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Percival J. Fryer and Frank E. Weston
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SECTION I

INTRODUCTION TO PRACTICAL WORK FOR TECHNICAL STUDENTS

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INTRODUCTION TO PRACTICAL WORK FOR TECHNICAL STUDENTS[1]

§ 1. In order to obtain proficiency in analytical and experimental work, several factors are necessary for success. These may be classified as:

- (*a*) Personal.
- (*b*) Those relating to apparatus.
- (*c*) Those concerning procedure.

§ 2. The **Personal factor** is of great importance in practical chemistry. It is essential for success to acquire the habits of *cleanliness* and *tidiness* in all work; attention should be paid to details, which *precludes hurry*, many operations requiring time for their performance if accurate results are to be obtained. Finally, the student must learn to *concentrate his mind* upon the work in hand and not attempt to do two or more operations at the same time. The result of this will be to generate that confidence in one's work which is the great secret of the successful worker.

§ 3. **Apparatus** plays a very important part in all analytical work. Generally speaking, there is a *right* way and *many wrong* ways of using apparatus. An endeavour should be made from the beginning to learn the correct method of using chemical apparatus, even the most simple. Measuring vessels should always be verified before they are used in carrying out any quantitative determinations, as much valuable time and annoyance will often thereby be saved. Complicated apparatus should be thoroughly examined and the *use of all parts ascertained* before its employment. Accurate results will only be obtained by repeated practice and careful attention to detail.

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[1] This introductory chapter is intended to serve as a reminder to students of practical chemistry, and to some degree, perhaps, as a ready reference for qualified analytical and technical chemists. The successful performance of the practical analytical work in connection with the examination of oils and fats requires a high degree of training and experience in chemical theory and practice. The authors therefore strongly deprecate the too common practice of students taking up the study of applied chemistry, without qualifying in the general practical and theoretical branches of chemistry and physics.

§ 4. Even after one has become expert in manipulations, concordant results are sometimes unobtainable in certain determinations. This arises from some **inherent fault** in the method used, which *theoretically* may appear quite sound, but in practice reveals an error which frequently, when working under a given set of conditions, remains constant, and hence can be allowed for. Such methods however should not be employed if better ones can be devised.

§ 5. In the practical work described in this treatise the authors have endeavoured to give such instructions as will as far as possible ensure the acquisition of the **correct method of working**—many operations being illustrated, so that the best method of manipulation is readily seen. **Students** are directed also to the succeeding special section on the correct use of simple apparatus and the preparation of standard solutions for analysis.

Before commencing any test involving the use of even simple apparatus and reagents, it is always advisable to make sure that all the solutions are ready and accurately adjusted. All apparatus should be fixed up in readiness before the work is commenced, so that complete attention can be given to the manipulations.

To this end, in the chemical work described in this book, a list of the apparatus and special reagents necessary is given, unless these are likely to be immediately available.

§ 6. Many of the determinations carried out in the chemical examination of an oil, fat, or wax require the use of **standard solutions.**

A standard solution is one of known strength. The strength chosen in each case depends upon the use to which the solution is to be put, but it is always closely related to the *equivalent weight* of the compound being used.

§ 7. A solution of unit strength is known as a **normal solution**, and is one that contains one-equivalent-gramme weight of the compound in 1000 c.c. of the solution.

Consider the following equation:

$$\underset{40}{NaOH} + \underset{36{\cdot}5}{HCl} = \underset{58{\cdot}5}{NaCl} + \underset{18}{H_2O}$$

It is seen that 40 grammes of NaOH neutralise 36·5 grammes of HCl, and since 36·5 grammes of HCl contain 1 gramme of replaceable H, 36·5 grammes of HCl is one-equivalent-gramme weight of hydrochloric acid, and 40 grammes of NaOH is one-equivalent-gramme weight of sodium hydrate. Hence a normal solution of hydrochloric acid contains 36·5 grammes of HCl per 1000 c.c.; and a normal solution of sodium hydrate contains 40 grammes of NaOH per 1000 c.c.

A normal solution is indicated by the letter N. placed before the chemical formula: thus **N. HCl** indicates a normal solution of hydrochloric acid.

§ 8. The following **table** gives a list of the compounds that will be required in the analysis of oils and fats, together with data concerning their equivalents, equivalent weights, etc.

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Substance	Formula	Molecular weight	Equivalent	Equivalent weight	Weight of substance in 1000 c.c. of a N. solution
Hydrochloric acid	HCl	36·5	1 molecule	36·5	36·5 grms
Sulphuric acid	H_2SO_4	98	$\frac{1}{2}$,,	49	49·0 ,,
Oxalic acid	$H_2C_2O_4 . 2H_2O$	126	$\frac{1}{2}$,,	63	63·0 ,,
Sodium hydrate	$NaOH$	40	1 molecule	40	40·0 grms
Potassium hydrate ...	KOH	56·1	1 ,,	56·1	56·1 ,,
Sodium carbonate	Na_2CO_3	106	$\frac{1}{2}$,,	53	53·0 ,,
Sodium thiosulphate ...	$Na_2S_2O_3 . 5H_2O$	248·1	1 molecule	248·1	248·1 grms
Potassium dichromate ...	$K_2Cr_2O_7$	294·2	$\frac{1}{6}$,,	49·0	49·0 ,,
Potassium permanganate	$KMnO_4$	158·1	$\frac{1}{5}$,,	31·6	31·6 ,,
Iodine monochloride ...	ICl	162·4	$\frac{1}{2}$ molecule	81·2	81·2 grms
Iodine	I_2	253·8	$\frac{1}{2}$,,	126·9	126·9 ,,

In many cases these solutions will be too strong, and therefore solutions containing $\frac{1}{2}$, $\frac{1}{5}$, $\frac{1}{10}$, etc. these weights are used. These solutions are termed seminormal, fifth-normal, and decinormal respectively, and are represented by the signs N/2, N/5, N/10, etc., or ·5N, ·2N, ·1N, etc. If solutions stronger than Normal are required multiples of Normal solutions are used, as 2N, 2·5N, 3N, etc.

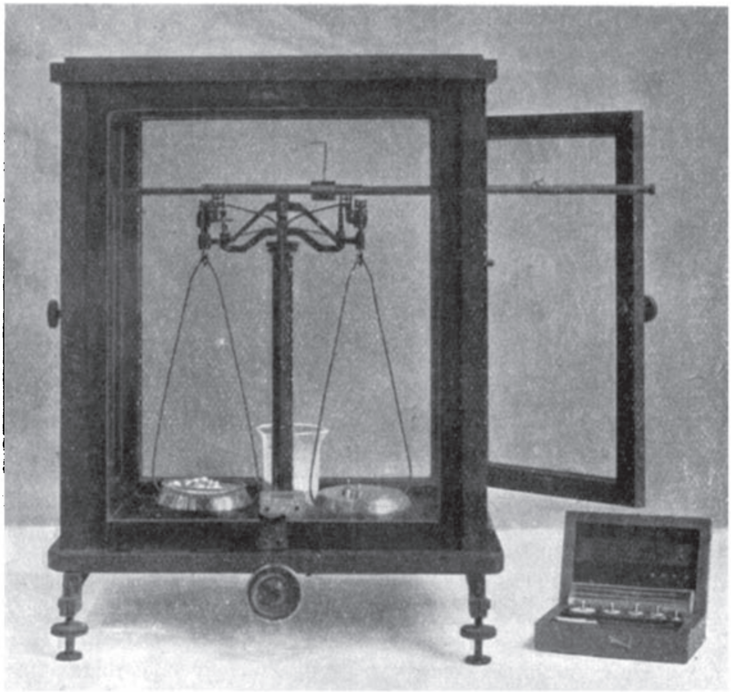

Fig. 1

PREPARATION OF STANDARD SOLUTIONS

§ 9. The simplest method of making a standard solution is to take a **known weight** of the substance required, dissolve in water, and make the solution up to the necessary volume by adding water to the required amount. This necessitates the following requirements:

(*a*) The use of a **pure** substance of *composition* corresponding to its *formula*.
(*b*) **Accuracy** in weighing.
(*c*) **Corrected** volumetric measuring apparatus.
(*d*) **Precision** in all the required manipulations.

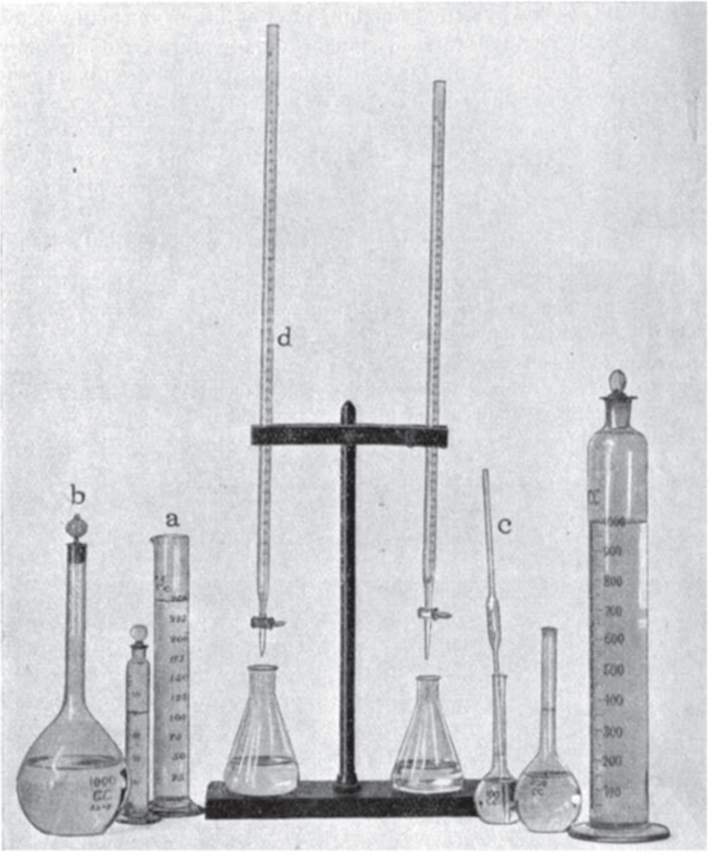

Fig. 2

Condition (*a*) is fulfilled by only a VERY FEW of the compounds that are required, and hence the method is limited in application. Of those

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mentioned in the preceding table, only the following are suitable for direct weighing:

1. Pure anhydrous SODIUM CARBONATE Na_2CO_3.
2. Pure crystallised OXALIC ACID $H_2C_2O_4 . 2H_2O$.
3. Pure recrystallised POTASSIUM DICHROMATE $K_2Cr_2O_7$.
4. Pure recrystallised POTASSIUM PERMANGANATE $KMnO_4$.

Condition (*b*) depends upon the use of a good balance and **skill in weighing**. A short-beam balance capable of carrying a total load of 200 grms and weighing to ·0005 grm will be found suitable. The illustration (fig. 1) is a photograph of a Sartorius short-beam balance.

Condition (*c*) requires **accurately calibrated** measuring vessels; these can be obtained quite easily at the chemical dealers. They should preferably be *checked* by actual weighing after additions of small volumes of pure recently-distilled water at standard temperature. An alternative method is comparison with a standardised apparatus. Fig. 2 is a photograph of the apparatus required. **a** represents a 250 c.c. *graduated cylinder* (50 c.c. and 1000 c.c. cylinders are also necessary), **b** is a 1000 c.c. *graduated flask* (50 c.c., 100 c.c., 250 c.c. flasks are also required), **c** shows *graduated pipettes*, 10, 20, and 50 c.c., and **d** represents a 50 c.c. *burette*.

Condition (*d*) is attained only by much practice and careful attention to detail.

§ 10. EXAMPLE OF DIRECT WEIGHING METHOD.—**Preparation of Decinormal (N/10) Sodium Carbonate.**

Apparatus required

balance sensitive to ½ milligramme
volumetric apparatus as in § 9
desiccator
watch glass
crucible
beakers
tripod stand and wire gauze
clay triangle
bunsen burner
wash bottle
funnel
small pestle and mortar
crucible tongs
250 c.c. Erlenmeyer flasks

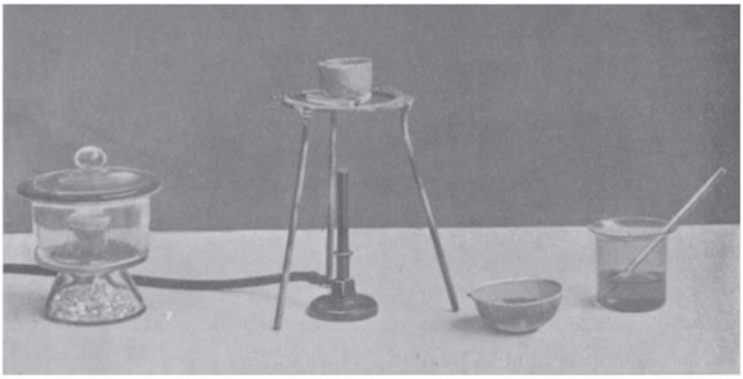

Fig. 3

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Materials

[A] Pure anhydrous SODIUM CARBONATE.
[B] DISTILLED WATER.

Procedure

1. Place about 10 grms of the well-powdered Na_2CO_3 [A] in a dry crucible and heat till bottom of crucible is dull red (fig. 3).
2. Transfer crucible to desiccator and allow to cool.
3. Take a clean dry watch glass, place on left pan of balance and accurately weigh it to ½ milligramme (fig. 1).
4. Add 5·3 grms to the weights already taken and weigh out this amount of the dry Na_2CO_3 on the watch glass, taking from the centre of the crucible.
5. Transfer the weighed Na_2CO_3 to a clean 250 c.c. beaker, rinsing off all the powder into the beaker with the wash bottle (fig. 4).

Fig. 4

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6. Add to the beaker about 100 c.c. of distilled water [B] and gently heat till all is dissolved.
7. Take the 1000 c.c. graduated flask, rinse it out with distilled water, and insert a funnel in it.

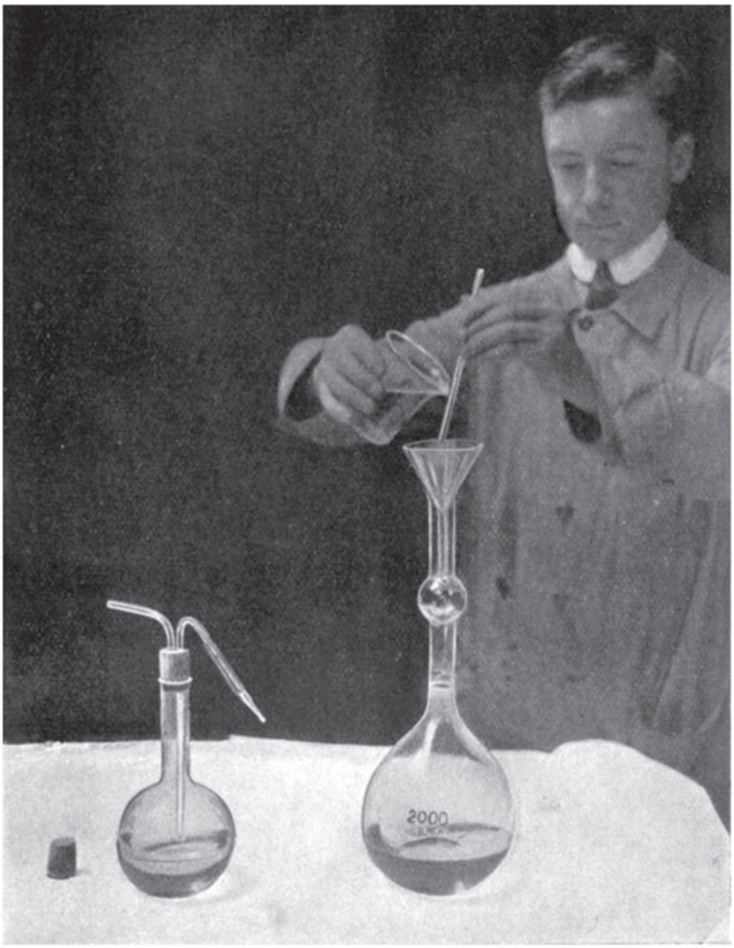

Fig. 5

8. Carefully pour the Na_2CO_3 solution from the beaker into the flask through the funnel, using a stirring rod to direct the stream of liquid (fig. 5), and rinse out beaker by holding it mouth downwards over the funnel and directing a jet of water up into the beaker from the wash bottle (see fig. 4).

9. Rinse down the funnel inside and out with the wash bottle, remove the funnel, and add distilled water until the mark is almost reached.
10. Test temperature of solution; if below 15·5° C., warm gently until this temperature is reached; if above, cool to the necessary degree by placing under a stream of water from the tap.
11. Add water to the mark on neck of flask; close with stopper, and mix contents by inverting flask several times, taking care that no leakage of the contents occurs. The solution so obtained is decinormal (**N/10**) **Sodium Carbonate.**

§ 11. N/10 solutions of oxalic acid, potassium dichromate, potassium permanganate and iodine are made in exactly the same manner, with the following exceptions:

The OXALIC ACID is not heated, but only well pressed between clean dry filter paper before weighing.

The POTASSIUM DICHROMATE is well powdered and heated in a crucible till it begins to fuse round the edges.

The POTASSIUM PERMANGANATE is well powdered and pressed between clean dry filter paper.

The IODINE is weighed in a stoppered weighing bottle, and is dissolved in potassium iodide solution or in alcohol.

In each case the WEIGHT TAKEN is $\frac{1}{10}$ part of the number of grammes for a normal solution, given in the foregoing table.

§ 12. Standard solutions of the **other compounds** mentioned in the table cannot be made by direct weighing, since the compound is

(1) known only in solution, or
(2) does not exist in a pure state.

Hence an **indirect method** has to be employed, involving the use of a standard solution made by direct weighing.

§ 13. EXAMPLE OF INDIRECT METHOD.—**Preparation of Decinormal (N/10) Hydrochloric Acid.**

Apparatus required

As before (§ 10) with addition of HYDROMETERS and 50 c.c. BURETTE IN STAND.

Materials

[A] Decinormal sodium carbonate.
[B] Pure concentrated hydrochloric acid.
[C] Methyl orange solution (1 per cent. in methylated spirit).
[D] Distilled water.

Procedure

1. Place 9 to 10 c.c. of the conc. HCl solution [B] in a 1000 c.c. cylinder and fill up with distilled water until the specific gravity is 1·02 (see p. 36). Mix by reversing several times. This gives a *slightly stronger* than N/10 solution.
2. The *exact strength* of this solution is now determined in the following manner:
   (i) Take a dry 10 c.c. pipette, rinse with the N/10 Na_2CO_3 [A] (which should be at 15·5° C. on testing with the thermometer: see § 10, above), and fill up to the mark with the same (fig. 6).

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(ii) Run the measured 10 c.c. into a clean Erlenmeyer flask and add 2—3 drops of the methyl orange solution. This turns the solution light yellow.

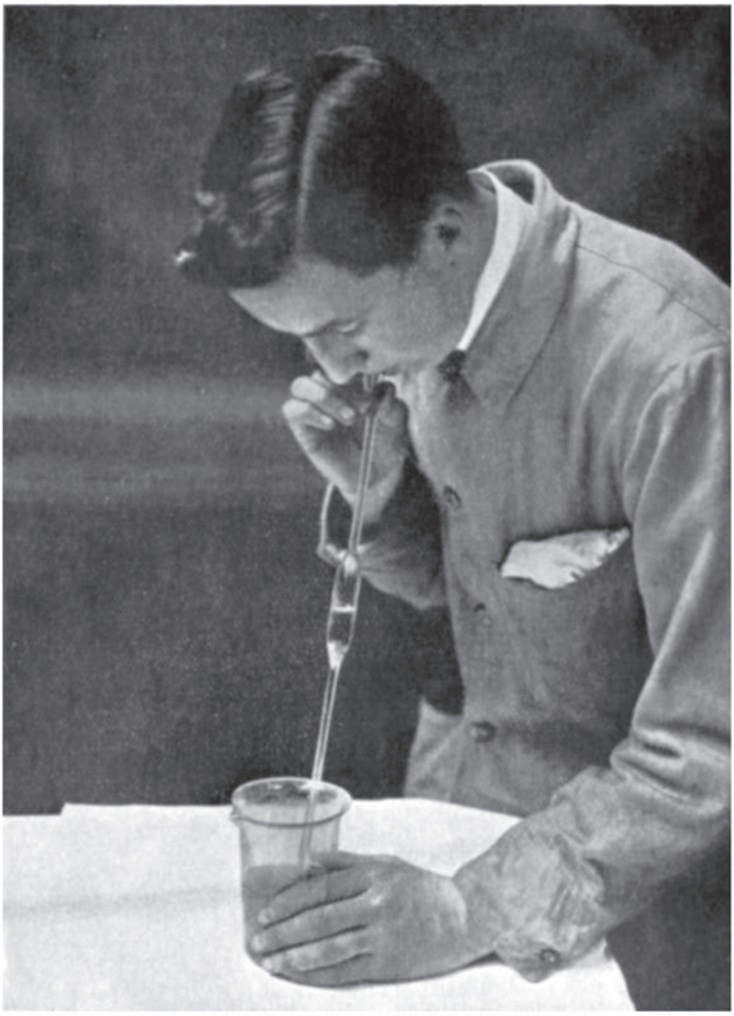

Fig. 6

(iii) Rinse out a 50 c.c. burette with the HCl solution (1) and then fill it to the top division with the same solution. The solution should be preferably at standard temperature (15·5° C.), or in any case at the *same* temperature as the N/10 Na_2CO_3.