 2 c.c. of 33 per cent. acetic acid; if CO is present the precipitate quickly becomes reddish and brown, whereas in presence of oxyhæmoglobin it is greyish-brown and the difference disappears slowly.

Another test is as follows: The suspected air is passed through a U tube containing periodic acid and maintained at 80° C. If CO is present iodine is liberated which is dissolved out in chloroform and estimated in a colorimeter scale. Periodic acid is not affected by any other elements present in the atmosphere.

CO is formed when combustion is incomplete; it is to be found in the air of rooms where iron or copper stoves are used, more particularly when made of cast iron and when the fuel is coke. It is also present in the air of furnace flues and also those from domestic fireplaces. Notable quantities may be found in the air near coke-ovens, brick-kilns and cement works.

Ozone (O_3).—In view of the antiseptic and germicidal properties of this gas, which has been applied to the purification of air and water, some mention of it should be made. It possesses marked irritating properties upon the mucous membrane of the eyes and nose and upon the respiratory tract. It is best prepared by passing electrical discharges through moist air; on this account it is present during and after thunderstorms. Ozone may be detected by exposing a moist starch and potassium iodide paper in the sample of air; a blue colour is produced due to the iodine liberated: $O_3 + 2KI + H_2O = 2KOH + I_2 + O_2$.

It should be noted that nitrous oxide and chlorine would also respond to this test.

Engler and Wild advise the use of manganese chloride papers; ozone makes them go brown owing to the formation of manganese dioxide. Ammonia and ammonium carbonate will also produce this, but if the papers are then moistened with fresh tincture of guaiacum a blue colour appears if the brown colour was due to ozone, whereas if it was produced by the ammonia no blue is obtained. Nitrous acid does not affect manganese papers.

PART III.—WATER ANALYSIS.

(A) Physical Tests.

(1) **The Reaction.**—This is taken by adding a few drops of neutral litmus to 100 c.c. of the sample. Carbonic acid will turn litmus solution red. Acid waters may attack lead pipes and also cause rust in iron pipes.

(2) **The Colour** may be ascertained by half filling the two-foot tube or stratum as it is called. The tube is then laid flat on the bench, and on looking through one half is colourless whilst the other half assumes the colour of the water. Good waters are usually of a pale blue or pale green colour.

(3) **The Odour** is obtained by warming about 100 c.c. of the water to about 37° C. in a beaker and covering with a watch-glass. Certain mineral waters contain sulphuretted hydrogen. A fishy odour is imparted by volvox, uroglena, &c., a grassy odour by anabæna, rivularia, &c., an aromatic odour by asterionella, diatoma, &c., sulphuretted hydrogen by beggiatoa and crenothrix.

(4) **The Sediment.**—It is important to examine microscopically any deposit or suspended matters, as valuable confirmatory evidence of pollution may not infrequently be obtained.

The following organisms are amongst the more common in :—

Impure Waters.

Vorticella.
Crenothrix.
Asterionella.
Diatoma.
Actinophrys.
Protococcus.
Euglena viridis.
Volvox.
Oscillaria.
Desmids.

Rotifera.
Ulothrix.
Algæ.
Spirilla.
Spirogyra.
Gammarus pulex.
Cyclops.
Daphnia.
Water bear.
Bomina longirostis.

Sewage.

Carchesium.
Oscillatoria nigra.
Leptomitus.
Sphærotilus natans.
Beggiatoa.
Ulva latissima.
Enteromorpha.
Anguillula.
Infusoria.

Water Standards.

The following figures expressed in parts per 100,000 will give the student some idea of the different classes of waters :—

	T.S.	CL.	Nitrates	Saline ammonia	Albuminoid ammonia	Oxygen absorbed
Good water	43—60	2—5	Up to ·5	·003—·008	·003—·008	Up to ·1
Upland surface	5—6	1·1	·05—·12	·002—·008	·005—·015	·1—·3
Rain water...	·5—2·9	0—·2	up to ·6	·02 —·2	·008—·02	...
Deep well...	43·7	5·1	·3—·5	Up to ·01	·003—·01	Slight
Spring ...	28·2	2·5	Up to ·5	Trace	...	Slight
Shallow well	...	...	High	Up to 2·75	Up to ·01	·1
River ...	9·67	·2	...	Up to ·01	...	...

Generally speaking, good waters are spring, deep well, upland surface ; suspicious waters are stored rain, surface water from cultivated land ; dangerous waters are shallow well and river to which sewage has access.

(B) Chemical Tests.

It is usual to express the results in water analyses either in parts per 100,000 or grains per gallon, which is equivalent to parts per 70,000, since there are 70,000 gr. in a gallon. 100 c.c. is a convenient quantity to work with for certain estimations, and providing one is using standard solutions of a strength 1 c.c. = ·001 grm. of the substance in question, the number of c.c. used in a titration gives the result in parts per 100,000. Similarly, if 70 c.c. of sample be taken, the actual titration gives the result in grains per gallon.

Example.—Cl in water. Took 100 c.c. sample ; required 3·4 c.c. of standard silver nitrate solution (1 c.c. = ·001 grm. of Cl).

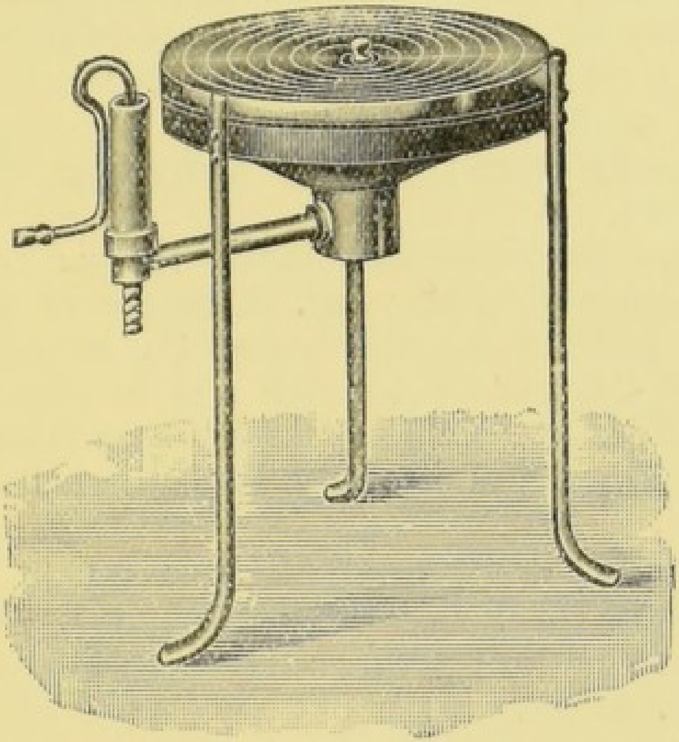
Hence : $\frac{3\cdot4 \times \cdot001 \times 100{,}000}{100} = 3\cdot4$ parts per 100,000, or if 70 c.c. of sample were taken and 2·4 c.c. of $AgNO_3$ solution used we should have :

$$\frac{2\cdot4 \times \cdot001 \times 70{,}000}{70} = 2.4 \text{ grains per gallon.}$$

It is, however, quite simple to convert one result into the other. To convert grains per gallon to parts per 100,000, multiply by ten and divide by seven.

Conversely, parts per 100,000 multiplied by seven and divided by ten gives grains per gallon.

(1) **Total Solids.**—Weigh a small porcelain dish. Measure out 200 c.c. of the sample in a graduated flask. Fill the dish and evaporate on the water bath, filling up the dish with more water from the graduated flask until all has been poured in. When all has been evaporated to dryness place the dish in a water oven for an hour, cool and weigh.



WATER BATH (constant level).

Example :—

Dish and total solids 27·848 grm.
,, alone 27·797
Wt. of residue = ·051 grm. × $\frac{100,000}{200}$ = 25·5 parts per 100,000.

(2) **Loss on Ignition.**—The dish from above is then carefully heated until all the organic matter has been burnt off. Allow to cool, then add a little ammonium carbonate solution. Dry and then heat just sufficiently to drive off the ammonia. During the first heating some of the carbonates present may be converted into oxides.

$CaCO_3 = CaO + CO_2$. In order to reform the carbonates we heat at a low temperature with ammonium carbonate, which gives off ammonia, water and carbonic acid: $(NH_4)_2\,CO_3 = 2NH_3 + H_2O + CO_2$.

The CO_2 thus liberated combines with any oxides present and reforms the carbonate: $CO_2 + CaO = CaCO_3$. The dish is then cooled and weighed.

Example: Dish + T.S. before 27·848
,, ,, ,, after 27·840
·008

$$\therefore \frac{\cdot 008 \times 100{,}000}{200} = 4 \text{ parts per } 100{,}000.$$

(3) **Chlorine.**—Chlorine is present in most waters as sodium chloride, or as the potassium or calcium salt. Its presence may be due to (*a*) the water having previously percolated strata composed of saline compounds, *e.g.*, greensand, sandstone; (*b*) pollution by organic matter, more especially urine; (*c*) deep wells near the sea-coast; (*d*) tidal rivers.

In a large porcelain dish place 100 c.c. of the water. Add a few drops of 5 per cent. chromate of potash. Run in from a burette standard $AgNO_3$ solution (1 c.c. = ·001 grm. of Cl) until a reddish tint is obtained. When all the chlorides present have been precipitated the $AgNO_3$ acts on the potassium chromate and forms silver chromate, which is red.

$$AgNO_3 + KCl = AgCl + KNO_3$$
$$2\,AgNO_3 + K_2CrO_4 = Ag_2CrO_4 + 2\,KNO_3$$

In order to make up the silver solution so that 1 c.c. = ·001 grm. of Cl, weigh out 4·79 grm. of $AgNO_3$ and make up 1 litre with distilled water. The amount is arrived at as follows:—

$$AgNO_3 + KCl = AgCl + KNO_3$$

i.e., 170 ,, combine with 35·5 Cl.
$\frac{170}{35\cdot 5}$,, ,, ,, 1 ,,

= 4·79 (4·7887). Now as 1 c.c. is required to be equal to ·001 grm. Cl, *i.e.*, 1 litre is required to be equal to 1 grm., hence 4·79 grm. $AgNO_3$ dissolved in 1 litre of water will give the solution required.

The amount of Cl usually found is about 1 to 3 parts per 100,000.

Example: 100 c.c. sample required 1·7 c.c. of standard $AgNO_3$ solution—

$$1\cdot 7 \times \cdot 001 \times \frac{100{,}000}{100} = 1\cdot 7 \text{ parts per } 100{,}000.$$

If it is required to express the result in terms of sodium chloride, multiply the Cl figures by 1·647. Cl × 1·647 = NaCl. This factor is derived from the ratio

$$\frac{NaCl}{Cl} = \frac{58\cdot 5}{35\cdot 5} = 1\cdot 647.$$

(4) **Nitrites.**—The presence of nitrites in water generally indicates recent organic pollution and that the oxidation of

the nitrogenous organic matter is still going on. During the decomposition of nitrogenous organic matter a large part of the nitrogen passes off in the gaseous state, the remainder combines with hydrogen and forms ammonia. Through the action of certain nitrifying organisms in the soil, the nitrogen becomes partially oxidized to nitrous acid (HNO_2) and forms nitrites with bases such as lime, soda and potash, usually present in water. The nitrites are eventually oxidized to nitrates. Sometimes the presence of nitrites may be due to the reduction of nitrates by ferrous salts or in deep wells due to the metal of the bore pipes. Nitrites are usually found in sewage effluent.

Test for Nitrites.—(1) Griess-Ilosvay test. In a Nessler cylinder place 100 c.c. of water, add six drops of dilute HCl, six drops of naphthylamine hydrochloride and six drops of sulphanilic acid. Stir with a glass rod. A pink colour develops if nitrites are present. If no coloration is apparent after fifteen minutes nitrites may be considered absent.

(2) To 100 c.c. of water in a Nessler cylinder add a few drops of dilute H_2SO_4, a crystal of KI and then mix well. Now add 5 c.c. of fresh starch solution. A blue colour forms if nitrites are present. When a nitrite is acidified with dilute H_2SO_4 in presence of KI the following reaction takes place:—

$$2\,KI + 2\,H_2SO_4 + 2\,NaNO_2 = K_2SO_4 + Na_2SO_4 + 2\,HNO_2 + 2\,HI.$$

The nitrous acid and hydriodic acid interact liberating iodine thus:—

$$2\,HI + 2\,HNO_2 = I_2 + 2\,NO + 2\,H_2O.$$

(3) *Griess Test.*—To 100 c.c. of water add a few drops of dilute HCl, then 5 c.c. of solution of metaphenylene-diamine [prepared by dissolving 5 grm. of metaphenylene-diamine in water, adding 4 c.c. conc. H_2SO_4 and making up to 1,000 c.c. Shake frequently and after twenty-four hours filter and decolorize with animal charcoal]. Mix and allow to stand. A yellowish orange colour indicates nitrites. This is due to the formation of Bismarck brown or triamido-azobenzene. The reagent should be colourless; if it is not so shake a little with animal charcoal and filter.

If it is desired to estimate nitrites quantitatively, the last method is used colorimetrically, using a standard solution of silver or sodium nitrite to match the sample. To make up a standard nitrite solution of a strength 1 c.c. = ·001 grm. of N as nitrite, weigh out 4·93 grm. $NaNO_2$ and make up to 1 litre with water. Since 14 grm. are contained in 69 grm. $NaNO_2$.

$\therefore$ 1 grm. N is contained in $\frac{69}{14} = 4{\cdot}93$ grm.

The result may be expressed in terms of the "acid radical," N_2O_3 — nitrous anhydride, from the ratio

$$\frac{N_2O_3}{N_2} = \frac{76}{28} = 2{,}714 \text{ (factor)}$$

(5) **Nitrates.**—Nitrates are usually present in waters. They are the final products of complete oxidation of organic nitrogenous matter. Sometimes their presence is due to the water having permeated strata containing nitrates, *e.g.*, from the oolite, red sandstones, &c.

QUALITATIVE TESTS.—(1) Fill a large test tube with the water, add a few c.c. of diphenylamine solution and pour conc. H_2SO_4 carefully down the side of the tube. A blue ring is formed at the junction of the two liquids, the acid being at the bottom of the tube.

(2) Repeat the above, using a solution of brucine in H_2SO_4. A red ring is formed at the junction of the two liquids which turns to yellow, then brown.

Brucine and diphenylamine both give reactions with nitrites. Metaphenylene-diamine does not react with nitrates. The picric acid test is reliable for nitrates.

QUANTITATIVE ESTIMATION OF NITRATES.

(1) Phenolsulphonic acid method, consisting in the conversion of the nitrates into picric acid, which is then estimated colorimetrically. Evaporate 50 c.c. of water to dryness on the water bath. Add 1 c.c. of phenolsulphonic acid, mix well with a small glass rod and heat on the boiling water bath for about a minute. Remove the basin, transfer the liquid to a 100 c.c. Nessler glass, washing out basin and rod with a little water. Add about 10 c.c. of concentrated ammonia, stir and make up to 100 c.c. with water. Shake well. A yellow coloration denotes the presence of nitrates. The phenol forms with the nitrates trinitrophenol, *i.e.*, picric acid. The addition of ammonia forms ammonium picrate, which has an intense yellow colour.

The following are the equations representing these reactions :—

$$\underset{\text{(Phenolsulphonic acid)}}{C_6H_4(OH)SO_2{\cdot}OH} + 3\,HNO_3 = \underset{\text{[Picric acid]}}{C_6H_2(OH)(NO_2)_3} + H_2SO_4 + 2\,H_2O$$

$$C_6H_2(OH)(NO_2)_3 + NH_4OH = \underset{\text{[Ammon. picrate]}}{C_6H_2ONH_4(NO_2)_3} + H_2O$$

The yellow colour is matched colorimetrically as follows: 5 c.c. of standard KNO_3 solution (1 c.c. = ·0001 grm. N) are evaporated to dryness and treated identically as above described. To prepare the standard KNO_3 solution weigh out ·722 grm. pure KNO_3 and make up to 1,000 c.c. with distilled water, since 14 grm. of N are contained in 101 grm. KNO_3 and ·1 grm. of N is contained in $\frac{101 \times \cdot 1}{14} = \cdot 722$ grm.

We now have two cylinders having a yellow colour. From the deeper of the two pour out liquid into a 100 c.c. graduated cylinder, until the tints are identical, and calculate the amount of N as nitrate as follows:—

Example:—

(i) *Where the standard is deeper than the sample of water*: Poured out 25 c.c.; quantity remaining in cylinder is 75 c.c., whence $\frac{75}{100} \times \cdot 0005 \times \frac{100,000}{50} = \cdot 75$ parts N as nitrate in 100,000 parts.

(ii) *Where the sample is darker than the standard*: *e.g.*, Poured out 25 c.c.; quantity remaining in the cylinder is 75 c.c. We should then have $\frac{100}{75} \times \cdot 0005 \times \frac{100,000}{50} = 1 \cdot 3$ parts per 100,000. The result may be expressed in terms of the "acid radical" N_2O_5 – nitric anhydride, by multiplying by 3·857 obtained from the ratio $\frac{N_2O_5}{N_2} = \frac{108}{28} = 3 \cdot 857$ (factor).

Other Methods for Estimation of Nitrates.

(2) *Zinc-copper Couple Method.*—This method is based on the fact that when zinc coated with copper is immersed in water an evolution of hydrogen takes place and zinc oxide is formed. If nitrates are present in the water the hydrogen reduces them first to nitrites and then combines with the nitrogen to form ammonia. The amount of the latter can then be estimated by distillation and Nesslerizing. Granulated zinc is first cleaned with HCl, then immersed for about ten minutes in 3 per cent. copper sulphate solution. The copper forms a black coating on the zinc. Wash well with distilled water, then place in a bottle of known capacity (about 300 c.c.) and fill up with a sample of water. Allow to stand for twenty-four hours in a dark place. The nitrates are converted into ammonia, which remains in solution.

Before proceeding to the distillation for the ammonia estimation it is prudent to test for nitrites on 5 c.c. with Griess's test in order to ensure that reduction is complete.

(3) *Indigo Method.*—When indigo and a nitrate are treated with fuming H_2SO_4, the nitric acid liberated decolorizes the indigo. This reaction is used for the estimation of nitrates. In a large test tube place 10 c.c. of sample and 1 c.c. of standard indigo solution (or more according to amount of nitrate present) and add 4 c.c. water. Pour in 20 c.c. of fuming H_2SO_4 and stir. If decoloration is produced try 2 c.c. of standard indigo, &c., always making up volume to 15 c.c. with water. The end point is reached when the blue colour ceases to be discharged. The standard indigo solution is made by comparison with a known solution of KNO_3. A suitable strength would be

1 c.c. indigo = ·00002745 grm. N as nitrate.

The Oxygen absorbed: Tidy's Process.—The object of this experiment is to estimate the oxidizable organic matter in the water. It is usual to do this test either for three hours at 37° C. or at 80° F. for four hours. The putrescent organic matter is chiefly oxidized in the first thirty minutes. Oxygen may also be taken up by nitrites, H_2S, ferrous salts, &c.

In a bottle of about 400 c.c. capacity with glass stopper, place 200 c.c. of the sample, add 10 c.c. of $KMnO_4$ solution (1 c.c. = ·0001 grm. of available oxygen) and 10 c.c. of H_2SO_4 (1 in 4). Place in an incubator at 37° C. for three hours.

There must always be an excess of $KMnO_4$; when the water is badly polluted the pink colour may quickly fade, and it is then necessary to add more $KMnO_4$, taking note of how many c.c. have been introduced for the whole of the experiment. At the expiration of the three hours at 37° C. there must always be a good pink colour.

In order to make the permanganate solution weigh out ·395 grm. of pure crystallized $KMnO_4$, dissolve in water and make up to 1,000 c.c.

From the equation given on p. 9 we see that

8 grm. of available oxygen are obtained from 31·6 grm. $KMnO_4$

·1 grm. of available oxygen is obtained from $\frac{31·6 \times ·1}{8}$ = ·395 grm.

After the three hours cool under the tap, add 5 c.c. of

10 per cent. KI, then run in from a burette sodium thiosulphate solution (1 grm. in 1,000 c.c.) until the yellow colour has nearly disappeared. Now add 5 c.c. of fresh starch solution and continue the addition of the thiosulphate until the blue colour has disappeared, shaking constantly. The following equations represent the reactions :—

$$3\,H_2SO_4 + 2\,KMnO_4 = 2\,MnSO_4 + K_2SO_4 + 3\,H_2O + 5O$$

$$10\,KI + 8\,H_2SO_4 + 2\,KMnO_4 = 2\,MnSO_4 + 6\,K_2SO_4 + 8\,H_2O + 5I_2$$

$$2\,Na_2S_2O_3 + I_2 = 2\,NaI \quad + \quad \underset{\text{(Sodium tetrathionate)}}{Na_2S_4O_6}$$

Blank Experiment.—The object of this is to find the strength of the thiosulphate in terms of oxygen. In a similar bottle as above place 200 c.c. of distilled water, add 5 c.c. of 10 per cent. KI solution, 10 c.c. H_2SO_4 (1 in 4) and 10 c.c. of $KMnO_4$ solution (1 c.c. = ·0001 grm. oxygen).

It is not necessary to incubate this bottle. Run in the thiosulphate solution from a burette until the yellow colour has nearly disappeared, then add 5 c.c. of fresh starch solution and continue the addition of the thiosulphate until the blue colour is discharged.

Example :—

Blank experiment required, 16·8 c.c. of thiosulphate
Sample of water ,, 10·9 ,, ,,
Difference = 5·9 ,, ,,

Now 16·8 c.c. of thiosulphate = 10 c.c. of $KMnO_4$
= ·001 grm. of oxygen
Hence 16·8 ,, ,, = ,, ,, ,,
5·9 ,, ,, $= \frac{\cdot 001}{16\cdot 8} \times 5\cdot 9 = \cdot 00035$

In 200 parts there are ·00035 parts of oxygen absorbed.
In 100,000 ,, ,, ,, $\frac{\cdot 00035 \times 100{,}000}{200} = \cdot 175$

Good waters usually absorb up to ·1 part per 100,000. Peaty waters usually have high oxygen absorption figures, hence water cannot be judged unsatisfactory on this figure alone.

Frankland classifies waters approximately as follows :—
O absorbed (parts per 100,000) :—

Not exceeding ·1 = good organic purity
,, ,, ·3 = medium ,,
,, ,, ·4 = doubtful ,,
More than ·4 = polluted

The above figures concern waters derived from upland surfaces; for waters derived from sources other than these the amounts should be halved.

Estimation of the Ammonias—Wanklyn's Process.

(1) FREE AND SALINE AMMONIA.—During the decomposition of nitrogenous organic matter a part of the nitrogen combines with hydrogen to form ammonia; the latter then combines with the saline constituents of the water, or it may remain in the free state. One of the chief nitrogenous substances in sewage is urea, which is converted into ammonium carbonate by the *Micrococcus ureæ* $CO(NH_2)_2 + 2H_2O = (NH_4)_2CO_3$. Generally speaking "the free and saline ammonia" is a product of the decomposition of animal matter.

As the amount of ammonia is often very small and its estimation of importance, it is essential to ensure that the condenser, flasks, reagents, &c., used in the processes are themselves ammonia-free. For this purpose a litre boiling flask, containing about 300 c.c. of ammonia-free water, is connected to a condenser and distilled. The distillate is collected in 50 c.c. of Nessler glasses, to each of which 2 c.c. of Nessler solution is added. Nessler's solution is composed of potassio-mercurio iodide with caustic potash. It gives a yellowish colour with small quantities of ammonia, whilst quite a reddish colour is obtained when a considerable amount is present.

$$2[HgI_2 \cdot 2KI] + 3KOH + NH_3 = 7KI + 3H_2O + \overset{\text{[\textit{Dimercurio-ammonium iodide}].}}{NHg_2I}$$

Distillation is continued until no colour is given by the addition of Nessler reagent after allowing to stand three minutes. It usually requires 100-150 c.c. The apparatus is now free from ammonia.

Pour away the water remaining in the flask and replace it by 500 c.c. of the sample under examination. Add a pinch of calcined sodium carbonate. This is to render the water alkaline, as ammonium salts are retained in acid solutions; all the ammonia present is converted into ammonium carbonate, which on boiling yields ammonia.

Connect up with the condenser and collect the distillate in 50 c.c. Nessler glasses. As soon as one is filled to the mark replace it with another. Add 2 c.c. of Nessler

solution to the first cylinder. Stir with a glass rod and allow to stand for three minutes. The coloration is now matched colorimetrically. In a 50 c.c. Nessler glass place ·5, 1 or 2 c.c. of standard ammonium chloride solution (1 c.c. = ·00001 grm. NH_3), according to the depth of tint in the distillate, fill up to the mark with ammonia-free water, add 2 c.c. Nessler solution, stir and allow to stand. Compare the tints. If the standard is too dark or too light in tint make a fresh one, using less or more ammonium chloride solution as may be necessary. Fresh standards must be made each time. The second and successive cylinders from the sample water are treated in a similar manner. As a rule the first two cylinders will contain all the ammonia and it is seldom necessary to collect three unless the water is polluted.

Example :—

1st cylinder required 2·5 c.c. ammonium chloride solution.
2nd „ „ ·5 „ „ „ „
3rd „ „ *nil.*

Total 3 c.c. ; hence amount of NH_3 in 500 c.c. water is 3 × ·00001 ; amount of NH_3 in 100,000 c.c. water is $\frac{3 \times \cdot 00001}{500} \times 100,000 = \cdot 006$ parts per 100,000.

The amount usually found in good waters does not exceed ·01 parts per 100,000.

The standard ammonium chloride solution is made by dissolving 3·15 grm. pure NH_4Cl in 1,000 c.c. water, because $\frac{NH_4Cl}{NH_3} = \frac{53 \cdot 5}{17} = 3 \cdot 15$ grm. ; ∴ 1 c.c. of this solution = ·001 grm. NH_3.

10 c.c. of the same solution are diluted to 1,000 c.c. ; hence 1 c.c. = ·00001 grm. NH_3.

Estimation of the Albuminoid Ammonia.—When nitrogenous organic matter is heated with an alkaline solution of potassium permanganate, it is oxidized and part of the nitrogen present is converted into ammonia.

50 c.c. of alkaline permanganate solution (prepared by boiling down to three-quarters of its bulk 200 grm. KOH and 8 grm. $KMnO_4$ with 1,000 c.c. water, and finally making up to 1 litre with ammonia-free water) are boiled in a beaker for fifteen minutes with 100 c.c. water. The hot liquid is then poured by means of a funnel into the liquid remaining in the distillation flask. A few pieces of pipe-stem are added to prevent bumping and the

liquid distilled over, the ammonia in the distillate being estimated as in the saline ammonia process. The albuminoid ammonia comes over much slower and it is usually necessary to distil three or four Nessler glasses before it is all distilled over.

Example: Four cylinders required 3, 2, 1, ·8 c.c. respectively of standard ammonium chloride solution. Total, 6·8 c.c. Hence amount of NH_3 in 500 parts of water is 6·8 × ·00001; amount of NH_3 in 100,000 parts water is $\frac{6{\cdot}8 \times {\cdot}00001 \times 100{,}000}{500}$ = ·013 part per 100,000. The amount usually found in good waters does not exceed ·01 part per 100,000.

Consideration of the Amounts estimated.—If the albuminoid ammonia greatly exceeds the saline, it indicates vegetable pollution. Animal pollution would be indicated by a fairly high saline ammonia, together with albuminoid ammonia exceeding ·005, high figures for chlorine and nitrates.

In upland surface and peaty waters the saline ammonia should not exceed ·005 parts per 100,000.

High figures for saline ammonia may be found in waters from the greensand, due to the reduction of nitrates by ferrous salts. Deep well waters may also contain considerable quantities of free ammonia owing to the reduction of nitrates by the metal of the bore pipes.

The Hardness.—The hardness or soap-destroying power of a water is mainly due to the presence of the bicarbonates, sulphates and chlorides of calcium and magnesium.

Estimation of Total Hardness.—In a stoppered bottle of about 150 c.c. capacity place 100 c.c. of the sample of water. Run in from a burette standard soap solution (1 c.c. = ·001 grm. $CaCO_3$), shaking vigorously until a permanent lather is obtained.

Example: 13·8 c.c. soap solution.

Hence 13·8 × ·001 $CaCO_3$ is contained in 100 c.c. water

$$\therefore \frac{13{\cdot}8 \times {\cdot}001 \times 100{,}000}{100} = 13{\cdot}8 \text{ parts per } 100{,}000.$$

The strength of the soap solution can be estimated by titrating it against standard $CaCO_3$ solution prepared by dissolving 1 grm. of pure calcite in dilute HCl, evaporating twice to dryness and making up to 1,000 c.c. 1 c.c. of this solution contains the equivalent of ·001 grm. $CaCO_3$. Pipette 10 c.c. into a small stoppered bottle, add distilled

water which has been previously boiled, then run in the soap solution till a permanent lather is obtained.

Example: 12 c.c. soap solution required.

10 c.c. $CaCO_3$ solution = ·01 grm. $CaCO_3$
12 c.c. soap ,, = ·01 ,, ,,
1 c.c. ,, ,, = $\cdot\frac{01}{12}$ = ·00083 grm. $CaCO_3$

In the estimation in the sample of water each c.c. of soap solution used would, therefore, have to be multiplied by ·00083.

The temporary hardness—*i.e.*, the hardness which can be removed by boiling—is due to the presence of the bicarbonates of lime and magnesia. On boiling CO_2 is driven off and the calcium and magnesium carbonates are precipitated according to the equation:—

$$\overset{[\textit{Bicarbonate}]}{CaH_2(CO_3)_2} = \overset{[\textit{Carbonate}]}{CaCO_3} + H_2O + CO_2$$

On a commercial scale the temporary hardness is removed by adding lime. The insoluble calcium carbonate formed settles out:—

$$\overset{[\textit{Bicarbonate}]}{CaH_2(CO_3)_2} + CaO = 2\,\overset{[\textit{Carbonate}]}{CaCO_3} + H_2O$$

With magnesium bicarbonate the reactions are:—

$$MgH_2(CO_3)_2 + CaO = MgCO_3 + CaCO_3 + H_2O$$

Magnesium carbonate is slightly soluble, so that more lime is added and magnesium hydrate is formed which is insoluble:—

$$MgCO_3 + CaO + H_2O = Mg(OH)_2 + CaCO_3.$$

The temporary hardness is obtained by subtracting the permanent from the total hardness.

Estimation of Permanent Hardness.—The permanent hardness, *i.e.*, the hardness which cannot be removed by boiling, is due to the presence of the sulphates and chlorides of calcium and magnesium. These salts are soluble in water and cannot be precipitated by boiling. For the estimation, boil 100 c.c. of the sample for fifteen minutes in a beaker. When cold, filter and make up the filtrate with distilled water to 100 c.c. Shake and transfer to bottle and continue as above.

Example: 4·2 c.c. soap = 4·2 parts per 100,000.

When a hard water is brought into contact with soap (a mixture of the sodium salts of the higher fatty acids, stearic, &c.) it forms insoluble calcium and magnesium compounds, the water thus being softened at the expense of the soap.

Poisonous Metals.—To 100 c.c. of water in a Nessler cylinder add a few drops of acetic acid, then pass in sulphuretted hydrogen (H_2S) for a minute or two. Place the cylinder on a white tile. A brownish black coloration denotes the presence of copper or lead.

Detection and Estimation of Copper.—To another 100 c.c. of water add a few drops of acetic acid, then 2 c.c. of a fresh solution of potassium ferrocyanide. A chestnut brown coloration is obtained. This is matched colorimetrically by means of standard copper solution (1 c.c. = ·0001 grm. Cu) just as in Nesslerizing in the ammonia estimations. The standard copper solution is made by dissolving 3·93 grm. $CuSO_4 \cdot 5H_2O$ in 1,000 c.c. water, and diluting ten times.

(2) **Detection and Estimation of Lead.**—If no reaction for copper is obtained, test for lead on another 100 c.c. of water. Acidify with acetic acid and add a few drops of potassium chromate. Stir with a glass rod. A yellow turbidity due to lead chromate indicates the presence of lead. If a fair amount is present a yellow precipitate is obtained.

Lead is estimated quantitatively by means of H_2S.

The brownish coloration obtained in the sample is matched colorimetrically with standard lead solution (1 c.c. = ·0001 grm. Pb) prepared by dissolving 1·831 grm. of crystallized lead acetate in 1,000 c.c. water and diluting ten times.

(3) **Detection and Estimation of Iron.**—If neither lead nor copper are found to be present test for iron by taking 100 c.c. water, add 5 c.c. of nitric acid (1 in 5), then 5 c.c. of 10 per cent. potassium sulphocyanide. A red coloration denotes iron. This is matched colorimetrically by means of standard iron solution (1 c.c. = ·0001 grm. Fe) made by dissolving 1 grm. of pure iron wire in dilute HCl, boil with nitric acid, evaporate, and make up to 1 litre. Dilute ten times for use.

If 100 c.c. of the sample produces a colour too intense to be conveniently matched, a smaller quantity should be taken and diluted to 100 c.c. with distilled water.

Test for Phosphates.—This test can conveniently be done on the residue from the "loss on ignition"; if this is not to hand evaporate 100 c.c. of water with 2 c.c. nitric

acid to dryness on a water bath. Take up with a little dilute nitric acid. Filter off any silica and calcium sulphate. Receive the filtrate, which should not be more than 2 to 3 c.c. in a small test tube. Add an equal bulk of ammonium molybdate solution, warm gently and allow to stand. A yellowish turbidity or precipitate denotes phosphates. Generally phosphates indicate sewage pollution or they are derived from organic matter. The absence of phosphates, however, does not mean that the water is free from pollution. According to Hehner more than ·05 parts P_2O_5 per 100,000 should be regarded with suspicion.

The Oxygen dissolved in Water—Winkler's Process.

This is a measure of the aeration of waters.

Measure the volume of a stoppered bottle of about 200 to 300 c.c. capacity. It should be completely filled. Now fill it slowly and carefully with the sample, taking care to exclude air-bubbles. Add 1 c.c. of 40 per cent. manganese chloride and then 3 c.c. of a solution containing 50 per cent. NaOH and 10 per cent. KI. As these solutions are very heavy, by holding the end of the pipette near the bottom of the bottle they settle down and immediatley form a precipitate of manganese hydrate.

$$MnCl_2 + 2NaOH = Mn(OH)_2 + 2NaCl.$$

Insert the stopper, excluding air-bubbles, and shake.

In the calculations 4 c.c. is subtracted from the volume of the bottle on account of the reagents added.

The manganous hydrate takes up the dissolved oxygen and forms manganic hydrate :—

$$2\,Mn(OH)_2 + O + H_2O = 2\,Mn(OH)_3$$

Allow the precipitate to settle for ten minutes, then run in 5 c.c. of conc. HCl, holding the pipette just above the precipitate :—

$$Mn(OH)_3 + 3HCl = MnCl_3 + 3H_2O$$

Insert the stopper carefully and shake.

A clear reddish brown liquid is obtained, due to the liberation of free iodine :—

$$2\,MnCl_3 + 2KI = 2KCl + I_2 + 2MnCl_2$$

Transfer the liquid to a flask. Rinse out with 10 c.c. water and run in from a burette sodium thiosulphate solution (4 grm. per 1,000 c.c.) until only a faint yellow colour remains, add 5 c.c. of fresh starch solution and continue the addition of the thiosulphate until the blue colour is discharged.

Blank Experiment with the Thiosulphate Solution.

In a flask place 50 c.c. of $KMnO_4$ solution (1 c.c. = ·0001 grm. oxygen), 20 c.c. of H_2SO_4 (1 in 4) and 10 c.c. of 10 per cent. KI. Titrate the liberated iodine as above:—

Example: Thiosulphate blank experiment 23 c.c.
,, sample of water 10·2 c.c.
50 c.c. $KMnO_4$ = 50 × ·0001 = ·005 grm. oxygen.
i.e. 23 c.c. thiosulphate = ·005 grm. oxygen
10·2 c.c. ,, $= \frac{\cdot 005 \times 10\cdot 2}{23} = \cdot 002142$
Volume of bottle = (206-4 c.c.) = 202
In 202 parts of sample there are ·002142 oxygen.
100,000 $\frac{\cdot 002142 \times 100{,}000}{202}$ = 1·06 parts per 100,000
A good water should have about 1·1 parts per 100,000.

Estimation of Dissolved Oxygen—Thresh's Process.

The apparatus required is as given in the figure.

C is a burette which is filled with sodium thiosulphate solution, 7·75 grm. per litre — 1 c.c. of this solution = ·25 mg. of oxygen.

1 c.c. $\frac{N}{10}$ thiosulphate = 1 c.c. $\frac{N}{10}$ oxygen
= ·0008 grm. ,,
1,000 c.c. ,, = ·8 ,, ,,
Sodium thiosulphate = $Na_2S_2O_3 \cdot 5\,H_2O$ = 248
Normal solution of ditto contains 248 grm. per litre.
$\frac{N}{10}$ solution of ditto contains 24·8 grm. per litre.
Now 24·8 grm. thiosulphate are equivalent to ·8 grm. oxygen.

7·75 grm. thiosulphate are equivalent to $\frac{\cdot 8 \times 7\cdot 75}{24\cdot 8}$ = ·25 grm. oxygen per litre, *i.e.*, 1 c.c. = ·25 mg. of oxygen.

D is a separating funnel holding 250 c.c. of water when filled to the lower of the two marks on the neck. The difference between the latter is 2 c.c., this being the volume of the reagents added (see below).

B is the gas exit and fitted with rubber tubing and a cork.

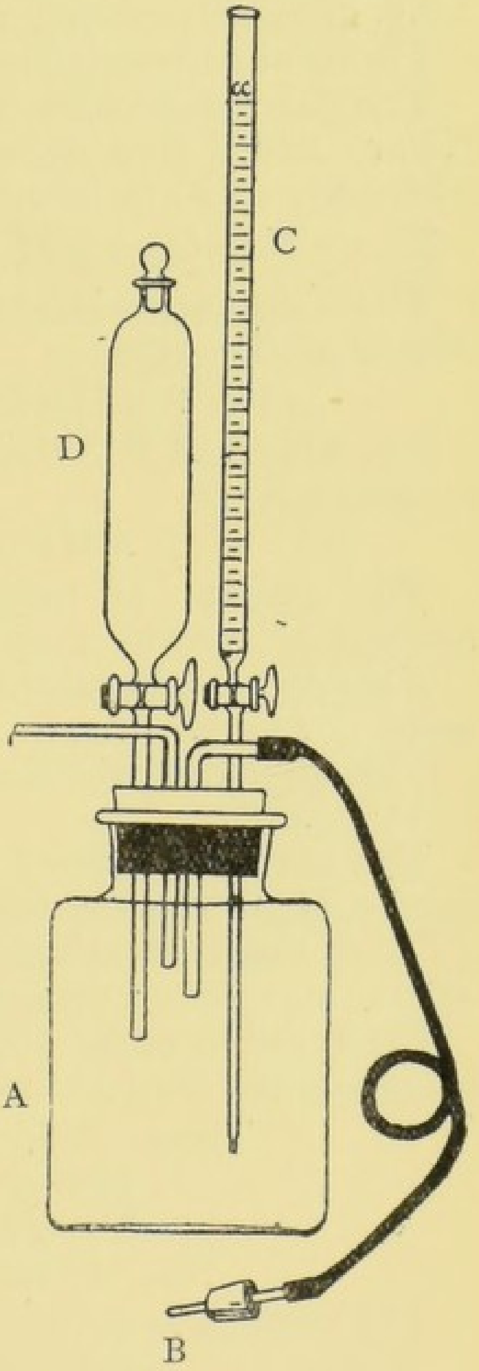
A is a large bottle fitted with a rubber bung having four holes. The fourth hole contains a small piece of glass tubing for the gas entrance.

Solutions required: (1) Sodium nitrite ·5 grm., pot. iodide 20 grm., distilled water, 100 c.c.

(2) Pure H_2SO_4 1 part, distilled water 3 parts.

(3) Fresh starch solution. The burette being filled, the tube *D* is filled up to the lower mark with the sample of water. 1 c.c. of the nitrite solution and 1 c.c. of H_2SO_4 (1 in 4) are then added. The stopper is carefully inserted, excluding air-bubbles. Invert the tube several times, pass it through the bung and allow to stand for fifteen minutes. A current of coal gas is now passed through the apparatus into the bottle and lighted at *B*. The flame is then extinguished, the stopper of the tube removed and the cork *B* rapidly inserted in its place, the gas being on continuously.

The solution containing the liberated iodine is now run into the bottle *A*. The cork *B* is now removed and the gas immediately lighted again at *B*. In this manner no air has access to the bottle *A*. The thiosulphate solution is now run in until the liquid is only faintly yellow. 1 c.c. of starch solution is now added through the tube *D* and the addition of the thiosulphate is continued until the blue colour is discharged. After a minute the latter colour reappears and it is necessary to add ·1 c.c. of thiosulphate solution. Note the burette reading.



THRESH'S APPARATUS.[1]

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[1] To be obtained from Messrs. A. Gallenkamp and other instrument makers.

As the reagents used contain dissolved oxygen it is necessary to do a blank experiment so as to ascertain how many c.c. of thiosulphate to subtract. After refilling the burette with the thiosulphate solution, and leaving the apparatus otherwise intact, run in successively into the bottle, through the tube *D*, 5 c.c. of the nitrite solution, 5 c.c. of starch solution and 5 c.c. of H_2SO_4 (1 in 4).

Titrate the liberated iodine with the thiosulphate solution.

The process depends on the fact that when H_2SO_4 and KI are added to a water containing a nitrite, nitrous and hydriodic acid are formed thus :—

$$2\,KI + 2\,NaNO_2 + 2\,H_2SO_4 = K_2SO_4 + 2\,HNO_2 + 2\,HI$$

These two acids interact, iodine is liberated with formation of nitric oxide :—

$$2\,HI + 2\,HNO_2 = I_2 + 2\,H_2O + 2\,NO$$

The liberated NO then acts as a carrier, more HI being decomposed :—

$$2\,HI + O = H_2O + I_2$$

The amount of iodine liberated corresponds to the amount of nitrous acid used.

Example: Thiosulphate solution required for experiment = 11 c.c.......(a).

Thiosulphate solution required for blank experiment = 12·2 c.c.

The latter is required for (5 + 5 + 5) = 15 c.c. of reagents used, and since in the actual experiment we used (1 + 1 + 1) 3 c.c. of reagents, it follows that for them we should require $\frac{12{\cdot}2}{5} = 2{\cdot}44$ c.c. of thiosulphate solution......(b).

From the formula ·969 a − b we get the number of milligrammes per litre of the sample.

Thus ·969 × 11 − 2·44
= 10·659 − 2·44
= 8·219 milligrammes per litre,
i.e., ·82 parts per 100,000.

The formula is arrived at as follows :—

Let a = c.c. of thiosulphate used in the experiment
b = " " required for the 3 c.c. of reagents used.
x = amount of dissolved oxygen in mg. per litre.

Then $x = 4 \times {\cdot}25\,[a - (b + {\cdot}031a)]$.

We multiply by 4 because the volume of the tube *D* has 250 c.c., and bringing this up to 1,000 c.c., we have 4 × 250.

If the capacity of *D* had been 300 c.c. we should have had $\frac{1000}{300}$.

Each c.c. of thiosulphate solution = ·25 mg. of oxygen. It is assumed that the solution is fully saturated with oxygen, 1 c.c. of which = ·031. If the solution had been made up twice as strong it would have been = ·062.

Simplifying the above equation we obtain

$$\begin{aligned} & a - (b + {\cdot}031a) \\ &= a - b - {\cdot}031\,a \\ &= {\cdot}969\,a - b \end{aligned}$$

Certificate of Analysis of a Sample of Water.

Date received:
From:
Particulars on Label: London tap water. Taken at Laboratory.

PHYSICAL CHARACTERS:
Colour in 2 ft. stratum: clear pale blue.
Suspended matters: *nil.*
Taste:
Odour when warmed to 37° C.: *nil.*
Reaction: alkaline.

CHEMICAL CHARACTERS:

	Parts per 100,000	Grains per gallon
Total solids dried at 100° C. ...	29·60	20·72
Loss on ignition (after re-carbonating)	2·50	1·75
Chlorine	2·1	1·47
,, × 1·647 = sodium chloride	3·45	2·41
Nitrites (as N_2O_3)...	*Nil*	
Nitrogen as nitrates	0·12	0·074
Saline ammonia	0·001	0·0007
Albuminoid Ammonia	0·007	0·0049
Oxygen absorbed in 3 hours at 37° C.	0·04	0·028
Hardness—Total	12·5	8·75
,, Temporary	8·0	5·60
,, Permanent	4·5	3·15
Poisonous metals—Lead	*Nil*	
Copper ...	*Nil*	

REMARKS.

From a consideration of the above data no exception can be taken to the use of this water for domestic purposes.

Sewage and Sewage Effluents.

The same processes used for water analysis are employed for the analysis of effluents and sewages, only it is necessary to dilute the effluent five times when estimating the ammonias. 100 c.c. are made up to 500 c.c. with ammonia free water. It is a good plan to collect 200 c.c. of distillate in a graduated flask, and after mixing well Nesslerize 50 c.c., multiplying the result by 4. As a precautionary measure, a further 50 c.c. should be collected and tested to see if any ammonia remains.

The most important estimations are: (1) The solids in suspension; (2) the rate at which the effluent after filtration absorbs dissolved oxygen.

The first is estimated by filtering through a weighed filter paper 1 litre of the effluent. After washing and drying the residue it is again weighed.

The second estimation is done by Winkler's process (*q.v.*).

Oxygen absorbed.—It is usual to determine this for three minutes and also for four hours at laboratory temperature. For this purpose 100 c.c. of the effluent are placed in a bottle, add 25 c.c. H_2SO_4 (1 in 4), then run in the permanganate solution during three minutes so that a faint pink coloration is always present. Read the burette. Now run in the remaining permanganate in the burette (making 50 c.c. in all) and allow to stand for four hours. After this period add the KI and continue as in water analysis.

The Royal Commission on Sewage Disposal recommended that an effluent (1) should not contain more than three parts per 100,000 of suspended matter. (2) That after being filtered through paper it should not absorb more than (*a*) ·5 part by weight per 100,000 of dissolved or atmospheric oxygen in twenty-four hours; (*b*) 1·0 part by weight per 100,000 of dissolved or atmospheric oxygen in forty-eight hours; (*c*) 1·5 part by weight per 100,000 of dissolved or atmospheric oxygen in five days.

The following are characteristics of an average effluent:—

(1) Cl does not exceed 10 parts per 100,000.

(2) Oxygen absorbed in four hours at 60° F. (or two hours at 80° F.) and the saline ammonia do not exceed 1·5 part per 100,000.

(3) The albumenoid ammonia should not exceed ·15 part per 100,000.

(4) A satisfactory effluent should be without fæcal odour and should be but slightly coloured or turbid.

(5) The final effluent must not be liable to putrefaction or secondary decomposition. Incubated at 37° C. for five days, it should not have any odour.

(6) Generally, high nitrates are a good feature, showing that the oxidation of a considerable amount of nitrogenous organic matter has taken place.

The following figures were obtained from analyses of sewage and effluents :—

	SEWAGE Parts per 100,000	EFFLUENT Parts per 100,000
Total solids	134	86·7
Solids in solution ...	99·3	82·8
,, suspension ...	34·6	3·87
Chlorine	11·0	10·0
Free and saline ammonia	9·6	1·68
Albuminoid ammonia ...	2·6	·29
Nitrites	trace	large trace
Nitrates	2·31	2·12
Oxygen absorbed in 4 hrs. at 60° F.	8·13	1·64
Dissolved oxygen absorbed in 24 hrs. ...	—	·44
Dissolved oxygen absorbed in 48 hrs. ...	—	·56
Dissolved oxygen absorbed in 5 days ...	—	1·42

For the guidance of students the following scheme will permit of a rapid analysis of a water.

Total Solids.—Evaporate 50 c.c. in a weighed porcelain dish.

Nitrates.—Evaporate 50 c.c. of sample to dryness and follow by evaporating 5 c.c. of standard KNO_3.

Oxygen absorbed.—*Ammonias :* Clean the apparatus by distillation of ammonia-free water. While this is proceeding estimate *chlorine*.

While the estimation of the *saline ammonia* is proceeding, start to boil the alkaline permanganate solution, so that there may be no delay in estimating the *albuminoid ammonia*. Nesslerize each cylinder as soon as it is collected. Employ any spare time by testing for *poisonous metals*.

If time permits estimate the total, temporary and permanent hardness.

PART IV.—ANALYSIS OF FOODS AND BEVERAGES.

Milk Analysis.

The average composition of pure cow's milk is as follows:—

Water			87·4
Fat		3·73	Total solids = 12·6
Lactose	4·50	= solids not fat 8·87	
Proteins	3·62		
Mineral matter	0·75		
			100·0

The specific gravity averages about 1032 at 15° C.

The mineral matter consists of the chlorides of K and Na, the citrates and phosphates of K, Mg, Ca, and a small percentage of lime combined with proteids.

Estimation of Total Solids.

Weigh a small flat-bottomed porcelain dish.

Pipette into it 10 c.c. of milk and re-weigh.

Evaporate to dryness on a water bath for one hour and then dry in a water oven for one to one and a quarter hours. Cool in a desiccator and weigh.

Calculate the percentage on 100 grm. milk.

Example:—

Dish + 10 c.c. milk	33·650	Dish + T.S. ...	24·741
Dish	23·437	Dish	23·437
Weight of milk...	10·213 grm.	Weight of T.S. =	1·304 grm.

100 grm. milk will therefore contain $\frac{1\cdot304 \times 100}{10\cdot213} = 12\cdot76$ per cent. total solids.

Estimation of the Ash or Mineral Matter.

The residue from above is heated on a porcelain triangle until the ash is white. When charred it should be stirred with a platinum wire or a glass rod to facilitate the burning of the organic matter.

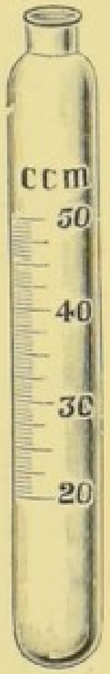
Cool in the desiccator and weigh.

Example :	
Dish + ash	23·513
Dish	23·437
Weight of ash	0.076

100 grm. of milk will therefore contain $\frac{100 \times \cdot076}{10\cdot213} = \cdot74$ per cent.

Estimation of Fat—Werner-Schmidt Process.

Pipette 10 c.c. of milk of known weight into a Stokes' tube, add 10 c.c. conc. HCl and heat cautiously till the liquid becomes dark brown.



Stokes' Tube.

When cold fill up to the 50 c.c. mark with ether.

Shake several times and allow the ether to settle out.

Read off the volume of the ether.

Pipette out 10 c.c. into a small weighed glass flask.

Evaporate the ether, dry in the water oven for an hour. Cool and weigh.

If a Stokes tube is not available then the milk is heated in a 6 × 1 in. test tube. This is transferred when cold to a stoppered graduated 100 c.c. cylinder. The tube is washed out with the ether, transferred to the cylinder and the volume made up to 50 c.c., and the process continued as above.

Example : Volume of ether 26 c.c.

Flask and fat, 23·571. In 10 c.c. ether there is ·15 of fat
Flask, 23·421 In 26 ,, ,, ,, $\frac{\cdot15 \times 26}{10} = \cdot39$
0·150
Amount of fat in 10·213 grm. milk (10 c.c.) = ·39
,, ,, 100 ,, ,, $= \frac{\cdot39 \times 100}{10\cdot231} = 3\cdot81$ per cent.

Solids not Fat.

Subtract the fat from the total solids.

Example : 12·76 − 3·81 = 8·95 per cent. S.N.F.

Colouring Matter.

(1) **Annatto** is largely used to impart a creamy appearance to milk. It is obtained from the seeds of the plant *Bixa orellana*.

It may be detected in milk by adding sodium bicarbonate to some milk in a porcelain dish and then immersing a strip of filter paper for twelve hours. The paper acquires a pink tint.

(2) **Coal tar dyes** are also employed. Their presence may be detected by adding a few c.c. of conc. HCl to some milk in a test tube. A pink colour is produced.

Preservatives in Milk.—The most common are boric acid and formaldehyde.

(1) *Test for Boric Acid.*—Acidify 3 c.c. of milk in a test-tube with dilute HCl. Mix well and place a drop on a turmeric paper. Dry the paper on a watch-glass in the water oven. A red spot is obtained if boric acid is present, which turns greenish black on the addition of caustic alkalies.

(2) *Test for Formaldehyde.*—In a test tube place 10 c.c. milk, add 10 c.c. conc. HCl and one drop of ferric chloride. Stir well with a glass rod and heat up to boiling. A violet coloration denotes formaldehyde.

Estimation of Boric Acid—Richardson and Walton Process.[1]

To 50 c.c. of milk add 5 c.c. of fresh 5 per cent. $CuSO_4$ solution. Stir well and heat to boiling for a few seconds. Filter and wash the coagulum four or five times with boiling water. To the cold filtrate add 2 c.c. of 1 per cent. neutralized solution of phenolphthalein in alcohol. Then add $\frac{N}{10}$ NaOH till a certain blue shade appears. Now add $\frac{1}{3}$ of the volume of glycerine and titrate with $\frac{N}{10}$ NaOH till the blue shade again appears. Read the burette. Under these conditions

$$1 \text{ c.c. } \tfrac{N}{10} \text{ NaOH} = \cdot 0071 \text{ grm. } H_3BO_3$$

Example: Required 6 c.c.

Hence 6 × ·0071 = ·0426 grm. H_3BO_3, and this is in 50 c.c. milk. In 100 c.c. milk = ·0426 × 2 = ·0852 per cent. boric acid.

Estimation of Lactose (Milk Sugar)—Pavy's Method.

This is conveniently estimated by using Pavy's method, which is a modification of Fehling's process. The copper oxide is retained in solution. A 300 c.c. boiling flask is fitted with a double bored cork. Through one hole a small

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[1] *Analyst*, 38, No. 445.

piece of glass tubing passes which is connected to a burette by a small piece of rubber tubing. Through the other hole a small piece of bent glass tubing is inserted and this is connected with rubber tubing to two Woulff's bottles containing dilute H_2SO_4 to absorb the ammoniacal vapours which will come off during the process.

50 c.c. of Pavy's solution (Pavy's solution is prepared by adding 300 c.c. of concentrated ammonia to 120 c.c. of the mixed Fehling solutions, 400 c.c. of 12 per cent. caustic soda are then added and the volume made up to 1,000 c.c. with water) measured in a graduated cylinder are placed in the flask; the burette is filled with the diluted milk solution made up as follows: 5 c.c. milk, 45 c.c. conc. NH_4OH and 50 c.c. H_2O. The Pavy solution is heated up to boiling point and the diluted milk solution is run in 1 c.c. at a time until the blue colour is discharged, the liquid being maintained at a gentle ebullition during the process. 50 c.c. Pavy solution = ·0481 lactose.

Example : Required 25 c.c.

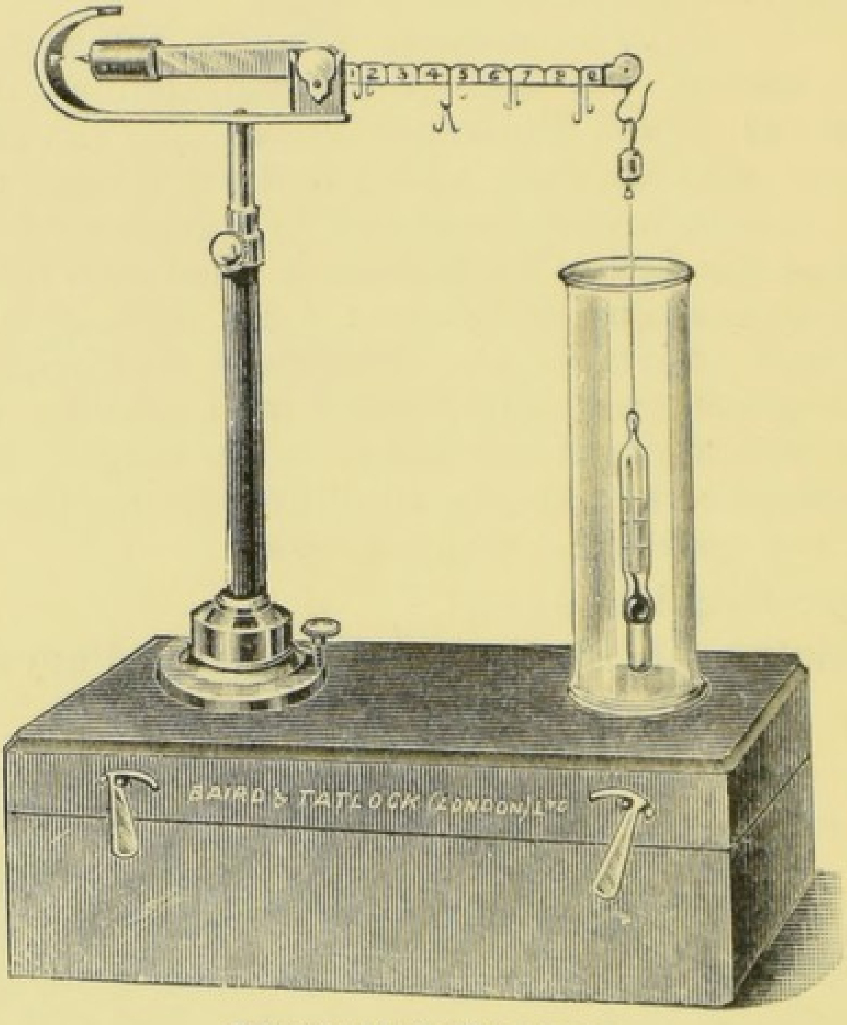
25 c.c. diluted milk = ·048 grm. lactose (50 c.c. Pavy solution)
100 c.c. ,, ,, $= \frac{·048 \times 100}{25}$
100 c.c. ,, ,, = 5 c.c. orginal milk
100 c.c. ,, ,, $= \frac{·048 \times 100 \times 100}{25 \times 5}$
= 3·84 per cent.

Fehling's Method.—Pipette 20 c.c. milk into a 100 c.c. graduated flask; add a few drops of acetic acid, mix well, place on a water bath for a few minutes, cool and make up to 100 c.c. with water. Shake well and filter. Fill up a burette with the filtrate.

Fehling's solution is prepared by dissolving: (A) 34·639 grm. pure sulphate of copper and making up a volume of 500 c.c. with water; (B) 150 grm. of potassio-tartate of soda (Rochelle salt) and 50 grm. of KOH are dissolved in water and made up to 500 c.c.

When required for use equal parts of A and B are mixed.

In a porcelain basin place 10 c.c. of Fehling's solution (5 c.c. of A and B). Add 40 c.c. of water. Boil, run in the diluted milk solution until a few drops of the liquid taken out cease to give a blue colour with the starch and KI indicator. For this latter purpose dissolve ·5 grm. of starch in water by boiling; 10 grm. of KI are then dissolved in water. The two solutions are mixed and made up to 100 c.c. This constitutes the indicator. A series of test tubes are prepared, each containing 2 c.c. of the indicator, and a drop of acetic acid. As long as any $CuSO_4$ remains unreduced, copper iodide and free iodine are produced so that the blue colour with the starch and iodine appear. As lactose reduces the Fehling somewhat slowly it is necessary to boil the liquid for a few minutes after the addition of each amount of diluted milk.



Westphal's Balance.

The Westphal Balance has a swinging arm resting on a knife edge. Part of the arm is graduated into nine divisions, at the end of which is a hook for suspending a glass plummet by thin platinum wire. At the other extremity of the arm is a metal pointer. When using the apparatus this point must be exactly opposite another point fixed on the frame of the balance. This is effected by turning the screw fixed at the base of the instrument whilst the plummet is immersed in distilled water at 15° C. When the apparatus is regulated fill the cylinder with the fluid under examination, and place on the different riders until the two points are opposite. The three riders indicate units, tens and hundreds, the latter being the largest rider. If the largest rider is on 8, the next one on 6, and the smallest one on 4, the specific gravity would be ·864.

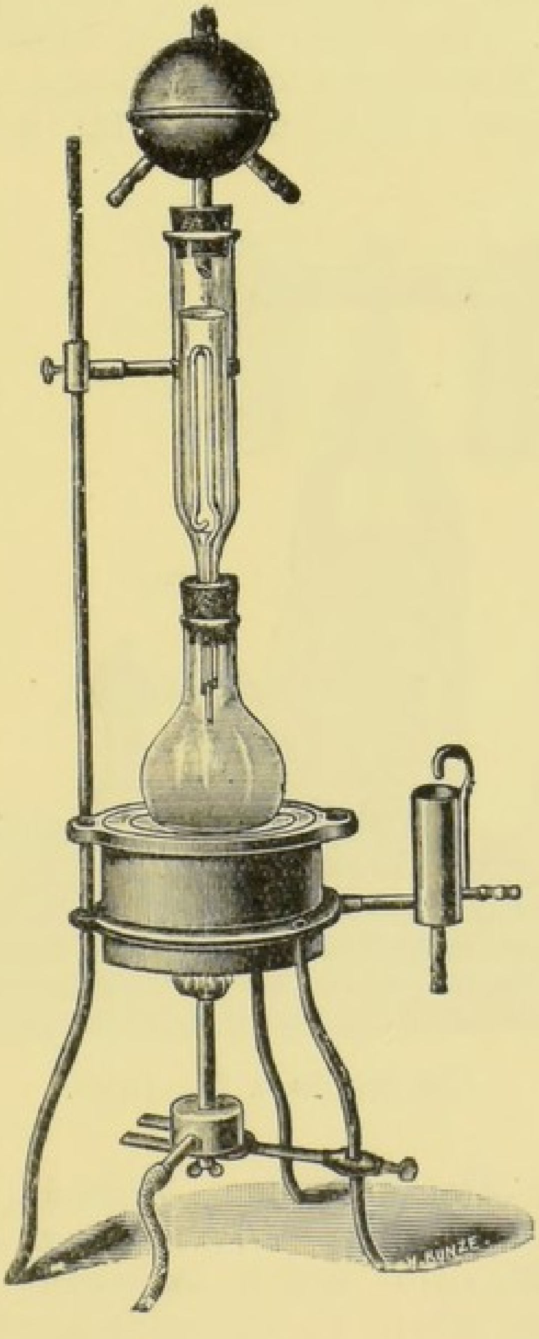
10 c.c. Fehling's solution = ·0678 grm. lactose.
= ·05 ,, glucose.
= ·0475 ,, cane sugar (after inversion).
= ·0807 ,, maltose.
= ·045 ,, dextrose from starch.

Example:—Required 7·2 c.c. of diluted milk solution.

7·2 c.c.................... = ·0678 grm. lactose.
100 c.c. .... = $·0678 \times \frac{100}{7·2}$,,

But this contains 20 c.c. sample of milk.

$$\therefore \text{100 c.c. ,, ,,} = \frac{·0678 \times 100 \times 100}{7·2 \times 20} = 4·7\% \text{ lactose.}$$

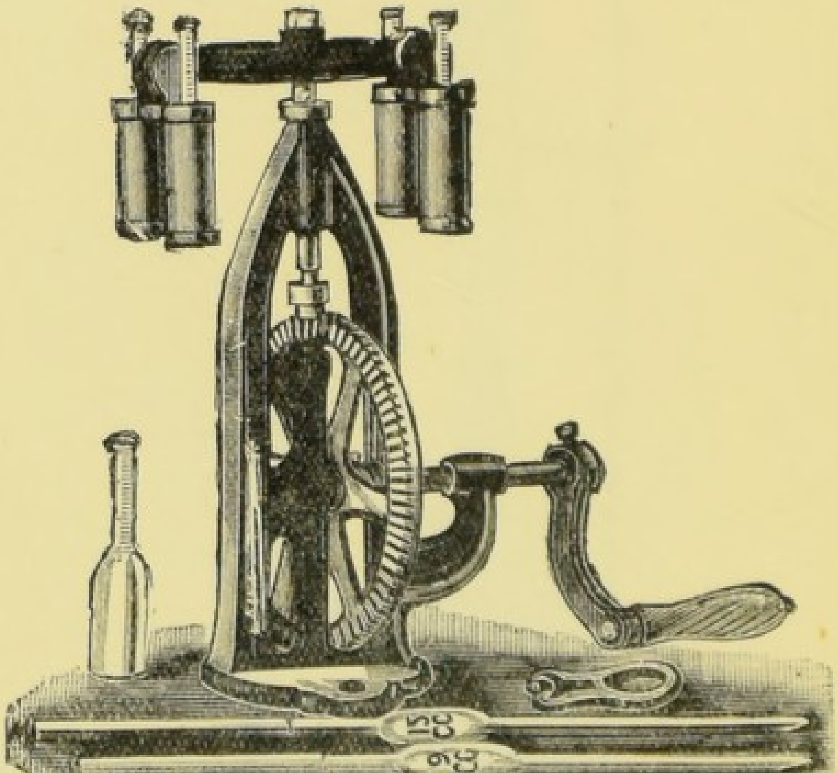


Soxhlet's Apparatus.

The specific gravity of milk may be determined by (*a*) Westphal's balance, (*b*) Sprengel tube, (*c*) S.G. bottle, (*d*) lactometer. The gravity of pure milk usually falls between 1028 and 1034.

Other Methods of Estimating Fat in Milk.

(1) **Adam's Process.**—Pipette 10 c.c. of milk of known weight on to a special fat-free strip of paper (Adam's coil). Hang it up to dry; roll it up and finally dry in a water oven for an hour. Extract in a Soxhlet apparatus with ether for about four hours, receiving the ether in a weighed extraction flask. The ether is then distilled off, and the flask dried in a water oven for one to one and a half hours and the increased weight due to fat is ascertained by weighing.



Centrifugal Machine.

(2) **Leffmann—Beam Process.**—Special bottles are used in this process, the necks of which are graduated from 0 to 80 divisions, 10 of which correspond to 1 per cent. of fat by weight when 15 c.c. milk are taken. In one of these bottles introduce 15 c.c. milk, add 3 c.c. of a mixture of equal parts of fusel oil and conc. HCl. Mix well, then run in slowly 9 c.c. of commercial H_2SO_4, shaking continuously. The liquid becomes hot and

assumes a dark brown colour. Fill up the bottle nearly to the zero mark with a hot mixture of 1 part of sulphuric acid and 2 parts of water. Whirl in a centrifuge for two minutes, then read off the number of divisions occupied by the fat, taking the bottom of the meniscus. Thus if the fat column is 36 divisions the percentage of fat in the milk is 3·6.

Knowing the specific gravity and the fat in a sample of milk the total solids can be calculated by using *Richmond's milk scale* or slide rule. On the right of the sliding part is a small arrow which is placed opposite the fat found. The gravity is next read off and opposite it the total solids will be indicated (see figure, p. 50).

The scale is based on the formula :—

$$\text{T.S.} = \cdot 25\,G + 1\cdot 2\,F + \cdot 14$$

G = the last two figures of the gravity and F is the fat.

Example : S.G. 1031. Fat 3·5

$$\begin{aligned}\text{T.S.} &= \cdot 25 \times 31 + 1\cdot 2 \times 3\cdot 5 + \cdot 14\\ &= 7\cdot 75 + 4\cdot 20 + \cdot 14\\ &= 12\cdot 09.\end{aligned}$$

Calculation of the Amount of Added Water and Deficiency in Fat.—These are obtained from two simple formulæ :—

(*A*) $\dfrac{8\cdot 5 - \text{S.N.F.}}{8\cdot 5} \times 100$ = percentage added water.

S.N.F. = solids not fat found in the sample, *e.g.*, 7·5.

Example : $\dfrac{8\cdot 5 - 7\cdot 5}{8\cdot 5} \times 100 = 11\cdot 7$ per cent. added water.

(*B*) $\dfrac{3 - F}{3} \times 100$ = percentage deficient in fat.

F = fat found in sample, *e.g.*, 2·7.

Example : $\dfrac{3 - 2\cdot 7}{3} \times 100 = 10$ per cent. deficient in fat.

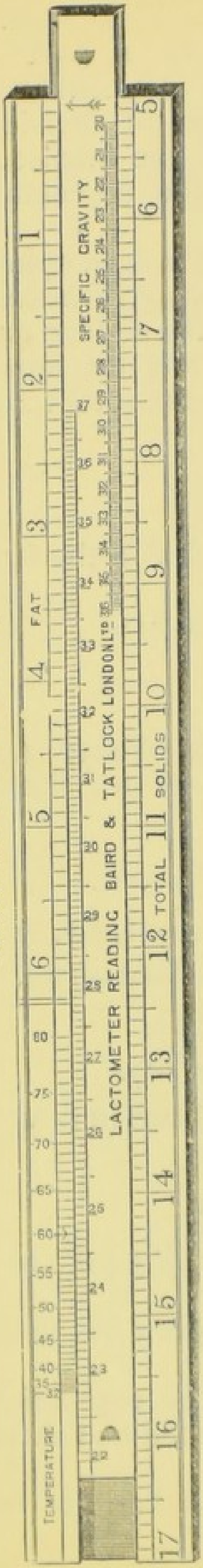
Regulations respecting Milk.

(1) The solids not fat must not be less than 8·5 per cent. (Sale of Milk Regulations, 1901).

(2) The milk-fat must not be less than 3 per cent. (Sale of Milk Regulations, 1901).

(3) No preservative is allowed in milk. The expression "milk" includes separated, skimmed, condensed and dried milk (Milk and Cream Regulations, 1912).

(4) In separated or skimmed milk the solids not fat



RICHMOND'S MILK SCALE.

must not be less than 8·7 per cent. (Sale of Milk Regulations, 1912).

Cream.—No preservative or thickening substance may be added to cream which contains less than 35 per cent. by weight of milk fat. The only preservatives permitted in cream containing 35 per cent. of milk fat or more are boric acid, borax, or a mixture of these, and hydrogen peroxide. In such cases the cream must bear a label stating the nature of the preservative, and that it does not exceed a certain percentage (Milk and Cream Regulations, 1912). The amount should not exceed ·25 per cent. H_3BO_3 from October to May, or 4 per cent. during the other months.

Confirmatory Tests for Formaldehyde.

Acidify 100 c.c. milk in a flask with a little dilute H_2SO_4. Connect up with a condenser and distil about 5 c.c.

(1) To 1 c.c. of the distillate add a drop of Schift's reagent. A pink colour appears in the presence of formaldehyde. Schiff's reagent is a solution of rosaniline the colour of which has been discharged by SO_2.

(2) To 1 c.c. of the milk distillate add 3 c.c. of a 10 per cent. solution of phloroglucine. Shake and add a few drops of KOH. A pink colour is obtained if formaldehyde is present. It may be necessary to warm the tube.

(3) 1 grm. phenylhydrazine hydrochloride and $1\frac{1}{2}$ grm. sodium acetate are dissolved in 10 c.c. H_2O. Four drops of this reagent and an equal amount of H_2SO_4 are added to 1 c.c. of the distillate. A green colour denotes formaldehyde.

Quantitative Estimation of Formaldehyde.

(1) **Ammonia Method (Legler).**—Distil 100 c.c. of milk after acidifying with dilute H_2SO_4. The distillate is then transferred to a pressure bottle, an excess of $\frac{N}{10}$

ammonia is then added and the bottle immersed in a boiling water bath for half an hour. At the same time and under identical conditions a blank experiment is made with the $\frac{N}{10}$ ammonia. Each bottle is then titrated with $\frac{N}{10}$ H_2SO_4, using methyl orange as indicator.

The reaction is as follows :—

$$4\,NH_3 + 6\,\overset{[\textit{Formaldehyde}]}{H\cdot COH} = 6\,H_2O + \overset{[\textit{Hexamethylene-tetramine}]}{N_4(CH_2)_6}$$

Whence—

68 grm. NH_3 are equivalent to 180 grm. formaldehyde.
∴ 17 ,, ,, ,, 45 ,, ,,
i.e., 45 ,, formaldehyde per litre = N solution.
4·5 ,, ,, ,, = $\frac{N}{10}$,,
∴ 1 c.c. of $\frac{N}{10}$ ammonia = ·0045 grm. formaldehyde.

(2) **Iodometric method (Romijn).**—The distillate obtained as above is transferred to a glass stoppered bottle. Add 30 c.c. of normal caustic soda (or potash) and then decinormal iodine from a burette until the fluid remains bright yellow. Shake vigorously for one minute. Add 40 c.c. of normal H_2SO_4 and titrate excess of iodine with $\frac{N}{10}$ thiosulphate.

1 c.c. $\frac{N}{10}$ iodine = ·0015 grm. formaldehyde.

Do a blank experiment with the reagents.

The equation is :—

$$\overset{[\textit{Formaldehyde}]}{CH_2O} + I_2 + 2\,NaOH = 2\,NaI + \overset{[\textit{Formic acid}]}{H\cdot COOH} + H_2O$$

Whence—

30 grm. formaldehyde are equivalent to 2 × 127 grm. iodine
∴ 15 ,, ,, ,, ,, 127 ,, ,,
i.e., Normal formaldehyde contains 15 grm. per litre
$\frac{N}{10}$,, ,, 1·5 ,, ,,
∴ 1 c.c. of $\frac{N}{10}$ iodine is equivalent to ·0015 grm. formaldehyde.

Table showing the Composition of Different Kinds of Milk.

Kind of milk	Water	Fat	Proteins	Ash	Lactose	Author
Cow	87·4	3·73	3·62	·75	4·5	Richmond.
Human... ...	87·41	3·78	2·29	·31	6·21	König.
Goat	85·71	4·78	4·29	·76	4·46	,,
Ewe	80·82	6·86	6·52	·89	4·91	,,
Mare	90·78	1·21	1·99	·35	5·67	,,
Ass	89·64	1·64	2·22	·51	5·99	,,
Sow	89·60	4·8	1·3	·9	3·4	Vieth.
Dried cow's milk	1·46	30·00	27·69	5·57	35·28	Author.
Humanized milk	88·98	3·36	1·22	·24	6·20	,,

For analyses of some dried milks and patent foods, see *Journ. of State Med.*, 1912, vol. xx, No. 11, p. 696.

Analysis of Condensed Milk.

A 10 per cent. solution is made and the analysis is made in the same way as ordinary milk. For the total solids and the proteins take 15 c.c. of the solution; for the fat, 10 c.c. The fat, ash, proteins and lactose subtracted from the total solids give the cane sugar. In the case of machine-skimmed milks it is advisable to use the Adams process owing to the small percentage of fat.

The following are the average figures of a full-cream condensed milk:—

Water	25·2 per cent.
Fat	11·0 ,,
Proteins	9·07 ,,
Ash	1·90 ,,
Lactose	13·40 ,,
Cane sugar	39·43 ,,

The Analysis of Butter.

Butter has the following approximate composition:—

Water	12·0 per cent.
Fat	85·0 ,,
Curd and salt	3·0 ,,
	100·00

Estimation of Water.

Weigh a porcelain dish with a glass rod. Weigh in it 20 grm. of butter. Place on a sand bath and heat with a rose burner, stirring continuously. The fat melts and becomes quite clear and the curd and salt settle down. Heating is continued until the curd commences to turn brown. This is best seen by removing the dish from time to time and scraping the froth aside, when the deposit can be examined. Cool and weigh. The loss in weight represents water.

Estimation of Fat.

Melt the contents of the dish and decant off the fat. Treat the residue three times with 25 c.c. successive portions of ether, stirring well and decanting the ether. The residue, consisting of curd and salt, is dried in the water oven and finally weighed.

The difference from the previous weight will give the fat.

Example :—

Water :

Dish + rod + butter	47·3500
Dish + rod	27·3500
Wt. of butter	20·0000 grm.
d + r + b (before drying)	47·3500
ditto (after)	45·0850
Loss	2·2650

2·265 × 5 = 11·32 per cent. water.

Curd and Salt :

d + r + curd and salt	27·965
d + r	27·350
	0·615

·615 × 5 = 3·07 per cent. curd and salt.

Fat :

d + r + fat + c + s	= 45·0850
d + r + curd and salt	= 27·9650
Wt. of fat removed	17·1200

17·12 × 5 = 85·6 per cent. fat.

Estimation of Salt (NaCl).

The curd and salt residue is carefully charred. The residue is dissolved in 20 c.c. of warm water, filtered and made up to 100 c.c. Shake and pipette out 25 c.c. into a porcelain basin, add a few drops of potassium chromate and run in $\frac{N}{10}$ $AgNO_3$ from a burette until a reddish tinge is apparent. 1 c.c. $\frac{N}{10}$ $AgNO_3$ = ·00585 NaCl.

Example : Required 10 c.c. silver nitrate solution.
∴ 10 × ·00585 × 4 = ·2340 is amt. of NaCl in 100 c.c
(*i.e.*, 20 grm. butter)
and ·234 × 5 = 1·17 = NaCl per cent.
Curd and salt found = 3·07 per cent.
NaCl = 1·17 ,,
∴ *Curd* = 1·90 ,,

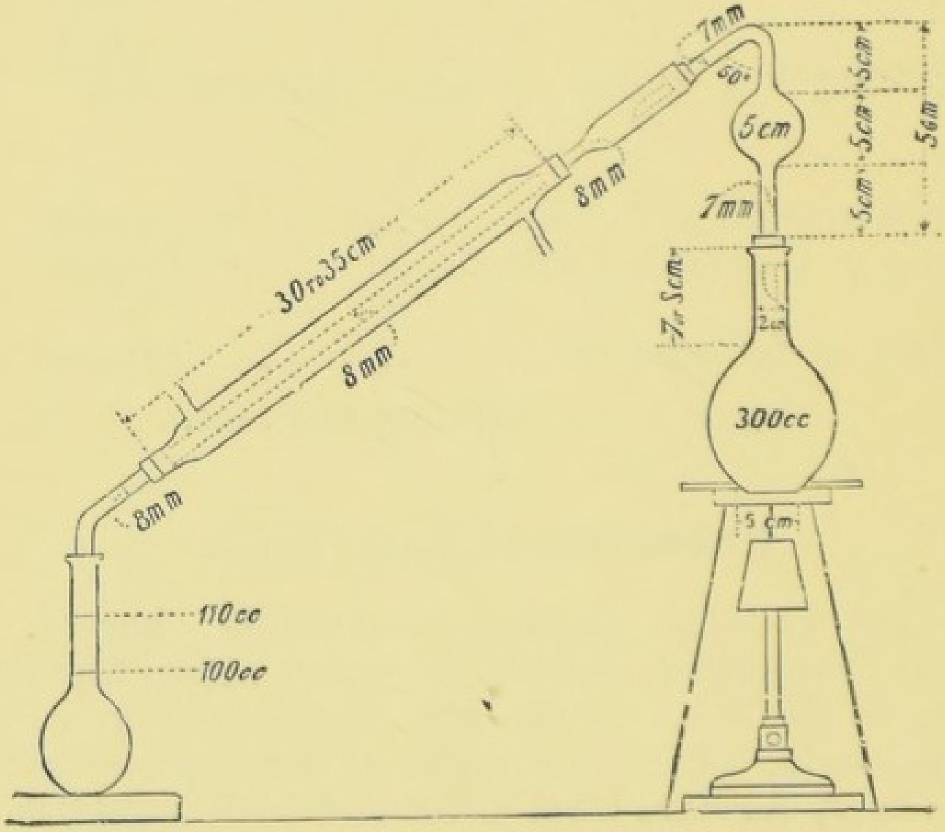
Adding up we get :—

Water	11·33
Fat	85·60
Salt	1·17
Curd	1·90
	100·00

The Reichert-Wollny Number.

This is the number of cubic centimetres of decinormal alkali required to neutralize the soluble volatile fatty acids contained in 5 grm. of filtered fat :—

In genuine butter it averages ...	24—32 c.c.
In margarine it averages	1—4 ,,
In coconut oil it averages	7 ,,


7mm
60°
5cm
5cm
5cm
5cm
5cm
7mm
8mm
30 to 35cm
8mm
7 or 5cm
2cm
300cc
5cm
8mm
110cc
100cc

Reichert-Wollny Apparatus.

In a 300 c.c. flask weigh 5 grm. of filtered fat, add 2 c.c. of 50 per cent. NaOH and 10 c.c. of 92 per cent. alcohol. Attach a long tube to the flask to act as a reflux condenser, and immerse in a boiling water bath for fifteen minutes. Remove the tube and evaporate off the alcohol on the water bath. A dry soap is obtained. This is dissolved in

100 c.c. of distilled water which has been boiled for ten minutes to expel the carbonic acid. Now add 40 c.c. of normal sulphuric acid, a few pieces of pipe-stem, connect up with a condenser and distil over 110 c.c. in a graduated flask. Shake well and filter off 100 c.c., add a few drops of phenolphthalein and titrate with $\frac{N}{10}$ NaOH.

The number of cubic centimetres increased by one-tenth, *i.e.*, $\times \frac{110}{100}$, gives the Reichert-Wollny Number.

It is sometimes necessary to do a blank experiment for the reagents used. This should not exceed ·3 c.c. of $\frac{N}{10}$ alkali, which for all practical purposes may be neglected.

The *soluble volatile fatty acids* in butter are butyric, caproic, caprylic and capric, and the *insoluble non-volatile fatty acids* are myristic, palmitic, stearic and oleic. These acids are not in a free state in butter, but are in combination with glycerine as glycerides. In the Reichert-Wollny process the NaOH saponifies (*i.e.*, transforms into soap) the glycerides, the alcohol keeping the fat in solution. Thus butyl glyceride + NaOH = glycerine + sodium butyrate. The soap composed of the sodium salts of the fatty acids is dissolved in hot water. The addition of the sulphuric acid liberates all the fatty acids of which the volatile ones distil over; on cooling the insoluble fatty acids become solid. Thus sodium butyrate + H_2SO_4 = butyric acid + sodium sulphate.

Sometimes the volatile fatty acids are returned in terms of butyric acid, in which case 1 c.c. $\frac{N}{10}$ NaOH = ·0088 grm. butyric acid [$CH_3—CH_2—CH_2—COOH$]. The usual amount in butter fat varies from 5—8 per cent.

Calculation of the Percentage of Foreign Fat from a given Reichert-Wollny Number.

Example: R.W. No. 17. Fat found by analysis = 85 per cent.

Allowing 2 for margarine we have:—

R.W. No. of 22 (*i.e.*, 24 − 2) = 100 of genuine butter fat.
∴ ,, ,, 15 (*i.e.*, 17—2) = $\frac{100 \times 15}{22}$ = 68·1 ,, ,,

100 fat in sample contain 68·1 butter fat.
∴ 85 ,, ,, ,, $\frac{68·1}{100} \times 85 = 57·88$ butter fat.
Hence 85 − 57·88 = 27·12 per cent. foreign fat.

The Valenta Number.

The Valenta number is the critical temperature at which a mixture of equal parts of butter fat and glacial acetic acid changes from clear to turbid, or *vice versâ*. 3 c.c. of filtered butter fat are run into a test tube and 3 c.c. of glacial acetic acid added. Stir with a thermometer and warm over a small flame. The mixture becomes clear. Allow it to cool, stirring continuously. Read off the temperature at which turbidity reappears. The test should be done two or three times to obtain concordant readings. A mixture of genuine butter fat and glacial acetic acid becomes clear at a temperature between 30°—40° C., whereas margarine under the same conditions does not clear until about 80° C.

The specific gravity of the fat may be ascertained with a special specific gravity bottle containing a thermometer. It is usually taken at 37·8° C. Pure butter fat averages about ·910 to ·9135, whereas margarine yields figures from ·9015 to ·906.

Test for Boric Acid.

Place some butter in a test tube, immerse in a beaker of hot water, decant off the fat. Treat the residue with ether. Pour off the ether. Add 5 c.c. of water, acidify with dilute HCl, mix well and place a spot on a turmeric paper. Dry in the water oven. A red spot, which is turned greenish black with caustic alkalies, indicates boric acid.

Estimation of Boric Acid. Thompson's Process.

In a porcelain dish weigh 20 grm. butter. Melt it on a sand bath, add phenolphthalein and then NaOH until a pink colour is obtained, stirring well. Drive off the water, remove the fat with ether, dry.

Thoroughly char the residue, avoiding too high a temperature. Take up with 20 c.c. hot water, and add about 5 c.c. conc. HCl till all but the carbon is dissolved. Transfer to a 100 c.c. graduated flask, washing out the dish with a little water, taking care that the volume does not exceed 50—60 c.c.

Add half a gramme of calcium chloride, a few drops of phenolphthalein and then 20 per cent. NaOH till a pink colour is obtained. Add 25 c.c. lime-water and make up

to 100 c.c. with water. All phosphoric acid has thus been precipitated as calcium phosphate.

Shake well and filter, receiving the filtrate into a 100 c.c. graduated cylinder. Allow to drain and read off the volume of filtrate. Transfer to a boiling flask and add dilute HCl till colourless. Add a few drops of methyl orange and continue the addition of the acid till a reddish colour is obtained. Bring up to the boil to expel carbonic acid. When cold add dilute NaOH till the red colour changes to yellow. Add 30 c.c. glycerine, forming glycero-boric acid, mix well and titrate with $\frac{N}{10}$ NaOH, using phenolphthalein as indicator, until a pink colour is obtained.

1 c.c. $\frac{N}{10}$ NaOH = ·0062 grm. H_3BO_3.

Example: Required 14 c.c. Volume of filtrate 95 c.c.

$\therefore 14 \times \cdot0062 \times \frac{100}{95}$, which is amount in 20 grm. butter.

$\therefore$ 100 grm. butter contain—

$$14 \times \cdot0062 \times \frac{100}{95} \times 5 = \cdot45 \text{ per cent. } H_3BO_3.$$

The usual amount does not exceed ·5 per cent.

MARGARINE is prepared from beef fat, mutton fat, lard, lard oil, cotton-seed oil, stearin, cocoanut oil, peanut oil, sesame oil and other oils and fats. They are churned up with milk to impart a taste, and colouring matter is frequently added either in the form of annatto or coal tar dyes.

When carefully prepared it is a wholesome substitute for butter, and probably is just as nutritious although perhaps not quite so easily digested.

Regulations concerning Butter and Margarine.

(i) Water not to exceed 16 per cent. In milk-blended butter the limit is 24 per cent., and this must be declared. The name of the article must be approved by the Board of Agriculture and Fisheries (Butter and Margarine Act, 1907).

(ii) The amount of butter fat in margarine must not exceed 10 per cent. (Sale of Food and Drugs Act, 1899).

(iii) The word "butter" shall mean the substance usually known as butter, made exclusively from milk or cream or both, with or without salt or other preservative, and with or without the addition of colouring matter (Margarine Act, 1887).

Estimation of Chicory in a Coffee Mixture.

(i) **By weighing the Extract.**—5 grm. of the dried sample are placed in a beaker and boiled for a quarter of an hour with 100 c.c. water. Allow to settle for two or three minutes. Decant off supernatant liquid through muslin, receiving filtrate in a 250 c.c. graduated flask. Add another 100 c.c. of water to the beaker, boil again for fifteen minutes, strain through muslin; rinse the residue with 10—20 c.c. water. The united liquids when cold are made up to the mark with distilled water. Shake well, filter off through paper about 70—80 c.c. Of this liquid pipette out 50 c.c., evaporate to dryness on the water bath in a weighed porcelain dish. [The lid of a Petri dish is very convenient and the evaporation is rapid.] Dry in the water oven for an hour, cool and weigh. Calculate the extract per cent.

The per cent. chicory = 2 (E — 25) where E = extract per cent.

This formula is obtained from the following equation:—

The percentage extracts of coffee and chicory are 25 and 75 respectively.

$$\begin{aligned} \text{Let } x &= \text{per cent. of chicory;} \\ \text{Then } 100 - x &\quad ,, \quad \text{of coffee.} \\ \frac{75x}{100} + \frac{25(100 - x)}{100} &= \text{E (extract)} \\ 75x + 2{,}500 - 25x &= 100\,\text{E} \\ 50x &= 100(\text{E} - 25) \\ \therefore x &= 2(\text{E} - 25) \end{aligned}$$

Example:

Dish + extract ... 27·632
Dish ... ... 27·098
∴ Weight of extract is ·534 in 50 c.c. solution
= 2·670 in 250 ,, ,,
i.e., in 5 grm. sample.
∴ 2·67 × 20 = 53·4 per cent. extract.
Hence per cent. chicory = 2 (53·4 — 25)
= 56·8 per cent.

(ii) **By taking the Specific Gravity of a 10 per cent. Decoction.**—10 grm. of the dried sample are just brought up to the boil with 100 c.c. water. Filter through paper and determine the S.G. of the filtrate at 15° C. by means of the S.G. bottle.

The S.G. of a 10 per cent. decoction of coffee is 1009·5
,, ,, ,, chicory ,, 1024·5

First weigh the bottle empty and quite dry. Then fill it with the 10 per cent. decoction at 15° C., and weigh. Rinse the bottle out and fill it with distilled water at 15° C., and weigh.

$$\text{The S.G.} = \frac{\text{wt. of decoction}}{\text{wt. of water}}$$

Example :—

Bottle + decoction...	40·375	Bottle + water ...	40·0
Bottle	15·000	Bottle	15·0
Wt. of decoction	25·375	Wt. of water ...	25·0

$$\therefore \text{S.G.} = \frac{25{\cdot}375}{25} = 1{\cdot}015$$

$$\begin{aligned} \text{Whence } 1015 - 1009{\cdot}5 &= 5{\cdot}5 \text{ chicory} \\ 1024{\cdot}5 - 1015 &= 9{\cdot}5 \text{ coffee} \\ &\overline{15{\cdot}0} \end{aligned}$$

$$\text{Chicory} = \frac{5{\cdot}5 \times 100}{15} = 36{\cdot}6 \text{ per cent.}$$

$$\text{Coffee} = \frac{9{\cdot}5 \times 100}{15} = 63{\cdot}3 \quad ,,$$

Composition of Tea and Coffee.

Tea.[1]		*Roasted Coffee.*[2]	
Water ...	5·81 per cent.	Water ...	1·13 per cent.
Extract ...	38·77 ,,	Caffeine ..	1·05 ,,
Tannin ...	14·87 ,,	Saccharine matter ...	·41 ,,
Theine ...	2·70 ,,	Caffeic acid	4·52 ,,
Ash ...	5·62 ,,	Fat and oil...	13·41 ,,
		Legumin and albumin...	13·13 ,,
		Dextrin ...	1·38 ,,
		Cellulose ...	47·42 ,,
		Ash ...	4·88 ,,

Analysis of Flour.

Flour has the following average composition[3] :—

Water	10·62 per cent.
Proteins (N × 5·7)	12·23 ,,
Oil, etc....	1·77 ,,
Crude fibre	2·36 ,,
Ash	1·82 ,,
Carbohydrate other than fibre	71·18 ,,

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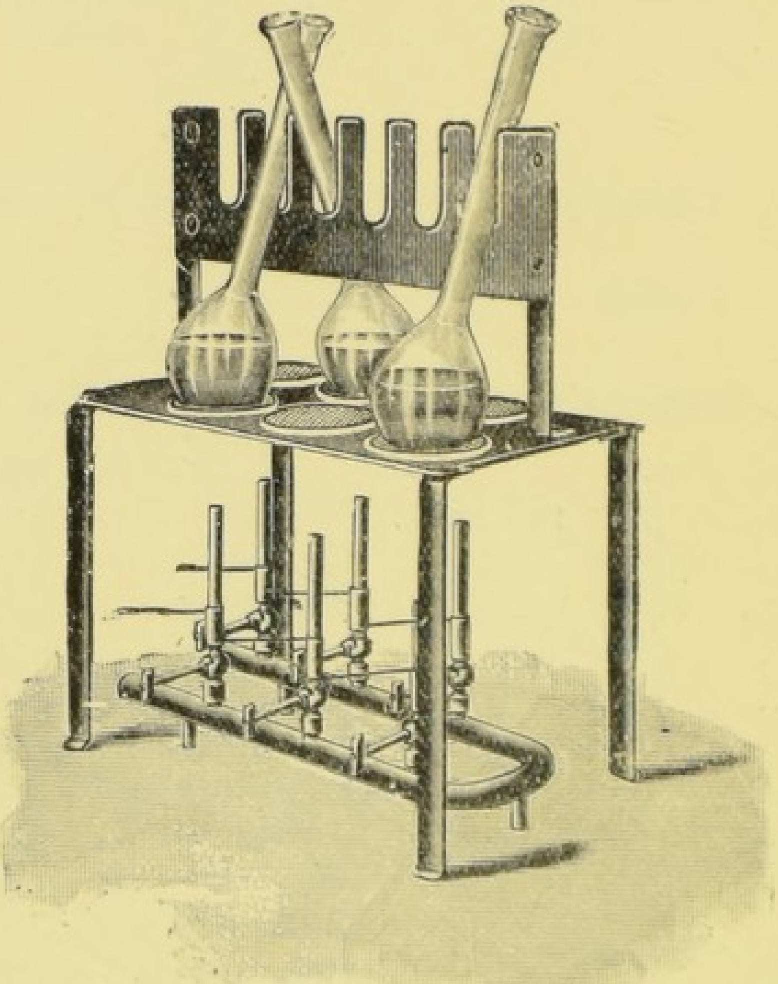
[1] Analysis by Geissler.

[2] Analysis by Bell.

[3] *U.S. Dept. Agricult.*, *Bulletin*, No. 13, p. 1189.

Estimation of Proteins. Kjeldahl's Process for estimating Nitrogen.

In a long-necked Kjeldahl's flask place 1 grm. of flour. Add 10 grm. powdered potassium sulphate, mix and add 20 c.c. of conc. H_2SO_4. Heat cautiously until the brisk reaction has ceased, then maintain the liquid at a gentle ebullition until it is colourless (two to three hours); 0·7 grm. HgO may be added in addition to the potassium sulphate.



KJELDAHL FLASKS.

In a 700 to 1,000 c.c. boiling flask place about 100 c.c. water; pour the contents of the Kjeldahl flask into it and wash out the latter with three successive quantities of water, using 25 c.c. each time. The united liquids are then rendered alkaline by the addition of 70 c.c. of 50 per cent. NaOH measured in a graduated cylinder (and 25 c.c. of 4 per cent. sodium sulphide is added if HgO has been used). Now add a fragment of zinc and connect up with a condenser. The liquid is then distilled and the distillate received

in a flask containing 50 c.c. of $\frac{N}{10}$ H_2SO_4. Distillation is continued until the distillate is free from ammonia (test with litmus paper). Add a few drops of methyl orange and titrate the distillate with $\frac{N}{10}$ NaOH until a straw yellow colour is obtained.

Kjeldahl's process consists in the conversion of nitrogen into ammonium sulphate. When boiled with caustic soda, ammonia is liberated and is estimated by receiving it in a known volume of $\frac{N}{10}$ H_2SO_4, the excess of which is titrated by $\frac{N}{10}$ NaOH.

Example: 35·1 c.c. $\frac{N}{10}$ NaOH

50 − 35·1 = 14·9

14·9 × ·0014 = ·02086 grm. N in 1 grm. flour.

= 2·086 ,, ,, 100 ,, ,,

N per cent. × 5·7 (factor) = proteins.

[100 parts protein contain 5·7 parts of N.]

∴ 2·086 × 5·7 = 11·89 per cent. proteins.

Flour usually contains from 10 to 13 per cent. proteins.

Estimation of Crude Gluten in Flour.

Weigh out 50 grm. flour, place in a large mortar and make a dough with about 25 c.c. water, which should be added gradually; place the dough in the palm of the hand and squeeze out the starch carefully under a stream of running water, taking care to keep the dough together as much as possible. When the water which runs away is quite clear, place the sticky gluten on a weighed watch glass and dry in the water oven until the weight is constant. In order to facilitate drying the gluten after an hour's drying should be cut up into small pieces. Gluten in flour varies from 8 to 10 per cent.

Test for Alum in Flour.

Alum exists normally in flour. It stops fermentation and improves taste and colour of bread. It is little employed to-day. It used to be used in quantities ranging from 4 to 9 grains per pound loaf.

Make a paste with a few grammes of flour in a porcelain dish; add 5 c.c. of 10 per cent. ammonium carbonate and 5 c.c. of freshly prepared tincture of logwood, prepared by treating logwood chips with alcohol. Mix well and add more flour if the paste is too wet. Dry in the water oven. At the same time prepare another lot of flour to which a pinch of alum has been added.

On drying, the flour without alum is practically colourless, whereas the one containing alum remains a permanent lavender colour.

Quantitative Estimation of Alum.

Incinerate $\frac{1}{4}$ lb. of flour or bread to a grey ash. Treat the ash with a little conc. HCl and boiling water. Evaporate to dryness on the water bath, and repeat this treatment a second time. Take up with boiling water, filter and wash the precipitate, consisting of silica, with boiling water. To the filtrate add 5 c.c. of ammonia solution. This precipitates all the phosphates. Then add 20 c.c. of conc. acetic acid. The phosphates of lime and magnesia are re-dissolved, those of iron and aluminium remaining insoluble. Filter and wash precipitate with boiling water. Dry, ignite, weigh (A). This gives iron and aluminium phosphates. Dissolve the residue in HCl, and after dilution estimate the iron colorimetrically as in water analysis. Convert the Fe into ferric phosphate by multiplying by the factor 2·7. Deduct this from the combined weight of iron and aluminium phosphates found above. Difference gives aluminium phosphate which, multiplied by 3·7, gives ammonium alum, *i.e.*, commercial alum.

Calculate result in grains per lb.

Kenwood states that if the amount of alum found represents more than 6 to 10 grains of alum per 4 lb. loaf, then adulteration is indicated.

Analysis of Beer.

Estimation of Alcohol.—In a 300 c.c. distillation flask place 100 c.c. of beer, measured in a graduated flask. Rinse out the measuring flask with 10—20 c.c. water, pour into the distillation flask, add a piece of pipe-stem, connect up with a condenser and distil over about 80 c.c. into a 100 c.c. graduated flask. Make up to the mark with water, shake well and take the specific gravity with the S.G. bottle (*see under* Coffee). Consult tables for the percentage of alcohol.[1] [2] Ordinary beer contains from 3·5 to 5 per cent. alcohol.

Estimation of the Extract.—The fluid remaining in the distillation flask is poured into a 100 c.c. graduated

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[1] Otto Hehner. Alcohol Tables, 1880.

[2] Thomas Stevenson. Tables of Spirit Gravities, 1888.

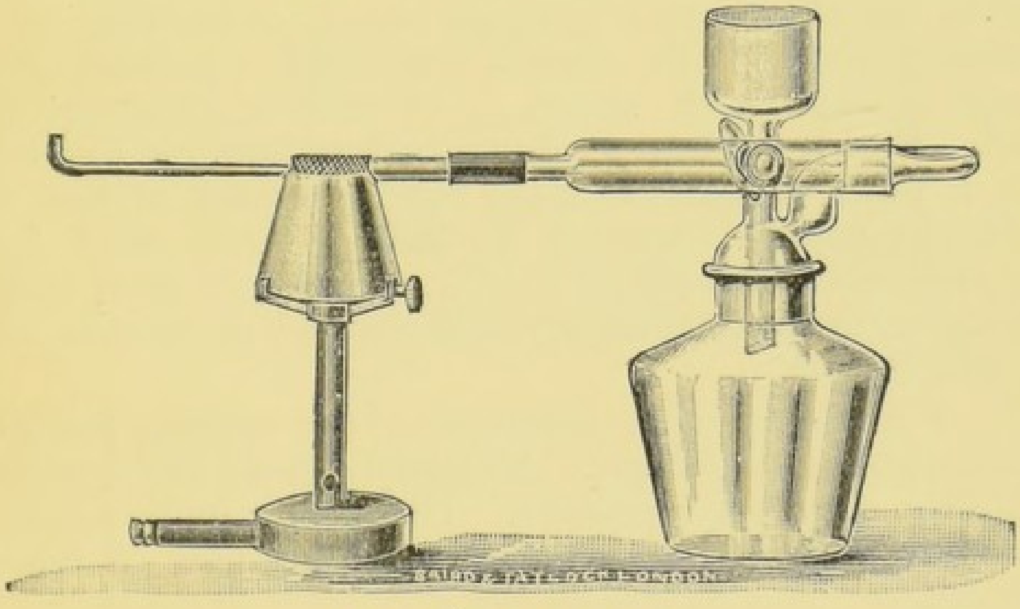
flask, together with the necessary washings, and finally made up to the mark with water. Shake well and determine the specific gravity with the S.G. bottle.

The percentage of extract is obtained by consulting tables (Schultz and Ostermann). The average is about 5 per cent.

Confirmation of the results by the S.G. method can be obtained by evaporating 25 c.c. of beer to dryness on a water bath in a weighed porcelain dish. After drying in the water oven for one hour, and then weighing, the extract per cent. can be calculated.

Test for Arsenic.

(1) Marsh's Test.—A small flask is fitted with a double bored rubber stopper. A small thistle funnel is inserted through one hole, and a piece of tubing which passes through the other hole is connected to a tube containing a coil of lead acetate paper (to retain H_2S), granular calcium chloride (for retaining moisture), and a piece of glass wool. At the end of this tube a finely drawn-out tube is inserted, and a small piece of copper gauze is wrapped round the end nearest the rubber stopper. The tube is supported by a special burner with an iron hood. About 10 grm. of zinc and 25 per cent. sulphuric acid, both arsenic-free, are placed in the flask, and when all the air has been expelled the hydrogen is lit at the drawn-out



Marsh's Apparatus.

point. The small burner is now lit and the flame directed on to the copper gauze. About 5 c.c. of the suspected fluid is now cautiously poured down the thistle funnel. If any arsenic is present a mirror of a brownish appearance is obtained in the cold part of the drawn-out tube. If nothing can be seen after half an hour arsenic may be considered absent.

As this test is very delicate the reagents should be tested to see that they are free from arsenic. The nascent hydrogen forms arsenuretted hydrogen with the arsenic present. This is decomposed by heat into arsenic and hydrogen, the former being deposited on the cold part of the tube.

Arsenic should never be present to the extent of more than $\frac{1}{100}$th grain per gallon.

(2) **Reinsch's Test.**—Place 100 c.c. of the suspected fluid in a boiling flask. Add 15 c.c. conc. HCl and a fragment of copper foil. Boil for half an hour to an hour, replacing the water evaporated during the boiling with distilled water. If arsenic is present it is deposited on the copper, which appears greyish black, according to the amount present. The supernatant fluid is poured off, the copper washed with water, dried with alcohol and then ether. Roll it up into as fine a coil as possible and insert it in a piece of narrow glass tubing sealed at one end. Heat the copper carefully by means of a small flame. The arsenic is oxidized to As_2O_3, which sublimes, forming octahedral or tetrahedral crystals which can be seen under the microscope.

Note: Antimony also deposits on the copper, but gives an amorphous sublimate. Mercury causes the copper to assume a silvery appearance. Under the microscope the sublimate will be seen to consist of globules of metallic mercury.

Estimation of Acetic Acid.

10 c.c. of beer are well diluted with water, a few drops of phenolphthalein added, and the solution titrated with $\frac{N}{10}$ NaOH till a permanent rose colour is obtained.

$$1 \text{ c.c. } \frac{N}{10} \text{ NaOH} = \cdot 006 \text{ grm. acetic acid.}$$

The average is about 0·1 per cent.

Test for Sulphites.

In a test tube place 10 c.c. beer, acidify with dilute HCl and place over the mouth of the tube a starch and potassium iodate paper. Heat the tube up to boiling. A blue colour is obtained if sulphurous acid is present.

Calculation of the Original Gravity of the "Wort."

When beer is exported a rebate is allowed. The duty on beer is calculated from the strength of the "wort" as indicated by its specific gravity. During fermentation the S.G. of the wort is diminished according to the amount of alcohol formed, the latter being approximately half the saccharine matters destroyed by fermentation.

Example :—	1000·0
S.G. of alcoholic distillate ...	995·6
Spirit indication	4·4
(Consulting tables) Gravity lost =	16·8
S.G. of de-alcoholised beer ...	1020·4
Original gravity of wort ...	1037·2

Spirits. Sykes's Hydrometer.

This consists of a metallic hydrometer with special weights. The sample is placed in a cylinder and the hydrometer immersed in the fluid. Different weights are then put on the bottom of the hydrometer until the meniscus comes within the graduated scale divisions on the instrument.

There is only one weight that will do this. The other ones will either be too light or too heavy. Take the temperature of the spirit.

Example: Weight and scale reading, 76·6; temperature, 63° F.

Consulting the tables which accompany the instrument the strength of the spirit is found to be 33·6° under proof.

This means that it contains 33·6 per cent. water
and 66·4 ,, proof spirit.

100·00

100 proof spirit contain 57·06 absolute alcohol by volume
or 49·24 ,, ,, ,, weight.

Hence 66·4 contain $\frac{66·4}{100} \times 57·06 = 37·88$ per cent. alcohol by volume.

This should be verified by actual distillation (as under Analysis of Beer).

Conversely, if the percentage of alcohol is known, we can calculate how much a spirit is over or under proof.

Example: A spirit contains 42 per cent. of alcohol by volume; how many degrees is it under proof?

57·06 of alcohol = 100 proof spirit
42 ,, ,, $= \frac{100 \times 42}{57·06} = 73·6.$

The composition of the spirit is therefore 73·6 proof spirit
26·4 water

100·0

(*i.e.*, 26·4° under proof.)

By 25° over proof is meant that if 25 parts of water are added to 100 parts of the spirit, 125 parts of proof spirit would be produced.

By the Sale of Food and Drugs Act Amendment Act, 1879, it is required that whisky, brandy and rum must not be more than 25° under proof, *i.e.*, shall not contain less than 75 per cent. proof spirit. Gin must not be more than 35° under proof, *i.e.*, shall not contain less than 65 per cent. proof spirit.

PART V.—DISINFECTANTS AND PRESERVATIVES.

Estimation of Carbolic Acid in Carbolic Powder.

In a hard glass retort place 50 grm. of the powder. Incline the retort, heat it directly with the flame, shaking from time to time, and receiving the distillate in a small flask. This will contain water, tar oils (if present), and phenols. When distillation is complete saturate the distillate with salt and decant off the supernatant fluid into a burette, washing out the flask with saturated salt solution. Allow the phenols and tar oils to separate. Read off their volume. Run off the aqueous part and treat (in the burette) the oily layer with twice its volume of 9 per cent. NaOH. The phenols are dissolved, forming phenates or carbolates. The tar oils are not dissolved and will settle out if present, and their volume can be read off. This result subtracted from the first burette reading will give the phenols. If no tar oils are present run off the phenate solution on to a small filter paper and receive the filtrate in a small flask. Acidify with concentrated HCl, saturate with salt and transfer supernatant liquid again to burette, wash out flask with saturated salt solution. Allow the phenols to separate and read off their volume.

The percentage of phenols multiplied by 1·05 = phenols by weight. A good powder should contain not less than 15 per cent. phenols and should be made with some inert substance (china clay, &c.) and not with lime or soda.

Estimation of Salicylic Acid in Lime Juice.

25 c.c. of lime-juice are shaken in a separating funnel with 25 c.c. of ether. The aqueous part is run off and the ether collected in a small flask. The aqueous portion is then treated again with a similar quantity of ether, and after separation the second ether portion is collected in the same flask. The united ether layers are then slowly evaporated. Air is pumped into the flask to remove the last traces of ether, and if salicylic acid is present crystals

may be seen. Dissolve in 40—50 c.c. warm water, and make up to 100 c.c. in a graduated flask. Shake well and pipette out 10 c.c. into a 100 c.c. Nessler glass, make up to the mark with water and add 2 c.c. of 1 per cent. ferric chloride. A violet colour is obtained in the presence of salicylic acid. This coloration is matched by means of standard salicylic acid solution (1 c.c. = ·00025 grm. of salicylic acid) in the same manner as poisonous metals in water. If 10 c.c. of the solution produces too intense a colour with the iron chloride, a less amount must be taken, and if the coloration is faint it is advisable to take more.

Example: Required 6·2 c.c. of standard salicylic acid for 10 c.c. of the diluted solution.

Salicylic acid
6·2 c.c. standard salicylic acid solution = 6·2 × ·00025 grm.
= ·00155 „
10 „ of diluted sample ... ... = ·00155 „
100 „ „ „ ... ... = ·0155 „
i.e., 25 c.c. of lime-juice ... = ·0155 „
∴ 100 „ „ ... = ·0620
Grm. per 100 c.c. × 87·5 = grains per pint
Whence ·062 grm. × 87·5 = 5·42 grains per pint

Some lime-juices and lime-juice cordials contain as much as 7 grains per pint of salicylic acid (·08 per cent.). For tests for citric and tartaric acids, see p. 7.

Qualitative Tests for Disinfectants.

(1) **Corrosive Sublimate. $HgCl_2$.**

Hg. (*a*) Sulphuretted hydrogen gives a black precipitate.

$$H_2S + HgCl_2 = HgS + 2\,HCl$$

(*b*) Potassium iodide gives a red precipitate, soluble in excess.

$$2\,KI + HgCl_2 = 2\,KCl + HgI_2$$
$$2\,KI + HgI_2 = HgK_2I_4$$

(*c*) Stannous chloride gives a white precipitate turning to grey.

$$SnCl_2 + 2\,HgCl_2 = 2\,HgCl + SnCl_4$$
$$2\,HgCl + SnCl_2 = 2\,Hg + SnCl_4$$

Cl. Add sodium carbonate solution, filter, acidify filtrate with nitric acid, then add silver nitrate. A white curdy precipitate is formed, soluble in ammonia.

$$2\ HgCl_2 + Na_2CO_3 = HgCO_3 \cdot HgO + 2\ NaCl$$
$$NaCl + AgNO_3 = AgCl + NaNO_3$$

(2) **Ferrous Sulphate. $FeSO_4$.**

Fe. (*a*) Potassium ferricyanide gives a deep blue precipitate known as Turnbull's blue.

$$3\ FeSO_4 + 2\ K_3\ Fe\ (CN)_6 = Fe_3\,[Fe(CN)_6]_2 + 3\ K_2SO_4$$

(*b*) Solution of ammonia gives a whitish precipitate which turns to dirty green and then brown.

$$FeSO_4 + 2\ NH_4OH = Fe(OH)_2 + (NH_4)_2\ SO_4$$

SO_4. Acidify with dilute HCl, add barium chloride. A white precipitate is formed, insoluble in all acids.

$$BaCl_2 + FeSO_4 = BaSO_4 + FeCl_2$$

(3) **Carbolic Acid. C_6H_5OH.**

(*a*) Ferric chloride gives a violet colour.

(*b*) Bromine water gives a yellowish-white precipitate of tribromophenol.

$$C_6H_5OH + 3\ Br_2 = C_6H_2Br_3OH + 3\ HBr$$

(*c*) Add a drop of nitric acid, warm, dilute with water, then add ammonia. A yellow colour due to ammonium picrate is obtained.

$$C_6H_5OH + 3\ HNO_3 = C_6H_2OH\ (NO_2)_3 + 3\ H_2O$$
$$C_6H_2OH(NO_2)_3 + NH_4OH = \overset{[\textit{ammon. picrate}]}{C_6H_2ONH_4(NO_2)_3} + H_2O$$

(4) **Hydrogen Peroxide. H_2O_2.**

(*a*) Add a few drops of potassium bichromate and a little dilute sulphuric acid, then 10 c.c. of ether. Shake and allow to stand. A blue layer settles out.

(*b*) Add 2 c.c. of dilute permanganate solution and acidify with dilute sulphuric acid. Oxygen is evolved and the colour of the permanganate is discharged.

$$5\ H_2O_2 + 3\ H_2SO_4 + 2\ KMnO_4 =$$
$$5\ O_2 + K_2SO_4 + 2\ MnSO_4 + 8\ H_2O$$

(5) **Zinc Chloride. $ZnCl_2$.**

Zn. (*a*) Add sodium acetate and pass in sulphuretted hydrogen. A white precipitate is produced.

$$H_2S + ZnCl_2 = ZnS + 2\ HCl$$

$$CH_3 - COONa + HCl = NaCl + CH_3COOH$$

(*b*) Sodium carbonate gives a white precipitate.

$$2\ ZnCl_2 + 2\ Na_2CO_3 + H_2O =$$
$$ZnCO_3 \cdot Zn(OH)_2 + CO_2 + 4\ NaCl$$

(*c*) Potassium chromate gives a yellow precipitate.

$$ZnCl_2 + K_2CrO_4 = 2\ KCl + ZnCrO_4$$

Cl_2. As under corrosive sublimate.

(6) **Potassium Permanganate. $KMnO_4$.**

(*a*) Add dilute sulphuric acid, then potassium iodide. The addition of starch solution gives a blue colour.

$$2\ KMnO_4 + 8\ H_2SO_4 + 10\ KI =$$
$$5\ I_2 + 6\ K_2SO_4 + 2\ MnSO_4 + 8\ H_2O$$

(*b*) Acidify with dilute sulphuric acid, add oxalic acid and heat. The red colour disappears.

$$5\ C_2H_2O_4 + 2\ KMnO_4 + 3\ H_2SO_4 =$$
$$2\ MnSO_4 + K_2SO_4 + 8\ H_2O + 10\ CO_2$$

(*c*) Acidify with dilute sulphuric acid and add sodium sulphite. The red colour is discharged.

$$5\ SO_2 + 2\ KMnO_4 + 2\ H_2O =$$
$$2\ MnSO_4 + K_2SO_4 + 2\ H_2SO_4$$

(7) **Bleaching Powder.**

(*a*) Mix a little in a test tube with water. Add dilute HCl and place a starch and potassium iodide paper over the mouth of the tube. Warm. A blue colour is produced on the paper.

$$Ca(OCl)_2 + 4\ HCl = 2\ Cl_2 + CaCl_2 + 2\ H_2O$$
$$Cl + KI = KCl + I$$

(*b*) A solution of bleaching powder will discharge the blue colour of a solution of iodine and starch.

Test for Calcium.—Warm a little with dilute HCl; filter. Add ammonia and ammonium oxalate. Boil. A white precipitate is formed, insoluble in acetic acid.

$$CaCl_2 + (NH_4)_2\ C_2O_4 = CaC_2O_4 + 2\ NH_4Cl$$

(8) **Formaldehyde. H.COH.**

(*a*) Add a little milk, 1 drop of ferric chloride and 10 c.c. conc. HCl. Warm. A violet colour is produced.

(*b*) Add a few c.c. of Schiff's reagent. A pink colour develops.

(*c*) Add 3 c.c. of 10 per cent. phloroglucin. Shake and add a few drops of KOH. A pink colour is obtained.

(*d*) Add 3 c.c. of conc. H_2SO_4 to a few drops of the liquid, then a crystal of morphine. A purple-red colour is obtained which turns to violet and finally blue.

(9) **Sulphites.**

(*a*) Acidify with dilute H_2SO_4 and place over the mouth of the test tube a starch and potassium iodate paper. Warm. The paper acquires a blue stain (SO_2 is evolved).

$$H_2SO_4 + Na_2SO_3 = SO_2 + H_2O + Na_2SO_4$$

$$2\,HIO_3 + 5\,SO_2 + 4\,H_2O = I_2 + 5\,H_2SO_4$$

(*b*) Acidify with dilute H_2SO_4 and add a few drops of potassium bichromate. A green colour is obtained due to the formation of chromium sulphate.

(*c*) The blue colour produced by adding iodine to starch is discharged on adding an acidified solution of a sulphite.

(10) **Benzoic Acid. C_6H_5COOH.**—If in a liquid, treat as under salicylic acid in lime-juice, and test the residue.

(i) Heat with CaO; an odour of benzene is produced.

(ii) Heat with dilute H_2SO_4 and gallic acid. When cold render alkaline with NaOH; a reddish violet colour changing to brown is obtained.

(iii) A little of the residue dissolved in water gives a buff precipitate with ferric chloride.

(11) **Copper Sulphate. $CuSO_4$.**

(i) Add excess of ammonia; a fine blue colour is obtained.

(ii) Fresh solution of potassium ferrocyanide gives a chestnut brown precipitate.

Test for SO_4 as under Ferrous Sulphate.

The Chemical Action of Disinfectants.

(1) **Oxidizing Agents.**—The nascent oxygen oxidizes organic matter into NH_3, H_2O, and CO_2, *e.g.*, permanganate of potash, ozone, nitrous fumes, bleaching powder, hydrogen peroxide.

(2) **Reducing Agents.**—*e.g.*, Sulphurous acid.

(3) The following act as **corrosives and coagulate albumen:** Carbolic acid, corrosive sublimate, formaldehyde, zinc chloride. They have a direct poisonous effect on the micro-organisms. The organisms are killed owing to the destruction of the albumen, or an albuminous layer may envelop the germs and so deprive them of nourishment.

(4) **Deodorants.**—Carbon, $KMnO_4$, bleaching powder, slaked lime, ferrous sulphate, copper sulphate, sulphurous acid.

PART VI.—MICROSCOPICAL WORK.

The student should be familiar with the appearance of :—

(1) Starches.
(2) Parasites, growths, &c.
(3) Water deposits.
(4) Fibres and hairs.

Good illustrations of these will be found in "A Compendium of Food-Microscopy," by E. G. Clayton, F.I.C., and other text-books of public health.

The following Local Government Reports should be consulted :—

(1) "The Application of Formaldehyde to Meat," by Drs. G. S. Buchanan and S. B. Schryver. *Food Reports*, No. 9.

(2) "The Use of Preservatives in Cream," by Dr. J. M. Hamill. *Food Reports*, No. 10.

(3) "Bleaching of Flour and the Addition of so-called 'Improvers' to Flour," by Dr. J. M. Hamill; and "The Chemical Changes produced in Flour by Bleaching," by Dr. G. W. Monier-Williams. *Food Reports*, No. 12.

(4) "The Nutritive Value of Bread made from different varieties of Wheat Flour," by Dr. J. M. Hamill. *Food Reports*, No. 14.

(5) "Condensed Milks, with Special Reference to their use as Infants' Foods," by Dr. F. J. H. Coutts. *Food Reports*, No. 15.

(6) "Methods of Detection of certain Proprietary Substances sold as Preservatives for Milk, Cream, &c," by Dr. G. W. Monier-Williams. *Food Reports*, No. 17.

(7) "The Colouring-matter of Flour and its Relation to Processes of Natural and Artificial Bleaching," by Dr. G. W. Monier-Williams. *Food Reports*, No. 19.

(8) "The Biological Properties of Milk, both of the Human Species and of Cows, considered in Special Relation to the Feeding of Infants," by Dr. Janet Lane-Claypon. *New Series*, No. 76.

(9) "The Available Data upon the Value of Boiled Milk," by Dr. Lane-Claypon. *New Series*, No. 63.

USEFUL DATA.

1 metre = 39·37 inches
1 inch = 25 millimetres
1 mm. = ·0393 inch
1 foot = 30 cm.
1 cm. = ·393 inch
1 kg. = 2⅕ lbs.
1 oz. = 28·34 grammes
1 gramme = ·0353 oz.
1 lb. = 453·2 grammes
1 grain = ·0648 gramme
1 gramme = 15·432 grains
1 oz. = 437·5 grains
1 lb. = 7,000 grains
1 ton = 1,015·6 kg.
1 drachm = 1·8 gramme
1 scruple = 20 grains
1 c.c. = 16·7 mimims
100 c.c. = 3½ fluid ozs.
1 litre = 1·76 pints
1 gallon = 4544 c.c.
1 gallon = 70,000 grains
1 pint = 568 c.c.
1 minim = ·06 c.c.
1 fluid oz. = 8 fluid drachms
1 pint = 20 fluid oz.
1 fluid drachm = 60 minims

Grammes per 100 c.c. × 4·375 = grains per fluid oz.
,, ,, ,, × 87·5 = ,, ,, pint.
,, ,, ,, × 700 = ,, ,, gallon.
,, ,, 100 grams × 70 = ,, ,, lb.

Examination Questions.

Practical Chemistry and Microscopy.

1. Examine the water A and write a report on the results of the examination as to its fitness for household purposes. (Camb., 1911.)

2. Examine the water sediments A and B and state your opinion, with reasons, as to the comparative safety of the two waters for potable purposes. (Camb., 1911.)

3. Describe with sketches the microscopic characters of *Ustilago segetum* and *Strongylus filaria* respectively. Where are these organisms found and what effect may they have on food value? (Camb., 1911.)

4. Name and describe the objects under the microscopes 1 to 4. (Camb., 1911.)

5. In the sample of milk determine (1) the specific gravity; (2) the percentage of fat; (3) the antiseptic (if any). From your analysis report as to its fitness for use as a food. (Oxford, 1912.)

6. Estimate the amount of poisonous metal in the sample of water. (Oxford, 1912.)

7. Examine the sample of water qualitatively and quantitatively so as to pass an opinion as to its fitness for drinking purposes. (Oxford, 1907.)

8. Determine the amount of gluten in the sample of flour. State the amounts of moisture, gluten and ash in good flour. (Oxford, 1907.)

9. Identify the gases contained in the air in the bottles marked 1, 2 and 3. (Oxford, 1907.)

10. In the sample of water estimate (1) chlorine as chlorides; (2) the equivalent of the chlorine as common salt; (3) the oxygen absorbed in ninety minutes from permanganate at the laboratory temperature; (4) the total hardness. State your opinion of the quality of the water as far as it can be given from these results. (Oxford, 1898.)

11. Determine by means of the specific gravity bottle the density and alcoholic strength of the alcohol supplied. (Oxford, 1898.)

12. Examine the butter for preservatives. (Oxford, 1898).

13. Determine the free and albuminoid ammonia in the sample of water. (Oxford, 1899.)

14. Examine the sample of water for ammonia, calcium salts and alkaline carbonates and sulphates. Determine the amount of sodium chloride and the hardness. (Oxford, 1900.)

15. Determine the percentage of citric acid in the sample of lime-juice. (Oxford, 1900.)

16. Determine the percentage of alcohol in the sample of wine. Test the sample for sugar, preservatives and metals. (Oxford, 1900.)

17. Estimate the proportion of nitrates in the sample of water. (Oxford, 1901.)

18. Submit the sample of beer to the Reinsch test for arsenic. (Oxford, 1901.)

19. Report on the purity of the coffee supplied. (Oxford, 1901.)

20. Examine the sample of water for evidence of animal contamination. (London, 1912.)

21. Demonstrate the presence of copper in the sample of tinned peas and make an approximate estimation of the amount of the metal present. (London, 1912.)

22. Estimate the amount of available chlorine in the disinfectant solution. Determine the amount of oxidizable organic matter in the sample of water. (London, 1912.)

23. Detect the preservative in the sample of milk. (Oxford, 1913.)

24. Determine the specific gravity of the sample of sand. (Oxford, 1913.)

25. Determine the alkalinity of B by means of the acid provided and express the result as grammes of NaOH per litre. (Oxford, 1911.)

26. Examine the sample of water for metallic impurities. (Oxford, 1911.)

27. Examine the sample of milk for boracic acid, salicylic acid, and formaldehyde. (Oxford, 1911.)

28. Estimate the nitrates in the sample of water. (Oxford, 1912.)

29. Determine the specific gravity of the sample of milk. (Oxford, 1912.)

30. Determine the volume of oxygen at N.T.P. dissolved in 1 litre of tap water by Winkler's process. (Oxford, 1912.)

31. Estimate the nitrates in the sample of water and state the result in parts per 100,000 of the acid radicle. (Oxford, 1912.)

32. Determine the strength of the sample of lime-water supplied. (London, 1908.)

33. Identify the given disinfectants and state the tests employed for their identification. (London, 1908.)

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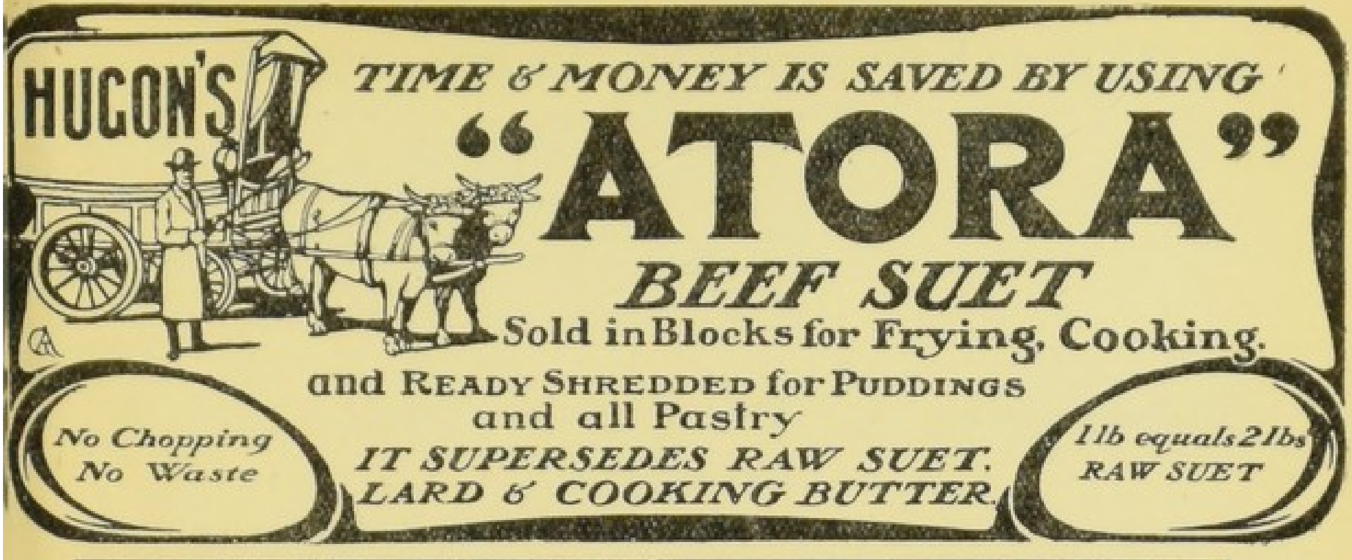
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