KANGLU KUMHAR GOVT. COLLEGE DURGUKONDAL

DIST. - U. B. KANKER (C.G.)

DEPARTMENT OF CHEMISTRY

TOPIC - VOLUMETRIC ANALYSIS

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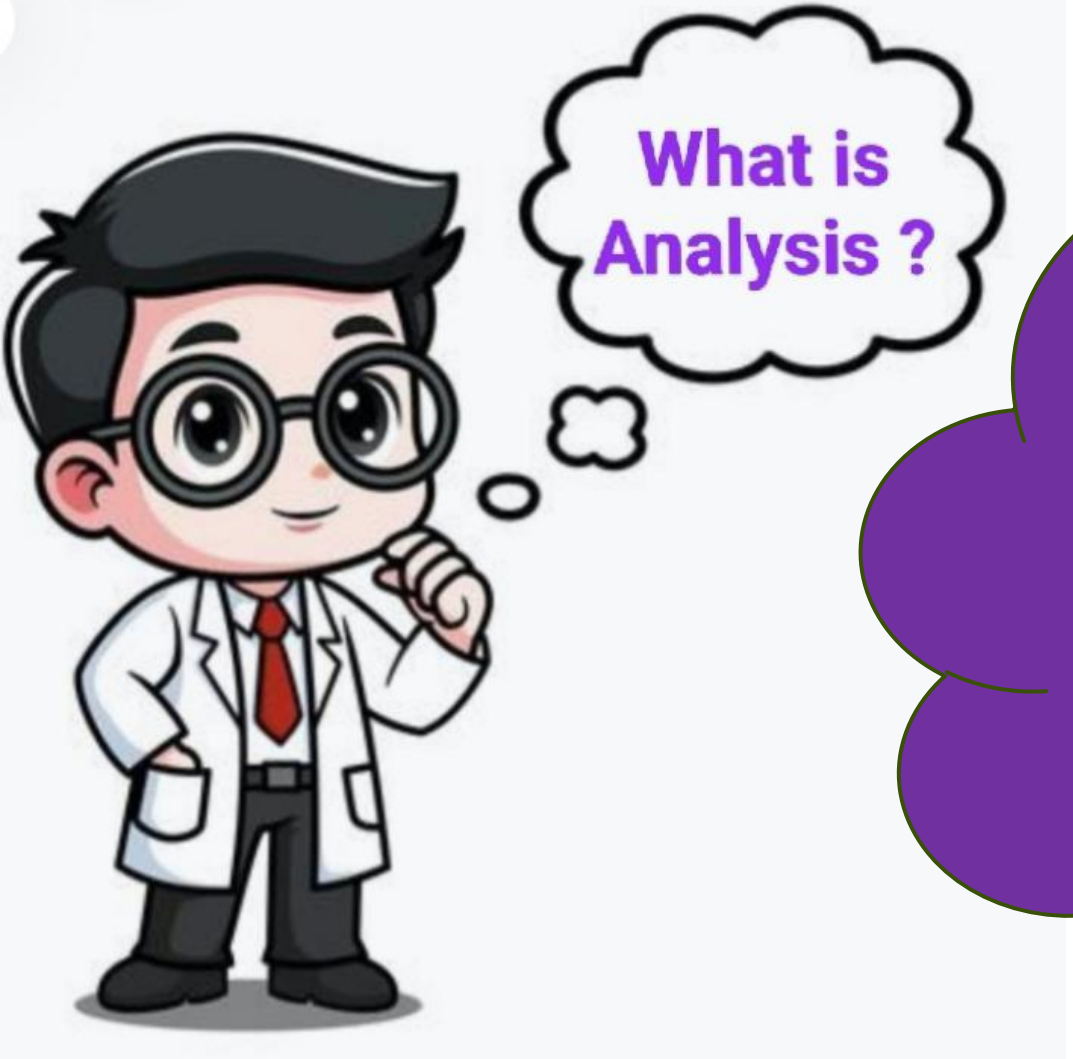
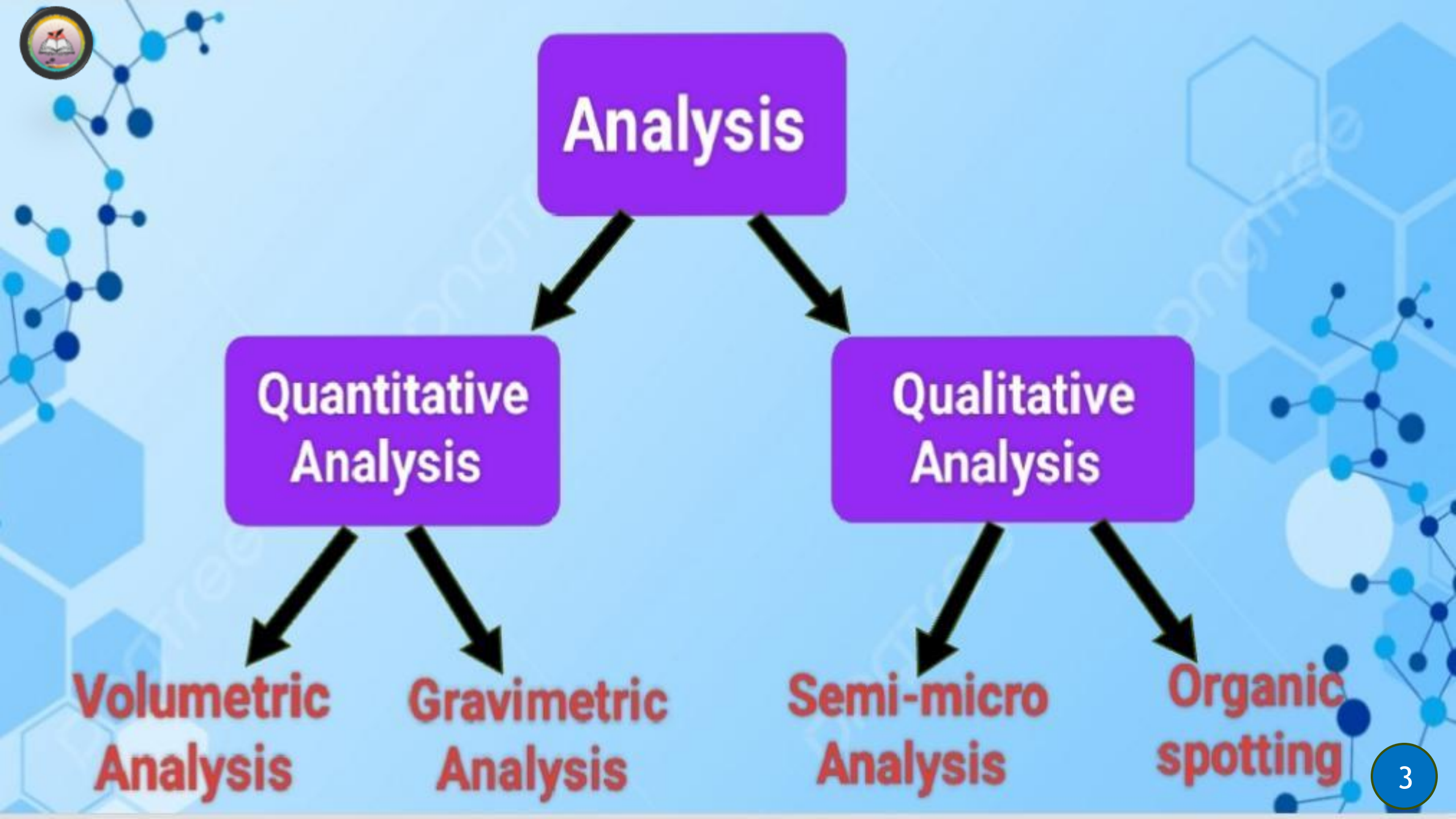
Content

Introduction



Analysis is the process of identifying, seperating and quantifying the components of a substance to understand its chemical composition , properties and structure.


Analysis
Quantitative Analysis
Qualitative Analysis
Volumetric Analysis
Gravimetric Analysis
Semi-micro Analysis
Organic spotting

INTRODUCTION

Volumetric Analysis **:-**

- Volumetric Analysis is a quantitative chemistry technique used to determine the unknown concentration of a solution by reacting it with a solution of precisely known concentration until the reaction is complete.

- It also known as titration .

Basic Terms In Volumetric Analysis

- *Titration* :-Titration is a technique in which a solution of known concentration is used to determine the concentration of unknown solution .

- *Titrant* :- The solution of known concentration which is usually taken in a burette .

- *Analyte* :- The solution whose concentration is to be determined and usually taken in conical flask.

- ***Standard Solution*** **:-** A solution whose concentration is accurately known.

- ***End Point*** **:-** The point in titration at which reaction between two solution is just completed and at which indicator can show sharp colour change.

- ***Equivalence Point*** **:-** The stage at which exactly equivalent amount of titrant is added to analyte is known as equivalence Point.

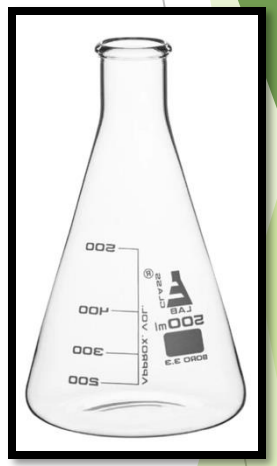
- ***Indicator*** **:-** A reagent is used to indicate the end point of a titration by bringing out clear visual colour change.

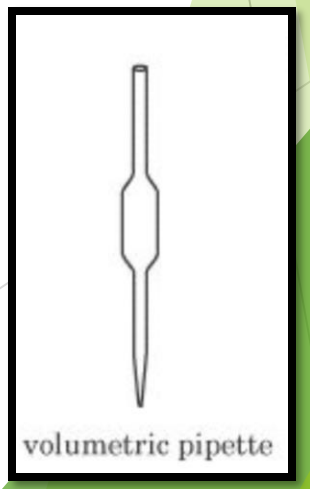
Apparatus Needed For Titration

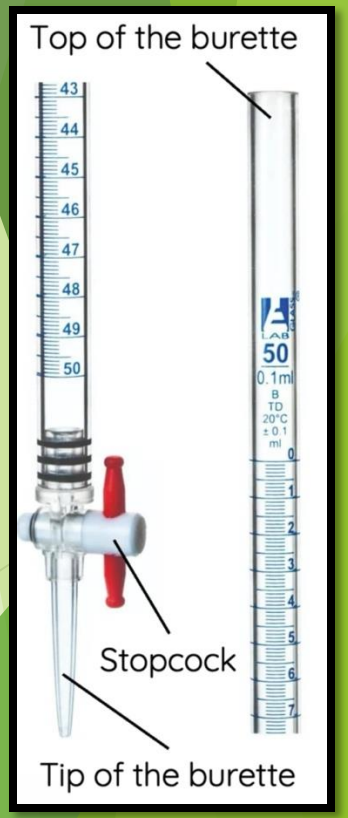
1. **Pipette** - For measuring accurate volumes of solutions.

2. **Burette** – For pouring measured volumes of solutions.

3. **Conical Flask** – For mixing two solutions and containing solution



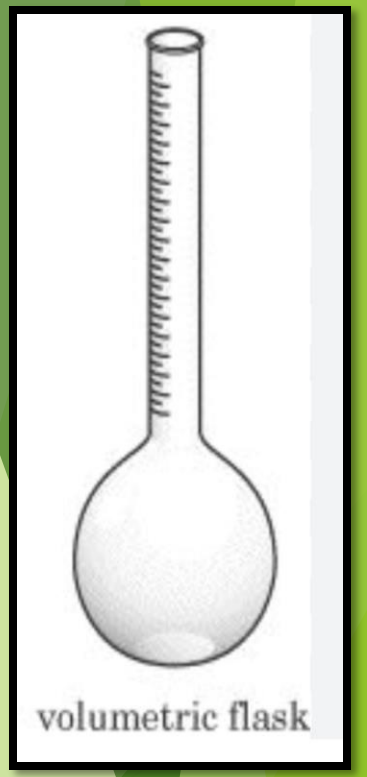




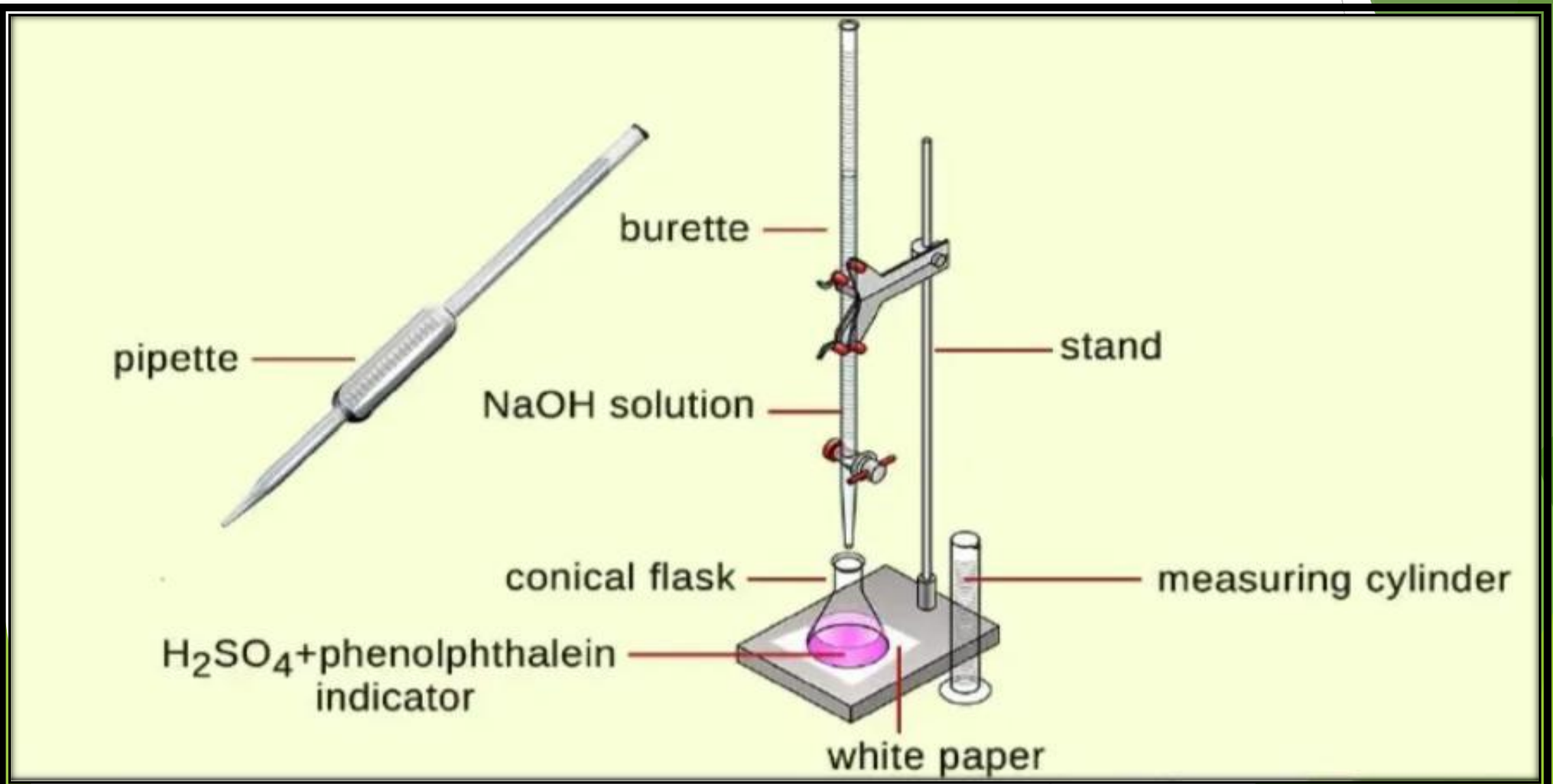
4. Volumetric Flask - A flask used to make up accurate volumes for standard solutions of known concentration.

5. Wash bottle - These contain distilled water for cleaning equipment and preparing solutions .

6. Funnel - For transfer of liquid without spilling.



Apparatus Needed For Titration



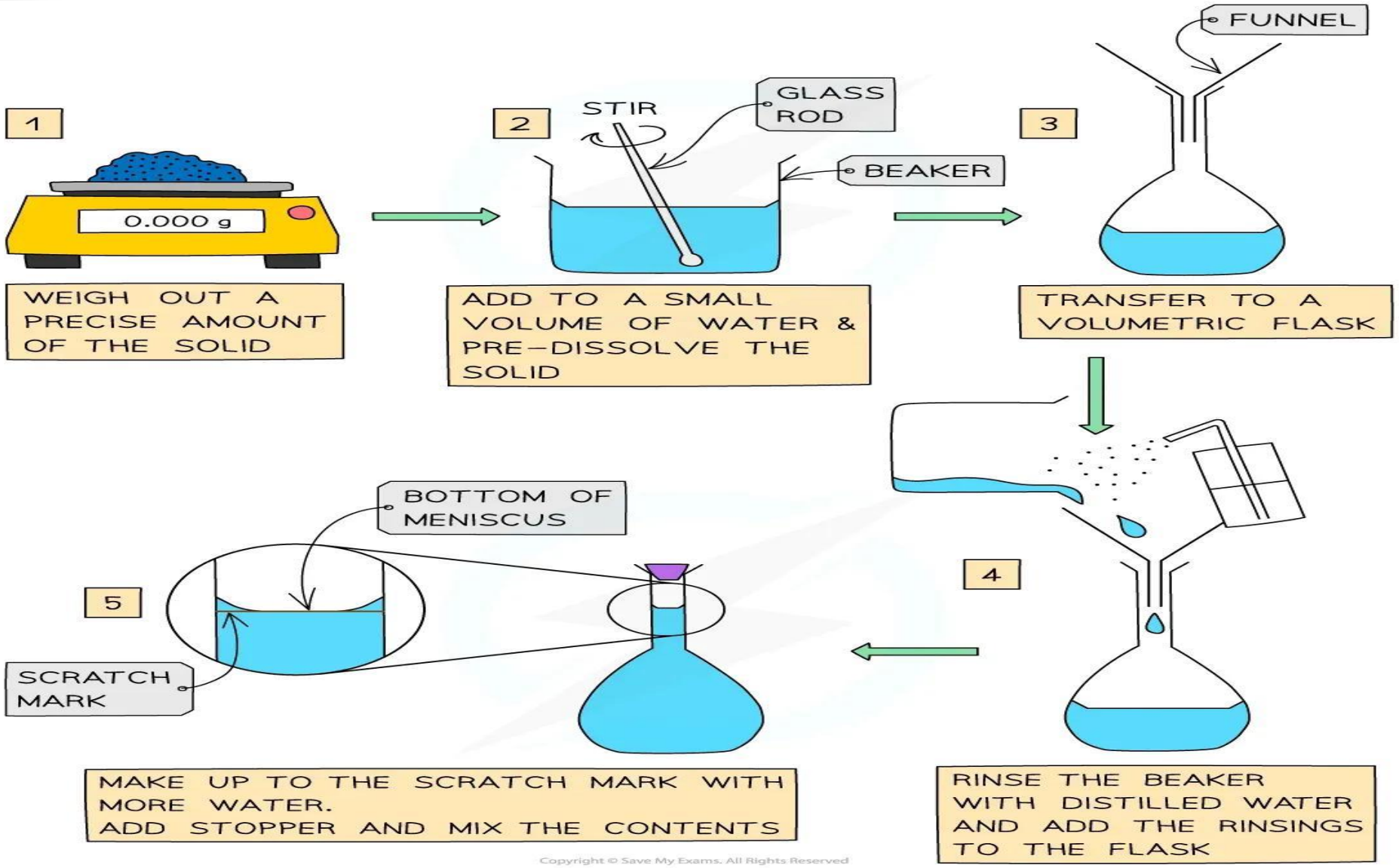
Primary Standard Solution

- A solution which can be prepared by directly weighing the solute and making it up to certain volume is a primary standard solutions .
- It is stable longer time under storage.
- It is in pure form.
- It's concentration does not change during the storage.
- ***Examples*** :- Oxalic acid, Potassium dichromate, Mohr salt

Secondary Standard Solutions

- The solution whose concentration is determined by titrating it with the primary standard solution and then can be used directly in Volumetric Analysis is the secondary standard solution.
- It's concentration changes during the storage.
- *Examples:-* Copper sulphate, Sodium hydroxide, Potassium permanganate, Sodium thiosulfate

How To Prepare Standard Solutions



General Principles Of Volumetric Analysis

A titrimetric method of analysis is based on the following chemical reaction –

$$A + B \longrightarrow \text{Product}$$

A molecule of analyte reacts with B molecules of titrant to give the product . The titrant is gradually added from the burette until the amount of B is chemically equivalent to the amount of A

Terms To Express Concentrations

Molecular weight:- **Molecular weight is the sum of atomic weights of all atoms in the molecule .**

Molecular weight = (Atomic weight of the element) × (no. of atoms of that element)

Unit – a.m.u. (atomic mass unit)

Equivalent weight **:- The ratio of atomic weight / molecular weight of compound to its valence is called as equivalent weight.**

$$Equivalent\ Weight = \frac{Molecular\ Weight}{No.of\ vaalancy/Charge}$$

[Valence = no. of replaceable hydrogen ions (H^+) or hydroxyl ion (OH^-)]

Molarity **(M) :- Molarity is a measure of solutions concentration defined as the number of moles of solute per litre of solution.**

$$M = \frac{Moles\ of\ Solute}{Volume\ of\ Solution\ (Litre)}$$

Unit – mole / L

Normality**(N) :- The normality of a solution represents the number of equivalents of solute contained in one litre of the solution .**

$$N = \frac{Gram\ Weight\ of\ Solute \times 100}{Gram\ Eq.Wt.of\ Solute \times Volume\ of\ Solution}$$

Unit – eq / L

Mass Percentage (w/w)% **:- The amount of solute in grams present in 100 grams of the solution is called "Mass Percentage" .**

$$\%(W/W) = \frac{Mass\ of\ Solute\ (gm) \times 100}{Mass\ of\ Solution\ (gm)}$$

Unit = Unit less

Volume Percentage (v/v)% **:- The volume of solute in ml present in 100 ml of the solution is called "Volume Percentage" .**

$$\%(V/V) = \frac{Mass\ of\ Solute\ (ml) \times 100}{Mass\ of\ Solution\ (ml)}$$

Mass-Volume Percentage **(w/v)% :- The mass of solute in grams present in 100 ml of the solution is called "Mass-Volume Percentage" .**

$$\%(W/V) = \frac{Mass\ of\ Solute\ (gm) \times 100}{Mass\ of\ Solution\ (ml)}$$

Unit = unit less

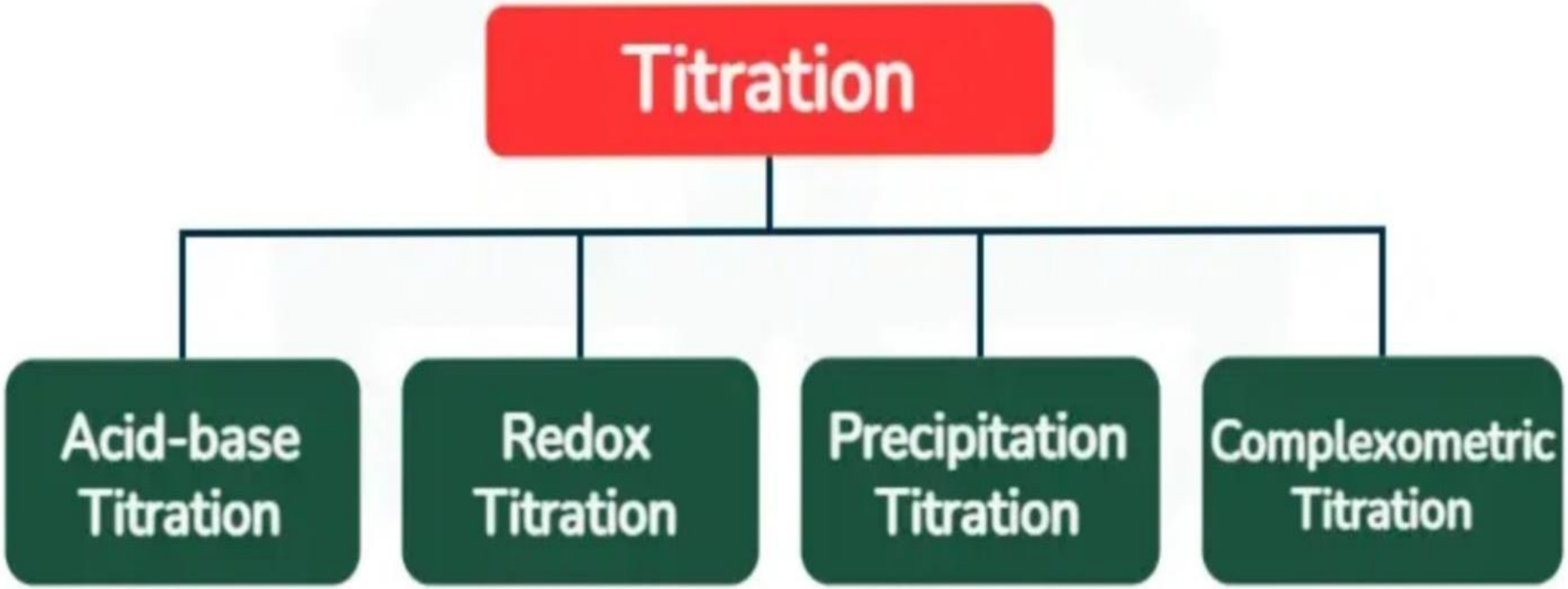
Parts per million **(ppm) :- The amount of a solute present in one million (10 lakh) parts of a solution is called "ppm" .**

$$ppm = \frac{Mass\ of\ Solute\ (gm) \times 106}{Mass\ of\ Solvent\ (gm)}$$

Formality **(F) :- The number of gram formula masses of solute dissolved in 1 litre of solution is called "Formality" .**

$$F = \frac{Mass\ of\ Solute\ (gm)}{Formula\ Mass\ of\ Solute \times Volume\ of\ Solution\ (Litre)}$$

Classification of Titration



(1) Acid – Base Titration

In this type of titration there is a reaction between an acid and base to form salt and water .

Basically the reaction is between the H+ ions of acid with OH- ions of the base to form nearly undissociated water .

$$HCl + NaOH \longrightarrow NaCl + H_2O$$

$$H^+ + OH^- \longrightarrow H_2O$$

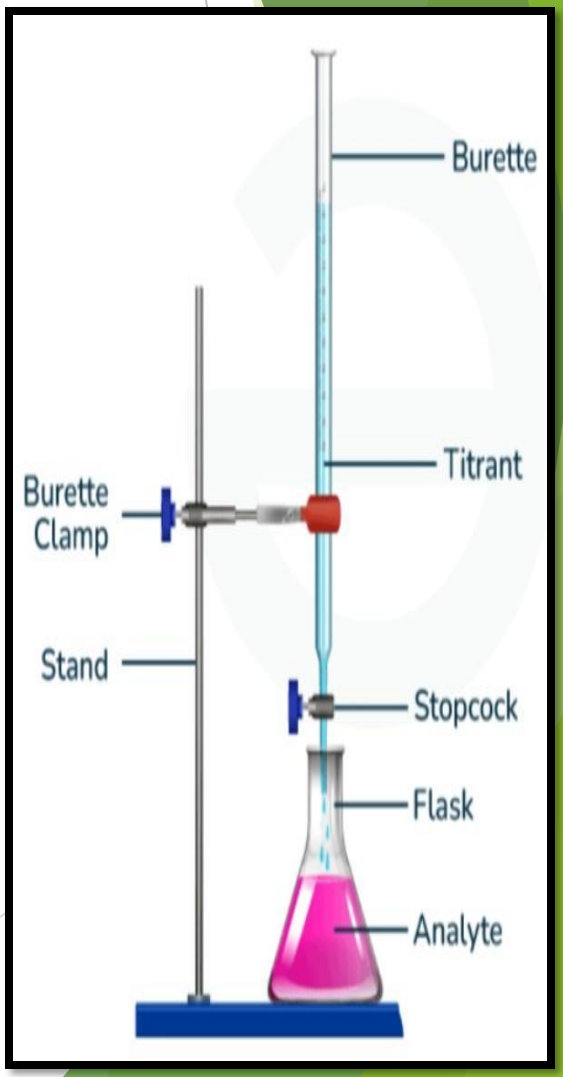
Types Of Acid – Base Of Titration

1) Titration of strong acid Vs strong base .

$$HCl + NaOH \longrightarrow NaCl + H_2O$$

2) Titration of strong acid Vs weak base .

$$HCl + NH_3OH \longrightarrow NH_4Cl + H_2O$$



3) Titration of weak acid Vs strong base .

$$CH_3COOH + NaOH \longrightarrow CH_3COONa + H_2O$$

4) Titration of weak acid Vs weak base.

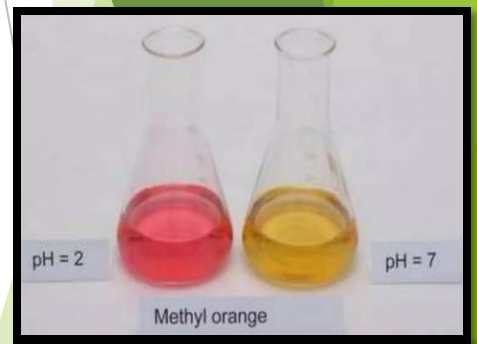
$$CH_3COOH + NH_3OH \longrightarrow CH_3COONH_4 + H_2O$$

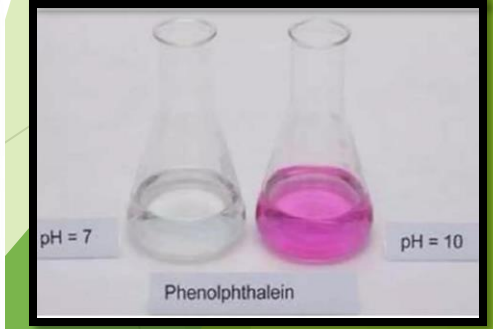
Acid - Base Indicators

Two essential characteristics of good indicator

- The color change must be clear and sharp.
- Indicate when the reaction is complete.

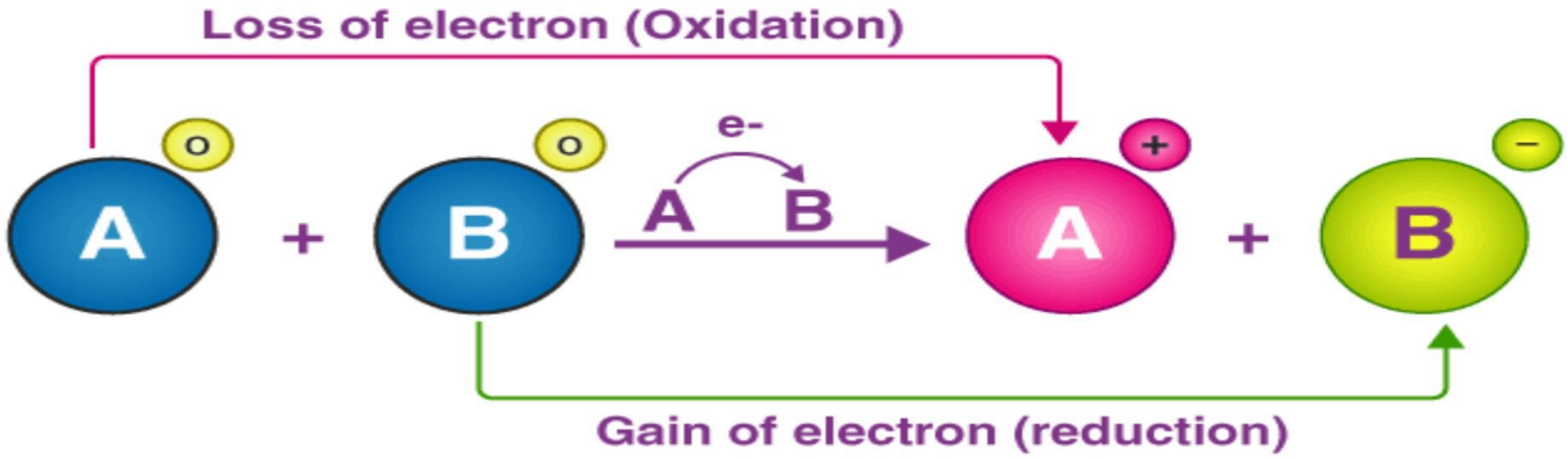
Indicator	pH range	Color change		Used in titration
		Acidic medium	Basic medium	
Methyl orange	3.2 – 4.4	Pink / orange	Yellow	SB vs SA, WB vs SA
Methyl red	4.8 – 6.0	Red	Yellow	WB vs SA
Phenolphthalein	8.2 – 10.0	Colorless	Pink	SA vs SB, WA vs SB





(2) Redox Titration

- Redox Titration are those titration that are based on the redox reaction. Redox reaction are those reaction in which both oxidation and reduction takes place .



Oxidation - Oxidation can be defined as three ways -

- Gain of Oxygen = $CO + O \longrightarrow CO_2$
- Loss of Hydrogen = $H_2S \longrightarrow S^{2-} + 2H^+$
- Loss of Electrons = $Fe \longrightarrow Fe^{2+} + 2e^-$

- Oxidation can be remembered by " LEO " (Loosing Electrons is Oxidation)

Reduction :- Reduction is just opposite to oxidation, it can be defined as :-

- Loss of Oxygen = $2HgO. \longrightarrow 2Hg + O_2$
- Gain of Hydrogen = $C + 4H \longrightarrow CH_4$
- Gain of Electrons = $Na^+ + 1e^- \longrightarrow Na$

- It can be remembered by "GER" (Gaining Electrons is Reduction)

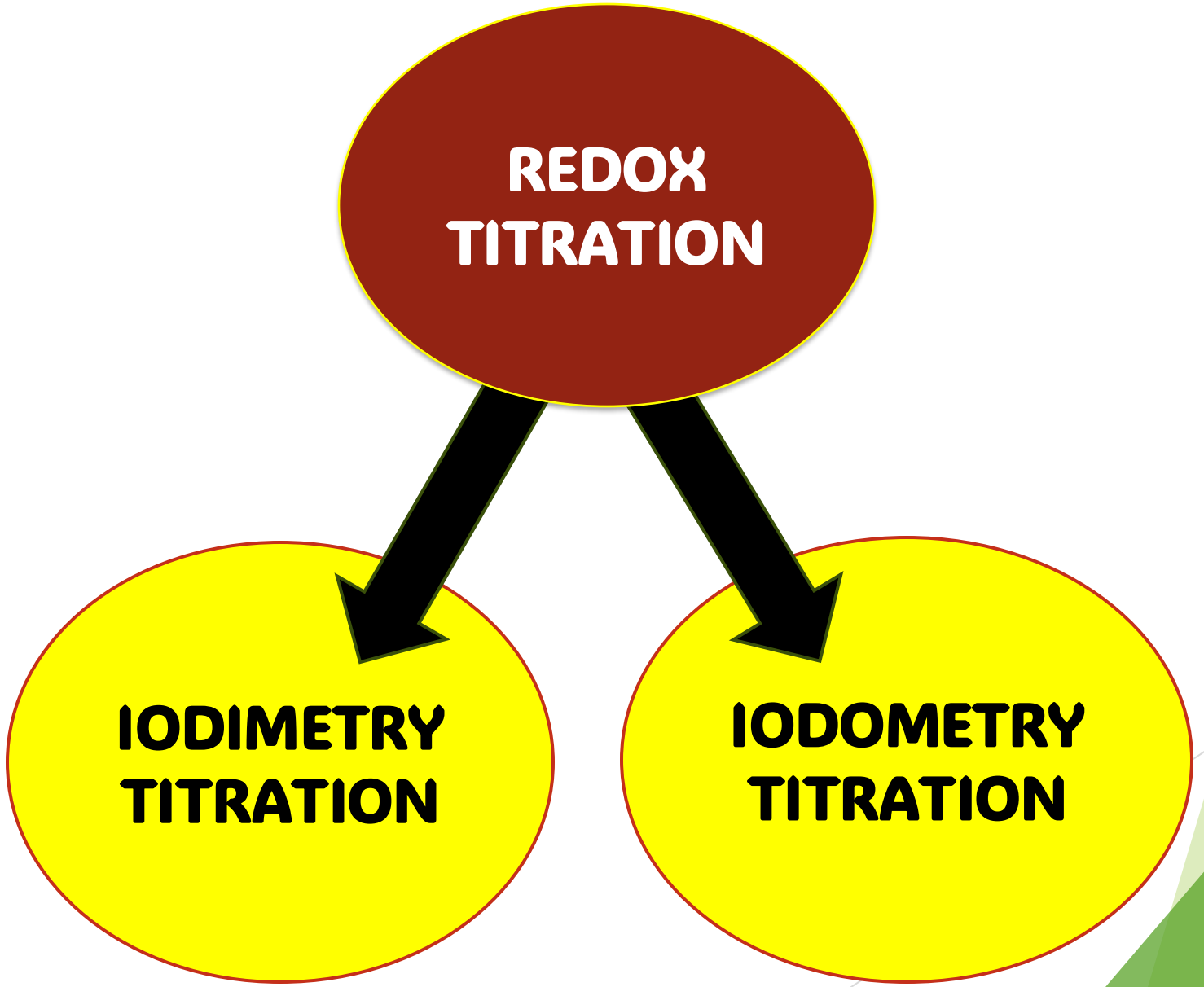
Oxidizing Agent

- Oxidizing agent are those substance which do oxidation of others and itself gets reduced.
- We can simply say substance which gain electrons are called as oxidizing agent.

Reducing Agent

- Reducing agents are those substance which do reduction of others and itself gets oxidized .
- We can simply say substance which loss / donates electrons are called reducing agents

TYPES OF REDOX TITRATION



IODIMETRY TITRATION

- Iodimetry is a type of Direct Titration. These are the titration in which free iodine is used.
- The titration in which standard iodine solution is used for the determination of reducing agents like Sodium thiosulfate ($Na_2S_2O_3$) is known as Iodimetry .
- End Point of the titration is determined by change in colour from Blue to colourless.
- PROCESS :- First iodine is mixed with KI because generally it is not easily soluble in water.

$$KI + I_2 \longrightarrow KI_3$$

- Now we use starch solution as an indicator, iodine will react with starch and forms iodine – starch complex which appears blue in colour.

$$I_2 + \text{Starch} \longrightarrow \text{Blue Colour}$$

- Now this standard iodine solution is titrated with reducing agent ($Na_2S_2O_3$) .

$$I_2 + 2S_2O_3^{2-} \rightleftharpoons S_4O_6^{2-} + 2I^-$$

- Now at the end point all iodine reacts with thiosulphate ions and converted into iodine and blue colour disappears .

$$I^- + \text{starch indicator} \longrightarrow \text{No Colour}$$

IODOMETRY TITRATION

- Iodometry is a type of indirect titration. These are the titration in which liberated iodine is used.

- When a standard solution of Sodium thiosulphate ($Na_2S_2O_3$) is used for the determination of liberated iodine using starch solution as an indicator then it is known as Iodometry .

- Iodometry is completed in two steps :

1. STEP - 1

- The first step is done by the reaction between the oxidizing agent ($K_2Cr_2O_7$) and excess KI and as a result of the reaction iodine gets liberated.

$$K_2Cr_2O_7 + 6KI + 7H_2SO_4 \longrightarrow Cr_2(SO_4)_3 + 4K_2SO_4 + 3I_2 + 7H_2O$$

2. STEP:- 2

- Starch solution is added in the liberated iodine which form blue colour.

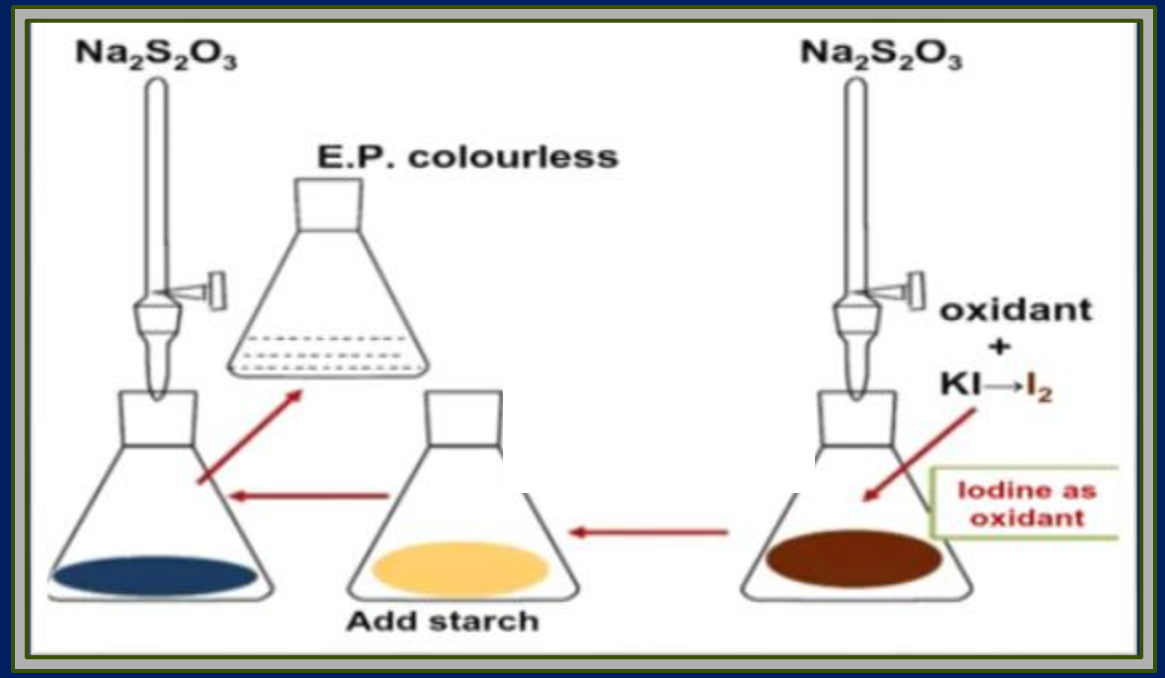
$$I_2 + \text{starch} \longrightarrow \text{Blue colour}$$

- Now we use standard sodium thiosulphate solution which reacts with liberated iodine in the solution.

$$I_2 + 2S_2O_3^{2-} \longleftrightarrow S_4O_6^{2-} + 2I^-$$

- Now at the end point all the liberated iodine reacts with thiosulphate and produce iodide ion , the indicator don’t show any reaction with iodide ion, hence blue colour of solution disappears (colour less) .

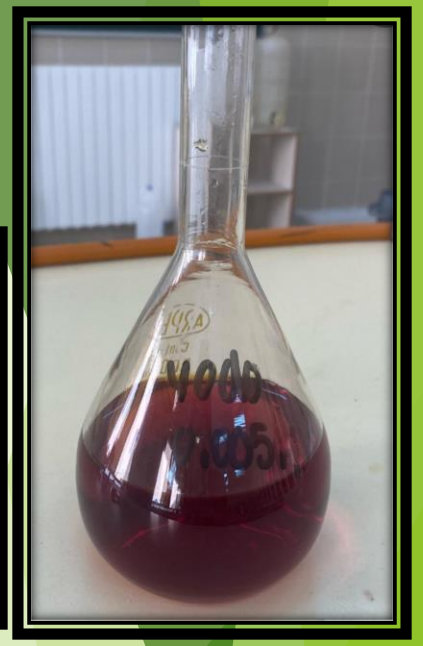
I^- + Indicator ⟶ Colourless



Redox Indicators

Self indicator

$KMnO_4$ - Pink
Iodine - Brown



External indicator

Potassium ferricyanide [$K_3Fe(CN)_6$] = Dark Blue

Internal Indicator

Name	Oxidized form	Reduced form
1) Ferroin	Pale Blue	Red
2) Diphenylamine	Violet / Blue	Colour less
3) Methylene Blue	Blue	Colour less
4) Starch Iodine	Blue	Colour less
5) Nitro ferroin	Pale Blue	Red

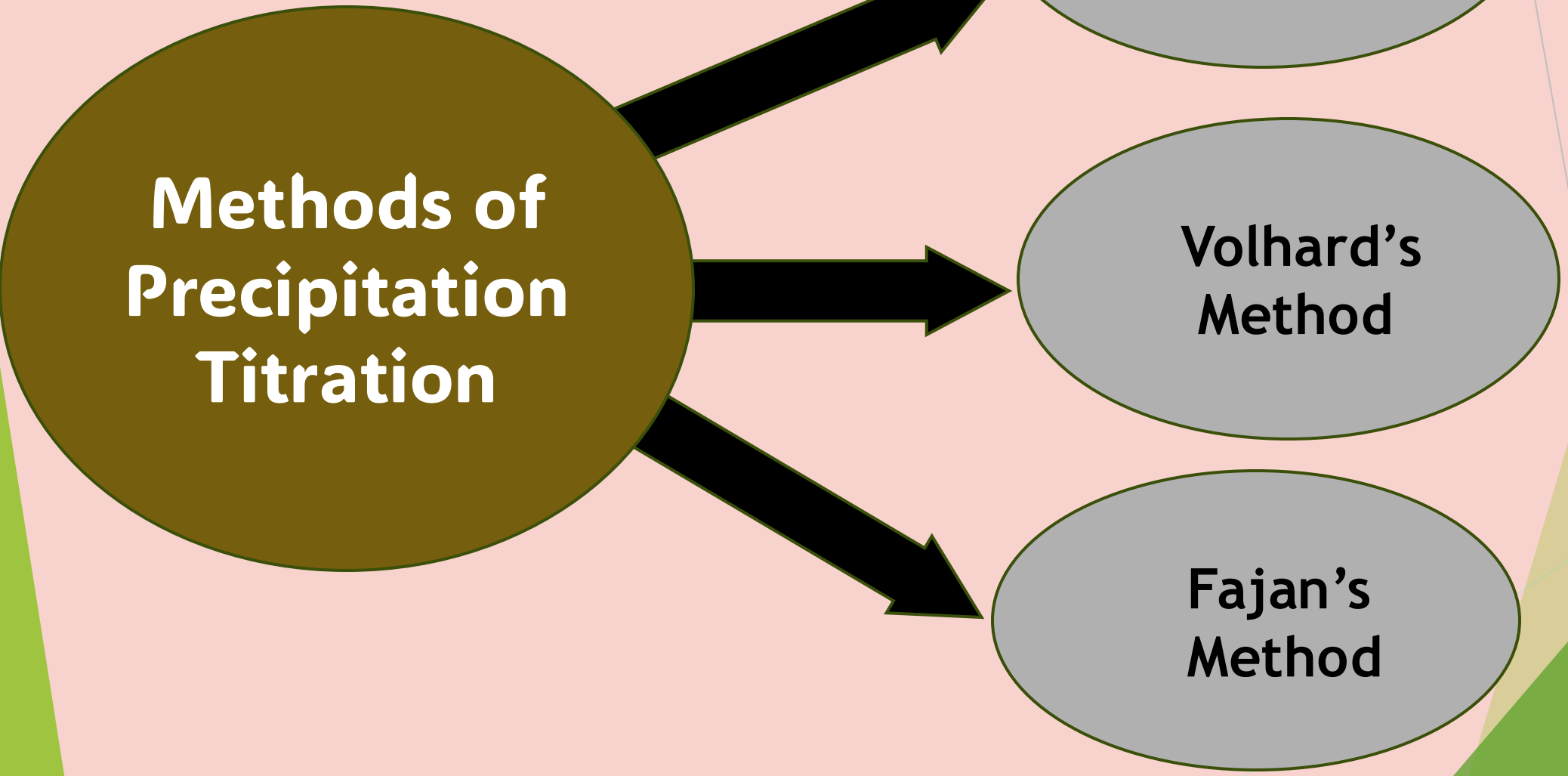
(3) PRECIPITATION TITRATION

- The word precipitation refers to formation of solid mass in a liquid. The solid formed is termed as Precipitate.

- Precipitation titration is a type of titration which involves the formation of Precipitate during titration.

- Since , precipitation titration is mainly used $AgNO_3$ (silver nitrate) as titrant. Hence it is also called "Argentometric Titration" .

$$NaCl + AgNO_3 \longrightarrow AgCl (\downarrow) + NaNO_3$$


Methods of Precipitation Titration
Mohr's Method
Volhard's Method
Fajan's Method

1. Mohr's Method

- The method was given by Sir Karl Friedrich Mohr.
- A precipitation titration in which Silver Nitrate is used as titrant and chromate ions as indicator is called as Mohr's Method.
- The method is mainly used for the determination of chlorides and bromides.

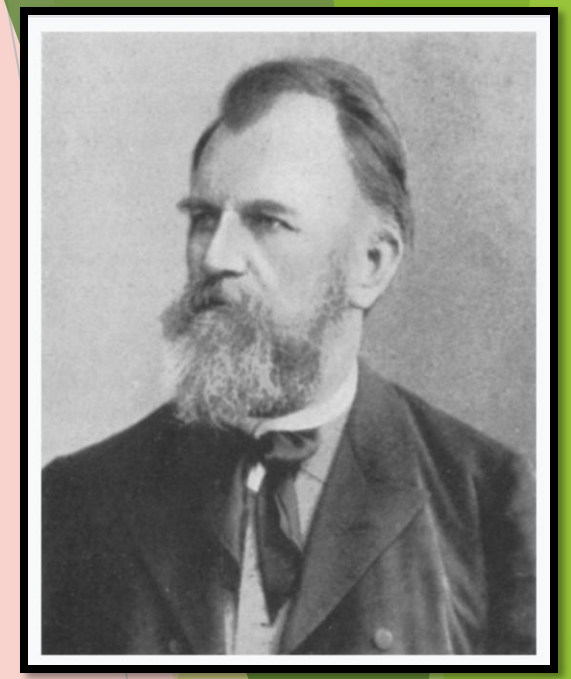
Conditions For Mohr's Method

In Mohr's Method titration must be carried out in a neutral medium neither acidic nor basic.

Because, In Basic Medium :- $Ag^+ + OH^- \longrightarrow AgOH$

In Acidic Medium :- $CrO_4^{2-} + H^+ \longrightarrow H_2CrO_4$

2. Volhard's Method



- The method was given by german scientist Jacob Volhard. This is an indirect method of titration involves back titration.
- Volhard's method is mainly used in the determination of Halides (Cl, Br, I).
- Precipitation titration by volhard's method complete in two steps.

Modified Volhard's Method

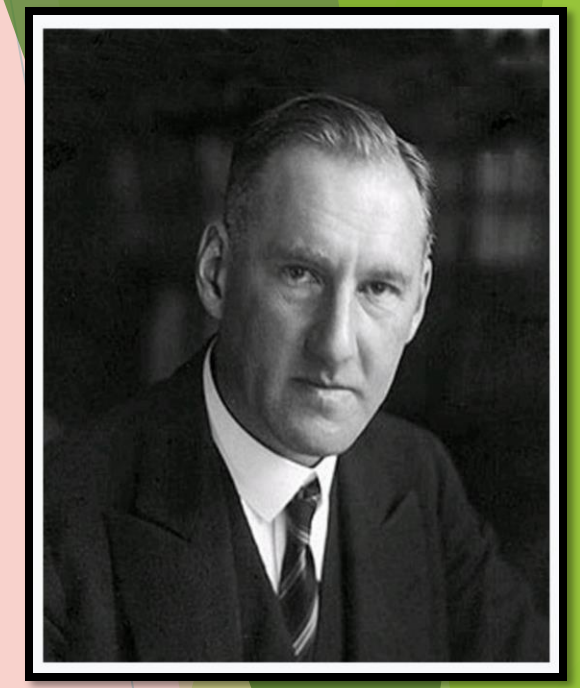
- In modified volhard's method we add some wetting agents like Chloroform, Nitrobenzene etc.

- Since , AgCl is also present in the analyte and it may solubilise which affect our end point, and addition of these wetting agents prevents the solubilisation of AgCl.

Conditions For Volhard's Method :-

The titration must be performed in a acidic medium, so that complex formed [$Fe(SCN)^{2+}$] will be stable .

Fajan's Method



- The method was given by Sir Kazimierz Fajan. The method is based upon the theory of adsorbtion indicators.

- The method is used for the determination of Halides (Cl,Br,I) using $AgNO_3$ as titrant and Fluorescein as indicator.

- At the end point, adsorbtion Indicators gets adsorb on the surface of AgCl and changes the colour of Precipitate.

Indicator For Precipitation Titration

Methods	Indicator	Colour
Mohr's Method	Pot. Chromate (K_2CrO_4)	Reddish ppt
Volhard's Method	Ferric ion (Fe^{3+})	Red colour
Fajan's Method	Fluorescein ($C_{20}H_{12}O_5$)	Pink / red

4) COMPLEXOMETRIC TITRATION

- Complexometric titration is a type of titration which is based on the complex formation between the analyte and titrant

- Complexometric titration are mainly used for the determination of Metal ions in a solution.

- Since EDTA (Ethylene diamene tetra acetic acid) is mainly used as titrant in complexometric titration, hence it is also called “EDTA Titration” .

Analyte : Metal ions
Titrant : Ligands or complexing agents (mainly EDTA)
Indicator : Metal ion indicator

Types of Ligands used in Complexometric Titration

Ligands are classified on the basis of number of donar groups :

Monodentate Ligands	**Donor atom : 1**	**e.g.- NH_3 , H_2O , etc.**
Bidentate Ligands	Donor atom : 2	e.g.- Ethylene diamene
Polydentate Ligands	Donor atom : More than 2	e.g.- EDTA

EDTA

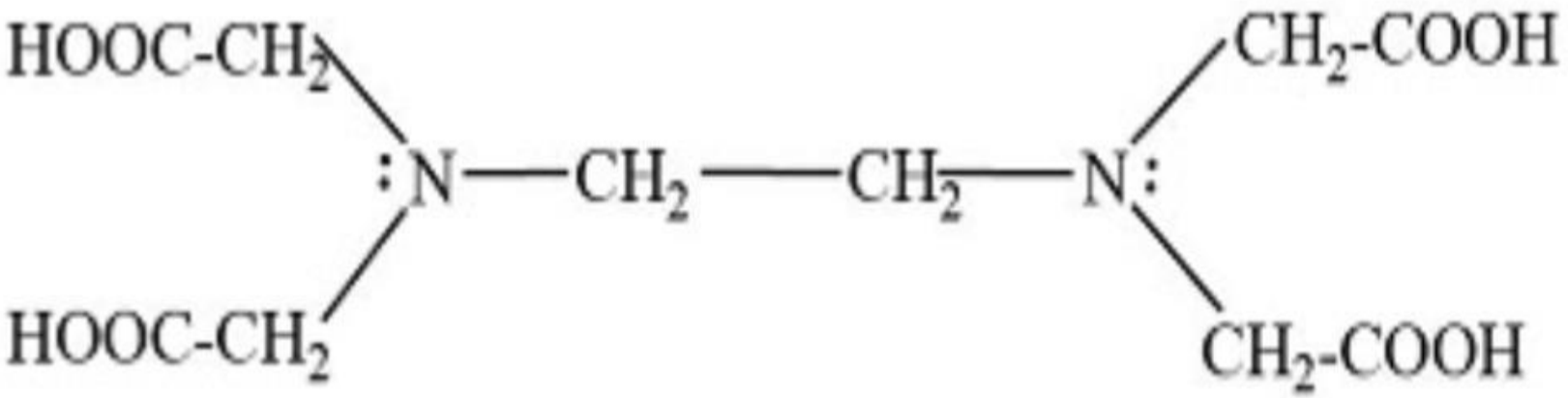
- EDTA stands for Ethylene diamene tetra acetic acid It is a multidentate ligand . It is widely used as titration in Complexometric Titrations .

- Generally it is insoluble in water, hence in Complexometric titration we use its salt Disodium EDTA which is water soluble.

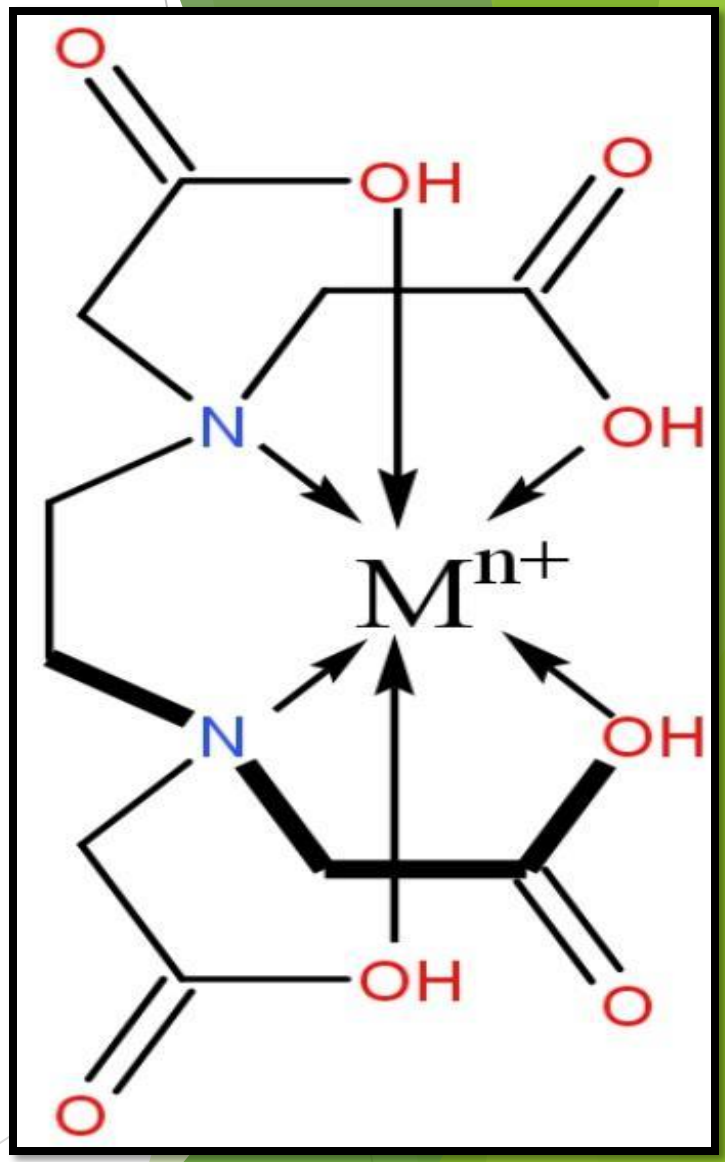
- Chemical Formula : $C_{10}H_{16}N_2O_8$

- Molecular weight : 292.24 g/ mol

Structure of EDTA



structure of EDTA



Classification Of Complexometric Titration

1. **Direct Titration** :- It is the simplest method of Complexometric titration. In this EDTA is mainly used as titrant.

- In this end point is simply determined by colour change due to indicator.

- It is not suitable for slow complexometric titrations.

2. Back Titration :- It is also known as Indirect Titration.

- In this titration is performed by taking excess amount of EDTA.
- In this first we add excess amount of EDTA in the analyte, then remaining amount of EDTA is again titrated by solution of second metal ion.

3. Replacement Titration :- This method is used when direct & indirect titration don't give sharp end point.

- In this titration , metal present in the analyte displaced another metal from metal - EDTA complex .

Indicators for complexometric titration

Indicators Name	Colour
Eriochrome Black T	Red
Catechol Violet	Bluish violet
Xylenol Orange	Red
Calmagite	Wine Red
Pyrocatechol Violet	Blue

Example of Acid-Base Titration Experiment

- **Object :-** To prepare N/10 NaOH in 100 ml .
- **Required chemicals :-** Oxalic acid , NaOH (solid) , phenolphthalein indicator .
- **The substance needed to make the solution**

$$W = \frac{ENV}{1000} = \frac{40 \times 0.1 \times 100}{1000} = 0.4 \text{ gm}$$

Quantity of NaOH = 0.4 gm

E = Equivalent Weight
N = Normality
V = Volume

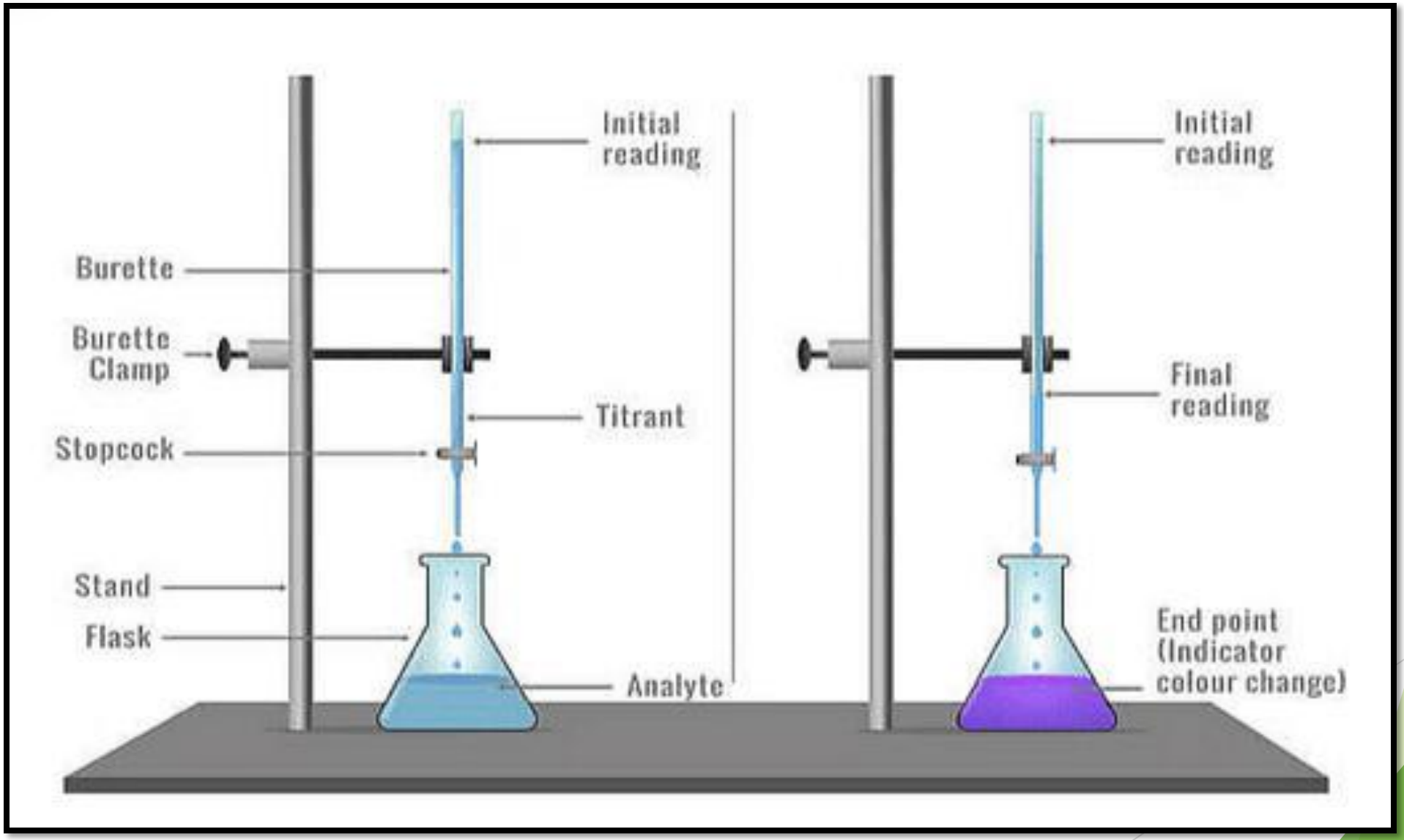
- **To prepare N/10 oxalic acid**

Mass of substance = 0.63gm
Substance dissolve in 100 ml water = 0.63 gm
Substance dissolve in 1000 ml water = 6.3 gm

$$\mathbf{N = \frac{W \times 1000}{E\,V}}$$

$$\text{Normality of oxalic acid} = \frac{0.63 \times 1000}{63 \times 100} = 0.1\text{N}$$

Titration


Initial reading
Burette
Burette Clamp
Titrant
Stopcock
Stand
Flask
Analyte
Initial reading
Final reading
End point (Indicator colour change)

Observation Table

Titration between Oxalic acid & NaOH :-

Volume of Oxalic acid (V_1)	Reading Of Burette		Volume of NaOH (V_2)
	Initial	Final	
10 ml	0.0 ml	10 ml	
10 ml	0.0 ml	10 ml	10 ml
10 ml	0.0 ml	10 ml	

Calculation :-

$$N_1V_1 = N_2V_2$$

(Oxalic acid) (NaOH)

$$N_1 V_1 = N_2V_2$$

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

$$N_2 = \frac{0.1 \times 10}{10} = 0.1N$$

Result :- The Normality of the prepared NaOH solution is 0.1N


Thank You