Semester–II

19BTU212 **CHEMISTRY PRACTICAL-II** **4H–2C**

Instruction Hours/week :L: 0 T:0 P:3 **Marks: Internal:40 External: 60** **Total:100**

Course Objective

The student on successful completion of the course should learn the principles of volumetric analysis and to estimate the compounds by acidimetry, alkalimetry and permanganometry.

Course Outcome

1. The student learnt the principles of quantitative analysis of inorganic compounds.
2. Learnt the estimation of sample present in a solution by volumetric analysis.

Volumetric analysis

A. Acidimetry & Alkalimetry

1. Estimation of sodium carbonate using standard sodium hydroxide.
2. Estimation of sodium hydroxide using standard sodium carbonate.
3. Estimation of sulphuric acid using standard oxalic acid.
4. Estimation of potassium permanganate using standard sodium hydroxide.

B. Permanganometry

1. Estimation of ferrous sulphate using standard Mohr's salt.
2. Estimation of oxalic acid using standard ferrous sulphate.
3. Estimation of calcium-direct method.

References:

1.Thomas, A.O. (2012). *Practical Chemistry for B.Sc. Main Students.* Cannanore: Kerala, Scientific Book Centre.

2. Ramasamy, R. (2011). *Allied Chemistry Practical Book*. Karur: Priya Publications.

3. Venkateswaran, V., Veeraswamy, R., & Kulandaivelu A. R. (2015). *Basic Principles of Practical Chemistry* (2nd edition). New Delhi: S. Chand Publications.

ESE Marks Allocation

Category	**Marks**
Experiment	40
Viva-Voce	10
Record	10
Total	**60**

KARPAGAM ACADEMY OF HIGHER EDUCATION

(Deemed to be University Established Under Section 3 of UGC Act 1956)

Coimbatore – 641 021.

LECTURE PLAN

DEPARTMENT OF CHEMISTRY

Staff Name : Dr. N. Kannapiran
Subject Name : Chemistry Practical-II
Sub.Code : 19BTU212
Semester : II
Class : I-B.Sc Biotechnology

S.No.	Lecture Duration Period	Topics to be Covered	Support Material
1	4	Introduction and Procedure writing	R1
2	4	Estimation of Sodium carbonate	R1
3	4	Estimation of Sodium hydroxide	R1
4	4	Estimation of Sulphuric acid	R2
5	4	Estimation of Potassium permanganate	R1
6	4	Estimation of Ferrous sulphate	R1
7	4	Estimation of Oxalic acid	R1
8	4	Estimation of Calcium	R1
9	4	Revision and Viva voce	
10	4	Model Practical Examination	
	Total No. of Hours Planned For Practical's = 40		

References:

R1. Venkateswaran, V., Veeraswamy, R., & Kulandaivelu A. R. (2015). *Basic Principles of Practical Chemistry* (2nd edition). New Delhi: S. Chand Publications.

R2. Thomas, A.O. (2012). *Practical Chemistry for B.Sc. Main Students.* Cannanore: Kerala, Scientific Book Centre.

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A Laboratory Manual on

VOLUMETRIC ANALYSIS

Dr. A. Thangamani
&
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Department of Chemistry

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Contents

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.

$$\text{Normality} = \frac{\text{Mass in grams per liter of solution}}{\text{Gram equivalent mass of the substance}}$$

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.

Example for iodimetry $2Na_2S_2O_3 + I_2 \rightarrow 2NaI + Na_2S_4O_6$

Example for iodometry $2CuSO4 + 4KI \rightarrow 2K_2SO_4 + Cu_2I_2 + I_2$

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

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^+ and OH^-) 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.

$$\text{Equivalent mass of an acid} = \frac{\text{Molecular mass of the acid}}{\text{Basicity of the acid}}$$

Acid	Molecular mass	Number of replaceable H atoms (Basicity)	Equivalent mass
HCl	36.5	1	36.5
HNO_3	63	1	63
H_2SO_4	98	2	49
$H_2C_2O_4.2H_2O$	126	2	63

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.

$$\text{Equivalent mass of an alkali} = \frac{\text{Molecular mass of the alkali}}{\text{Acidity of the alkali}}$$

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

Alkali	Molecular mass	Acidity	Equivalent mass
NaOH	40	1	40
KOH	56	1	56
$NaHCO_3$	84	1	84
$KHCO_3$	100	1	100
Na_2CO_3	106	2	53
K_2CO_3	138	2	69
$CaCO_3$	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 $KMnO_4$ 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.

$$\text{Equivalent mass of potassium permanganate in acid medium} = \frac{316 \times 8}{80} = 31.6$$

(b) Equivalent mass of $K_2Cr_2O_7$ 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.

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

(c) Equivalent mass of MnO_2 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 $= \frac{87 \times 8}{16} = 43.5$

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, $FeSO_4.7H_2O$

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 $FeSO_4.7H_2O$ is the same as its molecular mass i.e., 278.

(b) Equivalent mass of Mohr's salt, $FeSO_4(NH_4)_2SO_4.\ 6H_2O$

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

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

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

(d) Equivalent mass of sodium oxalate, $Na_2C_2O_4$

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

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

5. Equivalent mass of metal halides

$$\text{Equivalent mass of metal halide} = \frac{\text{Molecular mass of halide}}{\text{No. of halogen ion furnished by one molecule}}$$

Thus, equivalent mass of NaCl $= \frac{58.46}{1} = 58.46$

$$KCl = \frac{74.46}{1} = 74.46$$

(a) Equivalent mass of silver nitrate

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

Equivalent mass of silver nitrate $= \frac{AgNO_3}{1} = \frac{170}{1} = 170$

(b) Equivalent mass of Potassium thiocynate

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

Hence, equivalent mass of potassium thiocynate = $\frac{KCNS}{1}$ = 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., $Na_2S_2O_3.5H_2O$ = 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 = $\frac{As_2O_3}{4}$ = 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 = $\frac{Na_3AsO_3}{2}$ = 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 × Equivalent mass = Mass per litre of the solution

Estimation of Na_2CO_3 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 (V_1) (ml)	Burette reading		Volume of HCl (V_2) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of NaOH (V_1) = 20 ml

Normality of NaOH (N_1) = 0.1 N

Volume of HCl (V_2) =_____ml

Normality of HCl (N_2) = ?

According to Volumetric Principle,

$V_1N_1 = V_2N_2$

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

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

Normality of HCl (N_2) =_________N

Titration II:

Link HCl solution Vs unknown Na_2CO_3:

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.

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_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

$$\text{Formula} = \frac{\text{Weight}}{2} = \frac{106}{2} = 53$$

Similarly, NaOH reacts with HCl as follows.

$$NaOH + HCl \rightarrow NaCl + H_2O$$

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

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

Procedure:

Titration 1: Standardization of hydrochloric acid

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.

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

Calculation:

Volume of HCl (V_1) =_______ml

Normality of HCl (N_1) =_______N

Volume of Na_2CO_3 (V_2) = 20 ml

Normality of Na_2CO_3 (N_2) = ?

According to Volumetric Principle,

$V_1N_1 = V_2N_2$

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

$$N_2 = \frac{\qquad}{20}$$

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

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

$$= \frac{\_\_\_\_\_\_\_\_ \text{ x 100}}{1000}$$

=________g.

Titration 2: Estimation of unknown Na_2CO_3

Titration of standard HCl Vs unknown Na_2CO_3

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_2CO_3 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.

Estimation of NaOH using standard Na_2CO_3

Normality of standard Na_2CO_3 = 0.1N

Titration I:

Standard Na_2CO_3 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 (V_1) (ml)	Burette reading		Volume of HCl (V_2) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of Na_2CO_3 (V_1) = 20 ml

Normality of Na_2CO_3 (N_1) = 0.1 N

Volume of HCl (V_2) =______ml

Normality of HCl (N_2) = ?

According to Volumetric Principle,

$V_1N_1 = V_2N_2$

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

$$N_2 = \underline{20 \times 0.1}$$

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.

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

$$\text{Formula} = \frac{\text{Weight}}{2} = \frac{106}{2} = 53$$

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

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

Procedure:

Titration: Standardization of HCl

Titration of link HCl Vs standard Na_2CO_3

Exactly 20 ml of standard Na_2CO_3 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.

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

Calculation:

Volume of HCl (V_1) =________ml

Normality of HCl (N_1) =________N

Volume of NaOH (V_2) = 20 ml

Normality of NaOH (N_2) = ?

According to Volumetric Principle,

$V_1N_1 = V_2N_2$

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

$$N_2 = \frac{\quad}{20}$$

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

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

$$= \frac{\_\_\_\_\_\_\_\_ \text{ x 100}}{1000}$$

=________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.

Estimation of H_2SO_4 using standard oxalic acid

Normality of standard Oxalic acid = 0.1 N

Titration I:

Standard $H_2C_2O_4$ Vs link NaOH solution

Burette solution – NaOH solution

Pipette solution – Oxalic acid solution

Indicator – Phenolphthalein

End point – Appearance of pale pink colour.

S.No.	Volume of $H_2C_2O_4$ (V_1) (ml)	Burette reading		Volume of NaOH (V_2) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of oxalic acid (V_1) = 20 ml

Normality of oxalic acid (N_1) = 0.1 N

Volume of NaOH (V_2) =________ml

Normality of NaOH (N_2) = ?

According to Volumetric Principle,

$V_1N_1 = V_2N_2$

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

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

Normality of NaOH (N_2) =_______N

Titration II:

Unknown H_2SO_4 Vs standardized NaOH

Burette solution – H_2SO_4 solution

Pipette solution – NaOH solution

Indicator – Phenolphthalein

End point – Disappearance of pink colour.

Ex. No.:3

Estimation of sulphuric acid using standard oxalic acid

Aim:

To estimate the amount of sulphuric acid present in the whole of the given solution using a standard solution of oxalic acid containing 6.3 g of the substance in 1000 ml and an approximately decinormal solution of sodium hydroxide.

Principle:

NaOH is a strong base and it can react with oxalic acid and H_2SO_4 follows.

$2NaOH + H_2C_2O_4 \rightarrow Na_2C_2O_4 + 2H_2O$

$2NaOH + H_2SO_4 \rightarrow Na_2SO_4 + H_2O$

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

$$\text{Formula} = \frac{\text{Molecular weight}}{2} = \frac{H_2C_2O_4..2H_2O}{2}$$

$$= \frac{126}{2} = 63$$

Sulphuric acid is dibasic and its equivalent weight is

$$\text{Formula} = \frac{\text{Molecular weight}}{2} = \frac{98}{2} = 49$$

One molecule of H_2SO_4 reacts with two molecules of NaOH. Therefore equivalent weight of H_2SO_4 is 49.

Procedure:

Titration I: Standardization of NaOH

Titration of standard oxalic acid Vs NaOH solution

Exactly 20 ml of standard 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 NaOH taken in the burette till the appearance of pink colour. The titration was repeated for concordant value. Using this value, the strength of NaOH solution was calculated.

S.No.	Volume of NaOH (V_2) (ml)	Burette reading		Volume of H_2SO_4 (V_1) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of NaOH (V_2) = 20 ml

Normality of NaOH (N_2) =______N

Volume of H_2SO_4 (V_1) =______ml

Normality of H_2SO_4 (N_1) = ?

According to Volumetric Principle,

$V_1N_1 = V_2N_2$

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

$$N_1 = \frac{20 \times 0.1}{}$$

Normality of H_2SO_4 (N_1) =________N

Amount of H_2SO_4 in 1 litre of the given solution

= Normality x Equivalent weight

=________x 49

=________g

The amount of H_2SO_4 present in the whole of the given solution

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

$$= \frac{\_\_\_\_\_\_\_\_}{10}$$

=_______g.

Titration II: Estimation of unknown H_2SO_4

Titration of unknown H_2SO_4 Vs standard NaOH

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. Exactly 20 ml of standardized NaOH solution was pipetted out into a clean conical flask and one or two drops of phenolphthalein indicator was added. This solution was titrated against the made-up H_2SO_4 solution taken in the burette. The end point was the disappearance of pink colour. Repeat the titration for concordant value. Using this value, the strength and the amount of H_2SO_4 present in the whole of the given solution was calculated.

Result:

The amount of H_2SO_4 present in the whole of the given solution=_________g.

Estimation of $KMnO_4$ 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 acid (V_2) (ml)	Burette reading		Volume of NaOH (V_1) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of NaOH (V_1) =_______ml

Normality of NaOH (N_1) = 0.1 N

Volume of oxalic acid (V_2) = 20 ml

Normality of oxalic acid (N_2) = ?

According to Volumetric Principle,

$$V_1N_1 = V_2N_2$$

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

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

Normality of oxalic acid (N_2) =_________N

Titration II:

Unknown $KMnO_4$ Vs standardized oxalic acid solution

Burette solution – Unknown $KMnO_4$

Conical flask solution – 20 ml of oxalic acid solution + 20 ml of H_2SO_4

Indicator – Self

End point – Appearance of permanent pale pink colour.

Ex. No.: 4

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

$$\text{Formula} = \frac{\text{Molecular weight}}{2} = \frac{H_2C_2O_4..2H_2O}{2}$$

$$= \frac{126}{2} = 63$$

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

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

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

$$\text{Equivalent mass of } KMnO_4 = \frac{\text{Molecular weight}}{\text{Change in oxidizing state}}$$

$$= \frac{158}{5} = 31.6$$

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.

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

Calculation:

Volume of Oxalic acid (V_1) = 20 ml

Normality of Oxalic acid (N_1) =________N

Volume of $KMnO_4$ (V_2) =________ml

Normality of $KMnO_4$ (N_2) = ?

According to Volumetric Principle,

$V_1N_1 = V_2N_2$

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

$$N_2 = \frac{20 \times \_\_\_\_}{}$$

Normality of $KMnO_4$ (N_2) =________N

Amount of $KMnO_4$ present in 1 litre of the given solution

= Normality x Equivalent weight

=________x 31.6

=________g

Amount of $KMnO_4$ present in the whole of the given solution

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

$$= \frac{\_\_\_\_\_\_\_\_}{10}$$

=________g

Titration II: Estimation of unknown $KMnO_4$

Titration of unknown $KMnO_4$ Vs standardized oxalic acid

The given unknown $KMnO_4$ 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 $KMnO_4$ 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 $KMnO_4$ present in the whole of the given solution was calculated.

Result:

The amount of $KMnO_4$ present in the whole of the given solution =_________g.

Estimation of $FeSO_4$ using standard Mohr's salt

Normality of Standard Mohr's salt = 0.1 N

Titration 1:

Standard Mohr's salt Vs link $KMnO_4$ solution

Burette solution – Link $KMnO_4$ solution

Conical flask solution – 20 ml Mohr's salt + 20 ml of dilute H_2SO_4

Indicator – Self

End point – Appearance of permanent pale pink colour.

S.No.	Volume of Mohr's Salt (V_1) (ml)	Burette reading		Volume of $KMnO_4$ (V_2) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of Mohr's salt (V_1) = 20 ml

Normality of Mohr's salt (N_1) = 0.1 N

Volume of $KMnO_4$ (V_2) =_____ml

Normality of $KMnO_4$ (N_2) = ?

According to Volumetric Principle

$$V_1N_1 = V_2N_2$$

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

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

Normality of $KMnO_4$ (N_2) =______N

Titration II:

Unknown $FeSO_4$ Solution Vs Standardized $KMnO_4$

Burette solution – Link $KMnO_4$ solution

Conical flask solution – 20 ml $FeSO_4$ + 20 ml of dilute H_2SO_4

Indicator – Self

End point – Appearance of permanent pale pink colour.

Ex. No.: 5

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:

$FeSO_4$ exists in the crystalline state as, $FeSO_4.7H_2O$. It contains one Fe^{2+} ions such as ferrous ion is present in Mohr's salt, $[FeSO_4 (NH_4)_2SO_4.6H_2O]$ also. Both salts act as reducing agents because of the following tendency of Fe^{2+}.

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

Equivalent weight of $FeSO_4.7H_2O$ is 278 and that of Mohr's salt is 392. $KMnO_4$ is an oxidizing agent because MnO_4^- ion has the following tendency.

$MnO_4^- + 5e^- + 8H^+ \rightarrow Mn^{2+} + 4H_2O$

$$\text{Equivalent mass of } KMnO_4 = \frac{\text{Molecular weight}}{\text{Change in oxidizing state}}$$

$$= \frac{158}{5} = 31.6$$

Oxidation- reduction in these cases takes place in the presence of H^+ ions.

Procedure:

Titration I: Standardization of $KMnO_4$

Titration of standard Mohr's salt solution Vs link $KMnO_4$ 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 $KMnO_4$ 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 $KMnO_4$ solution was calculated.

S.No.	Volume of $FeSO_4$ (V_2) (ml)	Burette reading		Volume of $KMnO_4$ (V_1) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of $KMnO_4$ (V_1) =_____ml

Normality of $KMnO_4$ (N_1) = 0.1 N

Volume of $FeSO_4$ (V_2) = 20 ml

Normality of $FeSO_4$ (N_2) = ?

According to Volumetric Principle,

$V_1N_1 = V_2N_2$

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

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

Normality of $FeSO_4$ (N_2) =________N

Amount of $FeSO_4$ present in 1 litre of the given solution

= Normality x Equivalent weight

=________x 278

=________g

The amount of $FeSO_4$ in the whole of the given solution

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

$$= \frac{\_\_\_\_\_\_\_\_}{10}$$

=________g.

Titration 2: Estimation of unknown $FeSO_4$

Titration of unknown $FeSO_4$ Vs standardized $KMnO_4$ 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 $KMnO_4$ 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 $FeSO_4$ present in the whole of the given solution was calculated.

Result:

The amount of $FeSO_4$ present in the whole of the given solution =___________g.

Estimation of oxalic acid using standard $FeSO_4$

Normality of standard $FeSO_4$ = 0.1 N

Titration 1:

Standard $FeSO_4$ Vs link $KMnO_4$ solution

Burette solution – Link $KMnO_4$ solution

Conical flask solution – 20 ml of $FeSO_4$ + 20 ml of dilute H_2SO_4 solution

Indicator – Self

End point – Appearance of permanent pale pink colour.

S.No.	Volume of $FeSO_4$ (V_1) (ml)	Burette reading		Volume of $KMnO_4$ (V_2) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of $FeSO_4$ (V_1) = 20 ml

Normality of $FeSO_4$ (N_1) = 0.1 N

Volume of $KMnO_4$ (V_2) =_____ml

Normality of $KMnO_4$ (N_2) = ?

According to Volumetric Principle,

$$V_1N_1 = V_2N_2$$

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

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

Normality of $KMnO_4$ (N_2) =_________N

Titration II:

Unknown oxalic acid Vs Standardized $KMnO_4$

Burette solution – Link $KMnO_4$ solution

Conical flask solution – 20 ml oxalic acid + 20 ml of dilute H_2SO_4

Indicator – Self

End point – Appearance of pale pink colour.

Ex. No.:6

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 $FeSO_4$ 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 $FeSO_4$ is 278.

Procedure:

Titration I: Standardization of $KMnO_4$

Titration of standard $FeSO_4$ Vs link $KMnO_4$

Exactly 20 ml of standard $FeSO_4$ was pipetted out into a clean conical flask. About equal volume of dilute H_2SO_4 was added and the solution was titrated against $KMnO_4$ 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 $KMnO_4$ solution was calculated.

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

Calculation:

Volume of $KMnO_4$ (V_1) =________ml

Normality of $KMnO_4$ (N_1) = ________

Volume of oxalic acid (V_2) = 20 ml

Normality of oxalic acid (N_2) = ?

According to Volumetric Principle,

$V_1N_1 = V_2N_2$

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

$$N_2 = \frac{\quad \times \quad}{20}$$

Normality of oxalic acid (N_2) =________N

Amount of oxalic acid present in 1 litre of the given solution

= Normality x Equivalent weight

=________x 63

=________g

The amount of oxalic acid in the whole of the given solution

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

$$= \frac{\_\_\_\_\_\_\_\_}{10}$$

=________g.

Titration II: Estimation of unknown oxalic acid

Titration of unknown oxalic acid Vs standardized $KMnO_4$

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_2SO_4 was added and the mixture was heated to a bearable warmth. It was then titrated against the $KMnO_4$ 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.

Estimation of calcium

Normality of standard $CaCl_2$ = 0.1 N

Titration 1:

Standard $CaCl_2$ Vs EDTA solution

Burette solution – EDTA solution

Conical flask solution – 20 ml $CaCl_2$ + 100 ml of distilled water + 2 ml of buffer solution

Indicator – Erichrome Black–T

End point – Change of colour from wine red o blue.

S.No.	Volume of $CaCl_2$ (V_1) (ml)	Burette reading		Volume of EDTA (V_2) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of $CaCl_2$ (V_1) = 20 ml

Normality of $CaCl_2$ (N_1) = 0.1 N

Volume of EDTA (V_2) =_____ml

Normality of EDTA (N_2) = ?

According to Volumetric Principle,

$$V_1N_1 = V_2N_2$$

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

$$N_2 = \underline{20 \times 0.1}$$

Normality of EDTA (N_2) =________N

Titration II:

Unknown $CaCl_2$ Vs EDTA solution

Burette solution – EDTA solution

Conical flask solution – 20 ml of $CaCl_2$ + 100 ml of distilled water + 2 ml of buffer solution

Indicator – Erichrome Black-T

End point – Change of colour from wine red to blue.

Ex. No.:7

Estimation of calcium-Direct method

Aim:

To estimate the amount of calcium present in the whole of the given solution

Principle:

Calcium ion forms a wine red coloured complex with Erichrome Black-T (EBT) indicator. At the end point calcium ions are complexed with EDTA solution. The liberated metal ion indicator gives blue colour. A drop of Mg-EDTA complex is added before titration.

Procedure:

Titration I: Standardization of EDTA solution

Titration of standard $CaCl_2$ Vs EDTA solution

A standard solution of calcium carbonate is prepared by dissolving 1.25 g in 1:1 HCl into 250 ml standard flask using distilled water. The solution is shaken well for uniform concentration. 20 ml of this solution is pipetted out into a clean conical flask. This solution is diluted to 100 ml with distilled water. 1 ml of Mg-EDTA complex, about 2 ml of buffer solution and 2 mg of EBT indicator is added. The solution turns wine red colour. This is titrated against EDTA taken in the burette. The end point is the colour change from wine red to blue. The titration is repeated for concordant value. From this value, the strength of EDTA solution is calculated.

S.No.	Volume of $CaCl_2$ (V_2) (ml)	Burette reading		Volume of EDTA (V_1) (ml)	Concordant value (ml)
		Initial (ml)	Final (ml)		
1	20	0			
2	20	0			
3	20	0			

Calculation:

Volume of EDTA solution (V_1) =______ml

Normality of EDTA solution (N_1) = ______

Volume of $CaCl_2$ (V_2) = 20 ml

Normality of $CaCl_2$ (N_2) = ?

According to Volumetric Principle,

$V_1N_1 = V_2N_2$

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

$$N_2 = \frac{\quad \times \quad}{20}$$

Normality of $CaCl_2$ (N_2) =________N

Amount of calcium present in 1 litre of the given solution

= Normality x Equivalent weight

=________x 40

=________g

The amount of calcium in the whole of the given solution

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

$$= \frac{\_\_\_\_\_\_\_\_}{10}$$

=________g.

Titration II: Estimation of unknown calcium

Titration of unknown $CaCl_2$ Vs standardization EDTA solution

The given unknown calcium chloride solution is made upto 100 ml in a standard flask 20 ml of this solution is pipetted into a clean conical flask and diluted to 100 ml with distilled water. 1 ml of Mg-EDTA complex, 2 ml of buffer solution and 2 mg of EBT is added. The solution turns wine red colour. This is titrated against EDTA solution taken in the burette. The end point is change of colour from wine red to blue. This titration is repeated for concordant value. From this value, the strength and the amount of calcium present in the whole of the given solution was calculated.

Result:

The amount of calcium present in the whole of the given solution =___________g.