

**PERIYAR INSTITUTE OF DISTANCE EDUCATION
(PRIDE)**

**PERIYAR UNIVERSITY
SALEM - 636 011.**

**B.Sc. CHEMISTRY
SECOND YEAR
PRACTICAL – II : VOLUMETRIC ESTIMATION**

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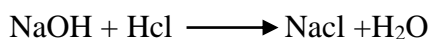
1.ESTIMATION OF SODIUM HYDROXIDE

AIM:

To estimate the amount of sodium hydroxide present in the whole of the given solution you are provided with sodium carbonate and approximately decinormal solution of hydrochloric acid as the link.

PRINCIPLE:

It is an acid base reaction. The neutralization takes place according to the equation.



PROCEDURE:

TITRATION:1

PREPARATION OF SODIUM CARBONATE SOLUTION:

About 1.5g of sodium carbonate are accurately weighed and transfer into a funnel placed over 250ml standard flask using glass rod. The crystals are carefully washed down into the flask by distilled water. The funnel is washed in to the flask. The crystals are dissolved and the solution is made up to the mark and shaken well for uniform concentration.

STANDARDISATION OF HCL SOLUTION:

The burette is first washed with tap water and then with distilled water. It is rinsed twice or thrice with the link Hcl solution. The burette is filled up to the zero mark without air gap in the nozzle. The pipette is washed with distilled water and then rinsing with sodium carbonate solution. Then exactly 20ml of standard sodium carbonate solution is pipette out in to clean conical flask. To this, a drop of methyl orange indicator is added. The solution becomes golden yellow in colour .It is titrated against hydrochloric taken in the burette. The end point is the appearance of pale pink colour. The burette reading is noted. The titration is repeated for concordant values. From the volume of Hcl solution, it's strength is calculated.

TITRATION:2

ESTIMATION OF SODIUM HYDROXIDE SOLUTION

The given NaOH solution is made up to in a 100ml standard flask. The solution is thoroughly shaken well to get a uniform concentrated solution. A 20ml pipette is washed with distilled water and then rinsed at least twice with the NaOH solution. Using the rinsed pipette, exactly 20ml of the made up solution is pipetted out in to clean conical flask . To this solution a drop of the phenolphthalein indicator is added . The solution turns pink in colour. It is titrated against standard Hcl solution is taken in the burette. Disappearance of

pink colour is the end point. The titration is repeated for concordant values. From the burette readings, the strength of sodium hydroxide and its amount is calculated.

WEIGHING OF SODIUM CARBONATE:

Object left	Accurate weight (g)
Weighing bottle + substance	_____
Weighing of empty bottle	

Accurate weight of sodium carbonate = _____ g

CALCULATION:

Normality of sodium carbonate = $\frac{\text{Weight of Na}_2\text{CO}_3 \times 1000}{\text{Equivalent weight of Na}_2\text{CO}_3}$

Equivalent weight of Na₂CO₃ = 53

Equivalent weight of NaOH = 40

Normality of sodium carbonate = ----- N

TITRATION :1

STANDARDISATION OF SODIUMCARBONATE SOLUTION:

Indicator --- Methyl Orange

End point --- Golden yellow to pale pink colour

SODIUMCARBONATE Vs HYDROCHLORIC ACID:

S.No	Volume of Na ₂ CO ₃ (ml)	Burette Reading		Concordant value of Hcl solution (ml)
		Initial (ml)	Final (ml)	

Calculation :

Volume of Sodium Carbonate (V1) =

Normality of Sodium Carbonate (N1) =

Volume of Hydrochloric solution (V2) =

$$\begin{aligned} \text{Normality of Hydrochloric solution} & \quad (N_2) = \\ & = \frac{V_1 N_1}{V_2} \end{aligned}$$

$$\text{Normality of Hcl solution} \quad (N_2) = \frac{V_2}{N}$$

TITRATION :2

ESTIMATION OF SODIUM HYDROXIDE SOLUTION

Indicator ----- Phenolphthaline

End point -----Disappearance of pink colour

Table :2

SODIUMHYDROXIDE Vs HYDROCHLORIC ACID

S.No	Volume of NaOH (ml)	Burette Reading		Concordant value of Hcl Solution (ml)
		Initial (ml)	Final (ml)	

Calculation :

$$\begin{aligned} \text{Volume of Hydrochloric solution} & \quad (V_2) = \\ \text{Normality of Hydrochloric solution} & \quad (N_2) = \\ \text{Volume of sodium hydroxide solution} & \quad (V_3) = \\ \text{Normality of sodium hydroxide solution} & \quad (N_3) = \\ & = \frac{V_2 N_2}{V_3} \end{aligned}$$

$$\text{Normality of sodium hydroxide solution} \quad (N_3) = \frac{V_2 N_2}{V_3} N$$

Amount of NaOH present in the whole of the given solution is,

$$= \frac{\text{Normality of NaoH} \times \text{Equivalent mass of NaoH}}{10}$$

$$= \text{—————} \text{g}$$

RESULT:

The amount of NaOH present in the whole of the given solution = ————— g.

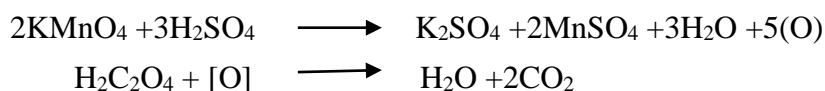
2. ESTIMATION OF OXALIC ACID

AIM:

To estimate the amount of Oxalic acid present in the whole of the given solution, being supplied with approximately deci normal solution of potassium permanganate and pure Oxalic acid crystals of AR quality.

PRINCIPLE:

The estimation is based on the reaction between potassium permanganate and Oxalic acid. Potassium permanganate oxidizes Oxalic acid in the presence of acid in hot condition.



Since a molecule of oxalic acid react with two equivalent of oxygen. It is equivalent mass is 63.

PROCEDURE:

TITRATION :1

STANDARDISATION OF POTASSIUM PERMANGANATE

SOLUTION:

About 1.5g of Oxalic acid crystals are accurately weighed and transfer into a funnel placed over 250ml standard flask. The crystals are carefully washed down into the flask by distilled water. The funnel is washed in to the flask. The crystals are dissolved and the solution made up to the mark and shaken well for uniform concentration.

Exactly 20ml of standard oxalic acid solution is pipetted out in to clean conical flask, about 20ml of dil. H_2SO_4 is added and the mixture is heated to 60°C - 80°C on a wire gauze. The solution is then titrated with potassium permanganate solution taken in the burette. The end point is the appearance of a pale pink colour. Burette reading is noted. The titration is repeated for concordant values.

TITRATION:2

ESTIMATION OF OXALIC ACID

The given Oxalic acid solution is made up to in a 100ml standard flask. Exactly 20ml of this solution is pipette out into a clean conical flask. To the solution about 20ml dil. H_2SO_4 is added it is heated to 60°C - 80°C on a wire gauze and the hot solution is titrated against potassium permanganate solution is taken in the burette . The end point is appearance of pale pink colour . The burette reading is noted. The titration is repeated for concordant values.

WEIGHING OF OXALIC ACID

<i>Object left</i>	<i>Accurate weight (g)</i>
Weighing bottle + substance	
Weighing of empty bottle	

Accurate weight of Oxalic acid = _____ g

CALCULATION:

Normality of Oxalic acid = $\frac{\text{Weight of Oxalic acid}}{\text{Equivalent weight of Oxalic acid}} \times \frac{1000}{250}$

Equivalent weight of Oxalic acid = 63

Normality of Oxalic acid (N_1) = _____ N

TITRATION :1

STANDARDISATION OF OXALIC ACID

Indicator ---- Self indicator

End point ---- Appearance of pale pink colour

Table :1

OXALIC ACID Vs POTASSIUM PERMANGANATE

<i>S.No</i>	<i>Volume of Oxalic acid (ml)</i>	<i>Burette Reading</i>		<i>Concordant value of KMnO4 Solution (ml)</i>
		<i>Initial (ml)</i>	<i>Final (ml)</i>	

Calculation :

Volume of Oxalic acid V_1 =

Normality of Oxalic acid (N_1) =

Volume of potassium permanganate (V_2) =

Normality of potassium permanganate (N_2) =

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

Normality of potassium permanganate (N2) = _____ N

TITRATION :1
ESTIMATION OF OXALIC ACID

Indicator ---- Self indicator

End point ---- Appearance of pale pink colour

Table :2

OXALIC ACID Vs POTASSIUM PERMANGANATE

<i>S. No</i>	<i>Volume of Oxalic acid</i> (ml)	<i>Burette Reading</i>		<i>Concordant value of KMnO4 Solution</i> (ml)
		Initial (ml)	Final (ml)	

Calculation:

Volume of potassium permanganate (V2) =

Normality of potassium permanganate (N2) =

Volume of Oxalic acid (V3) =

Normality of Oxalic acid (N3) =

$$= \frac{V_2 N_2}{V_3}$$

Normality of Oxalic acid (N3) = _____ N

Amount of Oxalic acid present in the whole of the given solution is,

$$= \frac{\text{Normality of Oxalic acid} \times \text{Equivalent mass of oxalic acid}}{10}$$

$$= \text{_____ g}$$

RESULT:

The amount of Oxalic acid present in the whole of the given solution is = _____ g

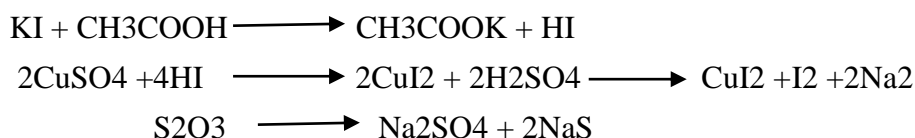
3. ESTIMATION OF COPPER

AIM:

To estimate the amount of copper present in the whole of the given solution being supplied with pure potassium dichromate crystals.

PRINCIPLE:

Copper is precipitated as cuprous iodide in acetic acid medium and an equivalent amount of iodine is liberated. The liberated iodine is titrated against sodium thio sulphate using starch as an indicator in a similar manner. The given copper sulphate solution is titrated against standard thio solution.



PROCEDURE:

TITRATION:1

SODIUM THIO SULPHATE Vs POTASSIUM DICHROMATE

About 1.25g of potassium dichromate are accurately weighed and transfer into a funnel placed over 250ml standard flask. Dissolved in distill water made upto zero mark. Exactly 20ml potassium dichromate of solution is pipette out in to a clean conical flask, about 20ml of dil.Hcl is added and mixed well . About 20ml of KI solution is added and mixture is titrated against thio solution drop by drop with constant shaking. When the dark colour becomes pale yellow, 2ml of freshly prepared starch solution is added, blue colour is obtained .The titration is continued till the end point is reached. The end point is the disappearance of blue colour leaving behind a green colour .The titration is repeated for concordant values. From the titre value, the strength of sodium thiosulphate is calculated.

TITRATION:2

ESTIMATION OF COPPER

The given unknown solution is made up to a 100ml standard flask. Exactly 20ml of the solution is pipette out into a clean conical flask and 20 ml potassium iodide is added and mixed well. It is titrated against sodium thio sulphate solution . When the dark brown colour becomes pale yellow 2 ml of starch solution is added and the titration is continued till a creamy white precipitate is obtained. A spoonful of ammonium thio cyanate is added and mixed well. If a blue colour is developed, the titration is continued till the blue colour is discharged.If there is no blue colour the burette reading is noted. The

titration is repeated for concordant values. The amount copper present in the whole of the given solution is calculated.

WEIGHING OF POTASSIUM DICHROMATE:

<i>Object left</i>	<i>Accurate weight (g)</i>
Weighing bottle + substance	_____
Weighing of empty bottle	

Accurate weight of potassium dichromate = _____ g

CALCULATION:

$$\text{Normality of potassium dichromate} = \frac{\text{Weight of potassium dichromate} \times 1000}{\text{Equivalent weight of potassium dichromate} \times 250}$$

Equivalent weight of potassium dichromate = 49

Equivalent weight of copper = 63.54

Normality of potassium dichromate (N₁) = _____ N

TITRATION :1

STANDARDISATION OF POTASSIUM DICHROMATE

Indicator --- Starch

End point --- Disappearance of blue colour

Table :1

OXALIC ACID Vs POTASSIUM PERMANGANATE

<i>S.No</i>	<i>Volume of potassium dichromate (ml)</i>	<i>Burette Reading</i>		Concordant value of sodium thio sulphate (ml)
		Initial (ml)	Final (ml)	

Calculation :

Volume of potassium dichromate V1) =

Normality potassium dichromate (N1) =

Volume of sodium thio sulphate (V2) =
 Normality sodium thio sulphate (N2) =

$$= \frac{V1N1}{V2}$$
 Normality of sodium thio sulphate (N2) = _____ N

Table :2

Indicator --- Starch

End point --- Disappearance of blue colour

COPPER Vs SODIUM THIO SULPHATE

S.No	Volume of Copper (ml)	Burette Reading		Concordant value of sodium thio sulphate (ml)
		Initial (ml)	Final (ml)	

Calculation :

Normality of sodium thio sulphate (N2) =
 Volume of sodium thio sulphate (V2) =
 Volume of copper (V3) =
 Normality of copper (N3) =

$$= \frac{V2N2}{V3}$$
 Normality of copper (N3) = _____ N

Amount of copper present in the whole of the given solution is,

$$= \frac{\text{Normality of copper} \times \text{Equivalent mass of copper}}{10}$$

= _____ g

RESULT:

The amount of copper present in the whole of the given solution = _____ g.

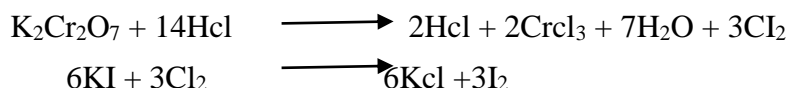
4. ESTIMATION OF POTASSIUM DICHROMATE

AIM:

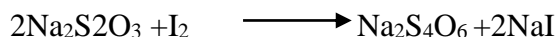
To estimate the amount of potassium dichromate present in the whole of the given solution being supplied with potassium dichromate crystals and an approximately deci normal solution of sodium thio sulphate..

PRINCIPLE:

In the presence of acid potassium dichromate liberate iodine from a solution of potassium iodide as per equation.



One molar mass of potassium dichromate liberated iodine is titrated against sodium thio sulphate using starch as indicator.



PROCEDURE:

STANDARDISATION OF POTASSIUM DICHROMATE

About 1.25g of potassium dichromate are accurately weighed and transfer into 250ml standard flask and the solution is made up to the mark. The solution is shaken well for uniform concentration.

TITRATION:1

ESTIMATION OF POTASSIUM DICHROMATE :

The burette is first washed with tap water and then with distilled water. It is rinsed twice or thrice with the link thio solution. The burette is filled up to the zero mark without air gap in the nozzle. Exactly 20ml of potassium dichromate solution is pipette out in to a clean conical flask. After this one test tube full of dil.Hcl is added, followed by 20ml 10% KI solution is added . The brown colour solution liberates iodine which is against thio sulphate taken in the burette. When the colour of solution becomes pale yellow, 2ml of freshly prepared starch solution is added and the titration is continued till end point is reached. The end point is the disappearance of blue colour, appearance of pale green colour, the end point is noted . The titration is repeated for concordant values. From the titre values the strength of thio sulphate is calculated.

TITRATION:2

ESTIMATION OF POTASSIUM DICHROMATE

The given potassium dichromate solution is made up to 100ml in a standard flask using funnel and glass rod. Exactly 20ml of the solution is pipette out into a clean conical flask. After this one test tube full of dil.HCl is added, followed by 20ml 10% KI solution is added. The brown colour solution liberates iodine which is against thio sulphate taken in the burette. When the solution becomes pale yellow 2 ml of freshly prepared starch solution is added and the titration is continued till the end point is reached. The end point is the disappearance of blue colour and appearance of green colour. The burette reading is noted. The titration is repeated for concordant values. From the titre value the strength and the amount of potassium dichromate is calculated.

WEIGHING OF POTASSIUM DICHROMATE:

<i>Object left</i>	<i>Accurate weight (g)</i>
Weighing bottle +substance	
Weighing of empty bottle	_____

Accurate weight of potassium dichromate = _____ g

Normality of potassium dichromate = $\frac{\text{Weight of potassium dichromate} \times 1000}{\text{Equivalent weight of potassium dichromate}} \times \frac{1000}{250}$

Equivalent weight of potassium dichromate = 49

Normality of potassium dichromate (N1) = _____ N

TITRATION :1
STANDARDISATION OF POTASSIUM

DICHROMATE

Indicator --- Starch

End point ---Blue colour change to pale green.

Table :1

POTASSIUM DICHROMATE Vs SODIUM THIO SULPHATE

S.No	Volume of potassium dichromate (ml)	Burette Reading		Concordant value of sodium thio sulphate (ml)
		Initial (ml)	Final (ml)	

Calculation :

Volume of potassium dichromate

V1) =

Normality potassium dichromate

(N1) =

Volume of sodium thio sulphate

(V2) =

Normality sodium thio sulphate

(N2) =

$$= \frac{V1N1}{V2}$$

Normality of sodium thio sulphate

(N2) = _____ N

TITRATION:2

ESTIMATION OF POTASSIUM DICHROMATE

Indicator ---Starch

End point --- Disappearance of blue colour

POTASSIUM DICHROMATE Vs SODIUM THIO SULPHATE

S.No	Volume of potassium dichromate (ml)	Burette Reading		Concordant value of sodium thio sulphate (ml)
		Initial (ml)	Final (ml)	

Calculation :

Volume sodium thio sulphate (V2) =

Normality of sodium thio sulphate (N 2) =

Volume of potassium dichromate (V3) =

Normality potassium dichromate (N3) =

$$= \frac{V_2 N_2}{V_3}$$

Normality of potassium dichromate (N3) = _____ N

Amount of copper present in the whole of the given solution is,

$$= \frac{\text{Normality of } \times \text{Equivalent weight of copper potassium dichromate}}{10}$$

$$= \text{_____ g}$$

RESULT:

The amount of potassium dichromate present in the whole of the given solution

$$= \text{_____ g}$$

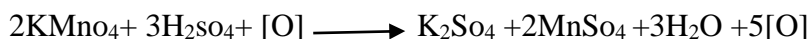
5. ESTIMATION OF FERROUS ION

AIM:

To estimate the amount of ferrous ion present in the whole of the given solution. You are provided with the sample of ferrous ammonium sulphate and approximately deci normal of KMnO_4 as link solution.

PRINCIPLE:

KMnO_4 act as an oxidizing agent, in presence of dil H_2SO_4 , KMnO_4 oxidize FeSO_4 in to $\text{Fe}_2(\text{SO}_4)_3$



Two molecular mass of both FeSO_4 and moles react with two equivalent of oxygen.

PROCEDURE:

TITRATION:1

STANDARDISATION OF POTASSIUM PERMANGANATE:

Above 10 gm of pure ferrous ammonium sulphate is weighed accurately using a chemical balance. The crystals carefully transferred into a 250 ml standard and distilled water is added up to the zero mark and the standard flask is shaken well for uniform concentration. The burette is first washed with tap water and rinse with distilled water and KMnO_4 . The same solution filled with the zero mark. The pipette is washed and rinse with distilled water and ferrous ammonium sulphate solution is pipette out into a clean conical flask Exactly 20 ml of this solution is pipette out into a clean conical flask. One test tube full of dil H_2SO_4 is added. The solution is titrated against KMnO_4 is taken in the burette. The end point is the appearance of pale pink color. The end point is noted. Titration is repeated for concordant value.

TITRATION:2

ESTIMATION OF FERROUS ION:

The given unknown solution is carefully transfer into a clean 100 ml standard flask, using distilled water is added and rinse with distilled water and transfer in to standard flask. The process repeated for two (or) three times. The solution is made up to the mark using distilled water. The solution is shaken well for concentration. Exactly 20 ml of this solution, one test tube full of dil. H_2SO_4 is added. The solution is titrated against $KMnO_4$ taken in the burette. The end point is appearance of pale pink color. The end point is noted. The titration is repeated for concordant value.

WEIGHING OF FAS:

<i>Object left</i>	<i>Accurate weight (g)</i>
Weighing bottle +substance	
Weighing of empty bottle	

Accurate weight of FAS = _____ g

CALCULATION:

$$\begin{aligned} \text{Normality of Ferrous ammonium sulphate} &= \frac{\text{Weight of FAS} \times 1000}{\text{Equivalent weight of FAS} \times 250} \\ &= \text{_____ N} \end{aligned}$$

$$\text{Equivalent weight of FAS} = 392$$

$$\text{Equivalent weight of ferrous ion} = 55.84$$

TITRATION:1

STANDARDISATION OF POTASSIUM PERMANGANATE:

Indicator --- Self indicator

End point --- appearance of pale pink color

FAS Vs KMnO_4 SOLUTION

S.No	Volume of FAS (ml)	Burette Reading		Concordant value of KMnO_4 (ml)
		Initial (ml)	Final (ml)	

Calculation:

Volume of FAS (V_1) =

Normality of FAS (N_1) =

Volume of Potassium Permanganate (V_2) =

Normality of Potassium Permanganate (N_2) =

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

Normality of KMnO_4 (N_2) = _____ N

TITRATION :2

ESTIMATION OF FERROUS ION:

Indicator --- Self indicator

End point --- Appearance of pale pink color

FERROUS ION Vs KMnO_4

S.No	Volume of Ferrous ion (ml)	Burette Reading		Concordant value of KMnO_4 (ml)
		Initial (ml)	Final (ml)	

Calculation:

Volume of Ferrous ion (V₃) =

Normality of Ferrous ion (N₃) =

Volume of Potassium Permanganate (V₂) =

Normality of Potassium Permanganate (N₂) =

$$N_3 = \frac{V_2 N_2}{V_3}$$

Normality of K₂MnO₄ (N₃) = _____ N

The amount of ferrous ion present in the whole of given solution is

$$= \frac{\text{Normality of ferrous ion} \times \text{equivalent weight of ferrous ion}}{10}$$

$$= \text{_____ g}$$

RESULT:

The amount of ferrous ion present in the whole of given solution is

$$= \text{_____ g}$$

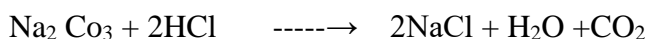
6. ESTIMATION OF SODIUM CARBONATE

AIM:

To estimate the amount of sodium carbonate present in the whole of given solution, being supplied with sodium hydroxide and an approximately decinormal HCL acid solution.

PRINCIPLE:

The estimation depends on the reaction between sodium carbonate between sodium carbonate and hydro chloric acid.



Since the carbonate react with two equivalents of hydrochloric acid solution . Equivalent mass of sodium carbonate is 53.

PROCEDURE:

TITRATION: 1

STANDARDISATION OF HCL ACID SOLUTION

About 1.0 gm of sodium Hydroxide is accurately weighed in a chemical balance and transferred into a 250 ml standard flask. Distilled water is added up to the mark and shaken well for uniform concentration. The burette is washed with tap water and rinse with distilled water and hydrochloric acid is filled in the burette. Exactly 20 ml of standard sodium hydroxide solution is pipette out into clean conical flask. Two drops of methyl orange as a indicator are added to the solution. It is titrated against hydrochloric acid solution taken in the burette. At the end point, the solution will change from golden yellow to red orange. The titration are repeated until concordant values are obtained.

TITRATION: 2

ESTIMATION OF SODIUM CARBONATE

The given sodium carbonate solution is carefully transferred into a 100 ml standard flask using fennel and glass rod. It is thoroughly shaken well uniform concentrated solution. Exactly 20 ml of this solution is pipette out in to a conical flask. To the solution, two drops of methyl orange indicator is added. It is titrated against the HCL acid solution. The end point is the change from golden yellow to red orange colour. From the titre values, the strength of sodium carbonate solution and hence its weight is calculated.

WEIGHING OF SODIUM CARBONATE:

<i>Object left</i>	<i>Accurate weight (g)</i>
Weighing bottle +substance	_____
Weighing of empty bottle	

Accurate weight of sodium carbonate = -----g

CALCULATION:

$$\begin{aligned} \text{Normality of sodium Hydroxide} &= \frac{\text{Weight of NaOH}}{\text{Equivalent mass of NaOH}} \times \frac{1000}{250} \\ &= \text{_____ N} \end{aligned}$$

Equivalent weight of Hydroxide = 40

TITRATION:1

STANDARDISATION OF HCL ACID SOLUTION

Indicator: Methyl orange

End point: golden yellow to red orange

Sodium Hydroxide Vs Hydrochloric solution

<i>S.No</i>	<i>Volume of NaOH (ml)</i>	<i>Burette Reading</i>		<i>Concordant value of HCL (ml)</i>
		<i>Initial (ml)</i>	<i>Final (ml)</i>	

Calculation:

Volume of Sodium hydroxide (V₁)=

Normality of Sodium hydroxide (N₁)=

Volume of HCL acid solution (V₂)=

Normality of HCL acid solution (N₂)=

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

Normality of HCL acid solution (N₂) = _____ N

TITRATION: 2
ESTIMATION OF SODIUM CARBONATE

Indicator: Methyl orange

End point: Golden yellow to red orange

Sodium carbonate Vs HCL solution

S.No	Volume of Na ₂ CO ₃ (ml)	Burette Reading		Concordant value of HCL solution(ml)
		Initial (ml)	Final (ml)	

Calculation:

Volume of Hcl solution (V₂)=

Normality of Hcl solution (N₂)=

Volume of Na₂CO₃ solution (N₃)=

Normality of Na₂CO₃ solution (N₃)=

$$N_3 = \frac{V_2 N_2}{V_3}$$

Normality of sodium carbonate (N₃) = _____ N

The amount of sodium carbonate is given in the whole of the given solution

= Normality Na₂CO₃ X equivalent mass of Na₂CO₃

$$= \frac{10}{\text{_____}} \text{ g}$$

RESULT:

The amount of sodium carbonate in the whole of the given solution is = _____ g

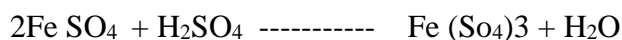
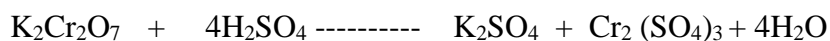
7. ESTIMATION OF FERROUS ION (USING INTERNAL INDICATOR)

AIM:

To estimate the amount of ferrous ion present in the whole of given solution, being supplied with ferrous ammonium sulphate and an approximately decinormal potassium dichromate solution.

PRINCIPLE:

Potassium dichromate is the link solution, The estimate is based on the reaction of potassium dichromate is acid medium with ferrous sulphate,



PROCEDURE:

About 10 gm of ferrous ammonium sulphates is weight accurately using chemical balance. The crystals are carefully transferred into a 250 ml standard flask-using funnel. About 10 ml of dil. Sulphuric acid is added and distilled water is added up to the mark. Then standard flask is shaken well for uniform concentration.

TITRATION: 1

Standard FAS Vs $\text{K}_2\text{Cr}_2\text{O}_7$

Exactly 20 ml of standard ferrous ammonium sulphate is pipette out into a clean conical flask One test tube of dilute Sulphuric acid is added followed by 3 drops of phosphoric acid and 4drops of diphenyl amine is added. It is titrated against potassium dichromate solution taken in the burette. The end point is the color change from green to violet. The titration is repeated for concordant value.

TITRATION:2

ESTIMATION OF FERROUS ION

The given unknown solution is made up to the 100 ml standard flask. The solution is shaken well for uniform concentration. Exactly 20 ml of solution is pipette out into a clean conical flask. Add 1 test dil. H_2SO_4 followed by 3 drops of phosphoric acid and 4 drops of dipheny ammine indicator. The solution is titrated against $\text{K}_2\text{Cr}_2\text{O}_7$ solution is taken in the burette. The end point is color change from green to violet color. The titration is repeated for concordant value.

WEIGHING OF FAS:

<i>Object left</i>	<i>Accurate weight (g)</i>
Weighing bottle +substance	_____
Weighing of empty +bottle	

Accurate weight of FAS = _____ g

CALCULATION:

Normality of Ferrous ammonium sulphate = $\frac{\text{Weight of FAS}}{\text{Equivalent weight of FAS}} \times \frac{1000}{250}$

= _____ g

Equivalent weight of FAS = 392

Equivalent weight of ferrous ion = 55.84

TITRATION : 1

Standardization of $K_2Cr_2O_7$

Indicator: Diphenyl amine

End point: Green color to violet color

Standard FAS Vs $K_2Cr_2O_7$

<i>S.No</i>	<i>Volume of FAS (ml)</i>	<i>Burette Reading</i>		<i>Concordant value of $K_2Cr_2O_7$ (ml)</i>
		<i>Initial (ml)</i>	<i>Final (ml)</i>	

Calculation:

$$\begin{aligned}
 \text{Volume of Ferrous ammonium sulphate} & \quad (V_1)= \\
 \text{Normality of Ferrous ammonium sulphate} & \quad (N_1)= \\
 \text{Volume of potassium dichromate} & \quad (V_2)= \\
 \text{Normality of potassium dichromate} & \quad (N_2)= \\
 & \quad (N_2) = \frac{V_1 N_1}{V_2} \\
 \text{Normality of potassium dichromate} & \quad (N_2) = \frac{\quad}{\quad} \quad N
 \end{aligned}$$

TITRATION :2**ESTIMATION OF FERROUS ION:****Indicator:** Di phenyl amine**End point:** Green color to violet color**Ferrous ion FAS Vs K₂Cr₂O₇**

<i>S.No</i>	<i>Volume of Ferrous ion (ml)</i>	<i>Burette Reading</i>		<i>Concordant value of (K₂Cr₂O₇) ml</i>
		<i>Initial (ml)</i>	<i>Final (ml)</i>	

Calculation:

$$\begin{aligned}
 \text{Volume of K}_2\text{Cr}_2\text{O}_7 & \quad (V_2) = \\
 \text{Normality of K}_2\text{Cr}_2\text{O}_7 & \quad (N_2) = \\
 \text{Volume of ferrous ion} & \quad (V_3) = \\
 \text{Normality of ferrous ion} & \quad (N_3) = \\
 & \quad (N_3) = \frac{V_2 N_2}{V_3} \\
 \text{Normality of ferrous ion} & \quad (N_3) = \frac{\quad}{\quad} \quad N
 \end{aligned}$$

The amount of ferrous ion given in the whole of the given solution is

$$= \frac{\text{Normality ferrous ion} \times \text{equivalent weight of ferrous ion}}{10}$$
$$= \text{————— g}$$

RESULT:

The amount of ferrous ion present in the whole of the given solution is

$$= \text{————— g}$$

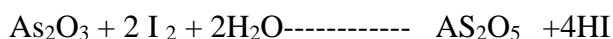
8. ESTIMATION OF ARSENIOS OXIDE

AIM:

To estimate the amount of Arsenious oxide present in the whole of given solution being supplied with Arsenious oxide.

PRINCIPLE:

Iodine oxidizes arsenious oxide. Since the reaction is reversible, all the hydrogen iodide formed must be removed as soon as formed. Sodium bicarbonate is added in excess, which removes hydrogen iodide, and it has no action on iodine. As arsenious oxide is not freely soluble in water, it is transferred into sodium arsenite.



PROCEDURE:

PREPARATION OF STANDARD ARSENIOS OXIDE SOLUTION:

About 1.2 gm of arsenious oxide is accurately weighed and transferred into a clean beaker carefully. A few drops of distilled water and 2 or 3 pellets of sodium hydroxide are added and stirred well to dissolve arsenious oxide. The clear solution is transferred into a 200 ml standard flask. The beaker is rinsed repeatedly with distilled water and transferred the washings into the flask and made up to the mark with distilled water and made it uniform. A small paper strip is placed in the stopper to prevent the stopper from stuck up.

TITRATION: 1

STANDARDISATION OF IODINE SOLUTION

The given iodine solution is taken in a clean and rinsed burette. Exactly 20 ml of the standard arsenious oxide is pipette out into a clean conical flask. A drop of phenolphthalein is added, followed by dilute hydrochloric acid drop by drop till the pink colour is discharged. About two spoonful of sodium bicarbonate is added and titrated against standard iodine solution using starch as an indicator. The end point is disappearance of blue color. The titration is repeated for concordant value and the amount of arsenious oxide in the whole of the given solution is calculated.

TITRATION: 2

ESTIMATION OF ARSENIOS OXIDE:

The whole of given solution is made up to 100 ml standard flask and shaken well for uniform concentration. Exactly 20ml of the made up solution is pipetted out in to clean conical flask. A drop of phenolphthalein is added followed by dilute hydrochloric acid drop by drop till the pink colour is discharged. About two spoonful of sodium bicarbonate is added and titrated

against standard iodine solution using starch as an indicator. The end of point is disappearance of blue color. The titration is repeated to get concordant values and the amount of arsenious oxide in the whole of the given solution is calculated.

WEIGHING OF ARSENIOS OXIDE

<i>Object left</i>	<i>Accurate weight (g)</i>
Weighing bottle substance	
Weighing of empty bottle	_____

Accurate weight of Arsenious oxide = _____ g

CALCULATION:

$$\text{Normality of Arsenious oxide} = \frac{\text{Weight of AS}_2\text{O}_3}{\text{Equivalent weight of AS}_2\text{O}_3} \times \frac{1000}{250}$$

$$\text{Equivalent weight of AS}_2\text{O}_3 = 49.45$$

TITRATION:1

Indicator: Starch

End point: appearance of blue color

Standard arsenic acid Vs Iodine solution

<i>S.No</i>	<i>Volume of Iodine solution (ml)</i>	<i>Burette Reading</i>		<i>Concordant value of Arsenious oxide (ml)</i>
		<i>Initial (ml)</i>	<i>Final (ml)</i>	

Calculation:

$$\begin{aligned}
 \text{Volume of Arsenious oxide} & \quad (V_1) = \\
 \text{Normality of Arsenious oxide} & \quad (N_1) = \\
 \text{Volume of Iodine solution} & \quad (V_2) = \\
 \text{Normality of Iodine solution} & \quad (N_2) = \\
 & \quad (N_2) = \frac{V_1 N_1}{V_2} \\
 \text{Normality of Iodine solution} & \quad (N_2) = \text{—————} N
 \end{aligned}$$

TITRATION: 2**ESTIMATION OF ARSENIUS OXIDE****Indicator:** Starch**End point:** appearance of blue color

<i>S.No</i>	<i>Volume of Iodine solution (ml)</i>	<i>Burette Reading</i>		<i>Concordant value of Arsenious oxide</i>
		<i>Initial (ml)</i>	<i>Final (ml)</i>	

Calculation:

$$\begin{aligned}
 \text{Volume of Iodine solution} & \quad (V_2) = \\
 \text{Normality of Iodine solution} & \quad (N_2) = \\
 \text{Volume of Arsenious oxide} & \quad (V_3) = \\
 \text{Normality of Arsenious oxide} & \quad (N_3) = \\
 & \quad (N_3) = \frac{V_2 N_2}{V_3} \\
 \text{Normality of Arsenious oxide} & \quad (N_3) = \text{—————} N
 \end{aligned}$$

The amount of Arsenious oxide present in the whole of the given solution

$$= \frac{\text{Normality of As}_2\text{O}_3 \times \text{equivalent mass of As}_2\text{O}_3}{10}$$
$$= \text{—————g}$$

RESULT:

The amount of Arsenious oxide present in the whole of the given solution

$$= \text{————— g}$$

9. ESTIMATION OF ZINC

AIM:

To estimate the amount of zinc present in the whole of the given zinc sulphate solution, being supplied with approximately decinormal potassium ferro cyanide solution and pure Zinc sulphate crystals.

PRINCIPLE:

Zinc ions and ferro cyanide ions react in neutral or acid medium as follows.



The Zinc ions get precipitated as protassium Zinc ferro cyanide. The end point of the reaction can be detected using internal indicator like diphenylamine. In a small quantity of potassium ferrocyanide is also added to the ferrocyanide solution. Then the end point is the colour change from blue to yellowish green. Since there is a change in Oxidation state of Zn to Zn²⁺ ions.

PROCEDURE:

TITRATION: 1

STANDARDISATION OF FERROCYANIDE SOLUTION:

1.4 gm of zinc sulphate is accurately weighed in a balance and transferred into 100 ml standard flask. Added distilled water up to the mark and shaken well for uniform concentration.

Exactly 20 ml of Zinc sulphate solution is pipette out into a conical flask. To the solution about 50 ml of 4N Sulphuric acid, 1 gm of ammonium sulphate and 2 to 4 drops of diphenylamine indicator are added and titrated against ferrocyanide solution taken in the burette. The end point is the color change from blue to yellowish green. The titration is repeated concordant values are obtained.

TITRATION:2

ESTIMATION OF ZINC:

The given salt solution is made up to 100 ml in a standard flask with distilled water. It shaken well for uniform concentration, exactly 20 ml of this solution is pipette out into a clean conical flask. About 50 ml of 4N sulfuric acid 1gm of ammonium sulphate and 2 to 4 drops diphenylamine indicator are added and the solution is titrated against the standard potassium ferrocyanide solution. The end point is the color change from blue to yellowish green. The titration is repeated to get concordant values.

WEIGHING OF ZINC SULPHATE:

<i>Object left</i>	<i>Accurate weight (g)</i>
Weighing bottle + substance	_____
Weighing of empty bottle	

Accurate weight of Zinc sulphate = _____ g

CALCULATION:

$$\text{Normality of Zinc sulphate} = \frac{\text{Weight of Znso}_4}{\text{Equivalent weight of Znso}_4} \times \frac{1000}{100}$$

$$= \text{_____ N}$$

Equivalent weight of ZnSO₄ = 143.78

Equivalent weight of Zn²⁺ ion = 65.38

TITRATION :1

STANDARDISATION OF FERROCYANIDE SOLUTION

Indicator : diphenylamine indicator

End point: Blue to yellowish green

ZnSO₄ Vs ferrocyanide solution

S.No	Volume of Znso ₄ solution (ml)	Burette Reading		Concordant value of Ferro cyanide solution (ml)
		Initial (ml)	Final (ml)	

Calculation:

$$\begin{aligned}
 \text{Volume of ZnSO}_4 \text{ solution} & \quad (V_1) = \\
 \text{Normality of ZnSO}_4 \text{ solution} & \quad (N_1) = \\
 \text{Volume of Ferro cyanide solution} & \quad (V_2) = \\
 \text{Normality of Ferro cyanide solution} & \quad (N_2) = \\
 N_2 & = \frac{V_1 N_1}{V_2} \\
 (N_2) & = \text{—————} N
 \end{aligned}$$

TABLE :2**Indicator :** diphenylamine indicator**End point:** Blue to yellowish green**Zinc solution Vs Ferrocyanide solution**

S.No	Volume of Zn ²⁺ solution (ml)	Burette Reading		Concordant value of Ferro cyanide solution (ml)
		Initial (ml)	Final (ml)	

Calculation:

$$\begin{aligned}
 \text{Volume of Ferrocyanide solution} & \quad V_1 = \\
 \text{Normality of Ferrocyanide solution} & \quad N_1 = \\
 \text{Volume of Zn}^{2+} \text{ solution} & \quad V_2 = \\
 \text{Normality of Zn}^{2+} \text{ solution} & \quad N_2 = \\
 N_2 & = \frac{V_1 N_1}{V_2} \\
 (N_2) & = \text{—————} N
 \end{aligned}$$

The amount of Zn²⁺ ion Present in the given solution.

$$\begin{aligned}
 & = \frac{\text{Normality Zn}^{2+} \text{ ion} \times \text{equivalent mass}}{10} \\
 & = \text{—————} \text{ g}
 \end{aligned}$$

RESULT:

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

$$= \text{—————} \text{ g}$$

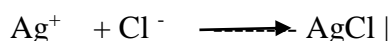
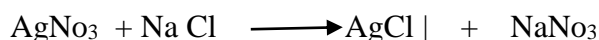
10. ESTIMATION OF CHLORIDE [MOHR'S METHOD]

AIM:

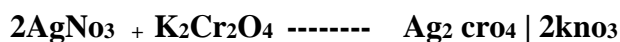
To estimate the amount of chloride present in the whole of the given sodium chloride solution being supplied with an approximately N/20 AgNO₃ solution and sodium chloride crystals of AR quality.

PRINCIPLE:

It is an example of precipitation reaction. The reaction between chloride and silver nitrate is direct and simple.



The completion of the reaction in this case is observed by employing K₂CrO₄ solution as the indicator. At the end point the yellow colour of chromate changes into reddish brown due to the reaction.



K₂Cr₂O₄ indicator will not be precipitated as Ag₂CrO₄ until all the chlorides in the solution have been precipitated as AgCl.

PROCEDURE:

TITRATION: 1

STANDARDISATION OF SILVER NITRATE SOLUTION

About 0.72 gm of NaCl is accurately weighed in the balance and transferred into a 250 ml standard flask and made up to mark with distilled water. The solution is shaken well for uniform concentration.

Exactly 20 ml of this standard NaCl solution is pipette out into a clean conical flask. 1 ml of 2% K₂CrO₄ indicator solution is added to it. The solution turns yellow in color. It is titrated against silver nitrate (AgNO₃) solution taken in the burette. During each addition of silver nitrate solution, the conical flask is shaken well. The faint reddish brown tinge is the end point. The end point is noted. The titration is repeated for concordant values. From the volume of silver nitrate solution, the strength of silver nitrate is calculated.

TITRATION:2

ESTIMATION OF CHLORIDE

The given chloride solution is made up to 100 ml in a standard flask using distilled water. The solution is shaken well for uniform concentration. Exactly 20 ml of this solution is pipette out into a clean conical flask. To this solution, 1 ml of 2% K_2CrO_4 indicator solution is added. It is titrated against $AgNO_3$, taken in the burette

The addition of $AgNO_3$ solution is continued until the solution produced a reddish brown tinge, the titration is repeated for concordant value. From the titre value, the strength of chloride and hence its amount is calculated.

<i>Object left</i>	<i>Accurate weight (g)</i>
Weighing bottle +substance	
Weighing of empty bottle	

Accurate weight of Sodium chloride = _____ g

CALCULATION:

$$\begin{aligned} \text{Normality of Sodium chloride} &= \frac{\text{Weight of NaCl}}{\text{Equivalent weight of NaCl}} \times \frac{1000}{250} \\ &= \text{_____} \text{N} \end{aligned}$$

$$\text{Equivalent weight of NaCl} = 58.50$$

TITRATION:1**Table.1****Indicator:** potassium chromate**End point:** faint reddish brown tinge**Sodium chloride Vs AgNO₃ solution**

<i>S.No</i>	<i>Volume of Nacl solution (ml)</i>	<i>Burette Reading</i>		<i>Concordant value of AgNO₃ (ml)</i>
		<i>Initial (ml)</i>	<i>Final (ml)</i>	

Calculation:

Volume of Nacl solution

 $V_1 =$

Normality of Nacl solution

 $N_1 =$ Volume of AgNO₃ solution $V_2 =$ Normality of AgNO₃ solution $N_2 =$

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

$$(N_2) = \frac{\quad}{\quad} N$$

TITRATION:2**Indicator:** potassium chromate**End point:** faint reddish brown tinge**Chloride ion solution Vs AgNO₃ solution**

S.No	Volume of Chloride ion solution (ml)	Burette Reading		Concordant value of AgNO₃ solution (ml)
		Initial (ml)	Final (ml)	

Calculation:

$$\begin{aligned} \text{Volume of AgNO}_3 \text{ solution} & \quad (V_2) = \\ \text{Normality of AgNO}_3 \text{ solution} & \quad (N_2) = \\ \text{Volume of Chloride solution} & \quad (V_3) = \\ \text{Normality of Chloride solution} & \quad (N_3) = \\ & \quad (N_3) = \frac{V_2 N_2}{V_3} \\ & \quad (N_3) = \text{—————} N \end{aligned}$$

The amount of Chloride ion present in the given solution is

$$\begin{aligned} & = \frac{\text{Normality chloride ion} \times \text{equivalent mass}}{10} \\ & = \text{—————} \text{ g} \end{aligned}$$

RESULT:

The amount of chloride ion present in the whole of the given solution

$$= \text{—————} \text{ g}$$