2018 - 2021 Syllabus Batch



# **KARPAGAM ACADEMY OF HIGHER EDUCATION**

(Deemed to be University) (Established Under Section 3 of UGC Act 1956) Coimbatore - 641 021. (For the candidates admitted from 2018 onwards)

# DEPARTMENT OF CHEMISTRY

#### SUBJECT NAME: ATOMIC STRUCTURE AND CHEMICAL BONDING PRACTICAL **SUBJECT CODE: 18CHU111 SEMESTER: I CLASS: I B.Sc CHEMISTRY**

# Scope

The course presents the practical knowledge in the in-organic chemistry, about the basics of atomic structure and chemical bonding. This involves quantitative techniques like volumetric analysis.

## **Objectives**

- To understand the principles of volumetric analysis. 1.
- To provide a versatile knowledge of preparations of solutions with appropriate 2. concentrations, titrations and to handle the respective apparatus while doing a titration.
- 3. To provide a knowledge about the calculations involved in the estimation of compounds using volumetric analysis,

# Methodology

Titrations, Volumetric analysis.

# Inorganic Chemistry - Volumetric Analysis

- 1. Estimation of sodium carbonate and sodium hydrogen carbonate present in a mixture.
- 2. Estimation of oxalic acid by titrating it with KMnO<sub>4</sub>.
- 3. Estimation of water of crystallization in Mohr's salt by titrating with KMnO<sub>4</sub>.
- 4. Estimation of Fe (II) ions by titrating it with K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> using internal indicator.
- 5. Estimation of Cu (II) ions iodometrically using  $Na_2S_2O_3$ .

# **Suggested Readings:**

# **Text Book:**

- 1. Svehla, G. (2012). Vogel's Qualitative Inorganic Analysis. Pearson Education.
- 2. Venkateswaran, V., Veerasamy, R. & Kulandaivelu, A. R. (2015). Basic Principles of Practical Chemistry, Sultan Chand & Sons

#### **Reference Book:**

Mendham, J. (2009) Vogel's Quantitative Chemical Analysis, Pearson. 1.



# **KARPAGAM ACADEMY OF HIGHER EDUCATION**

(Deemed to be University) (Established Under Section 3 of UGC Act 1956) Coimbatore - 641 021. (For the candidates admitted from 2018 onwards)

**DEPARTMENT OF CHEMISTRY** 

# SUBJECT NAME: ATOMIC STRUCTURE AND CHEMICAL BONDING PRACTICAL **SUBJECT CODE: 18CHU111**

# **SEMESTER: I**

# **CLASS: I B.Sc CHEMISTRY**

S.No.	Lecture	Topics to be Covered	Support Matarial/Dama Nam	
	Hour		Material/Page Nos	
1	2	General discussion about practicals, Issuing		
		apparatus and safety procedures to be		
		followed in the laboratory.		
2	2	Volumetric Analysis-Introduction-	TT1 70	
		Classification.	11:79	
3	2	Writing the experimental procedure.		
Δ	2	Demonstration for Estimation of sodium		
т	2	carbonate using standard sodium hydroxide	T1:102-103	
5	2	Estimation of a dium carbonate using		
3	Z	Estimation of socium carbonate using	T1:102-103	
-		standard sodium hydroxide.		
6	2	Estimation of oxalic acid by titrating it with	T1:108-109	
		KMnO <sub>4</sub>	111100 102	
7	2	Estimation of water of crystallization in	$T_{1,110,111}$	
		Mohr's salt by titrating with KMnO <sub>4</sub> .	11.110-111	
8	2	Estimation of Fe (II) ions by titrating it with	T1.125 126	
		K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> using internal indicator.	11:125-120	
9	2	Estimation of Cu (II) ions iodometrically	T1.125 126	
		using Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	11:135-130	
10	2	Viva-voce questions		
11	2			
11	<i>L</i>	Kevision.		
12	2	Model Practical Examination.		
	Total No.	of Hours Planned For Practical's = 24		

# **Suggested Readings:**

# **Text Book:**

T1: Venkateswaran, V., Veerasamy, R. & Kulandaivelu, A. R. (2015). Basic Principles of Practical Chemistry, Sultan Chand & Sons



# KARPAGAM ACADEMY OF HIGHER EDUCATION

(Deemed to be University) (Established Under Section 3 of UGC Act 1956) Coimbatore – 641 021. (For the candidates admitted from 2018 onwards) **DEPARTMENT OF CHEMISTRY** 

# SUBJECT NAME: ATOMIC STRUCTURE AND CHEMICAL BONDING PRACTICAL SUBJECT CODE: 18CHU111 SEMESTER: I CLASS: I B.Sc CHEMISTRY

# **VOLUMETRIC ANALYSIS**

**Introduction:** Quantitative chemical analysis is ordinarily done by two methods namely volumetric and gravimetric methods. Volumetric analysis involves estimation of a substance in solution by neutralization, reduction, oxidation or precipitation by means of another solution of accurately known strength. Gravimetric analysis involves estimation of a substance by process of weighing. Of the two methods of analysis, the volumetric analysis is much more rapidly carried out. Due to simplicity, accuracy and wide applicability, volumetric analysis is preferred to gravimetric analysis.

Volumetric analysis depends on measurements of the volumes of solutions of the interacting substances. A measured volume of the solution of a substance 'A' is allowed to react completely with the solution of definite strength of another substance 'B'. The volume of 'B' is noted. Thus we know the volumes of the two solutions used in the reaction and the strength of solution 'B'. From this data, we can find the strength of the solution 'A'.

**Titration:** The process of finding out the volumes of reagents required to bring out a definite reaction just to completion is termed as titration.

**End Point**: The end point of a reaction is the stage at which complete reaction takes place between two solutions. The end point is determined by an indicator which shows a marked color change at the completion of the chemical reaction.

**Standard Solution:** Volumetric analysis depends on the use of one standard solution; i.e., a solution of known strength or concentration. A standard solution is prepared by dissolving an accurately weighed amount of a substance called primary standard substance in a definite volume of the solution. The primarily standard substance should fulfill the following characteristics:-

- (a) It should be available in a high degree of purity.
- (b) It should be stable and unaffected by the atmosphere.
- (c) It should not be efflorescent or deliquescent.
- (d) It should be readily soluble in water and
- (e) Its solution in distilled water should not deteriorate on keeping.

Examples of primary standard substances are crystalline oxalic acid, anhydrous sodium carbonate, potassium dichromate, Mohr's salt, sodium chloride etc. If a substance does not conform to one or more of the above characteristics, its standard solution is prepared indirectly, i.e; preparing a solution of approximately higher concentration than the required one and subsequently determining its exact concentration by titrating it against a solution of a suitable primary standard substance. The solution is then diluted to such an extract so as to get the required concentration. Solution of potassium permanganate, inorganic acid, caustic alkalies etc. are prepared by indirect method.

**Normality:** The concentration of solution is generally expressed in terms of normality. A normal solution is a solution which contains one gram equivalent mass of the substance dissolved in one liter of a solution. Thus a normal (N) solution of sodium hydroxide contains 40 g of sodium hydroxide in one liter of the solution. 2N solution of sodium hydroxide contains 80 g of sodium hydroxide in one liter of the solution. A decinormal solution (0.1 N) of sodium hydroxide contains 4g of sodium hydroxide per liter of the solution.

Mass in grams per liter of solution

Normality =

Gram equivalent mass of the substance

#### **Classification of Volumetric Reactions:**

Volumetric reactions are classified under the following heads.

**1. Acidimetry and alkalimetry**: Acidimetry is the estimation of alkali solution using standard acid solution. Alkalimetry is the estimation of an acid solution using standard alkali solution.

e.g., 
$$HCl + NaOH \rightarrow NaCl + H_2O$$

**2. Oxidation-Reduction Titrations:** Here an oxidizing agent is estimation by titrating it with a standard reducing agent and vice-versa. In this type of reaction oxidation and reduction take place simultaneously.

e.g.,  $2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$  $5H_2C_2O_4 + 5[O] \rightarrow 5H_2O + 10CO_2$ 

**3. Iodimetry and iodometry**: Estimations using standard iodine solution are called iodimetry and those involving iodine liberated from the potassium iodide solution by a chemical reaction is iodomtery.

**4. Precipitation Titrations**: In this type of titration, the strength of a solution is determined by its complete precipitation with a standard solution of another substance.

e.g., 
$$AgNO_3 + NaCl \rightarrow AgCl + NaNO_3$$

**5.** Complexometric Titrations: This type of titration depends upon the concentration of ions (other than H<sup>+</sup> and OH<sup>-</sup>) to form a soluble ion or compound.

e.g., 
$$Ag^+ + 2CN^- \rightarrow [Ag(CN)_2]^-$$

The common complexing agent used in complexometric titration ethylene diamine tetra acetic acid (EDTA).

**Equivalent masses of compounds:** In volumetric analysis a knowledge of the equivalent mass of substance to be estimated and whose standard solutions are to be prepared should be known.

**1. Equivalent mass of an acid:** Equivalent mass of an acid is the number parts by mass of it which contains 1.008 parts by mass of replaceable hydrogen.

Equivalent mass of an acid = Molecular mass of the acid

Acid	Molecular mass	Number of replaceable	Equivalent mass
		H atoms (Basicity)	
		fi atoms (Busieny)	
HCl	36.5	1	36.5
HNO <sub>3</sub>	63	1	63
$H_2SO_4$	98	2	49
$H_2C_2O_4.2H_2O$	126	2	63

Basicity of the acid

**2. Equivalent mass of a base:** Equivalent mass of a base is the number parts by mass of it which will completely react with one equivalent of an acid.

Equivalent mass of an alkali = Molecular mass of the alkali

Acidity of the alkali Acidity is the number of hydrogen ions which react with one molecule of alkali.

		-	-
Alkali	Molecular mass	Acidity	Equivalent mass
NaOH	40	1	40
КОН	56	1	56
NaHCO <sub>3</sub>	84	1	84
KHCO <sub>3</sub>	100	1	100
Na <sub>2</sub> CO <sub>3</sub>	106	2	53
$K_2CO_3$	138	2	69
CaCO <sub>3</sub>	100	2	50

**3. Equivalent mass of an oxidizing agent:** Equivalent mass of oxidizing agent is numerically equal to the number of parts by mass of it which contains 8 parts by mass of available oxygen. Available oxygen means oxygen capable of being utilized for oxidation.

(a) Equivalent mass of KMnO<sub>4</sub> in acid medium. In acid medium two molecules of potassium permanganate give 5 atoms of oxygen for oxidation.

 $2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$ 

316 parts by mass of potassium permanganate give 80 parts by mass of oxygen for oxidation.

Equivalent mass of potassium permanganate in acid medium =  $316 \times 8$  = 31.6

#### (b) Equivalent mass of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in acid medium.

 $K_2Cr_2O_7 + 4H_2SO_4 \rightarrow K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3[O]$ 

294 parts by mass of potassium dichromate give 48 parts by mass of oxygen.

Equivalent mass of potassium dichromate in acid medium =  $\frac{294 \times 8}{48}$  = 49

#### (c) Equivalent mass of MnO<sub>2</sub> in acid medium.

 $MnO_2 + H_2SO_4 \rightarrow MnSO_4 + H_2O + [O]$ 

87 parts by mass of manganese dioxide give 16 parts by mass of oxygen.

Equivalent mass of manganese dioxide in acid medium =  $87 \times 8 = 43.5$ 

**4. Equivalent mass of a reducing agent:** Equivalent mass of a reducing agent is the number of parts by mass of it which can be oxidized by 8 parts (one equivalent) by mass of oxygen.

#### (a) Equivalent mass of ferrous sulphate, FeSO<sub>4</sub>.7H<sub>2</sub>O

In the presence of dilute sulphuric acid, ferrous sulphate is oxidized as follows:

$$2FeSO_4 + H_2SO_4 + O \rightarrow Fe_2(SO_4)_3 + H_2O$$

The equivalent mass of FeSO<sub>4</sub>.7H<sub>2</sub>O is the same as its molecular mass i.e., 278.

#### (b)Equivalent mass of Mohr's salt, FeSO4(NH4)2SO4. 6H2O

 $2FeSO_4(NH_4)_2SO_4 + H_2SO_4 + O \rightarrow Fe_2(SO_4)_3 + 2(NH_4)_2SO_4 + H_2O$ 

The equivalent mass of Mohr's salt is the same as its molecular mass i.e., 392.

# (c) Equivalent mass of oxalic acid, $H_2C_2O_4.2H_2O$

#### $H_2C_2O_4 + O \rightarrow H_2O + 2CO_2$

One equivalent of oxygen oxidizes half the molecular mass of oxalic acid. Therefore, the equivalent mass of oxalic acid is

$$\frac{H_2C_2O_4.2H_2O}{2} = \frac{126 = 63}{2}$$

#### (d) Equivalent mass of sodium oxalate, Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub>

 $Na_2C_2O_4 + H_2SO_4 + O \rightarrow Na_2SO_4 + 2CO_2 + H_2O$ 

Equivalent mass of sodium oxalate is half its molecular mass i.e., 67

#### 5. Equivalent mass of metal halides

Equivalent mass of metal halide = Molecular mass of halide No. of halogen ion furnished by one molecule

Thus, equivalent mass of NaCl = 58.46 = 58.46

$$\text{KCl} = 74.46 = 74.46$$

#### (a) Equivalent mass of silver nitrate.

$$AgNO_3 + NaCl \rightarrow NaNO_3 + AgCl$$

Equivalent mass of silver nitrate =  $AgNO_3 = 170 = 170$ 

# (b) Equivalent mass of Potassium thiocynate.

$$KCNS + AgNO_3 \rightarrow AgCNS + KNO_3$$

Hence, equivalent mass of potassium thiocynate = KCNS = 166

#### (c) Equivalent mass of sodium thiosulphate and iodine.

$$2Na_2S_2O_3+I_2 {\rightarrow} Na_2S_4O_6+2NaI$$

Equivalent mass of sodium thiosulphate is its own molecular mass i.e., Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.5H<sub>2</sub>O

= 248 and that of iodine is its own atomic mass i.e., 127.

#### (d) Equivalent mass of copper sulphate.

$$2CuSO_4 + 4KI \rightarrow Cu_2I_2 + 2K_2SO_4 + I_2$$

Therefore the equivalent mass of  $CuSO_4.5H_2O$  is its own molecular mass = 249.5.

#### (e) Equivalent mass of arsenious oxide.

$$As_2O_3 + 2H_2O + 2I_2 \leftrightarrow As_2O_5 + 4HI$$

Therefore the equivalent of arsenious oxide =  $As_2O_3$  = 49.

(f) Equivalent mass of sodium arsenite.

 $Na_3AsO_3 + I_2 + 2NaHCO_3 \rightarrow Na_3AsO_4 + 2NaI + 2CO_2$ 

Therefore the equivalent of sodium arsenite =  $Na_3AsO_3 = 98$ 

**Principle of volumetric analysis:** When two solutions compound react with each other, the product of volume and normality of one solution will be equal to the product of volume and normality of the other solution.

 $V \times N = V_1 \times N_1$  where V and N are the volume and normality of the first solution and  $V_1$  and  $N_1$  are the volume and normality of the second solution. Thus, if the volumes of the two solutions which compound react with each other are determined, and if the normality of one solution is known, the normality of the other solution can be calculated. By multiplying the normality factor by the equivalent mass, the mass of the substance in one liter of the solution is found out.

Normality  $\times$  Equivalent mass = Mass per litre of the solution

#### Ex. No.: 1

#### Estimation of sodium carbonate using standard sodium hydroxide

# Aim:

To estimate the amount of sodium carbonate present in the whole of the given solution using a standard solution of sodium hydroxide containing 4 g of substance in 1000 ml and an approximately decinormal solution of hydrochloric acid.

#### **Principle:**

The estimation depends on the reactions between Na<sub>2</sub>CO<sub>3</sub> and HCl and that of NaOH and HCl. Na<sub>2</sub>CO<sub>3</sub> reacts with HCl by a simple neutralization reaction.

 $Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2$ 

The end point of the reaction is determined by using methyl orange as indicator. Na<sub>2</sub>CO<sub>3</sub> is dibasic and its equivalent weight is

Formula =  $\underline{Weight} = \underline{106} = 53$ 2 Similarly, NaOH reacts with HCl as follows.

 $NaOH + HCl \rightarrow NaCl + H_2O$ 

The end point of this reaction is determined by using methyl orange as indicator. NaOH is monobasic and its equivalent weight is

Formula =  $\underline{\text{Weight}} = \underline{40} = 40$ 1

**Procedure:** 

Titration 1: Standardization of hydrochloric acid

1

Titration of standard NaOH Vs link HCl

Exactly 20 ml of standard NaOH solution was pipetted out into a clean conical flask, and a drop of methyl orange indicator was added. This solution was titrated against HCl taken in burette. The end point was the change of colour from golden yellow to pale pink. The titration was repeated for concordant value. Using this value, the strength of HCl solution was calculated.

# Estimation of Na<sub>2</sub>CO<sub>3</sub> using standard NaOH

Normality of standard NaOH = 0.1N

# **Titration I:**

Standard NaOH Vs link HCl solution:

Burette solution – Link HCl solution

Pipette solution – NaOH solution

Indicator – Methyl orange

End point – Change of colour from golden yellow to pale pink.

S No	Volume of NaOH	Burette reading		Volume of	Concordant
<b>5</b> .1 <b>N</b> 0.	$(\mathbf{V}_1)$ (ml)	Initial (ml)	Final (ml)	$HCl(V_2)(ml)$	value (ml)
1	20	0			
2	20	0			
3	20	0			

## **Calculation:**

Volume of NaOH (V1)	= 20 ml
Normality of NaOH (N1)	= 0.1 N

Volume of HCl ( $V_2$ ) = \_\_\_\_ ml

Normality of HCl  $(N_2) = ?$ 

According to Volumetric Principle,

$$\mathbf{V}_1\mathbf{N}_1 = \mathbf{V}_2\mathbf{N}_2$$

$$N_2 = \underline{V_1 \ N_1}$$

 $N_2 = \frac{V_2}{20 \times 0.1}$ 

Normality of HCl  $(N_2) =$ \_\_\_\_\_N

# **Titration II:**

Link HCl solution Vs unknown Na<sub>2</sub>CO<sub>3</sub>:

Burette solution	– Link HCl solution
Pipette solution	- Na <sub>2</sub> CO <sub>3</sub> solution
Indicator	– Methyl orange
End point	- Change of colour from golden yellow to pale pink.

=

S No	Volume of	Burette reading		Volume of	Concordant
5.INU.	$Na_2CO_3 (V_2) (ml)$	Initial (ml)	Final (ml)	$HCl(V_1)(ml)$	value (ml)
1	20	0			
2	20	0			
3	20	0			

Ν

# **Calculation:**

Volume of HCl ( $V_1$ ) = \_\_\_\_ ml

Normality of HCl (N1)

Volume of  $Na_2CO_3 (V_2) = 20 \text{ ml}$ 

Normality of  $Na_2CO_3(N_2) = ?$ 

20

According to Volumetric Principle,

 $V_1N_1 = V_2N_2$ 

 $\begin{array}{cc} N_2 & = \frac{V_1 \; N_1}{V_2} \end{array}$ 

 $N_2$ 

Normality of  $Na_2CO_3$  ( $N_2$ ) = \_\_\_\_N

Amount of  $Na_2CO_3$  present in 1 litre of the given solution

= Normality x Equivalent weight

= \_\_\_\_\_ x 53

= \_\_\_\_\_ g

The amount of  $Na_2CO_3$  present in the whole of the given solution

= Normality x Equivalent weight x 100 1000 <u>x 100</u> 1000 \_\_\_\_ g. = \_

#### Titration 2: Estimation of unknown Na<sub>2</sub>CO<sub>3</sub>

Titration of standard HCl Vs unknown Na<sub>2</sub>CO<sub>3</sub>

The given unknown solution was transferred to a 100 ml standard flask using a glass rod and funnel and was diluted upto the mark. The solution was shaken thoroughly for uniform concentration. The given pipette was washed with water and rinsed with the made up solution. Exactly 20 ml of this solution was pipetted out into a clean conical flask and a drop of methyl orange indicator was added and the solution had golden yellow. The solution was titrated against standardized HCl taken in the burette. The end point is the change of colour from golden yellow to pink. The final burette reading was noted. The titration was repeated for concordant value. Using the concordant value, the strength and the amount of Na<sub>2</sub>CO<sub>3</sub> present in the whole of the given solution was calculated.

#### **Result:**

The amount of  $Na_2CO_3$  present in the whole of the given solution is = \_\_\_\_\_ g.

#### Ex. No.: 2

#### Estimation of sodium hydroxide using standard sodium carbonate

## Aim:

To estimate the amount of sodium hydroxide present in the whole of the given solution using a standard solution of sodium carbonate containing 5.3 g of substance in 1000 ml and an approximately decinormal solutions of hydrochloric acid.

#### **Principle:**

The estimation depends on the reaction between  $Na_2CO_3$  and HCl and that of NaOH and HCl.  $Na_2CO_3$  reacts with HCl by a simple neutralization reaction.

 $Na_2CO_3 + 2HCl \rightarrow 2NaCl + H_2O + CO_2$ 

The end point of the reaction is determined by using methyl orange as indicator.  $Na_2CO_3$  is dibasic and its equivalent weight is

Formula =  $\frac{\text{Weight}}{2} = \frac{106}{2} = 53$ Similarly, NaOH reacts with HCl as follows.

 $NaOH + HCl \rightarrow NaCl + H_2O$ 

The end point of the reaction is determined by using phenolphthalein as indicator. NaOH is monobasic and its equivalent weight is

Formula = 
$$\underline{\text{Weight}}$$
 =  $\underline{40}$  = 40

# **Procedure:**

**Titration:** Standardization of HCl

Titration of link HCl Vs standard Na<sub>2</sub>CO<sub>3</sub>

Exactly 20 ml of standard Na<sub>2</sub>CO<sub>3</sub> solution was pipetted out into a clean conical flask and a drop of methyl orange indicator was added. This solution was titrated against HCl taken in the burette. The end point is the change of colour from golden yellow to pale pink. The titration was repeated for concordant value. Using this value, the strength of HCl solution was calculated.

# Estimation of NaOH using standard Na<sub>2</sub>CO<sub>3</sub>

Normality of standard  $Na_2CO_3 = 0.1N$ 

# **Titration I:**

Standard Na<sub>2</sub>CO<sub>3</sub> Vs link HCl solution

Burette solution – Link HCl solution

Pipette solution  $-Na_2CO_3$  solution

Indicator – Methyl orange

End point – Change of colour from golden yellow to pale pink.

S No	Volume of NaOH	Burette reading		Volume of	Concordant
5.INO.	$(V_1)$ (ml)	Initial (ml)	Final (ml)	$HCl(V_2)(ml)$	value (ml)
1	20	0			
2	20	0			
3	20	0			

## **Calculation:**

Volume of  $Na_2CO_3$  (V<sub>1</sub>) = 20 ml

Normality of  $Na_2CO_3(N_1) = 0.1 N$ 

Volume of HCl (V<sub>2</sub>) = \_\_\_\_ ml

Normality of HCl  $(N_2) = ?$ 

According to Volumetric Principle,

 $V_1N_1 \ = V_2N_2$ 

$$N_2 = \frac{V_1 N_1}{V_2}$$

 $N_2 = 20 \times 0.1$ 

Normality of HCl  $(N_2) =$ \_\_\_\_N

## **Titration II:**

Link HCl solution Vs unknown NaOH

Burette solution	- Link HCl solution
Pipette solution	– NaOH solution
Indicator	– Phenolphthalein

End point – Disappearance of pink colour.

S No	Volume of	Burette reading		Volume of	Concordant
<b>5</b> .1NO.	NaOH (V <sub>2</sub> ) (ml)	Initial (ml)	Final (ml)	$HCl(V_1)(ml)$	value (ml)
1	20	0			
2	20	0			
3	20	0			

ml

Ν

# **Calculation:**

Volume of HCl (V<sub>1</sub>) = \_\_\_\_\_

Normality of HCl (N1)

Volume of NaOH ( $V_2$ ) = 20 ml

Normality of NaOH  $(N_2) = ?$ 

According to Volumetric Principle,

 $V_1N_1 = V_2N_2$ 

 $N_2 = \frac{V_1 N_1}{V_2}$  $= \frac{20}{20}$ 

Normality of NaOH  $(N_2) =$ \_\_\_\_\_N

Amount of NaOH in 1 litre of the given solution

= Normality x Equivalent weight

= \_\_\_\_\_ x 40

= \_\_\_\_\_ g

The amount of NaOH present in the whole of the given solution

 $= \frac{\text{Normality x Equivalent weight x 100}}{1000}$  $= \frac{x 100}{1000}$  $= \underline{g}.$ 

#### Titration II: Estimation of NaOH

Titration of link HCl Vs unknown NaOH solution

The given unknown solution was transferred to a 100 ml standard flask using a glass rod and funnel and was diluted upto the mark and the solution was shaken thoroughly for uniform concentration. The given pipette was washed with water and rinsed with the made up solution. Exactly 20 ml of this solution was pipetted out into a conical flask and a drop of phenolphthalein indicator was added. The solution turns pink in colour. This solution was titrated against standardized HCl taken in the burette with the disappearance of pink colour. The final burette reading was noted. The titration was repeated for concordant value. Using the concordant value, the strength and the amount of NaOH present in the whole of the given solution was calculated.

#### **Result:**

The amount of NaOH present in the whole of the given solution is = \_\_\_\_\_ g.

Ex. No.: 3

# Estimation of potassium permanganate using standard sodium hydroxide

#### Aim:

To estimate the amount of potassium permanganate present in the whole of the given solution using a standard solution of sodium hydroxide containing 4 g of the substance in 1000 ml and an approximately decinormal solution of oxalic acid.

## **Principle:**

NaOH is a strong base and it reacts with oxalic acid to form sodium oxalate.

 $2NaOH + C_2H_2O_4 \rightarrow Na_2C_2O_4 + 2H_2O$ 

The end point of the reaction can be determined by using phenolphthalein as indicator. Oxalic acid is dibasic and its equivalent weight is

Formula =  $\frac{\text{Molecular weight}}{2} = \frac{126}{2} = 63$ 

 $KMnO_4$  is an oxidizing agent and it can react with oxalic acid in the presence of a mineral acid like,  $H_2SO4$ .

 $2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$ 

 $H_2C_2O4 + [O] \rightarrow H_2O + 2CO_2$ 

Equivalent mass of KMnO<sub>4</sub>

 $= \frac{\text{Molecular weight}}{\text{Change in oxidizing state}} = \frac{158}{5} = 31.6$ 

# **Procedure:**

**Titration I:** Standardization of oxalic acid Titration of standard NaOH Vs link oxalic acid solution

Exactly 20 ml of oxalic acid solution was pipetted out into a clean conical flask, and one or two drops of phenolphthalein indicator was added. This solution was titrated against standard NaOH taken in the burette. The end point was the appearance of permanent pale pink colour.

Repeated the titration for concordant value. Using the concordant value, the strength of oxalic acid solution was calculated.

# Estimation of KMnO<sub>4</sub> using standard NaOH

Normality of standard NaOH = 0.1N

# **Titration I:**

Titration of standard NaOH Vs link oxalic acid solution

Burette solution	– Standard NaOH solution
Conical flask solution	– 20 ml of oxalic acid solution

Indicator – Phenolphthalein

End point – Appearance of permanent pale pink colour.

S No	Volume of oxalic	Burette reading		Volume of	Concordant
5.110.	acid $(V_2)$ (ml)	Initial (ml)	Final (ml)	NaOH $(V_1)$ (ml)	value (ml)
1	20	0			
2	20	0			
3	20	0			

# **Calculation:**

Volume of NaOH (V<sub>1</sub>) = \_\_\_\_ ml

Normality of NaOH  $(N_1) = 0.1N$ 

Volume of oxalic acid  $(V_2) = 20$  ml

Normality of oxalic acid  $(N_2) = ?$ 

According to Volumetric Principle,

$$\mathbf{V}_1\mathbf{N}_1 = \mathbf{V}_2\mathbf{N}_2$$

$$N_2 = \frac{V_1 N_1}{V_2}$$
$$= \frac{x \ 0.1}{20}$$

Normality of oxalic  $(N_2) =$ \_\_\_\_\_N

# **Titration II:**

Unknown KMnO<sub>4</sub> Vs standardized oxalic acid solution

Burette solution	– Standard NaOH solution
Conical flask solution	- 20 ml of oxalic acid solution
Indicator	– Phenolphthalein
End point	– Appearance of permanent pale pink colour.

S No	Volume of oxalic	Burette reading		Volume of	Concordant
5.110.	acid $(V_1)$ (ml)	Initial (ml)	Final (ml)	$KMnO_4 (V_2) (ml)$	value (ml)
1	20	0			
2	20	0			
3	20	0			

# Calculation:

Volume of Oxalic acid (V<sub>1</sub>) = 20 mlNormality of Oxalic acid (N<sub>1</sub>) \_\_ N = Volume of KMnO<sub>4</sub> (V<sub>2</sub>) ml Normality of KMnO<sub>4</sub> (N<sub>2</sub>) = ? According to Volumetric Principle,  $\mathbf{V}_1\mathbf{N}_1 = \mathbf{V}_2\mathbf{N}_2$  $= \underline{V_1 \ N_1}$  $N_2$  $V_2$ =<u>20 x</u>\_ Normality of  $KMnO_4(N_2) =$ \_\_\_\_\_N Amount of KMnO<sub>4</sub> present in 1 litre of the given solution

= Normality x Equivalent weight

=\_\_\_\_\_ x 31.6

= \_\_\_\_\_ g

Amount of KMnO<sub>4</sub> present in the whole of the given solution

Normality x Equivalent weight x 100

1000

=\_\_\_\_\_

=\_\_\_\_g

**Titration II:** Estimation of unknown KMnO<sub>4</sub>

Titration of unknown KMnO4 Vs standardized oxalic acid

The given unknown KMnO<sub>4</sub> solution was transferred to a 100 ml of standard flask using a glass rod and funnel and was diluted upto the mark and shaken thoroughly for uniform concentration. Exactly 20 ml of standardized oxalic acid solution was pipetted out into a clean conical flask. About 20 ml of dilute  $H_2SO_4$  was added and the mixture was heated to bearable warmth. It was then titrated against the unknown KMnO<sub>4</sub> solution taken in the burette. The end point was the appearance of permanent pale pink colour. Repeat the titration for concordant value. Using this value, the strength and the amount of KMnO<sub>4</sub> present in the whole of the given solution was calculated.

# **Result:**

The amount of KMnO<sub>4</sub> present in the whole of the given solution=\_\_\_\_\_ g.

# Ex. No.: 4

#### Estimation of ferrous sulphate using standard Mohr's salt

# Aim:

To estimate the amount of ferrous sulphate present in the whole of the given solution using a standard solution of Mohr's salt, containing 39.18 g of the substance in 1000 ml and an approximately decinormal solution of potassium permanganate.

#### **Principle:**

FeSO<sub>4</sub> exists in the crystalline state as, FeSO<sub>4</sub>.7H<sub>2</sub>O. It contains one Fe<sup>2+</sup> ions such as ferrous ion is present in Mohr's salt, [FeSO<sub>4</sub> (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>.6H<sub>2</sub>O] also. Both salts act as reducing agents because of the following tendency of Fe<sup>2+</sup>.

$$Fe^{2+} \rightarrow Fe^{3+} + e^{-}$$

Equivalent weight of  $FeSO_{4.7}H_2O$  is 278 and that of Mohr's salt is 392. KMnO<sub>4</sub> is an oxidizing agent because MnO<sub>4</sub><sup>-</sup> ion has the following tendency.

$$MnO_4 - + 5e^- + 8H^+ \rightarrow Mn^{2+} + 4H_2C$$

Equivalent weight of KMnO<sub>4</sub> is

<u>Molecular weight</u> =  $\frac{158}{5} = 31.6$ Change in oxidizing state 5

Oxidation- reduction in these cases takes place in the presence of H<sup>+</sup> ions.

#### **Procedure:**

#### Titration I: Standardization of KMnO<sub>4</sub>

Titration of standard Mohr's salt solution Vs link KMnO<sub>4</sub> solution

Exactly 20 ml of standard Mohr's salt solution (FAS) was pipetted out into a clean conical flask and about equal volume of dilute  $H_2SO_4$  was added to it. This mixture was titrated against KMnO<sub>4</sub> solution taken in a burette. The end point was the appearance of permanent pale pink colour. The titration was repeated for concordant value. Using this value the strength of KMnO<sub>4</sub> solution was calculated.

# Estimation of FeSO<sub>4</sub> using standard Mohr's salt

Normality of Standard Mohr's salt = 0.1 N

# **Titration 1:**

Standard Mohr's salt Vs link KMnO<sub>4</sub> solution

Burette solution	– Link KMnO4 solution
Conical flask solution	-20 ml Mohr's salt $+20$ ml of dilute H <sub>2</sub> SO <sub>4</sub>
Indicator	– Self

= ?

End point – Appearance of permanent pale pink colour.

S.No.	Volume of Mohr's	Burette reading		Volume of KMnO <sub>4</sub>	Concordant
	Salt $(V_1)$ (ml)	Initial (ml)	Final (ml)	$(V_2)$ (ml)	value (ml)
1	20	0			
2	20	0			
3	20	0			

# **Calculation:**

Volume of Mohr's salt (V1)	= 20 ml
Normality of Mohr's salt (N1)	= 0.1 N
Volume of KMnO <sub>4</sub> (V <sub>2</sub> )	=ml

Normality of KMnO<sub>4</sub> (N<sub>2</sub>)

According to Volumetric Principle

 $V_1N_1 = V_2N_2$ 

 $\begin{array}{ll} N_2 & = \frac{V_1}{V_2} \frac{N_1}{V_2} \\ & = \frac{20 \ x \ 0.1} \end{array}$ 

Normality of  $KMnO_4(N_2) =$ \_\_\_\_N

# **Titration II:**

Unknown FeSO<sub>4</sub> Solution Vs Standardized KMnO<sub>4</sub>

Burette solution	– Link KMnO <sub>4</sub> solution				
Conical flask solution	-20 ml FeSO <sub>4</sub> + 20 ml of dilute H <sub>2</sub> SO <sub>4</sub>				
Indicator	– Self				
<b>F</b> 1 1					

End point – Appearance of permanent pale pink colour.

S No	Volume of FeSO	Burette	Burette reading		Concordant	
5.INO.	$(V_2)$ (ml)	Initial (ml)	Final (ml)	$(V_1)$ (ml)	value (ml)	
1	20	0				
2	20	0				
3	20	0				

# Calculation:

Volume of $KMnO_4(V_1)$	= ml
Normality of KMnO <sub>4</sub> (N <sub>1</sub> )	= 0.1 N
Volume of FeSO <sub>4</sub> (V <sub>2</sub> )	= 20 ml
Normality of FeSO <sub>4</sub> (N <sub>2</sub> )	= ?
According to Volumetric Principle,	
$\mathbf{V}_1\mathbf{N}_1 = \mathbf{V}_2\mathbf{N}_2$	
N <sub>2</sub> = $\frac{V_1 N_1}{V_2}$	
$=\frac{x 0.1}{20}$	
Normality of FeSO <sub>4</sub> $(N_2) =$	N
Amount of FeSO <sub>4</sub> present in 1 litre of	of the given solution

= Normality x Equivalent weight

= \_\_\_\_\_x 278

= \_\_\_\_\_ g

The amount of FeSO<sub>4</sub> in the whole of the given solution

 $= \frac{\text{Normality x Equivalent weight x 100}}{1000}$  $= \frac{10}{10}$  $= 2 \frac{10}{10} = 2 \frac{$ 

Titration 2: Estimation of unknown FeSO<sub>4</sub>

Titration of unknown FeSO<sub>4</sub> Vs standardized KMnO<sub>4</sub> solution

The given unknown  $FeSO_4$  solution was transferred to a 100 ml of standard flask using a glass rod and funnel and was then diluted upto the mark. The solution was shaken thoroughly for uniform concentration and about equal volume of dilute  $H_2SO_4$  was added. This solution was titrated against standardized KMnO<sub>4</sub> solution taken in the burette. The end point was the appearance of permanent pale pink colour. The titration was repeated for concordant value. Using this value, the strength and the amount of FeSO<sub>4</sub> present in the whole of the given solution was calculated.

### **Result:**

The amount of FeSO<sub>4</sub> present in the whole of the given solution=\_\_\_\_\_ g.

#### Ex. No.:5

## Estimation of oxalic acid using standard ferrous sulphate

# Aim:

To estimate the amount of oxalic acid present in the whole of the given solution using a standard solution of ferrous sulphate containing 27.8 g in 1000 ml and an approximately decinormal solution of potassium permanganate.

#### **Principle:**

The reaction between oxalic acid and  $KMnO_4$  and that of FeSO<sub>4</sub> and  $KMnO_4$  takes place in the presence of a mineral acid,  $H_2SO_4$ .

 $2KMnO_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5[O]$ 

 $H_2C_2O_4 + [O] \rightarrow H_2O + 2CO_2$ 

 $FeSO_4 + H_2SO_4 + [O] \rightarrow Fe(SO_4)_3 + H_2O$ 

Equivalent weight of crystalline oxalic acid is 63 and that of FeSO<sub>4</sub> is 278.

#### **Procedure:**

Titration I: Standardization of KMnO4

Titration of standard FeSO<sub>4</sub> Vs link KMnO<sub>4</sub>

Exactly 20 ml of standard FeSO<sub>4</sub> was pipetted out into a clean conical flask. About equal volume of dilute  $H_2SO_4$  was added and the solution was titrated against KMnO<sub>4</sub> solution taken in the burette. The end point was the appearance of permanent pink colour. Repeat the titration for concordant value. Using this value, the strength of the KMnO<sub>4</sub> solution was calculated.

# Estimation of oxalic acid using standard FeSO<sub>4</sub>

Normality of standard  $FeSO_4 = 0.1 N$ 

# **Titration 1:**

Standard FeSO<sub>4</sub> Vs link KMnO<sub>4</sub> solution

Burette solution	– Link KMnO <sub>4</sub> solution		
Conical flask solution	– 20 ml of FeSO <sub>4</sub> + 20 r	nl of dilute H <sub>2</sub> SO	4 solution
Indicator	– Self		

– Appearance of permanent pale pink colour.

S.No.	Volume of FeSO <sub>4</sub> (V <sub>1</sub> ) (ml)	Burette reading Initial (ml) Final (ml)		Volume of KMnO <sub>4</sub> (V <sub>2</sub> ) (ml)	Concordant value (ml)	
1	20	0				
2	20	0				
3	20	0				
	· .					٢.

#### Calculation:

End point

Volum	e of $FeSO_4(V_1)$	= 20 ml
Norma	lity of FeSO <sub>4</sub> (N <sub>1</sub> )	= 0.1 N
Volum	e of KMnO <sub>4</sub> (V <sub>2</sub> )	=ml
Norma	lity of KMnO4 (N2)	= ?
Accord	ling to Volumetric Principle,	
$V_1N_1$	$= \mathbf{V}_2 \mathbf{N}_2$	
$N_2$	$=\frac{V_1 N_1}{V_2}$	
	$= \frac{20 \times 0.1}{20 \times 0.1}$	
Norma	lity of KMnO <sub>4</sub> (N <sub>2</sub> ) =	_ N

#### **Titration II:**

Unknown oxalic acid Vs Standardized KMnO<sub>4</sub>

Burette solution – Link KMnO<sub>4</sub> solution

Conical flask solution $-20 \text{ ml} \text{ oxalic acid} + 20 \text{ ml} \text{ of dilute } H_2SO_4$ Indicator $-Self$						
End no	nd point Appearance of pale pink colour					
Volume Bure			tte reading Volume of Concordant			
S.No.	of oxalic $(V_2)$ (ml)	Initial (ml)	Final (ml)	$\frac{\text{KMnO}_4}{(\text{V}_1) \text{ (ml)}}$	value (ml)	
1	20	0				
2	20	0				
3	20	0				
Calcul	ation:					
Volum	e of KMnO4	( <b>V</b> <sub>1</sub> )	=	ml		
Norma	lity of KMn	O4 (N1)	=	_		
Volum	e of oxalic a	cid ( $V_2$ )	= 20 ml			
Norma	lity of oxalio	c acid (N <sub>2</sub> )	= ?			
Accord	ling to Volu	metric Princip	le,			
$\mathbf{V}_1\mathbf{N}_1$	$= V_2 N_2$					
$N_2$	$= \frac{V_1 N_1}{V}$				· ·	
	$V_2$	· · · · · · · · · · · · · · · · · · ·				
	20					
Norma	lity of oxalio	c acid $(N_2) = $ _	N			
Amour	nt of oxalic a	cid present in	1 litre of the	given solution	I	
	$= \mathbf{N}$	ormality x Equ	uivalent weig	ht		
= <u>x 63</u>						
=g						
The an	nount of oxa	lic acid in the	whole of the	given solution	l	
	= Normality x Equivalent weight x 100					

= \_\_\_\_\_g.

#### Titration II: Estimation of unknown oxalic acid

Titration of unknown oxalic acid Vs standardized KMnO4

The given unknown oxalic acid solution was transferred to a 100 ml standard flask using a glass rod and funnel and was diluted upto the mark. The solution was shaken thoroughly for uniform concentration. Exactly 20 ml of the solution was pipetted out into a clean conical flask. About 20 ml of dilute H<sub>2</sub>SO<sub>4</sub> was added and the mixture was heated to a bearable warmth. It was then titrated against the KMnO<sub>4</sub> solution taken in the burette. The end point was the appearance of permanent pale pink colour. Repeat the titration for concordant value. Using this value, the strength and the amount of oxalic acid present in the whole of the given solution was calculated.

#### **Result:**

The amount of oxalic acid present in the whole of the given solution=\_\_\_\_\_ g.