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B.Sc. CHEMISTRY

SECOND YEAR

PRACTICAL – II : VOLUMETRIC ESTIMATION

Prepared by :
V. Karthikeyan
Dept. of Chemistry
Sri Vidhya Mandir College
Katteri, Uthangarai
Krishnagiri – Dt.

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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.

$$NaOH + Hcl \longrightarrow Nacl + H_2O$$

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 = $\frac{\text{Weight of } Na_{22}CO3 \text{ X } 1000}{\text{Equivalent weight of } 250}$

Equivalent weight of Na2CO3 = 53 Na_2CO_3

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_2CO_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) =

Normality of Hydrochloric solution (N2) =

$= V1N1$

$\frac{}{V2}$

Normality of Hcl solution (N2) = ———— 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 :

Volume of Hydrochloric solution (V2) =

Normality of Hydrochloric solution (N2) =

Volume of sodium hydroxide solution (V3) =

Normality of sodium hydroxide solution (N3) =

$= \frac{V2N2}{V3}$

Normality of sodium hydroxide solution (N_3) = ———— N

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

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

= ———— 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.

$$2KMnO_4 + 3H_2SO_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 3H_2O + 5(O)$$

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

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

PROCEDURE:

TITRATION :1

STANDARDISATION OF POTASSIUM PERMONGANATE 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 dill. H_2SO_4 is added and the mixture is heated to 60^o c- 80^o 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^o c-80^o 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

Object left	*Accurate weight (g)*
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

S.No	*Volume of Oxalic acid* (ml)	*Burette Reading*		*Concordant value of KMnO4 Solution* (ml)
		Initial (ml)	Final (ml)	

Calculation :

Volume of Oxalic acid V1) =

Normality of Oxalic acid (N1) =

Volume of potassium permanganate (V2) =

Normality of potassium permanganate (N2) =

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

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

S. No	***Volume of Oxalic acid*** (ml)	***Burette Reading***		***Concordant value of KMnO4 Solution*** (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) =

$$= \frac{V2N2}{V3}$$

Normality of Oxalic acid (N3) = ———— N

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

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

= ———— 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 copeer sulphate solution is titrated against standard thio solution.

$$KI + CH3COOH \longrightarrow CH3COOK + HI$$

$$2CuSO4 + 4HI \longrightarrow 2CuI2 + 2H2SO4 \longrightarrow CuI2 + I2 + 2Na2$$

$$S2O3 \longrightarrow Na2SO4 + 2NaS$$

PROCEDURE:

TITRATION:1

SODIUM THIO SULPHATE Vs POTTASIUM 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 dis appearance 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 POTTASIUM DICHROMATE:

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

Accurate weight of potassium dichromate = ———— g

CALCULATION:

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

Equivalent weight of potassium dichromate = 49

Equivalent weight of copper = 63.54

Normality of potassium dichromate (N_1) = ———— N

TITRATION :1

STANDARDISATION OF POTASSIUM DICHROMATE

Indicator --- Starch

End point --- Disappearance of blue colour

Table :1

OXALIC ACID Vs POTASSIUM PERMANGANATE

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 $(N2) = \frac{\qquad}{\qquad}$ 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 $(N3) = \frac{\qquad}{\qquad}$ N

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

$$= \frac{\text{Normality of copper x Equivalentmass of copper}}{10}$$

$$= \frac{\qquad}{\qquad} \text{ g}$$

RESULT:

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

$= \frac{\qquad}{\qquad}$ 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.

$$K_2Cr_2O_7 + 14Hcl \longrightarrow 2Hcl + 2Crcl_3 + 7H_2O + 3CI_2$$

$$6KI + 3Cl_2 \longrightarrow 6Kcl + 3I_2$$

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

$$2Na_2S2O_3 + I_2 \longrightarrow Na_2S_4O_6 + 2NaI$$

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 POTTASIUM 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 POTTASIUM DICHROMATE

The given potassium dichromate solution is made up to100ml 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 poin t 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.

WEGHING OF POTTASIUM DICHROMATE:

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

Accurate weight of potassium dichromate = _____ g

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

Equivalent weight of potassium dichromate = 49

Normality of potassium dichromate (N1) = _____ N

TITRATION :1

STANDARDISATION OF POTTASIUM 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) =

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

TITRATION:2

ESTIMATION OF POTASSIUM DICHROMATE

Indicator ---Starch

End point --- Disappearance of blue colour

POTTASIUM 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) =

$$= \frac{V2N2}{V3}$$

Normality of potassium dichromate $(N3) = \frac{\quad\quad}{\quad\quad} N$

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

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

$$= \frac{\quad\quad}{\quad\quad} g$$

RESULT:

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

$$= \underline{\quad\quad} g$$

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 dill H_2SO_4 ,$KMnO_4$ oxidize $Feso_4$ in to $Fe_2(so_4)_3$

$2KMno_4 + 3H_2so_4 + [O] \longrightarrow K_2So_4 + 2MnSo_4 + 3H_2O + 5[O]$

$2FeSO_4 .N\ H_2So_4 + 5H_2so_4 + 5[o] \dashrightarrow 5\ Fe\ [SO_4]_3 + 10(NH_4)_2SO_4 + 5H_20$

$2FeSO_4 + H_2So_4 + [O] \dashrightarrow Fe\ [SO_4]_3 + H_20$

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. $H_2 SO_4$ 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:

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

Accurate weight of FAS = _____ g

CALCULATION:

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

= _____ N

Equivalent weight of FAS = 392

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) =

$$N2 = \frac{V1N1}{V2}$$

Normality of $KMno_4$ $(N_2) = \frac{\qquad\qquad}{\qquad\qquad}$ 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 (V_3) =

Normality of Ferrous ion (N_3) =

Volume of Potassium Permanganate (V_2) =

Normality of Potassium Permanganate (N_2) =

$$N3 = \frac{V2N2}{V_3}$$

Normality of $Kmno_4$ (N3) = _____ N

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

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

= _____ g

RESULT:

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

= _____ 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.

$Na_2 Co_3 + 2HCl \quad \text{-----}\rightarrow \quad 2NaCl + H_2O + CO_2$

Since the carbonate react with two equvalents 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 titrre values, the strength of sodium carbonate solution and hence its weight 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 Hydroxide = $\frac{\text{Weight of NaOH}}{\text{Equivalent mass of NaOH}} \times \frac{1000}{250}$

= _____ N

Equivalent weight of Hydroide = 40

TITRATION:1

STANDARDISATION OF HCL ACID SOLUTION

Indicator: Methyl orange

End point: golden yellow to red orange

Sodium Hydroxide Vs Hydrochloric solution

S.No	***Volume of NaOH (ml)***	***Burette Reading***		***Concordant value of* HCL** (ml)
		Initial (ml)	***Final*** (ml)	

Calculation:

Volume of Sodium hydroxide (V_1)=

Normality of Sodium hydroxide (N_1)=

Volume of HCL acid solution (V_2)=

Normality of HCL acid solution (N_2)=

$$N2 = \frac{V1N1}{V2}$$

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_2Co_3 (ml)***	***Burette Reading***		*Concordant value of* **HCL solution(ml)**
		Initial (ml)	***Final*** (ml)	

Calculation:

Volume of Hcl solution (V_2)=

Normality of Hcl solution (N_2)=

Volume of Na_2co_3 solution (N_3)=

Normality of Na_2co_3 solution (N_3)=

$$N3 = \frac{V2N2}{V3}$$

Normality of sodium carbonate (N3) = ———— N

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

$$= \frac{\text{Normality } Na_2Co_3 X \text{ equivalent mass of } Na_2Co_3}{10}$$

= ———— 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,

$K_2Cr_2O_7 + 4H_2SO_4$ ---------- $K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O$

$2FeSO_4 + H_2SO_4$ ---------- $Fe(So_4)3 + H_2O$

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 $K_2Cr_2O_7$

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 $K_2Cr_2O_7$ 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:

Object left	*Accurate weight (g)*
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 $K_2Cr_2O_7$

S.No	***Volume of FAS (ml)***	***Burette Reading***		***Concordant value of*** **$K_2Cr_2O_7$** (ml)
		Initial (ml)	***Final*** (ml)	

Calculation:

Volume of Ferrous ammonium sulphate (V_1)=

Normality of Ferrous ammonium sulphate (N_1)=

Volume of potassium dichromate (V_2)=

Normality of potassium dichromate (N_2)=

$$(N_2) = \frac{V1N1}{V2}$$

Normality of potassium dichromate $$(N2) = \frac{\quad}{\quad} N$$

TITRATION :2

ESTIMATION OF FERROUS ION:

Indicator: Di phenyl amine

End point: Green color to violet color

Ferrous ion FAS Vs $K_2Cr_2O_7$

S.No	***Volume of Ferrous ion (ml)***	***Burette Reading***		***Concordant value of*** **($K_2Cr_2O_7$)** ml
		Initial (ml)	***Final*** (ml)	

Calculation:

Volume of $K_2Cr_2O_7$ (V_2) =

Normality of $K_2Cr_2O_7$ (N_2) =

Volume of ferrous ion (V_3) =

Normality of ferrous ion (N_3) =

$$(N3) = \frac{V2N2}{V3}$$

Normality of ferrous ion $$(N3) = \frac{\quad}{\quad} N$$

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

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

= ———— g

RESULT:

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

= ———— g

8. ESTIMAION OF ARSENIOUS 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 bi carbonate 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.

$As_2O_3 + 2\ I_2 + 2H_2O$---------- AS_2O_5 +4HI

PROCEDURE:

PREPARATION OF STANDARD ARSENIOUS 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 hy dilute hydrochloric acid drop by drop till the pink colour is discharged. About two spoonful of sodium bi carbonate 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 ARSENIOUS 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 phenopthalein is added followed b 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 ARSENIOUS OXIDE

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

Accurate weight of Arsenious oxide = _____ g

CALCULATION:

$$\text{Normality of Arsenious oxide} = \frac{\text{Weight of } AS_2O_3}{\text{Equivalent weight of } AS_2O_3} \times \frac{1000}{250}$$

Equivalent weight of AS_2O_3 = 49.45

TITRATION:1

Indicator: Starch

End point: appearance of blue color

Standard arsenic acid Vs Iodine solution

S.No	***Volume of Iodine solution (ml)***	***Burette Reading***		***Concordant value of Arsenious oxide*** (ml)
		Initial (ml)	***Final*** (ml)	

Calculation:

Volume of Arsenious oxide (V_1) =

Normality of Arsenious oxide (N_1) =

Volume of Iodine solution (N_2) =

Normality of Iodine solution (N_2) =

$$(N2) = \frac{V1N1}{V2}$$

Normality of Iodine solution $(N2) = \frac{V1N1}{V2}$ N

TITRATION: 2

ESTIMATION OF ARSENIOUS OXIDE

Indicator: Starch

End point: appearance of blue color

S.No	***Volume of* Iodine solution *(ml)***	***Burette Reading***		***Concordant value of* Arsenious oxide**
		Initial (ml)	***Final*** (ml)	

Calculation:

Volume of Iodine solution (V_2) =

Normality of Iodine solution (N_2) =

Volume of Arsenious oxide (V_3) =

Normality of Arsenious oxide (N_3) =

$$(N3) = \frac{V2N2}{V3}$$

Normality of Arsenious oxide $(N_3) = \frac{V2N2}{V3}$ N

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

$$= \frac{\text{Normality of } As_2O_3 \text{ x equivalent mass of } As_2O_3}{10}$$

= _____g

RESULT:

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

= _____ 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.

$3Zn^{+2} + 2K_4[Fe(CN)_6] \longrightarrow K_2Zn_3[Fe(CN)_6]_2 + 6K^+$

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 Zn^{2+} 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:

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

Accurate weight of Zinc sulphate = _____ g

CALCULATION:

Normality of Zinc sulphate = $\frac{\text{Weight of } Znso_4 \times 1000}{\text{Equivalent weight of } Znso_4 \quad 100}$

= _____N

Equivalent weight of $ZnSO_4$ = 143 .78

Equivalent weight of Zn^{2+} ion = 65.38

TITRATION :1

STANDARDISATION OF FERROCYANIDE SOLUTION

Indicator : diphenylamine indicator

End point: Blue to yellowish green

$ZnSO_4$ 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:

Volume of $Znso_4$ solution (V_1) =

Normality of $Znso_4$ solution (N_1) =

Volume of Ferro cyanide solution (V_2) =

Normality of Ferro cyanide solution (N_2) =

$$N2 = \frac{V1N1}{V2}$$

(N2) = ———— N

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:

Volume of Ferrocyanide solution V_1=

Normality of Ferrocyanide solution N_1=

Volume of Zn^{2+}solution V_2=

Normality of Zn^{2+}solution N_2=

$$N2 = \frac{V1N1}{V2}$$

(N2) = ———— N

The amount of Zn^{2+} ion Present in the given solution.

$$= \frac{\text{Normality } Zn^{2+} \text{ ion x equivalent mass}}{10}$$

= ———— g

RESULT:

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

= ———— g

10.ESTIMATION OF CHLORIDE [MOHR'S METHOD]

AIM:

To estimate the amount of cholride present in the whole of the given sodium chloride solution being supplied with an approximately N/20 $AgNo_3$ solution and sodium chloride crystals of AR quality.

PRICIPLE:

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

$$AgNo_3 + Na\ Cl \longrightarrow AgCl\ | \ + \ NaNo_3$$

$$Ag^+ + Cl^- \longrightarrow AgCl\ |$$

The completion of the reaction in this case is observed by employing K_2CrO_4solution as the indicator. At the end point the yellow colour of chromate changes into reddish brown due to the reaction.

$$\mathbf{2AgNo_3 + K_2Cr_2O_4 \text{ -------- } Ag_2\ cro_4 \mid 2kno_3}$$

$K_2Cr_2O_4$ indicator will not be precipitated as $\mathbf{Ag_2\ cro_4}$ 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% K_2CrO_4 indicator solution is added to it. The solution turns yellow in color. It is titrated against silver nitrate ($AgNO_3$) solution taken in the burette. During each addition of silver nirate 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 valurs. 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 is 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.

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

Accurate weight of Sodium chloride = _____ g

CALCULATION:

Normality of Sodium chloride = $\frac{\text{Weight of NaCl}}{\text{Equivalent weight of NaCl}} \times \frac{1000}{250}$

= _____N

Equivalent weight of NaCl = 58.50

TITRATION:1

Table.1

Indicator: potassium chromate

End point: faint reddish brown tinge

Sodium chloride Vs $AgNO_3$ solution

S.No	***Volume of Nacl solution (ml)***	***Burette Reading***		***Concordant value of $AgNO_3$*** (ml)
		Initial (ml)	***Final*** (ml)	

Calculation:

Volume of Nacl solution V_1 =

Normality of Nacl solution N_1 =

Volume of $AgNO_3$solution V_2 =

Normality of $AgNO_3$solution N_2 =

$$N2 = \frac{V1N1}{V2}$$

(N2) = ~~N~~

TITRATION:2

Indicator: potassium chromate

End point: faint reddish brown tinge

Chloride ion solution Vs $Agno_3$ solution

S.No	**Volume of Chloride ion solution (ml)**	**Burette Reading**		**Concordant value of $AgNO_3$ solution** (ml)
		Initial (ml)	**Final** (ml)	

Calculation:

Volume of $AgNO_3$ solution (V_2) =

Normality of $AgNo_3$ solution (N_2) =

Volume of Chloride solution (V_3) =

Normality **of** Chloride solution (N_3) =

$$(N_3) = \frac{V2N2}{V3}$$

(N3) = ———— N

The amount of Chloride ion present in the given solution is

$$= \frac{\text{Normality chloride ion x equivalent mass}}{10}$$

=———— g

RESULT:

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

= ———— g