## Preparation and Standardisation of 0.1M Sodium thiosulphate :

## Sodium thiosulphate - M.Wt.- 248

Preparation of 0.1M Sodium thiosulphate Solution : Dissolve an accurately weighed amount of 25 gm of Sodium thiosulphate and 0.2 gm of sodium carbonate in sufficient amount of water to produce 1000 ml .
Principle : It is standardised by redox titration involving Iodometric methods using potassium bromate as oxidising agent. An accurately weighed amount of the primary standard potassium bromate is dissolved in water and the solution is made upto 250 ml with water. To this 50 ml is taken accurately and 20 gm of $\mathrm{KI} \& 30 \mathrm{ml}$ of 2 M HCl are added and the liberated Iodine is titrated with sodium thiosulphate solution using starch solution as indicator, until the blue colour disappears.
$\mathrm{KBrO}_{3}+6 \mathrm{KI}+6 \mathrm{HCl} \longrightarrow \mathrm{KBr}+\mathrm{KCl}+3 \mathrm{I}_{2} \uparrow+3 \mathrm{H}_{2} \mathrm{O}$

## Procedure:

Procedure for Standardisation of 0.1M Sodium thiosulphate Solution : Dissolve 0.2 gm of $\mathrm{KBrO}_{3}$ weighed accurately, in sufficient water to produce 250 ml . To 50 ml of this solution, add 2 gm of KI and 3 ml of 2 M HCl and titrate with Sodium thiosulphate solution using starch solution as indicator, until the blue colour disappears.
Equivalent Factor : Each ml of 0.1 M Sodium thiosulphate $\equiv 0.02784 \mathrm{gm}$ of $\mathrm{KBrO}_{3}$

## Calculation :

Molarity of Sodium thiosulphate $=\quad$ Weight taken*(pot. bromate) $\mathbf{x}$ Req. molarity

## Titration Volume x Equivalent factor

## Note : Weight taken*

Calculate the amount of potassium bromate that will be present in the volume pipetted out. (i.e., in 50 ml ) from the actual amount of weight of sample taken.

## Preparation and Standardisation of 0.1 N Sodium thiosulphate :

Sodium thiosulphate - M.Wt.- 248
Preparation of $\mathbf{0 . 1 N}$ Sodium thiosulphate Solution : Dissolve an accurately weighed amount of 25 gm of Sodium thiosulphate and 0.2 gm of sodium carbonate in sufficient amount of water to produce 1000 ml .
Principle : It is standardised by redox titration involving Iodometric methods using potassium bromate as oxidising agent. An accurately weighed amount of the primary standard potassium bromate is dissolved in water and the solution is made upto 250 ml with water. To this 50 ml is taken accurately and 2 gm of $\mathrm{KI} \& 3 \mathrm{ml}$ of 2 M HCl are added and the liberated Iodine is titrated with sodium thiosulphate solution using starch solution as indicator, until the blue colour disappears.


## Procedure :

Procedure for Standardisation of $\mathbf{0 . 1 N}$ Sodium thiosulphate Solution : Dissolve 0.2 gm of $\mathrm{KBrO}_{3}$ weighed accurately, in sufficient water to produce 250 ml . To 50 ml of this solution, add 2 gm of KI and 3 ml of 2 M HCl and titrate with Sodium thiosulphate solution using starch solution as indicator, until the blue colour disappears.
Equivalent Factor : Each ml of 0.1N Sodium thiosulphate $\equiv 0.02784 \mathrm{gm}$ of $\mathrm{KBrO}_{3}$

## Calculation :

Normality of Sodium thiosulphate $=$ Weight taken*(pot. bromate) x Req. normality

## Titration Volume x Equivalent factor

## Note : Weight taken*

Calculate the amount of potassium bromate that will be present in the volume pipetted out. (i.e., in 50 ml ) from the actual amount of weight of sample taken.

## Preparation and Standardisation of 1M Sulphuric Acid :

Sulphuric Acid - M.Wt - 98.07
Preparation of 1M Sulphuric Acid Solution : Dissolve an accurately measured volume of 54 ml of Sulphuric Acid in sufficient amount of water to produce 1000 ml .
Principle : It is an example of alkalimetry. When a strong acid is titrated with a strong base, the salt produced in the reaction is not hydrolysed and therefore the pH of the resultant solution at the endpoint is not $7.0 . \mathrm{H}_{2} \mathrm{SO}_{4}$, a strong acid, is standardised by titrating with a strong base,i.e., $\mathrm{Na}_{2} \mathrm{CO}_{3}$ (primary standard). The following reaction takes place when $\mathrm{Na}_{2} \mathrm{CO}_{3}$ is titrated with $\mathrm{H}_{2} \mathrm{SO}_{4}$.
$\mathrm{Na}_{2} \mathrm{CO}_{3}+\mathrm{H}_{2} \mathrm{SO}_{4} \longrightarrow \mathrm{Na}_{2} \mathrm{SO}_{4}+\mathrm{H}_{2} \mathrm{O}+\mathrm{CO}_{2}$

In this titration, for the detection of the end-point, methyl orange is used as an indicator.
Procedure :
Preparation of $\mathbf{1 M ~ N a} \mathbf{N O}_{2} \mathbf{C O}_{3}$ Solution : Dissolve an accurately weighed amount of 286.15 gm of $\mathrm{Na}_{2} \mathrm{CO}_{3}$ in sufficient amount of water to produce 1000 ml .
Procedure for Standardisation of $\mathbf{1} \mathbf{M ~ H}_{\mathbf{2}} \mathbf{S O}_{\mathbf{4}}$ : Pipette out exactly 10 ml of $1 \mathrm{M} \mathrm{Na} \mathbf{N O}_{2} \mathrm{CO} 3$ solution into a clean conical flask and add 2 or 3 drops of methyl orange indicator. Titrate the contents of the flask with $\mathrm{H}_{2} \mathrm{SO}_{4}$ until red colour is obtained. Repeat the titration for concordant values. Record the values in the tabular form...

Titration of $1 \mathrm{M} \mathrm{Na} \mathbf{N}_{2} \mathrm{CO}_{3}$ solution with $\mathrm{H}_{2} \mathrm{SO}_{4}$ solution

| S.No. | Volume of $1 \mathrm{M} \mathrm{Na}_{2} \mathrm{CO}_{3}$ <br> solution (ml) | Burette Reading |  | Vol. of $\mathrm{H}_{2} \mathrm{SO}_{4}$ rundown (ml) |
| :---: | :---: | :---: | :---: | :---: |
|  |  | Initial | Final |  |
| 1 | 10 | 0 |  |  |
| 2 | 10 |  |  |  |
| 3 | 10 |  |  |  |

The Molarity of HCl is calculated using the formula :
$\mathbf{M}_{1} \mathbf{V}_{\mathbf{1}}=\mathbf{M}_{2} \mathbf{V}_{\mathbf{2}}$
where, $\mathrm{V}_{1}=$ Volume of $1 \mathrm{M} \mathrm{Na} 2 \mathrm{CO}_{3}$ solution $=10 \mathrm{ml}$
$\mathrm{M}_{1}=$ Molarity of $\mathrm{Na}_{2} \mathrm{CO}_{3}$ solution $=1 \mathrm{M}$
$\mathrm{V}_{2}=$ Volume of $\mathrm{H}_{2} \mathrm{SO}_{4}$ solution rundown (Average Burette Reading)
$\mathrm{M}_{2}=$ Molarity of $\mathrm{H}_{2} \mathrm{SO}_{4}=$ ?

Therefore,


## Preparation and Standardisation of 0.1M Sulphuric Acid :

## Sulphuric Acid - M.Wt - 98.07

Preparation of 0.1M Sulphuric Acid Solution : Dissolve an accurately measured volume of 5.4 ml of Sulphuric Acid in sufficient amount of water to produce 1000 ml .
Principle : It is an example of alkalimetry. When a strong acid is titrated with a strong base, the salt produced in the reaction is not hydrolysed and therefore the pH of the resultant solution at the endpoint is not $7.0 . \mathrm{H}_{2} \mathrm{SO}_{4}$, a strong acid, is standardised by titrating with a strong base,i.e., $\mathrm{Na}_{2} \mathrm{CO}_{3}$ (primary standard). The folllowing reaction takes place when $\mathrm{Na}_{2} \mathrm{CO}_{3}$ is titrated with $\mathrm{H}_{2} \mathrm{SO}_{4}$.
$\mathrm{Na}_{2} \mathrm{CO}_{3}+\mathrm{H}_{2} \mathrm{SO}_{4} \longrightarrow \mathrm{Na}_{2} \mathrm{SO}_{4}+\mathrm{H}_{2} \mathrm{O}+\mathrm{CO}_{2}$

In this titration, for the detection of the end-point, methyl orange is used as an indicator.
Procedure :
Preparation of $\mathbf{0 . 1 M ~ N a} \mathbf{N}_{2} \mathrm{CO}_{3}$ Solution : Dissolve an accurately weighed amount of 28.62 gm of $\mathrm{Na}_{2} \mathrm{CO}_{3}$ in sufficient amount of water to produce 1000 ml .
Procedure for Standardisation of $\mathbf{0 . 1} \mathbf{M ~ H}_{\mathbf{2}} \mathbf{S O}_{\mathbf{4}}$ : Pipette out exactly 10 ml of $0.1 \mathrm{M} \mathrm{Na} \mathrm{Na}_{2} \mathrm{CO} 3$ solution into a clean conical flask and add 2 or 3 drops of methyl orange indicator. Titrate the contents of the flask with $\mathrm{H}_{2} \mathrm{SO}_{4}$ until red colour is obtained. Repeat the titration for concordant values. Record the values in the tabular form...

Titration of $0.1 \mathrm{M} \mathrm{Na} \mathbf{N a}_{2} \mathrm{CO}_{3}$ solution with $\mathrm{H}_{2} \mathrm{SO}_{4}$ solution

| S.No. | Volume of 0.1M <br> $\mathrm{Na}_{2} \mathrm{CO}_{3}$ solution (ml) | Burette Reading |  | Vol. of $\mathrm{H}_{2} \mathrm{SO}_{4}$ rundown (ml) |
| :---: | :---: | :---: | :---: | :---: |
|  |  | Initial | Final |  |
| 1 | 10 | 0 |  |  |
| 2 | 10 |  |  |  |
| 3 | 10 |  |  |  |

The Molarity of HCl is calculated using the formula :
$\mathbf{M}_{1} \mathbf{V}_{\mathbf{1}}=\mathbf{M}_{\mathbf{2}} \mathbf{V}_{\mathbf{2}}$
where, $\mathrm{V}_{1}=$ Volume of $0.1 \mathrm{M} \mathrm{Na}_{2} \mathrm{CO}_{3}$ solution $=10 \mathrm{ml}$
$\mathrm{M}_{1}=$ Molarity of $\mathrm{Na}_{2} \mathrm{CO}_{3}$ solution $=0.1 \mathrm{M}$
$\mathrm{V}_{2}=$ Volume of $\mathrm{H}_{2} \mathrm{SO}_{4}$ solution rundown (Average Burette Reading)
$\mathrm{M}_{2}=$ Molarity of $\mathrm{H}_{2} \mathrm{SO}_{4}=$ ?

Therefore,


## Preparation and Standardisation of 1N Sulphuric Acid :

Sulphuric Acid - M.Wt - 98.07
Preparation of 1N Sulphuric Acid Solution : Dissolve an accurately measured volume of 26.6 ml of Sulphuric Acid in sufficient amount of water to produce 1000 ml .
Principle : It is an example of alkalimetry. When a strong acid is titrated with a strong base, the salt produced in the reaction is not hydrolysed and therefore the pH of the resultant solution at the endpoint is not $7.0 . \mathrm{H}_{2} \mathrm{SO}_{4}$, a strong acid, is standardised by titrating with a strong base,i.e., $\mathrm{Na}_{2} \mathrm{CO}_{3}$ (primary standard). The folllowing reaction takes place when $\mathrm{Na}_{2} \mathrm{CO}_{3}$ is titrated with $\mathrm{H}_{2} \mathrm{SO}_{4}$.
$\mathrm{Na}_{2} \mathrm{CO}_{3}+\mathrm{H}_{2} \mathrm{SO}_{4} \longrightarrow \mathrm{Na}_{2} \mathrm{SO}_{4}+\mathrm{H}_{2} \mathrm{O}+\mathrm{CO}_{2}$

In this titration, for the detection of the end-point, methyl orange is used as an indicator.
Procedure :
Preparation of $1 \mathbf{N ~} \mathrm{Na}_{2} \mathrm{CO}_{3}$ Solution : Dissolve an accurately weighed amount of 53 gm of $\mathrm{Na}_{2} \mathrm{CO}_{3}$ in sufficient amount of water to produce 1000 ml .
Procedure for Standardisation of $\mathbf{1 N} \mathbf{H}_{\mathbf{2}} \mathbf{S O}_{\mathbf{4}}$ : Pipette out exactly 10 ml of $1 \mathrm{~N} \mathrm{Na}_{2} \mathrm{CO} 3$ solution into a clean conical flask and add 2 or 3 drops of methyl orange indicator. Titrate the contents of the flask with $\mathrm{H}_{2} \mathrm{SO}_{4}$ until red colour is obtained. Repeat the titration for concordant values. Record the values in the tabular form...

Titration of $1 \mathrm{~N} \mathrm{Na}_{2} \mathrm{CO}_{3}$ solution with $\mathbf{H}_{2} \mathrm{SO}_{4}$ solution

| S.No. | Volume of $1 \mathrm{~N} \mathrm{Na}_{2} \mathrm{CO}_{3}$ <br> solution (ml) | Burette Reading |  | Vol. of $\mathrm{H}_{2} \mathrm{SO}_{4}$ rundown (ml) |
| :---: | :---: | :---: | :---: | :---: |
|  |  | Initial | Final |  |
| 1 | 10 | 0 |  |  |
| 2 | 10 |  |  |  |
| 3 | 10 |  |  |  |

The Normality of HCl is calculated using the formula :
$\mathbf{N}_{1} \mathbf{V}_{\mathbf{1}}=\mathbf{N}_{2} \mathbf{V}_{\mathbf{2}}$
where, $\mathrm{V}_{1}=$ Volume of $1 \mathrm{~N} \mathrm{Na}_{2} \mathrm{CO}_{3}$ solution $=10 \mathrm{ml}$
$\mathrm{N}_{1}=$ Normality of $\mathrm{Na}_{2} \mathrm{CO}_{3}$ solution $=1 \mathrm{~N}$
$\mathrm{V}_{2}=$ Volume of $\mathrm{H}_{2} \mathrm{SO}_{4}$ solution rundown (Average Burette Reading)
$\mathrm{N}_{2}=$ Normality of $\mathrm{H}_{2} \mathrm{SO}_{4}=$ ?

Therefore,


## Preparation and Standardisation of 0.1N Sulphuric Acid :

## Sulphuric Acid - M.Wt - 98.07

Preparation of 0.1N Sulphuric Acid Solution : Dissolve an accurately measured volume of 2.7 ml of Sulphuric Acid in sufficient amount of water to produce 1000 ml .
Principle : It is an example of alkalimetry. When a strong acid is titrated with a strong base, the salt produced in the reaction is not hydrolysed and therefore the pH of the resultant solution at the endpoint is not $7.0 . \mathrm{H}_{2} \mathrm{SO}_{4}$, a strong acid, is standardised by titrating with a strong base,i.e., $\mathrm{Na}_{2} \mathrm{CO}_{3}$ (primary standard). The following reaction takes place when $\mathrm{Na}_{2} \mathrm{CO}_{3}$ is titrated with $\mathrm{H}_{2} \mathrm{SO}_{4}$.
$\mathrm{Na}_{2} \mathrm{CO}_{3}+\mathrm{H}_{2} \mathrm{SO}_{4} \longrightarrow \mathrm{Na}_{2} \mathrm{SO}_{4}+\mathrm{H}_{2} \mathrm{O}+\mathrm{CO}_{2}$

In this titration, for the detection of the end-point, methyl orange is used as an indicator.
Procedure :
Preparation of $0.1 \mathbf{N ~} \mathbf{N a}_{2} \mathrm{CO}_{3}$ Solution : Dissolve an accurately weighed amount of 5.3 gm of $\mathrm{Na}_{2} \mathrm{CO}_{3}$ in sufficient amount of water to produce 1000 ml .
Procedure for Standardisation of $\mathbf{0 . 1 N} \mathbf{H}_{\mathbf{2}} \mathbf{S O}_{\mathbf{4}}$ : Pipette out exactly 10 ml of $0.1 \mathrm{~N} \mathrm{Na} \mathrm{Na}_{2} \mathrm{CO} 3$ solution into a clean conical flask and add 2 or 3 drops of methyl orange indicator. Titrate the contents of the flask with $\mathrm{H}_{2} \mathrm{SO}_{4}$ until red colour is obtained. Repeat the titration for concordant values. Record the values in the tabular form...

Titration of $0.1 \mathrm{~N} \mathrm{Na}_{2} \mathrm{CO}_{3}$ solution with $\mathrm{H}_{2} \mathrm{SO}_{4}$ solution

| S.No. | Volume of 0.1N <br> $\mathrm{Na}_{2} \mathrm{CO}_{3}$ solution (ml) | Burette Reading |  | Vol. of $\mathrm{H}_{2} \mathrm{SO}_{4}$ rundown (ml) |
| :---: | :---: | :---: | :---: | :---: |
|  |  | Initial | Final |  |
| 1 | 10 | 0 |  |  |
| 2 | 10 |  |  |  |
| 3 | 10 |  |  |  |

## The Normality of HCl is calculated using the formula :

$\mathbf{N}_{\mathbf{1}} \mathbf{V}_{\mathbf{1}}=\mathbf{N}_{\mathbf{2}} \mathbf{V}_{\mathbf{2}}$
where, $\mathrm{V}_{1}=$ Volume of $0.1 \mathrm{~N} \mathrm{Na}_{2} \mathrm{CO}_{3}$ solution $=10 \mathrm{ml}$
$\mathrm{N}_{1}=$ Normality of $\mathrm{Na}_{2} \mathrm{CO}_{3}$ solution $=0.1 \mathrm{~N}$
$\mathrm{V}_{2}=$ Volume of $\mathrm{H}_{2} \mathrm{SO}_{4}$ solution rundown (Average Burette Reading)
$\mathrm{N}_{2}=$ Normality of $\mathrm{H}_{2} \mathrm{SO}_{4}=$ ?

Therefore,


## Preparation and Standardisation of 1M Potassium permanganate :

Potassium permanganate - M.Wt-158
Preparation of 1M Potassium permanganate Solution : Dissolve an accurately weighed amount of 158 gm of $\mathrm{KMnO}_{4}$ in 900 ml of water heat on water bath for 1 hour and then cool it, then filter it using a sintered-glass filter and add sufficient amount of water to produce 1000 ml .
Principle : In volumetric analysis many reactions involve the process of oxidation and reduction. An oxidising agent is estimated by titrating with a reducing agent and vice-versa. These titration are called redox titrations. The standardisation of $\mathrm{KMnO}_{4}$ is an example of redox titration. $\mathrm{KMnO}_{4}$ is a powerful oxidising agent and in acidic medium, it oxidises oxalic acid to $\mathrm{CO}_{2}$.

$$
2 \mathrm{KMnO}_{4}+3 \mathrm{H}_{2} \mathrm{SO}_{4}+5 \mathrm{H}_{2} \mathrm{C}_{2} \mathrm{O}_{4} \longrightarrow \mathrm{~K}_{2} \mathrm{SO}_{4}+\mathbf{2} \mathrm{MnSO}_{4}+\mathbf{8 \mathrm { H } _ { 2 } \mathrm { O }}+\mathbf{1 0 \mathrm { CO } _ { 2 }}
$$

The temperature is maintained at $60^{\circ}-70^{\circ} \mathrm{C}$ during the titration because, the reaction proceeds slowly at room temperature. No indicator is required as $\mathrm{KMnO}_{4}$, is a self-indicator.

## Procedure :

Preparation of 1M Oxalic acid Solution : Dissolve an accurately weighed amount of 126.07 gm of Oxalic acid in sufficient amount of water to produce 1000 ml .
Procedure for Standardisation of $\mathbf{1 M} \mathbf{K M n O}_{4}$ : Pipette out exactly 10 ml of NM Oxalic acid solution into a clean conical flask and add 10 ml of dil. $\mathrm{H}_{2} \mathrm{SO}_{4}$. Warm the contents of the flask to $70^{\circ} \mathrm{C}$ and titrate with $\mathrm{KMnO}_{4}$ solution. Continue the titration until a faint pink colour is produced. Repeat the titration for concordant values. Enter the values in a tabular form...

Titration of 1M Oxalic acid solution with $\mathrm{KMnO}_{4}$ solution

| S.No. | Volume of 1M Oxalic <br> acid solution (ml) | Burette Reading |  | Vol. of $\mathrm{KMnO}_{4}$ rundown (ml) |
| :---: | :---: | :---: | :---: | :---: |
|  |  | Initial | Final |  |
| 1 | 10 | 0 |  |  |
| 2 | 10 |  |  |  |
| 3 | 10 |  |  |  |

The Molarity of $\mathrm{KMnO}_{4}$ is calculated using the formula :

## $\mathbf{M}_{1} \mathbf{V}_{\mathbf{1}}=\mathbf{M}_{\mathbf{2}} \mathbf{V}_{\mathbf{2}}$

where, $\mathrm{V}_{1}=$ Volume of 1 M Oxalic acid solution $=10 \mathrm{ml}$
$\mathrm{M}_{1}=$ Molarity of Oxalic acid solution $=1 \mathrm{M}$
$\mathrm{V}_{2}=$ Volume of $\mathrm{KMnO}_{4}$ solution rundown (Average Burette Reading)
$\mathrm{M}_{2}=$ Molarity of $\mathrm{KMnO}_{4}=$ ?

Therefore,


## Preparation and Standardisation of 0.1M Potassium permanganate :

Potassium permanganate - M.Wt-158
Preparation of 0.1 M Potassium permanganate Solution : Dissolve an accurately weighed amount of 15.8 gm of $\mathrm{KMnO}_{4}$ in 900 ml of water heat on water bath for 1 hour and then cool it, then filter it using a sintered-glass filter and add sufficient amount of water to produce 1000 ml .
Principle : In volumetric analysis many reactions involve the process of oxidation and reduction. An oxidising agent is estimated by titrating with a reducing agent and vice-versa. These titration are called redox titrations. The standardisation of $\mathrm{KMnO}_{4}$ is an example of redox titration. $\mathrm{KMnO}_{4}$ is a powerful oxidising agent and in acidic medium, it oxidises oxalic acid to $\mathrm{CO}_{2}$.

$$
\mathbf{2} \mathrm{KMnO}_{4}+3 \mathrm{H}_{2} \mathrm{SO}_{4}+\mathbf{5} \mathrm{H}_{2} \mathrm{C}_{2} \mathrm{O}_{4} \longrightarrow \mathrm{~K}_{2} \mathrm{SO}_{4}+\mathbf{2} \mathrm{MnSO}_{4}+\mathbf{8} \mathrm{H}_{2} \mathrm{O}+10 \mathrm{CO}_{2}
$$

The temperature is maintained at $60^{\circ}-70^{\circ} \mathrm{C}$ during the titration because, the reaction proceeds slowly at room temperature. No indicator is required as $\mathrm{KMnO}_{4}$, is a self-indicator.

## Procedure :

Preparation of 0.1M Oxalic acid Solution : Dissolve an accurately weighed amount of 12.6 gm of Oxalic acid in sufficient amount of water to produce 1000 ml .
Procedure for Standardisation of $\mathbf{0 . 1 M}$ KMnO $_{4}$ : Pipette out exactly 10 ml of 0.1 M Oxalic acid solution into a clean conical flask and add 10 ml of dil. $\mathrm{H}_{2} \mathrm{SO}_{4}$. Warm the contents of the flask to $70^{\circ} \mathrm{C}$ and titrate with $\mathrm{KMnO}_{4}$ solution. Continue the titration until a faint pink colour is produced. Repeat the titration for concordant values. Enter the values in a tabular form...

Titration of 0.1M Oxalic acid solution with $\mathrm{KMnO}_{4}$ solution

| S.No. | Volume of 0.1M <br> Oxalic acid solution <br> $(\mathrm{ml})$ | $\|2\|$ | Vol. of $\mathrm{KMnO}_{4}$ rundown (ml) |  |
| :---: | :---: | :---: | :---: | :---: |
|  | Initial | Final |  |  |
| 1 | 10 | 0 |  |  |
| 2 | 10 |  |  |  |
| 3 | 10 |  |  |  |

The Molarity of $\mathrm{KMnO}_{4}$ is calculated using the formula :

## $\mathbf{M}_{1} \mathbf{V}_{1}=\mathbf{M}_{2} \mathbf{V}_{\mathbf{2}}$

where, $\mathrm{V}_{1}=$ Volume of 0.1 M Oxalic acid solution $=10 \mathrm{ml}$
$\mathrm{M}_{1}=$ Molarity of Oxalic acid solution $=0.1 \mathrm{M}$
$\mathrm{V}_{2}=$ Volume of $\mathrm{KMnO}_{4}$ solution rundown (Average Burette Reading)
$\mathrm{M}_{2}=$ Molarity of $\mathrm{KMnO}_{4}=$ ?

Therefore,
$\mathbf{M}_{\mathbf{2}}=\frac{\mathbf{M}_{\mathbf{l}} \mathbf{V}_{\mathbf{1}}}{\mathbf{V}_{\mathbf{2}}}$

## Preparation and Standardisation of 1N Potassium permanganate :

Potassium permanganate - M.Wt-158
Preparation of 1N Potassium permanganate Solution : Dissolve an accurately weighed amount of 31.6 gm of $\mathrm{KMnO}_{4}$ in 900 ml of water heat on water bath for 1 hour and then cool it, then filter it using a sintered-glass filter and add sufficient amount of water to produce 1000 ml .
Principle : In volumetric analysis many reactions involve the process of oxidation and reduction. An oxidising agent is estimated by titrating with a reducing agent and vice-versa. These titration are called redox titrations. The standardisation of $\mathrm{KMnO}_{4}$ is an example of redox titration. $\mathrm{KMnO}_{4}$ is a powerful oxidising agent and in acidic medium, it oxidises oxalic acid to $\mathrm{CO}_{2}$.

$$
\mathbf{2} \mathrm{KMnO}_{4}+3 \mathrm{H}_{2} \mathrm{SO}_{4}+\mathbf{5} \mathrm{H}_{2} \mathrm{C}_{2} \mathrm{O}_{4} \longrightarrow \mathrm{~K}_{2} \mathrm{SO}_{4}+\mathbf{2} \mathrm{MnSO}_{4}+\mathbf{8} \mathrm{H}_{2} \mathrm{O}+10 \mathrm{CO}_{2}
$$

The temperature is maintained at $60^{\circ}-70^{\circ} \mathrm{C}$ during the titration because, the reaction proceeds slowly at room temperature. No indicator is required as $\mathrm{KMnO}_{4}$, is a self-indicator.

## Procedure :

Preparation of 1N Oxalic acid Solution : Dissolve an accurately weighed amount of 63.035 gm of Oxalic acid in sufficient amount of water to produce 1000 ml .
Procedure for Standardisation of $\mathbf{1 N} \mathbf{K M n O}_{4}$ : Pipette out exactly 10 ml of 1 N Oxalic acid solution into a clean conical flask and add 10 ml of dil. $\mathrm{H}_{2} \mathrm{SO}_{4}$. Warm the contents of the flask to $70^{\circ} \mathrm{C}$ and titrate with $\mathrm{KMnO}_{4}$ solution. Continue the titration until a faint pink colour is produced. Repeat the titration for concordant values. Enter the values in a tabular form...

Titration of 1N Oxalic acid solution with $\mathrm{KMnO}_{4}$ solution

| S.No. | Volume of 1N Oxalic <br> acid solution (ml) | Burette Reading |  | Vol. of $\mathrm{KMnO}_{4}$ rundown (ml) |
| :---: | :---: | :---: | :---: | :---: |
|  |  | Initial | Final |  |
| 1 | 10 | 0 |  |  |
| 2 | 10 |  |  |  |
| 3 | 10 |  |  |  |

The Normality of $\mathrm{KMnO}_{4}$ is calculated using the formula :
$\mathbf{N}_{1} \mathbf{V}_{\mathbf{1}}=\mathbf{N}_{\mathbf{2}} \mathbf{V}_{\mathbf{2}}$
where, $\mathrm{V}_{1}=$ Volume of 1 N Oxalic acid solution $=10 \mathrm{ml}$
$\mathrm{N}_{1}=$ Normality of Oxalic acid solution $=1 \mathrm{~N}$
$\mathrm{V}_{2}=$ Volume of $\mathrm{KMnO}_{4}$ solution rundown (Average Burette Reading)
$\mathrm{N}_{2}=$ Normality of $\mathrm{KMnO}_{4}=$ ?

Therefore,


## Preparation and Standardisation of 0.1 N Potassium permanganate :

Potassium permanganate - M.Wt-158
Preparation of 0.1 N Potassium permanganate Solution : Dissolve an accurately weighed amount of 3.16 gm of $\mathrm{KMnO}_{4}$ in 900 ml of water heat on water bath for 1 hour and then cool it, then filter it using a sintered-glass filter and add sufficient amount of water to produce 1000 ml .
Principle : In volumetric analysis many reactions involve the process of oxidation and reduction. An oxidising agent is estimated by titrating with a reducing agent and vice-versa. These titration are called redox titrations. The standardisation of $\mathrm{KMnO}_{4}$ is an example of redox titration. $\mathrm{KMnO}_{4}$ is a powerful oxidising agent and in acidic medium, it oxidises oxalic acid to $\mathrm{CO}_{2}$.

$$
2 \mathrm{KMnO}_{4}+3 \mathrm{H}_{2} \mathrm{SO}_{4}+\mathbf{5} \mathrm{H}_{2} \mathrm{C}_{2} \mathrm{O}_{4} \longrightarrow \mathrm{~K}_{2} \mathrm{SO}_{4}+\mathbf{2} \mathrm{MnSO}_{4}+\mathbf{8} \mathrm{H}_{2} \mathrm{O}+10 \mathrm{CO}_{2}
$$

The temperature is maintained at $60^{\circ}-70^{\circ} \mathrm{C}$ during the titration because, the reaction proceeds slowly at room temperature. No indicator is required as $\mathrm{KMnO}_{4}$, is a self-indicator.

## Procedure :

Preparation of 0.1N Oxalic acid Solution : Dissolve an accurately weighed amount of 6.3 gm of Oxalic acid in sufficient amount of water to produce 1000 ml .
Procedure for Standardisation of $\mathbf{0 . 1 N} \mathbf{K M n O}_{4}$ : Pipette out exactly 10 ml of 0.1 N Oxalic acid solution into a clean conical flask and add 10 ml of dil. $\mathrm{H}_{2} \mathrm{SO}_{4}$. Warm the contents of the flask to $70^{\circ} \mathrm{C}$ and titrate with $\mathrm{KMnO}_{4}$ solution. Continue the titration until a faint pink colour is produced. Repeat the titration for concordant values. Enter the values in a tabular form...

Titration of 0.1 N Oxalic acid solution with $\mathrm{KMnO}_{4}$ solution

| S.No. | Volume of 0.1N Oxalic <br> acid solution (ml) | Burette Reading |  | Vol. of $\mathrm{KMnO}_{4}$ rundown (ml) |
| :---: | :---: | :---: | :---: | :---: |
|  |  | Initial | Final |  |
| 1 | 10 | 0 |  |  |
| 2 | 10 |  |  |  |
| 3 | 10 |  |  |  |

The Normality of $\mathrm{KMnO}_{4}$ is calculated using the formula :
$\mathbf{N}_{1} \mathbf{V}_{\mathbf{1}}=\mathbf{N}_{\mathbf{2}} \mathbf{V}_{\mathbf{2}}$
where, $\mathrm{V}_{1}=$ Volume of 0.1 N Oxalic acid solution $=10 \mathrm{ml}$
$\mathrm{N}_{1}=$ Normality of Oxalic acid solution $=0.1 \mathrm{~N}$
$\mathrm{V}_{2}=$ Volume of $\mathrm{KMnO}_{4}$ solution rundown (Average Burette Reading)
$\mathrm{N}_{2}=$ Normality of $\mathrm{KMnO}_{4}=$ ?

Therefore,


## Preparation and Standardisation of 0.1M Ceric ammonium sulphate :

Ceric ammonium sulphate - M.Wt - 632.53
Preparation of 0.1 M Ceric ammonium sulphate : Dissolve 63.25 gm of Ceric ammonium sulphate with the aid of gentle heat, in a mixture of 30 ml of sulphuric acid and 500 ml of water. Cool, filter the solution if turbid and dilute to 1000 ml with water.
Principle : Ceric ammonium sulphate in sulphuric acid medium can function as strong oxidising agent and have a high oxidation potential. Sufficient sulphuric acid is to be used to prevent hydrolysis and precipitation of basic salts.
Ceric ammonium sulphate, a salt of suitable solubility for the preparation of the standard solution has the approximate formula $\mathrm{Ce}\left(\mathrm{SO}_{4}\right)_{2} .2\left(\mathrm{NH}_{4}\right)_{2} \mathrm{SO}_{4} .2 \mathrm{H}_{2} \mathrm{O}$, but the solution has to be standardised against a solution of Arsenic trioxide $\left(\mathrm{As}_{2} \mathrm{O}_{3}\right)$. Since Ceric ammonium sulphate is a strong Oxidising agent, we can determine various reducing substances by simple titration.
In presence of reducing agent, it undergoes reduction to the cerous state for example.

$$
2 \mathrm{Ce}\left(\mathrm{SO}_{4}\right)_{2}+2 \mathrm{FeSO}_{4} \longrightarrow \mathrm{Ce}_{2}\left(\mathrm{SO}_{4}\right)_{3}+\mathrm{Fe}_{2}\left(\mathrm{SO}_{4}\right)_{3}
$$

Procedure for Standardisation of 0.1M Ceric ammonium sulphate : Weigh accurately about 0.2 gm of Arsenic trioxide, previously dried at $105^{\circ} \mathrm{C}$ for 1 hour, and transfer to a 500 ml conical flask. Wash down the inner walls of the flask with 25 ml of a $8 \% \mathrm{w} / \mathrm{v}$ solution of NaOH , Swirl to dissolve, add 100 ml of water and mix.

Add 30 ml of dil. sulphuric acid, 0.15 ml of Osmic acid solution, 0.1 ml of ferroin sulphate solution and slowly titrate with the ceric ammonium sulphate solution until the pink colour is changed to a very pale blue colour.
Equivalent factor : Each ml of 0.1 M Ceric ammonium sulphate $\equiv 0.04946 \mathrm{gm}$ of $\mathrm{As}_{2} \mathrm{O}_{3}$

## Calculation :

Molarity of Ceric ammonium sulphate $=\quad$ Weight taken $\left(\mathrm{Ce}_{\left(\mathrm{SO}_{4}\right)_{2} \cdot 2\left(\mathrm{NH}_{4}\right) 2 \mathrm{SO}_{4} .2 \mathrm{H}_{2} \mathrm{O}}\right) \times$ Req. molarity

## Preparation and Standardisation of 0.1N Ceric ammonium sulphate :

Ceric ammonium sulphate - M.Wt - 632.53
Preparation of 0.1 N Ceric ammonium sulphate : Dissolve 63.25 gm of Ceric ammonium sulphate with the aid of gentle heat, in a mixture of 30 ml of sulphuric acid and 500 ml of water. Cool, filter the solution if turbid and dilute to 1000 ml with water.
Principle : Ceric ammonium sulphate in sulphuric acid medium can function as strong oxidising agent and have a high oxidation potential. Sufficient sulphuric acid is to be used to prevent hydrolysis and precipitation of basic salts.
Ceric ammonium sulphate, a salt of suitable solubility for the preparation of the standard solution has the approximate formula $\mathrm{Ce}\left(\mathrm{SO}_{4}\right)_{2} .2\left(\mathrm{NH}_{4}\right)_{2} \mathrm{SO}_{4} .2 \mathrm{H}_{2} \mathrm{O}$, but the solution has to be standardised against a solution of Arsenic trioxide $\left(\mathrm{As}_{2} \mathrm{O}_{3}\right)$. Since Ceric ammonium sulphate is a strong Oxidising agent, we can determine various reducing substances by simple titration.
In presence of reducing agent, it undergoes reduction to the cerous state for example.

$$
2 \mathrm{Ce}\left(\mathrm{SO}_{4}\right)_{2}+2 \mathrm{FeSO}_{4} \longrightarrow \mathrm{Ce}_{2}\left(\mathrm{SO}_{4}\right)_{3}+\mathrm{Fe}_{2}\left(\mathrm{SO}_{4}\right)_{3}
$$

Procedure for Standardisation of $\mathbf{0 . 1} \mathbf{N}$ Ceric ammonium sulphate : Weigh accurately about 0.2 gm of Arsenic trioxide, previously dried at $105^{\circ} \mathrm{C}$ for 1 hour, and transfer to a 500 ml conical flask. Wash down the inner walls of the flask with 25 ml of a $8 \% \mathrm{w} / \mathrm{v}$ solution of NaOH , Swirl to dissolve, add 100 ml of water and mix.

Add 30 ml of dil. sulphuric acid, 0.15 ml of Osmic acid solution, 0.1 ml of ferroin sulphate solution and slowly titrate with the ceric ammonium sulphate solution until the pink colour is changed to a very pale blue colour.
Equivalent factor : Each ml of 0.1 N Ceric ammonium sulphate $\equiv 0.04946 \mathrm{gm}$ of $\mathrm{As}_{2} \mathrm{O}_{3}$

## Calculation :

Normality of Ceric ammonium sulphate $=$ Weight taken ( $\mathrm{Ce}_{\left.\left(\mathrm{SO}_{4}\right)_{2} \cdot 2\left(\mathrm{NH}_{4}\right) 2 \mathrm{SO}_{4} \cdot 2 \mathrm{H}_{2} \mathrm{O}\right)}$ x Req. normality

