B.Sc. Programme
(Chemistry Major)

Laboratory Manual

Volumetric Analysis

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Weight Calculation :

Weight of weighing bottle with substance =

Weight of weighing bottle after transferring the substance =

Weight of oxalic acid in 200 ml =

Equivalent weight of oxalic acid =

Normality of oxalic acid $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

$= \frac{\quad \text{x } 5}{6\,3}$

Normality of oxalic acid = N

Titration – I

Standard Oxalic acid Vs Potassium permanganate

S. No.	Volume of Oxalic acid (ml)	Burette reading (ml)		Volume of $KMnO_4$ (ml)	Indicator
		Initial	Final		

ESTIMATION OF FERROUS AMMONIUM SULPHATE

Aim

To estimate the amount of ferrous ammonium sulphate present in the whole of the given solution being supplied with pure crystals of oxalic acid and approximately 0.05N solution of potassium permanganate as the link.

Principle

Potassium permanganate oxidises $FeSO_4$ into $Fe_2(SO_4)_3$ in the presence of dil.H_2SO_4

$$KMnO_4 + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O$$

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

Since, one molecule of ferrous ammonium sulphate loses only one electron. Its equivalent is,

$$\frac{\text{Molecular mass}}{1} = \frac{392}{1} = 392$$

Procedure :

Preparation of standard oxalic acid solution :

Accurately about 0.63g of oxalic acid is weighed in a chemical balance. It is transferred into a 200ml standard flask and dissolved in distilled water. The solution is made upto the mark and shaken well.

Volume of oxalic acid V_1 =

Normality of oxalic acid N_1 =

Volume of potassium permanganate V_2 =

Normality of potassium permanganate N_2 =

$$= \frac{20 \text{ x}}{\qquad}$$

Normality of potassium permanganate N_2 = N

Titration – II

Made up Ferrous Ammonium Sulphate Vs Standardised potassium permanganate

S. No.	Volume of ferrous ammonium sulphate (ml)	Burette reading (ml)		Volume of $KMnO_4$ (ml)	Indicator
		Initial	Final		

Standardization of Potassium permanganate :

The given potassium permanganate solution is taken in a clean and rinsed burette. 20ml of standard oxalic acid is pipetted out into a clean conical flask by means of a washed and rinsed pipette. A test tube full of (20ml) dilute sulphuric acid is added and the solution is heated to bearable warmth. The hot solution is titrated against potassium permanganate taken in the burette. The end point is the appearance of pale permanent pink colour. The titrations are repeated to get concordant values. From the titre value, the strength of potassium permanganate solution is calculated.

Estimation of Ferrous Ammonium Sulphate

The given ferrous ammonium sulphate solution is made upto 100ml in a standard flask with distilled water. The flask is shaken well to get uniform concentration. 20ml of this solution is pipetted out into a conical flask by using a washed and rinsed pipette. A test tube full of dilute sulphuric acid is added and the solution is titrated against potassium permanganate taken in the burette. The end point is the appearance of pale permanent pink colour. The titrations are repeated to get concordant values. From the titre value, the strength of ferrous ammonium sulphate and hence its amount is calculated.

Volume of potassium permanganate V_1 =

Normality of potassium permanganate N_1 =

Volume of ferrous ammonium sulphate V_2 =

Normality of ferrous ammonium sulphate N_2 =

$$= \frac{x}{20}$$

Normality of ferrous ammonium sulphate N_2 = N

Calculation :

Equivalent weight of ferrous ammonium sulphate = 392

Weight / litre of ferrous ammonium sulphate = Normality x Equivalent weight

= 392 x

= g

The amount of ferrous ammonium sulphate in 100ml = $\frac{\quad}{10}$

= g

Result

The amount of ferrous ammonium sulphate present in the whole of the given solution = g

Weight Calculation :

Weight of weighing bottle with substance =

Weight of weighing bottle after transferring the substance =

Weight of Ferrous ammonium sulphate in 100 ml =

Equivalent weight of Ferrous ammonium sulphate = 392

Normality of Ferrous ammonium sulphate $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

$= \frac{\quad \text{x } 10}{392}$

Normality of Ferrous ammonium sulphate = N

Titration – I

Standard Ferrous Ammonium Sulphate Vs Potassium permanganate

S. No.	Volume of Ferrous Ammonium Sulphate (ml)	Burette reading (ml)		Volume of $KMnO_4$ (ml)	Indicator
		Initial	Final		

ESTIMATION OF OXALATE ION

Aim

To estimate the amount of oxalate ion present in the whole of the given solution being supplied with pure ferrous ammonium sulphate crystals and approximately 0.05N solution of potassium permanganate as the link.

Principle

Potassium permanganate oxidises oxalate ion according to the following equation

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

$$C_2O_4^{2-} \longrightarrow 2CO_2 + 2e^-$$

Since an oxalate ion loses two electrons. Its equivalent mass is,

$$\frac{\text{Molecular mass}}{2} = \frac{88}{2} = 44$$

Volume of ferrous ammonium sulphate V_1 =

Normality of ferrous ammonium sulphate N_1 =

Volume of potassium permanganate V_2 =

Normality of potassium permanganate N_2 =

N_2 =

Normality of potassium permanganate N_2 = N

Titration – II

Made up oxalic acid Vs Standardised potassium permanganate

S. No.	Volume of ferrous madeup oxalic acid (ml)	Burette reading (ml)		Volume of $KMnO_4$ (ml)	Indicator
		Initial	Final		

Procedure

Preparation of Standard Ferrous Ammonium Sulphate Solution

Accurately about 1.96g of ferrous ammonium sulphate is weighed and transferred into a 100ml standard flask. It is dissolved in dilute sulphuric acid and the solution is made upto the mark using distilled water. The standard flask is shaken well.

Standardization of potassium permanganate

20ml of standard ferrous ammonium sulphate is pipetted out into a clean conical flask after preliminary washing and rinsing. A test tube full of (20ml) dilute sulphuric acid is added and the solution is titrated against potassium permanganate taken in the burette. The end point is the appearance of pale permanent pink colour. The titrations are repeated to get concordant values. From the titre value, the strength of potassium permanganate solution is calculated.

Volume of $KMnO_4$	V_1	=	
Normality of $KMnO_4$	N_1	=	
Volume of oxalic acid	V_2	=	
Normality of oxalic acid	N_2	=	
	N_2	=	
Normality of oxalic acid	N_2	=	N

Calculation :

Equivalent weight of oxalate ion = 44

Weight / litre of oxalate solution = Normality x Equivalent weight

= x 44

= g

Weight of oxalate ion in 100ml of oxalate solution = $\frac{\quad}{10}$

= g

Estimation of Oxalate Ion

The given Oxalate solution is made upto 100ml in a standard flask with distilled water, 20ml of this solution is pipetted out into a colonical flask, added one test tube full of dil. H_2SO_4 and heated to bearable warmth. The hot solution is titrated against $KMnO_4$ taken in the burette. The end point is the appearance of pale permanent pink colour. The titrations are repeated to get concordant values.

Result

The amount of oxalate ion present in the whole of the given solution = g

Weight Calculation :

Weight of weighing bottle with substance =

Weight of weighing bottle after transferring the substance =

Weight of Oxalic acid in 200 ml =

Equivalent weight of Oxalic acid = 63

Normality of Oxalic acid $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

$= \frac{\text{x } 5}{63}$

Normality of Oxalic acid = N

Titration – I

Standard Oxalic acid Vs Potassium permanganate

S. No.	Volume of Oxalic acid (ml)	Burette reading (ml)		Volume of $KMnO_4$ (ml)	Indicator
		Initial	Final		

ESTIMATION OF CALCIUM – DIRECT METHOD

Aim

To estimate the amount of calcium present in the whole of the given solution, being provided with oxalic acid crystals and approximately 0.05N solution of potassium permanganate as the link.

Principle

Calcium in calcium chloride is precipitated as calcium oxalate in ammoniacal medium using ammonium oxalate. The precipitate is filtered and dissolved in hot dilute sulphuric acid and the liberated oxalic acid is titrated against potassium permanganate solution.

$$CaCl_2 + (NH_4)_2\, C_2O_4 \longrightarrow CaC_2O_4 + 2NH_4Cl$$

$$CaC_2O_4 + H_2SO_4 \longrightarrow CaSO_4 + H_2C_2O_4$$

$$2KMnO_4 + 2H_2SO_4 + H_2C_2O_4 \longrightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 2CO_2$$

$$\text{Equivalent mass of calcium} = \frac{\text{Atomic mass}}{2} = 20.04$$

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 =

Normality of potassium permanganate N_2 = N

Titration – II

Made up calcium chloride solution Vs standardized potassium permanganate

S. No.	Volume of calcium chloride solution (ml)	Burette reading (ml)		Volume of $KMnO_4$ (ml)	Indicator
		Initial	Final		

Procedure

Preparation of standard oxalic acid solution

A standard solution of oxalic acid is prepared by weighing about 0.63g of it accurately, dissolving it in distilled water and making upto 200ml in a standard flask.

Standardization of potassium permanganate solution

The given potassium permanganate solution is taken in a clean and rinsed burette. Exactly 20ml of the standard oxalic acid is pipetted out into a clean conical flask, added equal volume of dilute sulphuric acid and heated to bearable warmth. The hot solution is then titrated against potassium permanganate taken in the burette. The end point is the appearance of pale permanent pink colour. The titrations are repeated to get concordant values.

Estimation of Calcium

The given calcium chloride solution is made upto 100ml in a standard flask with distilled water. 20ml of this solution is pipetted out into a clean 250ml beaker provided with a glass rod and watch glass. A drop by methyl orange is added and the solution is neutralized by the addition of ammonium hydroxide drop by drop. Diluted with water and heated to boiling. About 20ml of ammonium oxalate is taken in a boiling tube and heated to 60-70°C. The hot ammonium oxalate solution is added slowly to the calcium solution with constant stirring. Ammonium hydroxide is then added till the mixture is ammoniacal. The precipitate is digested over a steam bath for half an hour.

Volume of Potassium permanganate	V_1	=
Normality of Potassium permanganate	N_1	=
Volume of calcium solution	V_2	=
Normality of calcium solution	N_2	=

N_2 =

Normality of calcium solution N_2 = N

Calculation :

Equivalent weight of calcium = 20.04

Weight / litre of calcium = Normality x Equivalent weight

= x 20.04

= g

Weight of calcium in 100ml of the given solution =

= g

A Whatmann No.42 filter paper is placed in a clean funnel in such a way that there is no air space. The contents of the beaker are tested for completion of precipitation by adding a drop of ammonium oxalate along the sides of the beaker. If there is no turbidity, the precipitation is completed. Using a glass rod, the clear supernatant liquid is transferred into the filter paper and the filtrate is collected in a conical flask. The precipitate is washed with distilled water containing ammonia and finally the precipitate is transferred completely into the filter paper.

A clean conical flask is placed below the funnel and the filter paper is pierced at the apex by a glass rod. The precipitate on the filter paper is dissolved by adding hot dilute sulphuric acid. If necessary, the filter paper is transferred into the conical flask and some more dilute sulphuric acid is added. The solution is heated to bearable warmth and titrated against potassium permanganate taken in the burette. The end point is the appearance of pale permanent pink colour. A duplicate is performed simultaneously.

Result

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

= a) g b) g

Weight Calculation :

Weight of weighing bottle with substance =

Weight of weighing bottle after transferring the substance =

Weight of Oxalic acid in 200 ml =

Equivalent weight of Oxalic acid = 63

Normality of Oxalic acid $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

$= \frac{\text{x } 5}{63}$

Normality of Oxalic acid = N

Titration – I

Standard Oxalic acid Vs Potassium permanganate

S. No.	Volume of Oxalic acid (ml)	Burette reading (ml)		Volume of $KMnO_4$ (ml)	Indicator
		Initial	Final		

ESTIMATION OF CALCIUM – INDIRECT METHOD

Aim

To estimate the amount of calcium present in the whole of the given solution, being provided with pure crystals of oxalic acid and approximately 0.05N solution of potassium permanganate as the link.

Principle

Calcium in calcium chloride solution is precipitated by adding a measured excess of oxalic acid. It is filtered and the unreacted oxalic acid is estimated by titrating with standardized potassium permanganate solution. From the difference in titre values the equivalent of oxalic acid required for calcium solution and hence its weight is calculated.

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 =

Normality of potassium permanganate N_2 = N

Titration – II

Made up calcium chloride solution Vs standardised potassium permanganate

S. No.	Volume of calcium chloride solution (ml)	Burette reading (ml)		Volume of $KMnO_4$ (ml)	Indicator
		Initial	Final		

Procedure

Preparation of standard oxalic acid solution

A standard solution of oxalic acid is prepared by weighing about 0.63g of it accurately, dissolving it in distilled water and making upto 200ml in a standard flask.

Standardization of potassium permanganate solution

Potassium permanganage is taken in a washed and rinsed burette. Exactly 20ml of oxalic acid is pipetted out into a clean conical flask. Added equal volume of dilute sulphuric acid and heated to bearable warmth. The hot solution is titrated against potassium permanganate taken in the burette. The end point is the appearance of pale permanent pink colour. The titrations are repeated to get concordant values.

20ml of N oxalic acid ≡ ml of potssium permanganate

40ml of N oxalic acid ≡ ml of potssium permanganate

= ml of potssium permanganate

Volume of Potassium permanganate ≡ unused oxalic acid

= ml

Volume of Potassium permanganate ≡ used oxalic acid

=

= ml

ml of N of potassium permanganate = 20 ml of calcium chloride solution

Normality of calcium solution = $\frac{\qquad}{20}$

= N

Calculation :

Equivalent weight of calcium ion = 20.04

Weight / litre of calcium = Normality x Equivalent weight of calcium ion

= x 20.04

= g

Weight of calcium ion present in 100ml of the given solution =

= g

Estimation of Calcium

The given calcium chloride solution is made upto 100ml in a standard flask. Exactly 20ml of it is pipetted out into a clean 250ml beaker provided with a glass rod and watch glass. The solution is diluted and heated to boiling. Into the boiling solution exactly 40ml of oxalic acid is pipetted out to precipitate calcium as calcium oxalate. Ammonium hydroxide is then added and the precipitate is digested over a steam bath for half an hour.

A Whatmann No.42 filter paper is placed in a clean funnel. The clear supernatant liquid is transferred into the filter paper and the filtrate is collected in a conical flask. The precipitate is washed many times with distilled water and the washings are also collected in the same conical flask. To the contents of the conical flask, 20ml of dilute sulphuric acid is added and heated to bearable warmth. The hot solution is titrated against potassium permanganate, taken in the burette. The end point is the appearance of pale permanent pink colour. A duplicate is performed simultaneously.

Result

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

(a) g

(b) g

Weight Calculation :

Weight of weighing bottle with substance =

Weight of weighing bottle after transferring the substance =

Weight of potassium dichromate in 200 ml =

Equivalent weight of potassium dichromate = 49

Normality of potassium chromate $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

$= \frac{\quad \text{x } 5}{49}$

Normality of potassium dichromate = N

Titration – I

Standard Potassium dichromate Vs sodium thiosulphate solution

S. No.	Volume of potassium dichromate (ml)	Burette reading (ml)		Volume of thiosulphate (ml)	Indicator
		Initial	Final		

ESTIMATION OF COPPER

Aim

To estimate the amount of copper present in the whole of the given solution, being supplied with pure crystals of potassium dichromate and approximately 0.05 N sodium thiosulphate solution.

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 thiosulphate.

$$2CuSO_4 + 4KI \longrightarrow 2CuI_2 + 2K_2SO_4$$

$$2CuI_2 \longrightarrow Cu_2I_2 + I_2$$

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

Equivalent mass of copper = Atomic mass of copper = 63.54

Volume of potassium dichromate V_1 =

Normality of potassium dichromate N_1 =

Volume of sodium thiosulphate V_2 =

Normality of sodium thiosulphate N_2 =

=

Normality of sodium thiosulphate N_2 = N

Titration – II

Made up copper sulphate solution Vs standardised sodium thiosulphate

S. No.	Volume of copper sulphate (ml)	Burette reading (ml)		Volume of thiosulphate (ml)	Indicator
		Initial	Final		

Procedure

Preparation of standard potassium dichromate solution

A standard solution of potassium dichromate is prepared by weighing about 0.49g of it accurately, dissolving it in distilled water and making upto 200ml in a standard flask.

Standardisation of Sodium Thiosulphate

The burette is filled with thio solution after washing and rinsing. Exactly 20ml of the standard potassium dichromate solution is pipetted out into a clean conical flask. To this 10ml of dilute hydrochloric acid and 10ml of 10% potassium iodide solution are added and the liberated iodine is titrated against thio sulphate solution. When the solution becomes straw yellow in colour, 1ml of freshly prepared starch solution is added and the titration is continued. The end point is the disappearance of blue colour and appearance of green colour. The titrations are repeated to get concordant values.

Volume of sodium thiosulphate	V_1	=
Normality of sodium thiosulphate	N_1	=
Volume of copper sulphate	V_2	=
Normality of copper sulphate	N_2	=
	N_2	=
Normality of copper sulphate	N_2	= N

Calculation :

Equivalent weight of copper = 63.54

Weight / litre of copper sulphate = Normality x Equivalent weight

= x 63.54

= g

Amount of copper sulphate present in whole of the given solution =

= g

Estimation of Copper

The given copper sulphate solution is made upto 100ml in a standard flask. Exactly 20ml of this solution is pipetted out into a clean conical flask and ammonium hydroxide is added till a pale blue precipitate is formed. The precipitate is dissolved by adding acetic acid and two drops of acetic acid is added in excess. To this solution 10ml of 10% potassium iodide solution is added and the liberated iodine is titrated against thiosulphate. When the dark brown colour becomes straw yellow, 1ml of starch solution is added and the titration is continued. The end point is the disappearance of blue colour. The titrations are repeated to get concordant values.

Result

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

Weight Calculation :

Weight of weighing bottle with substance =

Weight of weighing bottle after transferring the substance =

Weight of oxalic acid in 200 ml =

Equivalent weight of oxalic acid = 63

Normality of oxalic acid $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

$= \frac{\quad \text{x } 5}{63}$

Normality of oxalic acid = N

Titration – I

Standard Oxalic acid Vs potassium permanganate

S. No.	Volume of oxalic acid (ml)	Burette reading (ml)		Volume of $KMnO_4$ (ml)	Indicator
		Initial	Final		

PERCENTAGE PURITY OF PYROLUSITE

Aim

To estimate the amount of pure pyrolusite in 100g of the sample using standard oxalic acid and an approximately 0.05N potassium permanganate solution as the link.

Principle

The pyrolusite (MnO_2) oxidises oxalic acid in the presence of dilute sulphuric acid to carbon dioxide and water

$$MnO_2 + 4H^+ + 2e^- \longrightarrow Mn^{2+} + 2H_2O$$

$$C_2O_4^{2-} \longrightarrow 2CO_2 + 2e^-$$

Hence the equivalent mass of MnO_2

$$= \frac{\text{Molecular mass}}{2} = \frac{86.94}{2} = 43.47$$

Volume of oxalic acid	V_1	=	
Normality of oxalic acid	N_1	=	
Volume of potassium permanganate	V_2	=	
Normality of potassium permanganate	N_2	=	
		=	
Normality of potassium permanganate	N_2	=	N

Titration – II

Excess Oxalic acid Vs standardised potassium permanganate

S. No.	Volume of oxalic acid (ml)	Burette reading (ml)		Volume of $KMnO_4$ (ml)	Indicator
		Initial	Final		

PROCEDURE

Preparation of standard oxalic acid

A standard solution of oxalic acid is prepared by weighing about 0.63g of it accurately, dissolving it in distilled water and making upto 200ml in a standard flask.

Standardisation of potassium permanganate

Potassium permanganate solution is taken in a clean and rinsed burette. Exactly 20ml of standard oxalic acid is pipetted out into a clean conical flask and equal volume of dilute sulphuric acid is added. The contents of the conical flask is heated to bearable warmth and titrated against potassium permanganate. The end point is the appearance of pale permanent pink colour. The titrations are repeated to get concordant values.

20ml of oxalic acid ≡ ml of potassium permanganate

40ml of oxalic acid ≡ 2 x ml of potassium permanganate

= ml of potassium permanganate

Volume of Potassium permanganate ≡ unused oxalic acid

=

= ml

100 ml of N of potassium permanganate = used up oxalic acid [100ml of oxalic acid – 100ml of pyrolusite].

100ml of N potassium permanganate = 43.47 g of N pyrolusite

ml of N potassium permanganate = $\frac{43.47 \text{ x} \quad \text{x}}{1000}$

= g of pure MnO_2

0.05g of pyrolusite contain g of pure pyrolusite

100 g of pyrolusite contain = $\frac{\text{x } 100}{0.05}$ g

= %

Determination of percentage purity of Pyrolusite

About 0.1g of manganese dioxide is weighed accurately and transferred into a clean conical flask. Exactly 40ml of oxalic acid is pipetted out into the conical flask and three test tubes of dilute sulphuric acid ($\simeq$ 60ml) is added. The flask is heated gently till all the black particles get dissolved. Care is taken not to allow the mixture to boil, otherwise oxalic acid will volatilise. When the reaction is over, the unreacted oxalic acid is titrated against potassium permanganate taken in the burette. The end point is the appearance of pale permanent pink colour. A duplicate is performed simultaneously.

Result

The percentage purity of pyrolusite =

Weight Calculation :

Weight of weighing bottle with substance =

Weight of weighing bottle after transferring the substance =

Weight of oxalic acid in 200 ml =

Equivalent weight of oxalic acid = 63

Normality of oxalic acid $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

$= \frac{\quad\quad \text{x } 5}{63}$

Normality of oxalic acid = N

Titration – I

Standard Oxalic acid Vs Potassium Permanganate

S. No.	Volume of Oxalic acid (ml)	Burette reading (ml)		Volume of $KMnO_4$ (ml)	Indicator
		Initial	Final		

ESTIMATION OF HYDROGEN PEROXIDE

Aim

To estimate the volume strength of hydrogen peroxide, being supplied with pure crystals of oxalic acid and an approximately decinormal potassium permanganate solution as the link

Principle

In acid medium potassium permanganate oxidises hydrogen peroxide as follows

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

$$H_2O_2 \longrightarrow 2H^+ + O_2 + 2e^-$$

So, the equivalent mass of H_2O_2,

$$= \frac{\text{Molecular mass}}{2} = \frac{34}{2} = 17$$

Volume of oxalic acid	V_1	=	
Normality of oxalic acid	N_1	=	
Volume of potassium permanganate	V_2	=	
Normality of potassium permanganate	N_2	=	
		=	
Normality of potassium permanganate	N_2	=	N

Titration – II

Made up Hydrogen peroxide Vs standardised potassium permanganate

S. No.	Volume of hydrogen peroxide (ml)	Burette reading (ml)		Volume of $KMnO_4$ (ml)	Indicator
		Initial	Final		

Procedure

Preparation of standard oxalic acid solution

A standard solution of oxalic acid is prepared by weighing about 0.63g of it accurately, dissolving it in distilled water and making upto 200ml in a standard flask.

Standardisation of potassium permanganate

Potassium permanganate in solution is taken in a clean and rinsed burette. Exactly 20ml of standard oxalic acid is pipetted out into a clean conical flask and added equal volume of dilute sulphuric acid. The contents of the conical flask is heated to bearable warmth and titrated against potassium permanganate taken in the burette. The end point is the appearance of pale permanent pink colour. The titrations are repeated to get concordant values.

Volume of potassium permanganate V_1 =

Normality of potassium permanganate N_1 =

Volume of hydrogen peroxide V_2 =

Normality of hydrogen peroxide N_2 =

N_2 =

Normality of hydrogen peroxide N_2 = N

Calculation :

Equivalent weight of hydrogen peroxide = 17

Weight / litre of hydogen peroxide solution = Normality x Equivalent weight

= x 17

= g

Weight / 200 ml of hydrogen peroxide solution = $\frac{\quad}{5}$

= g

$$2H_2O_2 \rightarrow 2H_2O + O_2$$

2 x 34 g of H_2O_2 will liberate 32 g of O_2

g of H_2O_2 will liberate $\frac{32 \text{ x}}{68}$

= g

32 g of oxygen at NTP will occupy 22400 ml

_____g of oxygen at NTP will occupy $\frac{22400 \text{ x}}{32}$

= ml

Estimation of Hydrogen Peroxide

The given hydrogen peroxide is made upto 100ml in a standard flask with distilled water. Exactly 20ml of this solution is pipetted out into a clean conical flask. About 20ml of dilute sulphuric acid is added to it and titrated against standardized potassium permanganate taken in the burette. The end point is the appearance of pale permanent pink colour. The titrations are repeated to get concordant values.

Result

Volume strength of hydrogen peroxide = ml

Weight Calculation :

Weight of weighing bottle with substance =

Weight of weighing bottle after transferring the substance =

Weight of oxalic acid in 200 ml =

Equivalent weight of oxalic acid = 63

Normality of oxalic acid $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

$= \frac{\quad \times 5}{63}$

Normality of oxalic acid = N

Titration – I

Standard Oxalic acid Vs potassium permanganate

S. No.	Volume of Oxalic acid (ml)	Burette reading (ml)		Volume of $KMnO_4$ (ml)	Indicator
		Initial	Final		

ESTIMATION OF NITRITE ION

Aim

To estimate the amount of nitrite ion present in the whole of the given solution using standard oxalic acid and approximately 0.05N potassium permanganate solution as the link.

Principle

Nitrite is oxidized to nitrate by a warm acidified solution of potassium permanganate

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

$$NO_2^- + H_2O \longrightarrow NO_3^- + 2H^+ + 2e^-$$

Hence, the equivalent mass of nitrite,

$$= \frac{\text{Molecular mass}}{2} = \frac{46}{2} = 23$$

Volume of oxalic acid	V_1	=	
Normality of oxalic acid	N_1	=	
Volume of potassium permanganate	V_2	=	
Normality of potassium permanganate	N_2	=	
		=	
Normality of potassium permanganate	N_2	=	N

Titration – II

Standardised potassium permanganate Vs made up nitrite solution

S. No.	Volume of $KMnO_4$ (ml)	Burette reading (ml)		Volume of sodium nitrite solution (ml)	Indicator
		Initial	Final		

Procedure

Preparation of standard oxalic acid solution

About 0.63g of oxalic acid is weighed accurately, transferred into a 200ml standard flask and dissolved in distilled water. The solution is made upto 200ml.

Standardisation of potassium permanganate solution

Exactly 20ml of oxalic acid is pipetted out into a clean conical flask. Equal volume of dilute sulphuric acid is added and heated to bearable warmth. The hot solution is titrated against potassium permanganate taken in the burette. The end point is the appearance of pale permanent pink colour. The titrations are repeated to get concordant values.

Volume of potassium permanganate	V_1	=
Normality of potassium permanganate	N_1	=
Volume of sodium nitrite solution	V_2	=
Normality of sodium nitrite solution	N_2	=
		=
Normality of sodium nitrite solution	N_2	= N

Calculation :

Equivalent weight of nitrite ion = 23

Weight / litre of nitrite solution = Normality x Equivalent weight

= x 23

= g

Weight of nitrite ion present in the whole of the given solution = g

Estimation of Nitrite Ion

The given nitrite solution is made upto 100ml in a standard flask. The made up nitrite solution is taken in a clean and rinsed burette. Exactly 20ml of potassium permanganate solution is pipetted out into a clean beaker. Equal volume of dilute sulphuric acid is added and heated to 40°C after diluting the solution to 100ml. The tip of the burette is allowed to remain under the surface of potassium permanganate and the nitrite solution is added drop by drop. The solution is stirred using a glass rod after addition of each drop of nitrite solution. The end point is the disappearance of pink colour. The titrations are repeated to get concordant values.

Result

The amount of nitrite present in the whole of the given solution = g

Weight Calculation :

Weight of weighing bottle with substance =

Weight of weighing bottle after transferring the substance =

Weight of potassium chloride in 100 ml =

Equivalent weight of potassium chloride = 74.46

Normality of potassium chloride $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

$= \frac{\quad \text{x } 10}{74.46}$

Normality of potassium chloride = N

Titration – I

Standard Potassium chloride Vs silver nitrate

S. No.	Volume of potassium chloride (ml)	Burette reading (ml)		Volume of silver nitrate (ml)	Indicator
		Initial	Final		

ESTIMATION OF CHLORIDE ION – MOHR'S METHOD

Aim

To estimate the amount of chloride present in the whole of the given solution, being supplied with an approximately 0.05N silver nitrate solution and pure crystals of potassium chloride

Principle

It is an example of precipitation reaction

$$AgNO_3 + Cl^- \longrightarrow AgCl\downarrow + NO_3^-$$

The completion of the reaction is observed by using potassium chromate as the indicator. The yellow colour of potassium chromate changes into reddish brown due to the formation of silver chromate.

$$2AgNO_3 + K_2CrO_4 \longrightarrow Ag_2CrO_4\downarrow + 2KNO_3$$

Volume of potassium chloride	V_1	=	
Normality of potassium chloride	N_1	=	
Volume of silver nitrate solution	V_2	=	
Normality of silver nitrate solution	N_2	=	
		=	
Normality of silver nitrate solution	N_2	=	N

Titration – II

Made up chloride solution Vs standardised silver nitrate

S. No.	Volume of chloride solution (ml)	Burette reading (ml)		Volume of silver nitrate (ml)	Indicator
		Initial	Final		

Procedure

Preparation of standard potassium chloride solution

About 0.375 g of potassium chloride is accurately weighed and it is transferred into a 100ml standard flask. The crystals are dissolved in distilled water and made upto the mark.

Standardisation of Silver Nitrate Solution

Exactly 20ml of potassium chloride solution is pipetted out into a clean conical flask and 2ml of 5% potassium chromate solution is added as indicator. This solution is titrated against silver nitrate solution taken in the burette. During each addition of silver nitrate solution, the conical flask is shaken well. Nearing the end point, the silver chloride gets coagulated at the bottom of the conical flask. The addition of silver nitrate is done drop by drop until the supernatant solution gets reddish brown tinge. The titrations are repeated to get concordant values.

Volume of silver nitrate solution	V_1	=
Normality of silver nitrate solution	N_1	=
Volume of chloride solution	V_2	=
Normality of chloride solution	N_2	=

N_2 =

Normality of chloride solution N_2 = N

Calculation :

Equivalent weight of chloride = 35.46

Weight / litre of chloride = Normality x Equivalent weight

= x 35.46

= g

Weight of chloride present in the whole of the given solution = $\frac{\quad}{10}$

= g

Estimation of Chloride

The given chloride solution is made upto 100ml in a standard flask with distilled water. Exactly 20ml of this solution is pipetted out into a clean conical flask and 2ml of 5% potassium chromate is added. This solution is titrated against standardized silver nitrate solution taken in the burette. The end point is the appearance of reddish tinge. The titrations are repeated to get concordant values.

Result

The amount of chloride present in the whole of the given solution = g

Weight Calculation :

Weight of weighing bottle with substance =

Weight of weighing bottle after transferring the substance =

Weight of potassium chloride in 100 ml =

Equivalent weight of potassium chloride = 74.46

Normality of potassium chloride $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

$= \frac{\quad \text{x } 10}{74.46}$

Normality of potassium chloride = N

Titration – I

Standard Potassium chloride Vs silver nitrate

S. No.	Volume of potassium chloride (ml)	Burette reading (ml)		Volume of silver nitrate (ml)	Indicator
		Initial	Final		

Volume of potassium chloride V_1 =

Normality of potassium chloride N_1 =

Volume of silver nitrate V_2 =

Normality of silver nitrate N_2 =

=

Normality of silver nitrate N_2 = N

ESTIMATION OF COMMERCIAL HYDROCHLORIC ACID

- VOLHARD'S METHOD

Aim

To determine the normality of commercial hydrochloric acid by Volhard's method. Being supplied with 0.05N solution of silver nitrate ammonium, thiocyanate and pure crystals of potassium chloride.

Principle

Volhard's method is applicable to the estimation of chloride in neutral (or) acid medium. In this method the known volume of chloride is treated with known excess of silver nitrate solution. The unreacted silver nitrate is treated with standard ammonium thiocyanate solution. From the titre value, the volume of thiocyanate equivalent to the silver nitrate that has reacted with chloride solution is calculated from which the strength of chloride solution and hence the weight of chloride in the whole of the given solution is calculated.

$$AgNO_3 + HCl \longrightarrow AgCl\downarrow + HNO_3$$

$$AgNO_3 + NH_4CNS \longrightarrow AgCNS + NH_4NO_3$$

The indicator used is ferric alum solution. At the end point a reddish brown colour developes in the solution.

Titration – II

Standardised silver nitrate Vs Ammonium thiocyanate

S. No.	Volume of silver nitrate (ml)	Burette reading (ml)		Volume of Ammonium thiocyanate (ml)	Indicator
		Initial	Final		

Volume of silver nitrate V_1 =

Normality of silver nitrate N_1 =

Volume of Ammonium thiocyanate V_2 =

Normality of Ammonium thiocyanate N_2 =

=

Normality of Ammonium thiocyanate N_2 = N

20 ml N silver nitrate ≡ ml of Ammonium thiocyanate

40 ml N silver nitrate ≡ x 2 of Ammonium thiocyanate

= ml

Titration – III

Made up hydrochloric acid Vs standardised ammonium thiocyanate

S. No.	Volume of hydrochloric acid (ml)	Burette reading (ml)		Volume of ammonium thiocyanate (ml)	Indicator
		Initial	Final		

Procedure

Standardisation of Silver Nitrate Solution

Exactly 20ml of potassium chloride solution is pipetted out into a clean conical flask and 2ml of 5% potassium chromate solution is added as indicator. This solution is titrated against silver nitrate solution taken in the burette. During each addition of silver nitrate solution, the conical flask is shaken well. Nearing the end point, the silver chloride gets coagulated at the bottom of the conical flask. The addition of silver nitrate is done drop by drop until the supernatant solution gets reddish brown tinge. The titrations are repeated to get concordant values.

Standardization of Ammonium Thiocyanate

Exactly 20ml of the standardised silver nitrate solution is pipetted out into a clean conical flask and 5ml of dilute nitric acid and one ml ferric alum indicator are added. The solution is then titrated against ammonium thiocyanate solution taken in the burette. The end point is the appearance of reddish tinge. The titrations are repeated to get concordant values.

Estimation of Hydrochloric Acid

One ml of commercial hydrochloric acid is made upto 200ml in a standard flask with distilled water. 20ml of this solution is pipetted out into a clean beaker provided with a glass rod and watch glass. About 5ml of dilute nitric acid is added and 40ml of silver nitrate solution is pipetted out into the same beaker. The mixture is stirred well and the precipitated silver chloride is filtered through Whatmann No.41 filter paper. The precipitate is washed well with cold water. The filtrate and washing are collected in a conical flask. About one ml of ferric alum is added to the filtrate, and it is titrated against standardised ammonium thiocyanate solution taken in the burette. The end point is the appearance of reddish tinge. A duplicate is performed side by side.

Volume of Ammonium thiocyanate ≡ unused silver nitrate

= ml

Volume of Ammonium thiocyanate ≡ used up silver nitrate

=

= ml

used up silver nitrate = $\frac{\text{ml of Ammonium thiocyanate}}{\text{20ml of made up hydrochloric acid}}$

Normality of chloride solution = $\frac{\qquad\qquad}{20}$

= N

Equivalent weight of chloride = 35.46

Weight / litre of chloride solution = Normality x Equivalent weight

= x 35.46

= g

Weight of chloride present in 200ml

of chloride solution = $\frac{\qquad\qquad}{5}$

= g

_____ g of chloride present in 1ml of commercial hydrochloric acid

1 ml of commercial hydrochloric acid contains _____ g of chloride ion.

Weight of chloride ion present in 1000ml of commercial hydrochloric acid = x 1000

= g

Normality of commercial hydrochloric acid = $\frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

= $\frac{\qquad\qquad}{35.46}$

= N

Result

The normality of the commercial hydrochloric acid is = _____ N

Weight Calculation :

Weight of weighing bottle with substance =

Weight of weighing bottle after transferring the substance =

Weight of zinc sulphate in 100ml =

Equivalent weight of zinc sulphate = 143.77

Normality of zinc sulphate $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

$= \frac{\text{x } 10}{143.77}$

Normality of zinc sulphate = N

Titration – I

Standard zinc sulphate Vs potassium ferrocyanide solution

S. No.	Volume of zinc sulphate solution (ml)	Burette reading (ml)		Volume of potassium ferrocyanide solution (ml)	Indicator
		Initial	Final		

ESTIMATION OF ZINC

Aim

To estimate the amount of zinc present in the whole of the given solution, being supplied with approximately 0.05N potassium ferrocyanide solution and pure zinc sulphate crystals.

Principle

Zinc ions and ferrocyanide 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 potassium zinc ferrocyanide. In order to make the reaction as oxidation – reduction a small quantity of potassium ferricyanide is added to the ferrocyanide solution. Equivalent mass of zinc = atomic mass of zinc = 65.38

Volume of zinc sulphate	V_1	=	
Normality of zinc sulphate	N_1	=	
Volume of potassium ferrocyanide	V_2	=	
Normality of potassium ferrocyanide	N_2	=	
		=	
Normality of potassium ferrocyanide	N_2	=	N

Titration – II

Made up zinc sulphate solution Vs standardised potassium ferroyanide

S. No.	Volume of zinc sulphate solution (ml)	Burette reading (ml)		Volume of potassium ferrocyanide solution (ml)	Indicator
		Initial	Final		

Procedure

Preparation of standard zinc sulphate solution

A decinormal solution of zinc sulphate is prepared by weighing about 0.725g of zinc sulphate accurately, dissolving it in distilled water and making upto 100ml in a standard flask.

Standardization of Ferrocyanide Solution

Exactly 20ml of zinc sulphate solution is pipetted out into a clean conical flask. To that solution about 20ml of dilute sulphuric acid, one gram of ammonium sulphate and 2 to 3 drops of diphenylamine indicator are added. The solution is titrated against potassium ferrocyanide solution taken in the burette. The end point is the colour change from blue to green. The titrations are repeated to get concordant values.

Volume of potassium ferrocyanide V_1 =

Normality of potassium ferrocyanide N_1 =

Volume of made up zinc sulphate V_2 =

Normality of made up zinc sulphate N_2 =

=

Normality of made up zinc sulphate N_2 = N

Calculation :

Equivalent weight of zinc = 65.38

Weight / litre of zinc = Normality x Equivalent weight

= x 65.38

= g

Weight of zinc present in the whole of the given solution =

= g

Estimation of Zinc

The given zinc salt solution is made upto 100ml in a standard flask. Exactly 20ml of this solution is pipetted out into a clean conical flask. To that solution about 20ml of dilute sulphuric acid, one gram of ammonium sulphate and 2 to 3 drops of diphenylamine indicator are added and titrated against potassium ferrocyanide solution taken in the burette. The end point is the colour change from blue to green. The titrations are repeated to get concordant values.

Result

The amount of zinc present in the whole of the given solution = g

Titration – I

Hard water Vs standardised EDTA solution

S. No.	Volume of hard water (ml)	Burette reading (ml)		Volume of EDTA solution (ml)	Indicator
		Initial	Final		

Strength of EDTA =

1000ml of 1M EDTA ≡ 40.08 g of Ca^{2+} ions

ml of 0.01M EDTA = $\frac{40.08 \times 0.01 \times}{1000 \times 1}$

ml of 0.01M EDTA = $\times 10^{-3}$ g of Ca^{2+}

60ml of water contains g of Ca^{2+} ions.

10^6ml of water contains $\frac{\times 10^{-3} \times 10^6}{60}$ g of Ca^{2+} ions.

= g of Ca^{2+} ions.

40.08g of Ca^{2+} ions present in 100.08g of $CaCO_3$

$\therefore$ g of Ca^{2+} ions present in $\frac{\times 100.08}{40.08}$ g of $CaCO_3$

= g of $CaCO_3$

= PPM

DETERMINATION OF TOTAL HARDNESS OF WATER

Aim

To determine the total hardness of water, being supplied with standard EDTA solution.

Principle

The hard water contains calcium and magnesium ions. As these ions can readily form complexs with EDTA, they are estimated as a whole by titrating against EDTA solution using Eriochrome Black – T as indicator. The total hardness of water is expressed in terms of parts per millions (PPM) of calcium carbonate.

Procedure

Exactly 60ml of the given sample of water is pipetted out into a clean conical flask. To this 5 ml of buffer solution of pH = 10 and a pinch of Eriochrome Black – T are added and titrated against standard EDTA solution taken in the burette. The end point is the colour change from wine red to blue. The titration is repeated to get concordant value.

Result

The total hardness of water = ppm

Weight Calculation :

Weight of weighing bottle with substance =

Weight of weighing bottle after transferring the substance =

Weight of potassium dichromate in 200 ml =

Equivalent weight of potassium dichromate = 49

Normality of potassium dichromate $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

$= \frac{\quad \times 5}{49}$

Normality of potassium dichromate = N

Titration – I

Ferrous Ferric mixture Vs Standard Potassium dichromate

S. No.	Volume of ferrous ferric mixture (ml)	Burette reading (ml)		Volume of standard $K_2Cr_2O_7$ (ml)	Indicator
		Initial	Final		

ESTIMATION OF FERROUS AND FERRIC IRON IN A MIXTURE

Aim

To estimate the amount of ferrous and ferric iron in a mixture, being supplied with pure crystals of potassium dichromate.

Principle

The amount of ferrous iron in a mixture of ferrous and ferric iron is determined by direct titration with standard potassium dichromate solution. The ferric iron is reduced to ferrous iron by means of stannous chloride and the total ferrous iron is estimated using potassium dichromate.

$$K_2Cr_2O_7 + 5H_2SO_4 + 2FeSO_4 \longrightarrow Cr_2(SO_4) + K_2SO_4 + Fe_2(SO_4)_3 + 5H_2$$

$$2Fe_2(SO_4)_3 + SnCl_2 + 2HCl \longrightarrow FeSO_4 + SnCl_4 + H_2SO_4$$

Volume of potassium dichromate V_1 =

Normality of potassium dichromate N_1 =

Volume of ferrous ferric mixture V_2 =

Normality of ferrous ferric mixture N_2 =

=

Normality of ferrous ferric mixture N_2 = N

Equivalent weight of ferrous iron = 55.85

Weight / Litre of ferrous iron = x 55.85

= g

Titration – II

Ferrous – Ferric mixture after reduction Vs standardised potassium dichromate

S. No.	Volume of ferrous ferric mixture (ml)	Burette reading (ml)		Volume of standard $K_2Cr_2O_7$ (ml)	Indicator
		Initial	Final		

Procedure

Preparation of Standard Potassium Dichromate Solution

Accurately about 0.5g of potassium dichromate, is weighed in a chemical balance, transferred to a 200ml standard flask. It is dissolved in distilled water and made upto the mark.

Estimation of Ferrous Iron

Exactly 20ml of the given ferrous-ferric mixture is pipetted out into a clean conical flask. Added 20ml of a mixture of sulphuric acid and phosphoric acid and three drops of diphenyl amine indicator and titrated versus standard potassium dichromate solution taken in the burette. The end point is the appearance of bluish violet colour. The titrations are repeated to get concordant values.

Volume of potassium dichromate	V_1	=
Normality of potassium dichromate	N_1	=
Volume of ferrous-ferric mixture	V_2	=
Normality of ferrous-ferric mixture	N_2	=
		=

Normality of ferrous-ferric mixture N_2 = N

Calculation :

Equivalent weight of ferrous iron = 55.85

Weight / litre of ferrous iron = Normality x Equivalent weight

= x 55.85

= g

Amount of ferric iron present in the whole of the given solution = (Amount of total ferrous iron)– (Amount of ferrous ion)

=

Amount of ferric iron present in whole of the given solution = g

Reduction of Ferric Iron to ferrous Iron

Exactly 20ml of the given ferrous – ferric mixture is pipetted out into a clean conical flask and 5ml of concentrated hydrochloric acid is added to it. The solution is heated to boiling, and to the hot solution freshly prepared stannous chloride is added till the yellow colour of the solution disappears. Two drops of stannous chloride is added in excess. The solution is diluted to about 150ml, cooled and then 10ml of a saturated solution of mercuric chloride is added to it. A silky white precipitate is formed (If a black precipitate is formed or no precipitate, the reduction is incomplete and the solution is discarded).

The reduced solution containing ferrous iron is mixed with 20ml of mixture of sulphuric acid and phosphoric acid and three drops of diphenyl amine indicator, and titrated against standard potassium dichromate as described above.

Result

1. The amount of ferrous iron present in the whole of the given solution = g

2. The amount of ferric iron present in the whole of the given solution = g

Weight Calculation :

Weight of arsenious oxide in 100 ml =

Equivalent weight of arsenious oxide = 49.45

Normality of arsenious oxide $= \frac{\text{Weight / Litre}}{\text{Equivalent weight}}$

$= \frac{\text{x } 10}{49.45}$

Normality of arsenious oxide = N

Titration – I

Standard arsenious oxide Vs Iodine solution

S. No.	Volume of arsenious oxide (ml)	Burette reading (ml)		Volume of iodine solution (ml)	Indicator
		Initial	Final		

ESTIMATION OF ARSENIOUS OXIDE

Aim

To estimate the amount of arsenious oxide present in the whole of the given solution, being supplied with pure arsenious oxide and approximately 0.05N iodine solution.

Principle

Iodine oxidises arsenious oxide. Since the reaction is reversible, the hydrogen iodide formed must be removed 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 dissolved in sodium hydroxide to form sodium arsenite.

$$As_2O_3 + 2H_2O \longrightarrow As_2O_5 + 4H^+ + 4e^-$$

$$2I_2 + 4e^- \longrightarrow 4I^-$$

$$\text{Equivalent mass of } As_2O_3 = \frac{\text{Molecular mass}}{4} = \frac{197.82}{4} = 49.45$$

Volume of arsenious oxide solution	V_1	=	
Normality of arsenious oxide solution	N_1	=	
Volume of iodine solution	V_2	=	
Normality of iodine solution	N_2	=	
		=	
Normality of iodine solution	N_2	=	N

Titration – II

Made up Arsenious oxide solution Vs standardised Iodine solution

S. No.	Volume of arsenious oxide (ml)	Burette reading (ml)		Volume of iodine solution (ml)	Indicator
		Initial	Final		

Procedure

Preparation of Standard Arsenious Oxide Solution

Accurately about 0.25g of arsenious oxide is weighed in a beaker and a few drops of distilled water is added to make it into a paste. Then sodium hydroxide pellets are added one by one and stirred well to dissolve arsenious oxide. The clear solution is transferred into a 100ml standard flask and the beaker is washed repeatedly with distilled water and the washings are transferred into the flask. The solution is made upto the mark with the distilled water.

Standardization of Iodine Solution

The given iodine solution is taken in a clean and rinsed burette. The standard arsenious oxide solution is filled in another burette and exactly 20ml of it is buretted out into a clean conical flask. A drop of phenolphthalein is added and then the solution is neutralized with dilute hydrochloric acid and 3g of sodium bicarbonate is added, shaken well to dissolve the solid and then added 1ml of starch. The solution is titrated against iodine solution and the end point is the appearance of blue colour. The titrations are repeated to get concordant values.

Volume of iodine solution V_1 =

Normality of iodine solution N_1 =

Volume of made up arsenious oxide V_2 =

Normality of made up arsenious oxide N_2 =

N_2 =

Normality of made up arsenious oxide N_2 = N

Calculation :

Equivalent weight arsenious oxide = 49.45

Weight / litre of arsenious oxide = Normality x Equivalent weight

= x 49.45

= g

Amount of arsenious oxide in 100 ml = $\frac{\quad}{10}$

= g

Estimation of Arsenious Oxide

The given arseniuos oxide solution is made upto 100ml in a standard flask with distilled water. Exactly 20ml of this solution is buretted out into a clean conical flask. A drop of phenolphthalein is added and the solution neutralised with dilute hydrochloric acid. About 3g of sodium bicarbonate and one ml starch are added and the solution is titrated against iodine. The end point is the appearance of blue colour. The titrations are repeated to get concordant values.

Result

The weight of arsenious oxide present in the whole of the given solution = g