

EXPERIMENT-13

- (a) Preparation of solution of oxalic acid and ferrous ammonium sulphate of known molarity by weighing (non-evaluative). Use of chemical balance to be demonstrated.
- (b) A study of (i) acid-base and (ii) redox titrations (single titration only). Both the solutions to be provided.
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13.1 OBJECTIVES

After performing this experiment, you should be able to:

- *handle chemical balance properly;*
- *weigh chemicals accurately using a chemical balance;*
- *handle burette and pipette properly;*
- *pipette out a given solution correctly with the help of a pipette.*
- *prepare standard solutions of oxalic acid and ferrous ammonium sulphate;*
- *detect the sharp end point;*
- *perform acid-base (oxalic acid and sodium hydroxide) and, redox, (ferrous ammonium sulphate and potassium permanganate) titrations;*
- *calculate the strength of the given solution by using appropriate formula.*

13.2 WHAT YOU SHOULD KNOW

Volumetric analysis is quantitative analysis in which the results are expressed in a certain definite volume. It involves the use of at least one solution of known strength. It may be prepared by dissolving a definite amount of a solute in a solvent to get a known volume of solution. The volume of this solution which reacts with a volume of the solution of unknown strength of another substance is determined. The process is known as titration.

13.2.1 Handling the apparatus

In volumetric analysis, we deal with volumes of solutions. Therefore, these should be measured correctly. The glass apparatus must be clean and free from grease etc. Errors due to parallax in

reading the level of a solution in burette, pipette and volumetric flask should be avoided. To read the volume in the container shown in Figure (13.1) the point shown by the arrow is the correct position.



Fig. 13.1 : *Correct way to note down the burette readings*

13.2.2 Pipette

Pipettes are available in various capacities (volumes) and a pipette of required volume is used during titration for delivering a liquid out of the pipette. It should be held vertically with its tip in contact with the wall of vessel. After the flow of liquid has ceased, a very small solution of the liquid remains in the lower portion of the pipette. It should not be blown out. For this, hold the bulb of the pipette in left hand palm with its upper tip closed with a finger. Touch the lower tip of pipette to the wall of the vessel. Handling of a pipette and correct way to drain out the solution are shown in set of figures 13.2a and 13.2b.



Fig. 13.2(a) : *Pipette*

(b) Handling of a pipette **(c) Correct way to drain out the solution**

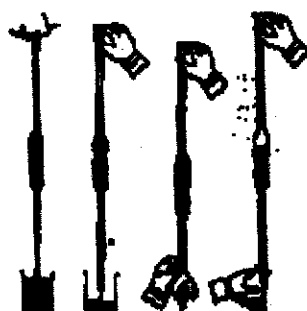


Fig. 13.3 : *Measuring of solution with a pipette*

13.2.3 Chemical Balance

Introduction

A chemical balance is commonly used in chemical laboratories to weigh an exact amount of the substance. Many chemical reactions are happened with exact mass of the substance. Each molecule or atoms of the substance has its own importance. Hence it is necessary to use exact mass of the substance in each experiment therefore, the trip balance is not useful for reaction concern. Chemical balance is used for weighing accurate mass of the substance.

Various kind of balance are used in the chemical laboratories are shown in the Figs. 13.4, 13.5, 13.6.

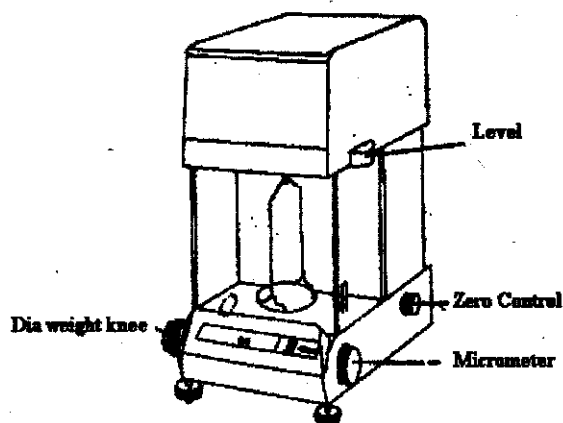


Fig. 13.4: Single Pan Balance

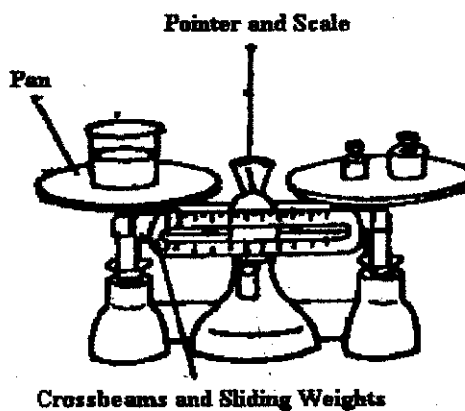


Fig. 13.5 : Rough Balance

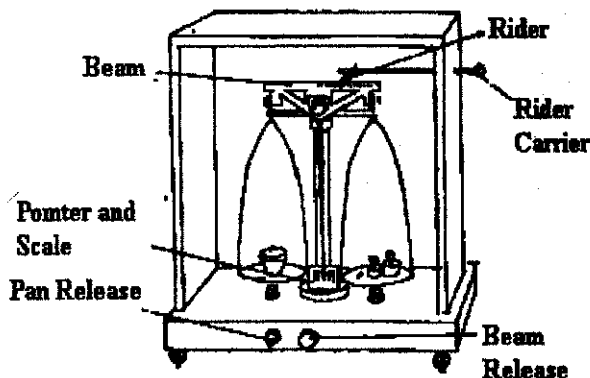


Fig. 13.6 : Analytical Balance

The analytical balance is an instrument for determining mass of a body by equilibrium of weights suspended from the opposite side of a bar having a fulcrum at its centre. It may be regarded as a rigid beam having a fulcrum at its centre and two arms of equal length. The chemical balance shown in the Fig. (13.7).

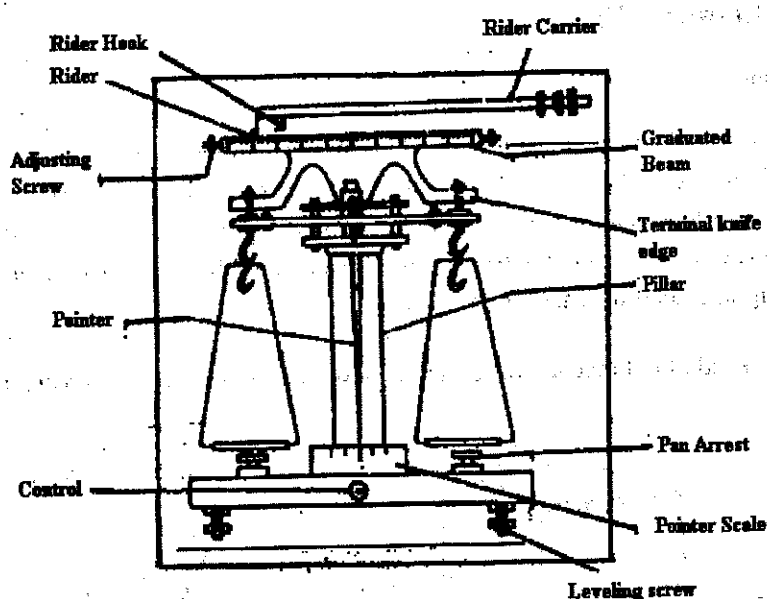


Fig. 13.7: A chemical balance

A chemical balance consists of a light-weight beam having a mounted knife edge at its centre. It rests on a pure corundum plate attached to the pillar. There are two knife edges mounted on the two ends of the beam which are equidistant from the central knife-edge. The two ends of the beam also have adjusting screws for Zero-adjustment. The centre of the beam is attached to a pointer which moves over the scale at the foot of the pillar. There are two levelling screws at the base and one plumb-line suspended from the column. The beam is divided into 100 divisions, i.e. 50 divisions on each side of the fulcrum.

The whole arrangement is enclosed in a wooden case with glass sides.

Weight-Box

A weight box is a wooden box having grooves of various sizes into which are placed different weights ranging from 1 gram to 100 grams as shown Fig. 13.8a. These weights are made up of brass coated with nickel or chromium. Each weight is nearly cylindrical having a knob at its one end with the help of which it can be lifted with forceps.

The following is the order of weights placed in a weight box.

100 g, 50 g, 20 g, 20 g

10g, 5g, 2g, 2g, 1g

The weights are placed in the following sequence in a weight box.

Fractional weight box

Fractional weights are made up of aluminium or brass coated with chromium or nickel that range from 1 milligrams to 500 milligrams as shown Fig. 13.8b. The fractional weights less than 10 milligrams are not used, instead the use of rider is recommended.

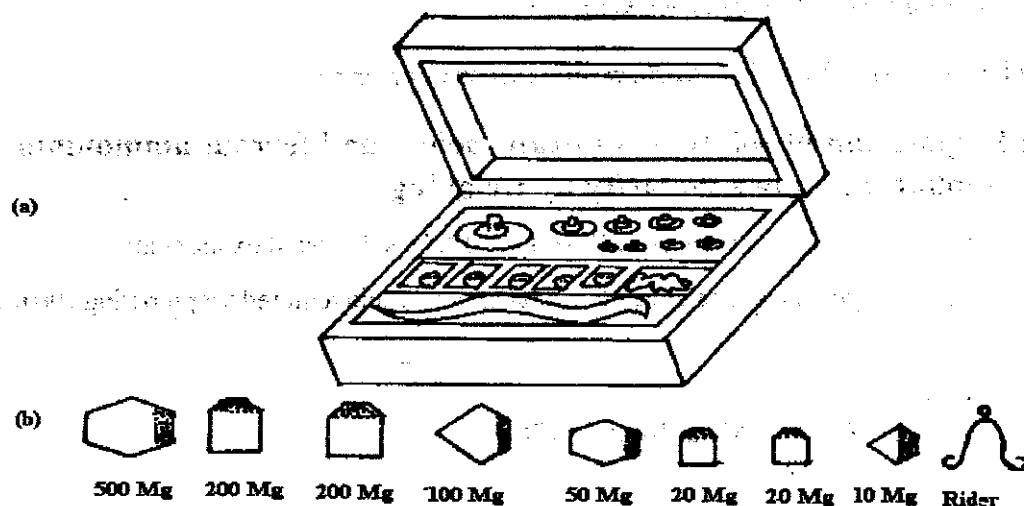


Fig : 13.8 (a) A weight box, (b) Fractional weights

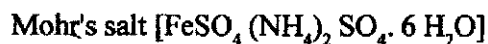
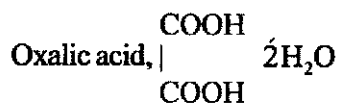
Primary Standard :

The standard solution of some substances can be prepared directly by weighing. These substances are available in their pure forms and do not undergo chemical change on storing. These are known as primary standards.

The important properties of primary standards are as follows:

- (i) They are easily available in pure and dry conditions.
- (ii) They should not undergo any chemical change with air, oxygen and carbon dioxide.
- (iii) They do not possess hygroscopic, deliquescent and efflorescent properties.
- (iv) They are easily soluble when added to the solvent (normally water).
- (v) They normally possess high molecular mass so that weighing errors are negligible.
- (vi) The standard solution of these substances should react in a stoichiometric ratio with the volumetric titrants.
- (vii) They don't react with impurities which are present in the solution to be titrated.

Examples of Primary Standard Substances



Secondary Standards

Many chemical substances do not possess the primary standard properties (properties given above). Therefore, they cannot be used to prepare standard solutions. However, the solution of this type of substance are first prepared of approximate strength and then standardized by titrating with a solution of a primary standard.

Example of Secondary Standard Substances

Sodium hydroxide (NaOH), potassium permanganate (KMnO₄).

13.2.4 Preparation of solutions of oxalic acid and ferrous ammonium sulphate of known molarity by weighing

Suppose you are asked to prepare 100 ml of 1 Molar solution of oxalic acid;

As a first step you have to calculate the mass of oxalic acid required for preparing 100 ml of solution.

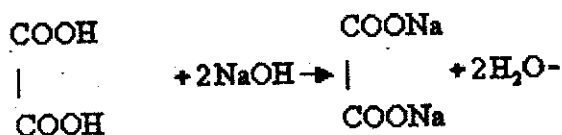


126 grams of oxalic acid is required to prepare 1 litre solution of molarity, 1M.

To prepare 0.1 Molar 100 ml solution, you dissolve 1.26 grams of the oxalic acid in distilled water and make it upto 100 ml. Similarly, to prepare 0.1 M solution of ferrous ammonium sulphate, 3.92 grams of ferrous ammonium sulphate is dissolved in distilled water so as to get 100 ml solution.

(a) Titration of oxalic acid solution against sodium hydroxide

In this acid-base titration, oxalic acid is completely neutralised by the base (NaOH) according to the following reaction.



According to this reaction, two moles of sodium hydroxide neutralises one mole of the acid for complete neutralisation. The end point is indicated by phenolphthalein indicator. It is colourless in acid medium and pink in the basic medium.

Now using the empirical relation you can calculate the strength of sodium hydroxide

$$a_1 M_1 V_1 = a_2 M_2 V_2$$

where,

a_1 = Acidity of sodium hydroxide = 1

M_1 = Molarity of sodium hydroxide = ?

V_1 = Volume of sodium hydroxide consumed (burette reading)

a_2 = Basicity of oxalic acid = 2

M_2 = Molarity of oxalic acid = 0.1M (known)

V_2 = Volume of oxalic acid taken for titration =

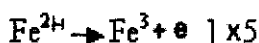
Values of a_1 , V_1 , a_2 , M_2 , V_2 are known. By using the above relation you can calculate M_1

$$M_1 = \frac{a_2 M_2 V_2}{a_1 V_1}$$

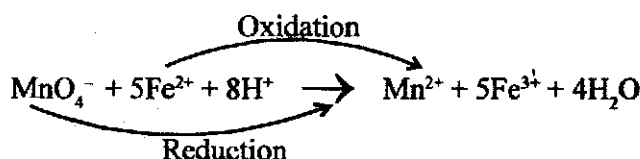
(b) Titration of Ferrous Ammonium Sulphate solution against Potassium Permanganate solution

Potassium permanganate is an oxidising agent, both in alkaline and acidic medium. Oxidation is always accompanied by reduction. In this titration, the ferrous ion is oxidised to ferric ion by permanganate ion, $[\text{Mn(VII)}]$ and at the same time permanganate ion is reduced to manganese (II) $[\text{Mn(II)}]$.

The following reactions take place



The over all reaction is



According to the stoichiometric relation, one mole of the oxidising agent, potassium permanganate, reduces 5 moles of reducing agent, ferrous ammonium sulphate.

Potassium permanganate is a self indicator, a pink colour appears and persists at the end point. To calculate the strength, the following relation is used:

$$a_2 M_1 V_1 = a_1 M_2 V_2$$

Here a_1 and a_2 are the change in oxidation number in oxidant and reductant respectively. For permanganate solution, $a_1 = 5$, M_1 and V_1 are its molarity and volume $a_2 = 1$ for ferrous ammonium sulphate, M_2 and V_2 are its molarity and volume respectively.

13.3 MATERIALS REQUIRED

(1) Apparatus :

Analytical balance,
Weight-box, Measuring flasks,
Beaker, Glass rod, Conical flask,
Funnel, Burette stand, Wire gauze,
Burette, Pipette

(2) Chemicals :

Oxalic acid, Sodium hydroxide,
Ferrous ammonium sulphate (Mohr's salt),
Phenolphthalein, Distilled water,
Potassium permanganate,
Sulphuric acid (dilute).

13.4 HOW TO PERFORM THE EXPERIMENT

To Prepare standard solutions of oxalic acid and ferrous ammonium sulphate, the following steps are followed

Weigh the required amount of substance (section 13.2) using an analytical balance. The substance should be weighed in a weighing bottle.

Transfer the weighed substance into a standard flask (100 ml) and dissolve in minimum amount of distilled water. Make the volume of solution upto the mark in the standard flask. Shake it by tilting the flask upside down. In case of ferrous ammonium sulphate, before making the solution upto the mark, dilute sulphuric acid (≈ 15 ml) should be added to prevent the hydrolysis.

13.4.1 Acid-base titration

Take a, clean burette, rinse it with the given sodium hydroxide solution and clamp it vertically in a burette stand. Fill the burette with sodium hydroxide solution. Ensure that no air bubbles are there in the stop cock. Note the initial reading (V_1). Rinse a clean 20 ml pipette with standard oxalic acid solution and then pipette out 20 ml of the given standard oxalic acid solution into a clean conical flask. Add a few drops phenolphthalein indicator. Hold the conical flask (as shown in figure 13.9) just below the nozzle of the burette and using your left hand, release sodium hydroxide solution into the conical flask drop by drop. Continuously swirl the contents of the conical flask smoothly and continue addition of NaOH solution dropwise till the appearance of pink colour. Note down the reading of the lower meniscus of NaOH solution. This is the final reading, (V_2). Repeat the titrations to get at least two concordant readings.

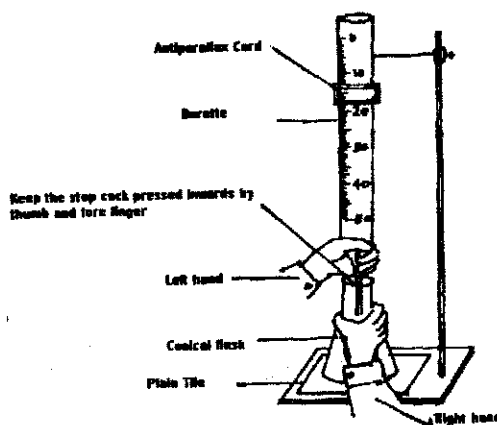


Fig. 13.9 : Carrying out a titration

13.4.2 Titration of ferrous ammonium sulphate and potassium permanganate

Take a clean and dry burette and rinse with the given potassium permanganate solution. Fit the burette on stand. Fill with potassium permanganate solution and remove the air bubbles.

Rinse a clean, dry pipette with given Mohr's salt solution. Then pipette out 20 ml of Mohr's salt solution and transfer into a clean 250 ml conical flask. Add approximately 20 ml of dilute sulphuric acid. Titrate it by gently swirling the conical flask. (Refer the figure 13.9) A permanent pink colour appears at the end point. Repeat the titration to get at least two concordant readings.

13.5 PRECAUTIONS

- Handle the Chemical Balance carefully.
- While preparing standard solutions add distilled water into the flask slowly. (Otherwise you will easily miss the mark).
- Chemicals should be pure (LR or AR).
- Apparatus used should be clean and dry.

- (v) Only 2 to 3 drops of indicator solution should be used.
- (vi) At least two concordant readings should be taken for calculations.
- (vii) While titrating, continuous swirling of the solution is required, to avoid incomplete reaction.

13.6 OBSERVATIONS

Preparation of standard solutions

(i) Oxalic acid

Mass of empty weighing tube =

Mass of weighing tube + substance =

Mass of substance =

(ii) Ferrous ammonium sulphate

Mass of empty weighing tube =

Mass of weighing tube + substance =

Mass of substance =

TITRATION

- (i) **Oxalic acid Vs. Sodium hydroxide.** Burette solution, sodium hydroxide. End point ; colourless to pink colour. Burette reading.

S.No.	Initial reading	Final reading	Volume of NaOH used (ml)
1			
2			
3			
4			

Volume of oxalic acid solution taken in each titration, $V_1 = 20$ ml

Molarity of oxalic acid = $M_1 =$

Volume of NaOH consumed (concurrent reading) = V_2

Molarity of Sodium hydroxide = $M_2 = ?$

The relation

$$a_2 M_1 V_1 = a_1 M_2 V_2$$

$$a_1 = 2$$

$$a_2 = 1$$

$$M_2 = \frac{a_2 M_1 V_1}{a_1 V_2}$$

Molarity of the given sodium hydroxide is..... mol l⁻¹

(ii) **Ferrous ammonium sulphate Vs. potassium permanganate.**

Burette solution : Potassium permanganate

End Point : Colourless to pink colour.

Burette readings:

S.No.	Initial readings	Final reading	Volume of KMnO ₄ consumed (ml)
1			
2			
3			

Relation of volume and molarity

$$a_2 M_1 V_1 = a_1 M_2 V_2$$

where a_1 and a_2 are changes in oxidation state (oxidation number), with respective species

Volume of Mohr's salt solution taken = $V_1 = 20$ ml

(pipette readings)

Molarity of standard mohr's salt solution is $M_1 =$

Change in oxidation state of Mn in MnO_4^- is $a_2 = 5$

Volume of $KMnO_4$ consumed = V_2

Molarity of $KMnO_4$ is $M_2 =$

By substituting, a_1 , a_2 , M_1 , V_1 and V_2 , M_2 can be calculated.

Concentration of the given potassium permanganate solution is = mol l⁻¹

13.7 CONCLUSION

- (i) Strength of sodium hydroxide and potassium permanganate are determined by titrating them with oxalic acid and ferrous ammonium sulphate solutions respectively. Their respective Molarities are..... and

13.8 CHECK YOUR UNDERSTANDING

1. *State the need of rinsing the burette and pipette with respective solutions to be used in the titration.*
.....
2. *Why should pure chemicals be used for preparing a standard solution ?*
.....
3. *Why should only the lower meniscus be taken into consideration while reading the level of solution.*
.....
4. *How the last (final) drop of solution in pipette can be taken out ?*
.....
5. *Why the secondary standard cannot be used in the standard solution preparation ?*
.....

13.9 A NOTE FOR THE TEACHERS

- (i) Teacher should demonstrate to the students the method to handle an Analytical Balance.
- (ii) Teacher should ensure that the chemicals are pure enough and that distilled water is used in the titration. The appearance of the end point and the concept of concurrent readings should be made clear.
- (iii) The reaction which is occurring in the titration should be explained to the students.

13.10 CHECK YOUR ANSWERS

Ans.1 To avoid error in the calculations.

Ans.2 The impurities may react and interfere in the reaction. Change in weights will lead to errors in the calculations.

Ans.3 The solution level in the burette is always in the curved manner (concave). Therefore if we take the upper meniscus, there will always be a loss in the volume of the solution.

Ans.4 After releasing all the solution just dip the edge of the pipette in the conical flask solution.

- Ans.5
- (a) Secondary standards are not available in the pure and dry forms.
 - (b) It may undergo reaction with solvents
 - (c) It may undergo hydration and alter its own weights during the process.
 - (d) It may react with air or oxygen and carbon dioxide.