

VOLUMETRIC ANALYSIS

(LAB MANUAL)

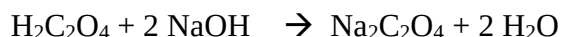
ESTIMATION OF HYDROCHLORIC ACID

AIM:

To estimation the amount of Hydrochloric acid present in the whole of the given solution. You are provided with a standard Oxalic acid solution.

PRINCIPLE:

The neutralisation of Hydrochloric acid by Oxalic acid takes place according to the equation.



PROCEDURE:

Titration - I

STANDARDISATION OF OXALIC ACID

About 1.525g of Oxalic acid according weighted and dissolved in 250ml of Standard measuring flask.

The burette is washed well with water and rinsed with hydrochloric acid and filled with the same hydrochloric acid upto Zero mark. The 20ml pipette is washed well with water and then rinsed with Oxalic acid solution. Exactly 20ml of the Standard Oxalic acid solution is pipette out into a clean conical flask. To that one (or) two drops of Phenolphthalein indicator is added. The solution turns to yellow colour. This solution is titrated against NaOH taken in the burette. The colour end point is appearance of pale pink colour. The titrations are repeated to get concordant value. From the titrate value the strength NaOH can be calculated.

Titration - I

STANDARDISATION OF OXALIC ACID

Oxalic acid Vs NaOH

Sl.No.	Volume of Oxalic acid (ml)	Burette Reading		Volume of NaOH (ml)	Concordant Value (ml)	Indicator
		Initial (ml)	Final (ml)			

Volume of Oxalic acid (V_1) = 20ml.

Normality of Oxalic acid (N_1) = -----N.

Volume of NaOH (V_2) =-----ml.

Normality of NaOH (N_2) = ?

$$V_1 N_1 = V_2 N_2$$

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

Titration II

ESTIMATION OF HYDROCHLORIC ACID

A given solution of Hydrochloric acid is completely transferred into a 100ml Standard measuring flask and the solution is made up to the mark with distilled water and shaken well for uniform concentration. The burette is filled with the same NaOH up to the Zero mark. 20ml of HCl solution is pipetted out into a clean conical flask. A drop of Phenolphthalein indicator is added to this solution the solution turns to Pink colour and it is titrated against NaOH taken in the burette. The end point is the disappearance of Pink colour. The titration is repeated to get concordant value. From this value the normality and amount HCl present in the whole of the given solution is determined.

Eq.wt of HCl = 36.5g

Titration II

ESTIMATION OF HYDROCHLORIC ACID

HCl Vs NaOH

Sl.No.	Volume of HCl (ml)	Burette Reading		Volume of NaOH (ml)	Concordant Value (ml)	Indicator
		Initial (ml)	Final (ml)			

Volume of NaOH (V_1) = 20ml.

Normality of NaOH (N_1) = -----N.

Volume of HCl (V_2) = -----ml.

Normality of **HCl** (N_2) = ?

$$V_1 N_1 = V_2 N_2$$

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

$$= \text{-----}N.$$

$$\left\{ \begin{array}{l} \text{The amount of HCl present} \\ \text{in the} \\ \text{whole of the given solution} \end{array} \right\} = \text{Normality} \times \text{Equivalent weight} \times 1/10.$$

$$= \text{-----}g.$$

RESULT:

The amount of hydrochloric acid present in the whole of the given solution= -----.

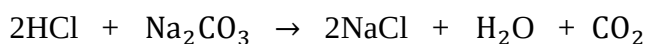
ESTIMATION OF SODIUM CARBONATE

AIM:

To estimate the amount of Na_2CO_3 present in the whole of the given solution you are provided with pure crystals of Sodium Carbonate and approximately decinormal solution of HCl.

PRINCIPLE:

The neutralization of hydrochloric acid by the Sodium Carbonate takes place according to the equation.



PROCEDURE

TITRATION - I

STANDARDISATION OF SODIUM CARBONATE

About 1.3g of Na_2CO_3 crystals are weighted and made upto 250ml of standard measuring flask, the crystals are dissolved with distilled water and the solution is made upto the zero mark. The solution is shaken well for uniform concentration. The burette is washed with water and linked with HCl and filled with same HCl acid upto zero mark. 20ml of solution is Na_2CO_3 is ppt out into a clean conical flask. A drop of methyl orange indicator is added to this solution turns to yellow colour and it is titrated against HCl acid taken in the burette till the yellow colour change to pink. The end point is noted. The titration is repeated to get concordant value from this value the normality of HCl is calculated.

TITRATION - I

STANDARDISATION OF SODIUM CARBONATE

Na_2CO_3 Vs HCl

Sl.No.	Volume of Na_2CO_3 (ml)	Burette Reading		Volume of HCl (ml)	Concordant Value (ml)	Indicator
		Initial (ml)	Final (ml)			

Volume of *standard* Na_2CO_3 (V_1) = 20ml.

Normality of standard Na_2CO_3 (N_1) = -----N.

Volume of HCl (V_2) =-----ml.

Normality of HCl (N_2) = ?

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 N_1}{V_2}$$
$$= \text{-----N.}$$

TITRATION - II

ESTIMATION OF SODIUM CARBONATE

The given solution is Sodium Carbonate is completely transferred into 100ml standard measuring flask and the solution is made for uniform concentration. The burette is mark. 20ml of Sodium Carbonate solution is a drop of methyl orange an indicator to this solution. The solution turns yellow colour and taken in the burette fill the yellow colour change into the pink. The end point is noted. The titration is repeated to get concordant value from this value the strength and the amount of Na_2CO_3 present in the whole of the given solution is calculated.

Equivalent weight of Sodium Carbonate = 53 g/eq.

TITRATION - II

ESTIMATION OF SODIUM CARBONATE

Na_2CO_3 Vs HCl

Sl.No.	Volume of Na_2CO_3 (ml)	Burette Reading		Volume of HCl (ml)	Concordant Value (ml)	Indicator
		Initial (ml)	Final (ml)			

Volume of HCl (V_1) = -----ml.

Normality of HCl (N_1) = -----N.

Volume of Na_2CO_3 (V_2) = 20ml.

Normality of Na_2CO_3 (N_2) = ?

$$V_1 N_1 = V_2 N_2$$

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

$$= \text{-----}N.$$

$$\left\{ \begin{array}{l} \text{The amount of } \mathbf{Na_2CO_3} \text{ present} \\ \text{in the} \\ \text{whole of the given solution} \end{array} \right\} = \text{Normality} \times \text{Equivalent weight} \times 1/10.$$

$$= \text{-----}g.$$

RESULT:

The amount of Sodium Carbonate present in the whole of the given solution=-----.

ESTIMATION OF OXALIC ACID

AIM:

To estimate the amount of the oxalic acid present in the hole of the given solution being supplied with approximately decinormal solutions of Pottassium Permanganate and pure FeSO_4 solutions crystals of quantity.

PRINCIPLE:

The estimation is based on the reaction between KMnO_4 oxides Oxalic acid in the presence of acid and while hot.



PROCEDURE:

TITRATION-I

STANDAREDISATION OF KMnO_4 SOLUTION

About 1.525g of OXALIC ACID crystals are accurately weighted transferred into a funnel placed over a 250ml, standard flask. The funnel is well washed into a flask. The crystals are dissolved and solutions made upto the mark and shaken well exactly 20ml of the standard OXALIC ACID solution is ppt. The solution is the titrated with KMnO_4 solution taken in a burette. H_2SO_4 is added it is taken and then heated to 60°C to 80°C . The hot solution is titrated against standard KMnO_4 solution taken in the burette the end point is the appearance of pink colour. The titrated is repeated until the concordant value are obtained from this value strength and amount of Oxalic acid is calculated

Sl.No.	Volume of OXALIC ACID (ml)	Burette Reading		Volume of KMnO_4 (ml)	Concordant Value (ml)	Indicator
		Initial (ml)	Final (ml)			

Volume of OXALIC ACID (V_1) = 20ml.

Normality of OXALIC ACID (N_1) = -----N.

Volume of KMnO_4 (V_2) =-----ml.

Normality of KMnO_4 (N_2) = ?

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 N_1}{V_2}$$
$$= \text{-----N.}$$

TITRATION-II

ESTIMATION OF OXALIC ACID

The burette is filled with same KMnO_4 is a solution upto zero mark. The given Oxalic acid solution is made upto 100ml in a standard flask. 20ml to this solution is pipette out in a conical flask 60 this solution is out in a conical flask. H_2SO_4 is added it is taken and then heated to 60°C to 80°C . The hot solution is titrated against standard KMnO_4 solution taken in the burette the end point is the appearance of pink colour. The titrated is repeated until the concordant value are obtained from this value strength and amount of Oxalic acid is calculated.

Equivalent weight of Oxalic acid = 63g/eq.

Equivalent weight of Potassium Permanganate = 31.6 g/eq.

Sl.No.	Volume of Oxalic Acid (ml)	Burette Reading		Volume of KMnO_4 (ml)	Concordant Value (ml)	Indicator
		Initial (ml)	Final (ml)			

Volume of KMnO_4 (V_1) =-----ml.

Normality of KMnO_4 (N_1) = -----N.

Volume of **Oxalic Acid** (V_2) = 20ml.

Normality of **Oxalic Acid** (N_2) = ?

$$V_1 N_1 = V_2 N_2$$

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

$$= \text{-----}N.$$

$$\left\{ \begin{array}{l} \text{The amount of } \mathbf{Oxalic\ Acid} \text{ present} \\ \text{in the} \\ \text{whole of the given solution} \end{array} \right\} = \text{Normality} \times \text{Equivalent weight} \times 1/10.$$

$$= \text{-----}g.$$

RESULT:

The amount of Oxalic acid present in the whole of the given solution = -----.

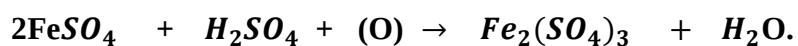
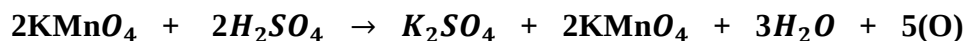
ESTIMATION OF FERROUS SULPHATE

AIM:

To estimate the amount of FeSO_4 present in the whole of the given solution. You are provided with pure crystals of Ferrous ammonium sulphate and a known solution of KMnO_4 .

PRINCIPLE:

The neutralization between FeSO_4 and KMnO_4 in dilute Sulphuric acid takes place according to the equation.



PROCEDURE:

TITRATION-I

STANDARDISATION OF KMnO_4

About 3.9204 g of pure Ferrous Ammonium Sulphate crystals are weighed and made up to 250 ml in a standard measuring flask by dissolving the crystals with distilled water the solution is shaken well for a uniform concentration. The burette is washed with water and Potassium permanganate and filled up to the zero mark. 20 ml of dilute H_2SO_4 is added. The solution is then titrated against Potassium Permanganate taken in the burette. The end point is appearance of Pale Pink colour. The titration is repeated to get constant value from this value KMnO_4 is calculated.

Sl.No.	Volume of FAS (ml)	Burette Reading		Volume of KMnO_4 (ml)	Concordant Value (ml)	Indicator
		Initial (ml)	Final (ml)			

Volume of FAS (V_1) = 20 ml.

Normality of **FAS** (N_1) = -----N.

Volume of **KMnO₄** (V_2) =-----ml.

Normality of **KMnO₄** (N_2) = ?

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 N_1}{V_2}$$
$$= \text{-----N.}$$

TITRATION-II

ESTIMATION OF **FeSO₄**

The burette is filled with **KMnO₄**. The given **FeSO₄** solution is made up to 100ml in SMF. Exactly pipette out into a clean conical flask, and 20ml of dilute **H₂SO₄** is added to it. The solution is against Potassium Permanganate taken in the burette. The solution is against Potassium Permanganate taken in the burette. The end point is appearance of Pale Pink colour. The titration is repeated to get constant value from this value **FeSO₄** is calculated.

Equivalent weight of **FeSO₄** = 278 g/eq.

Equivalent weight of **FAS** = 392 g/eq.

Sl.No.	Volume of FeSO₄ (ml)	Burette Reading		Volume of KMnO₄ (ml)	Concordant Value (ml)	Indicator
		Initial (ml)	Final (ml)			

Volume of **KMnO₄** (V_1) = -----ml.

Normality of **KMnO₄** (N_1) = -----N.

Volume of **FeSO₄**(V₂) = 20ml.

Normality of **FeSO₄**(N₂) = ?

$$V_1 N_1 = V_2 N_2$$

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

$$= \text{-----N.}$$

$\left\{ \begin{array}{l} \text{The amount of FeSO}_4 \text{ present} \\ \text{in the} \\ \text{whole of the given solution} \end{array} \right\} = \text{Normality} \times \text{Equivalent weight} \times 1/10.$

$$= \text{-----g.}$$

RESULT:

The amount of FeSO₄ present in the whole of the given solution = -----.

ESTIMATION OF KMnO_4

AIM:

To estimate the amount of Potassium permanganate present on the whole of the given solution you are provided with 0.1 standard $\text{K}_2\text{Cr}_2\text{O}_7$ and a link solution of $\text{Na}_2\text{S}_2\text{O}_3$

PRINCIPLE:

The neutralization between FeSO_4 and KMnO_4 of dilute Sulphuric acid taken place according to the equation.



PROCEDURE:

TITRATION-I

STANDARDISATION OF $\text{Na}_2\text{S}_2\text{O}_3$

About 1.225g of pure crystals $\text{K}_2\text{Cr}_2\text{O}_7$ are weighed and made up to 250ml in a standard measuring flask by dissolving the crystals with diluted water the solution is shaken well for a uniform concentration. The 20ml of $\text{K}_2\text{Cr}_2\text{O}_7$ solution is pipette out into a clean conical flask, and 20ml of 2N H_2SO_4 is added to it. The solution is against $\text{Na}_2\text{S}_2\text{O}_3$ taken in the burette. 2ml of Starch indicator added to the solution. The solution is against $\text{Na}_2\text{S}_2\text{O}_3$ taken in the burette. The end point is disappearance of dark blue colour. The titration is repeated to get constant value from this value is determined.

Sl.No.	Volume of $\text{K}_2\text{Cr}_2\text{O}_7$ (ml)	Burette Reading		Volume Of $\text{Na}_2\text{S}_2\text{O}_3$ (ml)	Concordant Value (ml)	Indicator
		Initial (ml)	Final (ml)			

Volume of **K₂Cr₂O₇** (V_1) =-----ml.

Normality of **K₂Cr₂O₇** (N_1) = -----N.

Volume of **Na₂S₂O₃** (V_2) = 20ml.

Normality of **Na₂S₂O₃** (N_2) = ?

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 N_1}{V_2}$$
$$= \text{-----N.}$$

TITRATION-II

ESTIMATION OF **KMnO₄**

The given **KMnO₄** solution is made upto 100ml SMF. The burette is washed with water filled with **Na₂S₂O₃**. 20ml of **KMnO₄** solution is pipette out into a clean conical flask and 2N **H₂SO₄** is added and is titrated against **KMnO₄** taken in the burette. The end point is the disappearance of permanganate pale pink colour from the titrated value the strength and the amount of **KMnO₄**

Sl.No.	Volume of KMnO₄ (ml)	Burette Reading		Volume of Na₂S₂O₃ (ml)	Concordant Value (ml)	Indicator
		Initial (ml)	Final (ml)			

present in the whole of the given solution is calculated.

Equivalent weight of **KMnO₄** = 31.6 g/eq.

Volume of **Na₂S₂O₃** (V_1) = -----ml.

Normality of **Na₂S₂O₃** (N_1) = -----N.

Volume of **KMnO₄** (V_2) =-----ml.

Normality of **KMnO₄**(*N*₂) = ?

$$V_1 N_1 = V_2 N_2$$

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

$$= \text{-----}N.$$

$$\left\{ \begin{array}{l} \text{The amount of } \mathbf{KMnO_4} \text{ present} \\ \text{in the} \\ \text{whole of the given solution} \end{array} \right\} = \text{Normality} \times \text{Equivalent weight} \times 1/10.$$

$$= \text{-----}g.$$

RESULT:

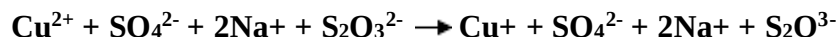
The amount of **KMnO₄** *present in the hole of the given solution* = -----.

ESTIMATION OF COPPER SULPHATE

AIM:

To estimate the amount of CuSO_4 present in the whole of the given solution. You are provided with pure crystals of CuSO_4 and a known solution of $\text{Na}_2\text{S}_2\text{O}_3$. **PRINCIPLE:**

The neutralization between CuSO_4 and $\text{Na}_2\text{S}_2\text{O}_3$ in dilute Sulphuric acid takes place according to the equation.



PROCEDURE:

TITRATION-I

STANDARDISATION OF COPPER SULPHATE

About 2g of pure CuSO_4 crystals are weighed and made up to 250ml in a standard measuring flask by dissolving the crystals with distilled water the solution is shaken well for a uniform concentration. The burette is washed with water and filled with $\text{Na}_2\text{S}_2\text{O}_3$ up to the zero mark. 2N H_2SO_4 is added. The solution is titrated against 5ml of $\text{Na}_2\text{S}_2\text{O}_3$. The solution is added 2ml of starch indicator. The solution is titrated against $\text{Na}_2\text{S}_2\text{O}_3$. The end point is disappearance of dark blue colour. The titration is repeated to get constant value from this value copper sulphate is calculated.

Sl.No.	Volume of CuSO_4 (ml)	Burette Reading		Volume of $\text{Na}_2\text{S}_2\text{O}_3$. (ml)	Concordant Value (ml)	Indicator
		Initial (ml)	Final (ml)			

Volume of CuSO_4 (V_1) = 20ml.

Normality of CuSO_4 (N_1) = -----N.

Volume of $\text{Na}_2\text{S}_2\text{O}_3$. (V_2) =-----ml.

Normality of $\text{Na}_2\text{S}_2\text{O}_3$ (N_2) = ?

$$V_1 N_1 = V_2 N_2$$

$$N_2 = \frac{V_1 N_1}{V_2}$$
$$= \text{-----}N.$$

TITRATION-II

ESTIMATION OF CuSO_4

The burette is filled with $\text{Na}_2\text{S}_2\text{O}_3$. The given solution CuSO_4 is made up to 100ml in SMF. Exactly 20ml of this solution pipette out into a clean conical flask, and 2N H_2SO_4 is added. The solution is titrated with 5ml of $\text{Na}_2\text{S}_2\text{O}_3$. The solution is added 2ml of starch indicator. The solution titrated against $\text{Na}_2\text{S}_2\text{O}_3$. The end point is disappearance of dark blue colour. The titration is repeated to get constant value from this value copper sulphate is calculated.

Equivalent weight of $\text{Na}_2\text{S}_2\text{O}_3$. = 250 g/eq.

Equivalent weight of CuSO_4 = 79.2g/eq .

Sl.No.	Volume of CuSO_4 (ml)	Burette Reading		Volume Of $\text{Na}_2\text{S}_2\text{O}_3$ (ml)	Concordant Value (ml)	Indicator
		Initial (ml)	Final (ml)			

Volume of $\text{Na}_2\text{S}_2\text{O}_3$ (V_1) = -----ml.

Normality of $\text{Na}_2\text{S}_2\text{O}_3$ (N_1) = -----N.

Volume of CuSO_4 (V_2) = 20ml.

Normality of CuSO_4 (N_2) = ?

$$V_1 N_1 = V_2 N_2$$

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

$$= \text{-----}N.$$

$$\left\{ \begin{array}{l} \text{The amount of CuSO}_4 \text{ present} \\ \text{in the} \\ \text{whole of the given solution} \end{array} \right\} = \text{Normality} \times \text{Equivalent weight} \times 1/10.$$

$$= \text{-----}g.$$

RESULT:

The amount of CuSO₄ present in the whole of the given solution = -----.