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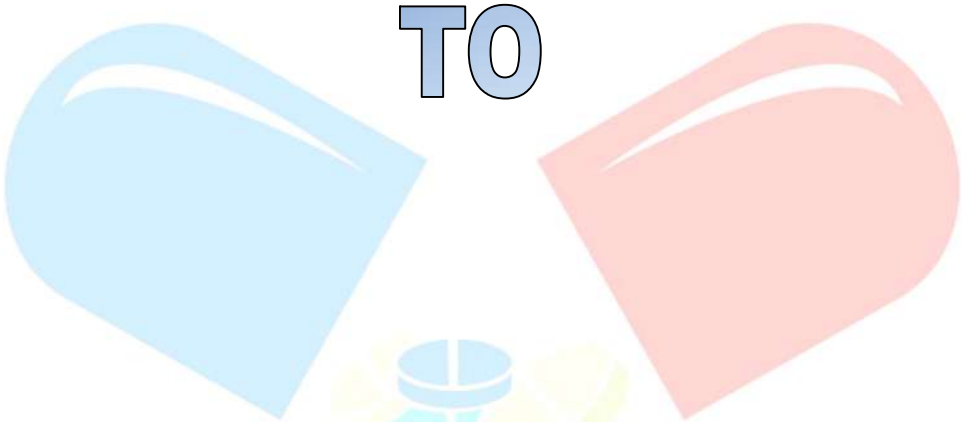

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PHARMACEUTICAL ANALYSIS I

UNIT 3

TOPIC :

- **Complexometric titration :** Classification, metal ion indicators, masking and demasking reagents, estimation of Magnesium sulphate, and calcium gluconate.

Complexometric titration

→ Complexometric titration chelatometry is a form of volumetric analysis in which the formation of a coloured complex is used to indicate the end point of a titration

→ Cl- are particularly useful for the determination of a mixture of different metal ions in solution

Principle

- In complexometric titration the metal ions are titrated with a complexing or a chelating agent
- This method is an analytical application of a complexation reaction
- This method involves transforming simple ion into a complex ions and determing the equivalence point using metal indicatoror electrometrically
- This method is also termed as chilometric titration, chilometry titration, chilometric titration and EDTA titration Ethylene Diamine Titra Acetic Acid

Theory

- Complexometric titration involves the disappearance of the free metal ions as they are changed into complexia
- In any complexation reaction can be used as a volumetric techniques provided
- The reaction reaches equilibrium rapidly after each portion of titrant is added
- A complete titration indicator capable of locating equivalence point with fair accuracy is available

Classification

Complexometric titration following the four types

- **Direct titration :** This method is similar to acid base titration and involve adding the standard solution and chelon solution to the metal ion solution till the end point is attrived

- **Back titration :** In this method excess of standard EDTA solution is added to the metal solution and the excess is back titration with a standard solution of a second metal ion

- **Replacement titration :** In this method the metal to be analysed quantitatively displaces the metal from the Complex.

- **Indirect titration :** This is also known as alkalimetric titration. It is used for determination of anion which do not react with EDTA chelate protons from disodium EDTA are displaced by a heavy metal and titrated with sodium alkali

Application of complexometric titration

- Determination of permanent and temporary hardness of water separately
- Determination of total hardness of water
- Determination of magnesium and silicon dioxide in magnesium trisilicate
- Determination of calcium and lead in a mixture
- Determination of chromium 111 and Iron 111 in mixture kinetic masking
- Determination of manganese in the presence of Ironferromanganese
- Determination of lead and tin in a mixture
- Determination of phosphates

Metal ion indicators

- These are dyes which show one colour in the presence of metal ion and no colour or different colour in absence of metal ion.

Properties

- ✓ Metal ion indicator should be sufficiently stable. It should not dissociate otherwise colour change is not observed.
- ✓ The indicator must be sensitive to metal ion concentration
- ✓ The colour reaction should be specific or atleast selective.
- ✓ All above requirements, must be fulfilled within the pH range at which titration is performed.
- ✓ The colour reaction should be such that before the end point the solution is strongly coloured.

Masking Agents

→ Masking agents are used in complexometric titration (like EDTA titration) to block or hide certain metal ions, so they don't interfere with the titration of the metal you want to measure.

Example:

- If both calcium and magnesium are present in a sample, and you want to measure only magnesium, you can mask calcium using phosphate or oxalate.

Demasking Agents

→ Demasking agents are chemicals that remove or break the bond between a metal ion and its masking agent, so that the masked metal ion becomes free and can now react in the titration (usually with EDTA).

Example:

- You **masked** Fe^{3+} using **fluoride (F^-)**.
- Now, you want to **estimate Fe^{3+}**.
- So you add **boric acid or H^+ ions** to **demask** it (break the complex).
- Now Fe^{3+} is free and can react with EDTA.

Estimation of Magnesium Sulphate ($MgSO_4 \cdot 7H_2O$)

Principle:

- Mg^{2+} reacts with EDTA to form a stable complex.
- Eriochrome Black T is used as the metal ion indicator.
- Endpoint: Color change from wine red to blue.

Procedure:

1. Dissolve accurately weighed **magnesium sulphate** sample in water.
2. Add a few drops of **EBT indicator**.
3. Adjust pH to ~10 using **ammonia-ammonium chloride buffer**.
4. Titrate with **0.01 M EDTA** till the color changes from wine red → blue.

Reaction:

$$\mathbf{Mg^{2+} + EDTA^{4-} \rightarrow Mg\text{-}EDTA^{2-}}$$

Calculation:

Amount of $MgSO_4$ \ cdotp$7H_2O$ = V x N x 246.48 / 1000

- **V = Volume of EDTA used**
- **N** = Normality of EDTA
- **246.48** = Molar mass of $MgSO_4 \cdot 7H_2O$

Estimation of Calcium Gluconate

Principle:

- Calcium ions (Ca^{2+}) in calcium gluconate react with EDTA to form a stable Ca-EDTA complex.
- Murexide is used as the indicator.
- Endpoint: Color change from pink to purple/violet.

Procedure:

1. Dissolve calcium gluconate sample in water.
2. Add a few drops of **murexide indicator**.
3. Adjust pH to around 12 using **NaOH** or buffer.
4. Titrate with **0.01 M EDTA** until the pink color turns violet.

Reaction:

$$\mathbf{Ca^{2+} + EDTA^{4-} \rightarrow Ca\text{-}EDTA^{2-}}$$

Calculation:

Amount of Calcium Gluconate = $V \times N \times 430.37 \; / \; 1000$

- **V = Volume of EDTA used**
- **N** = Normality of EDTA
- **430.37** = Molar mass of calcium gluconate