

SNS COLLEGE OF PHARMACY AND HEALTH SCIENCES

*Affiliated To The Tamil Nadu Dr. MGR Medical University, Chennai Approved by Pharmacy Council of
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Coimbatore -641035

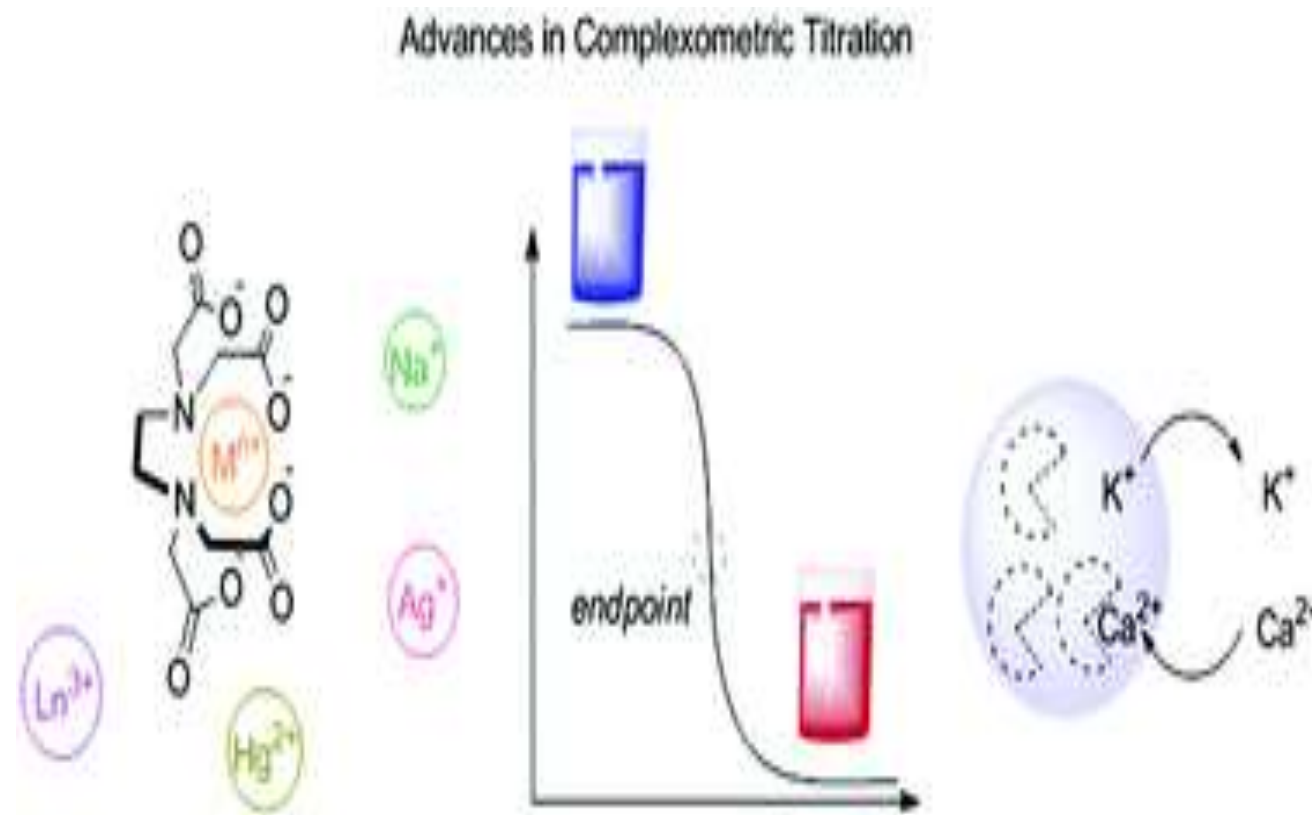
PROGRAM: B.PHARM I YEAR / I SEM.

COURSE NAME : BP102T. PHARMACEUTICAL ANALYSIS -UNIT III.

SUB TOPIC : COMPLEXOMETRIC TITRATION:

Classification, metal ion indicators, masking and demasking reagents, estimation of Magnesium sulphate, and calciumgluconate.

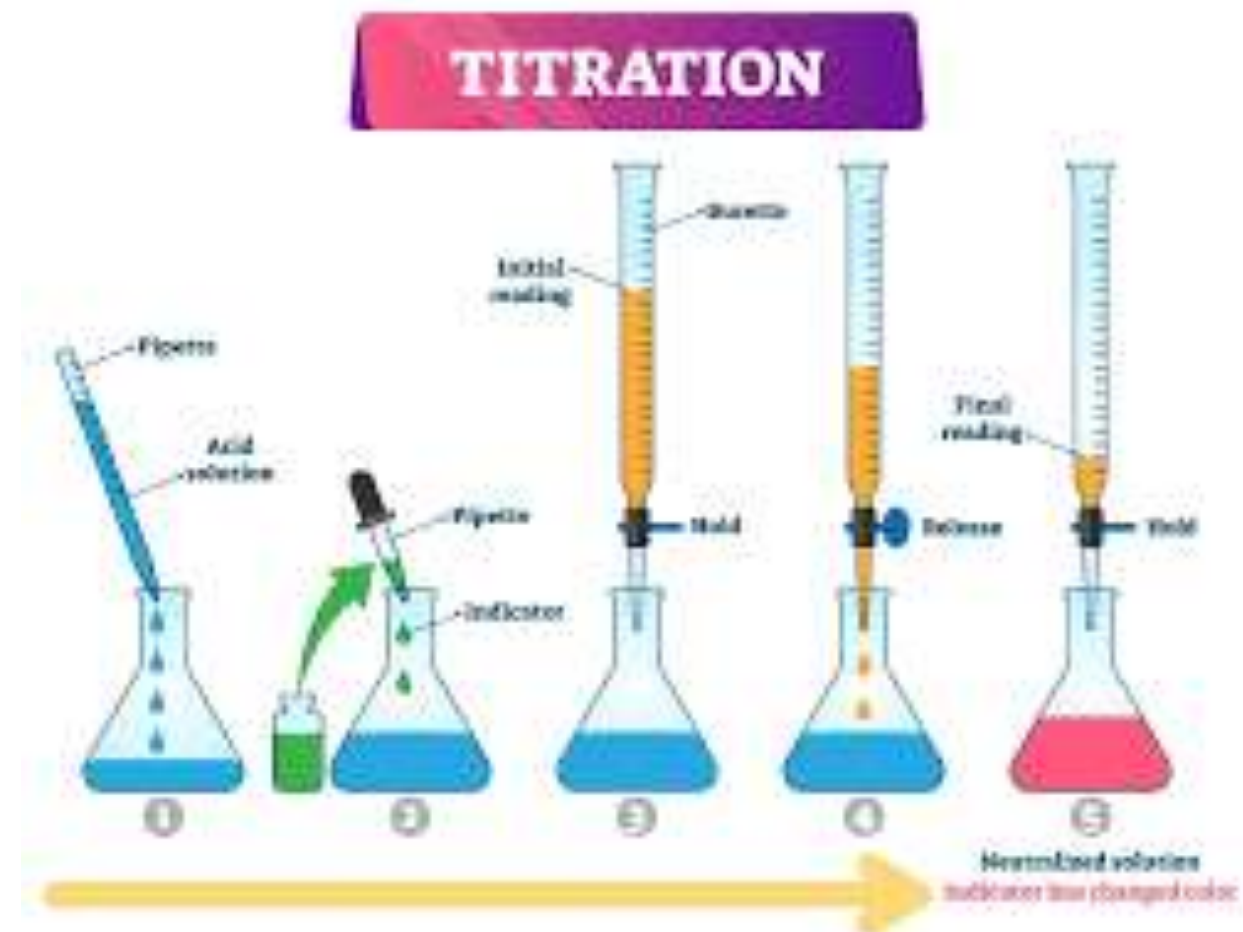
Complexometric titrations: Metal ions are quantitatively determined by forming a stable, soluble complex with a chelating agent.



Complexometric Titration-Classification:



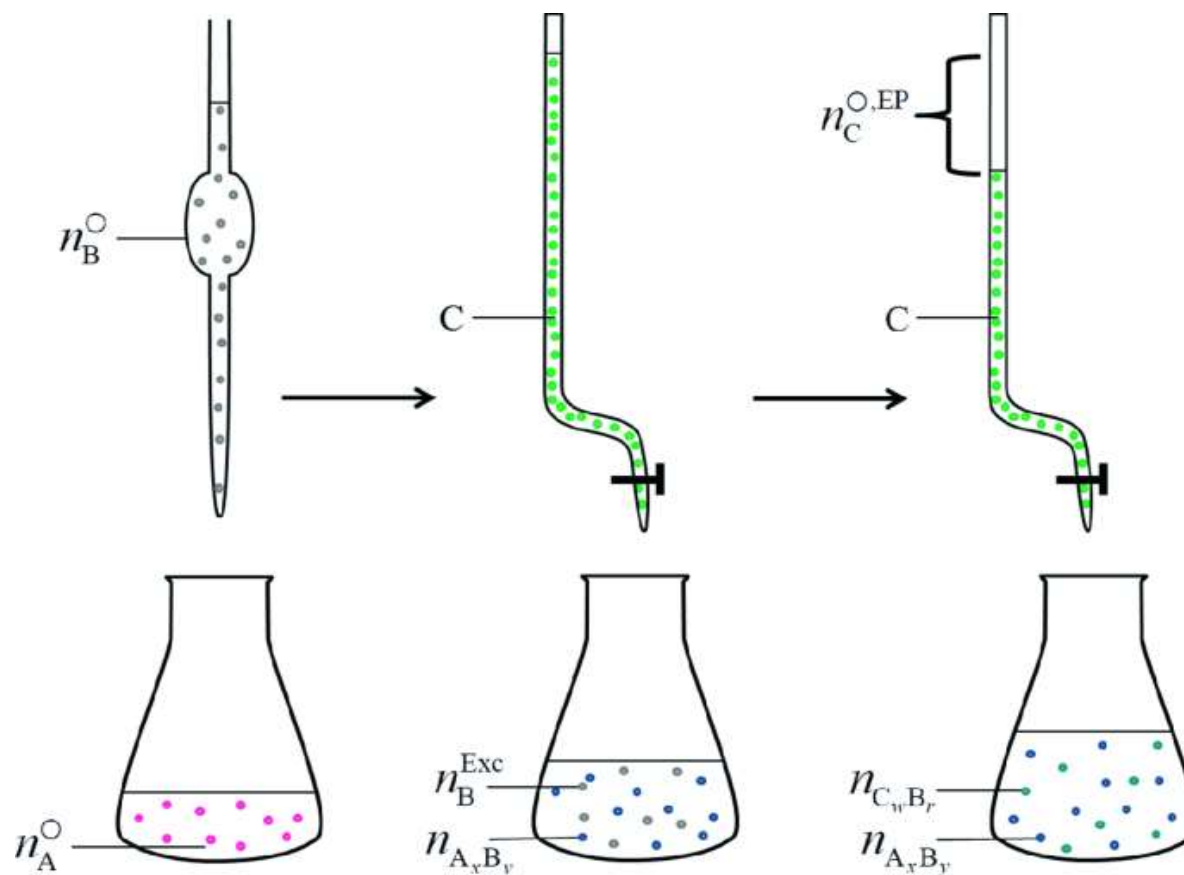
Complexometric Titration-Classification:



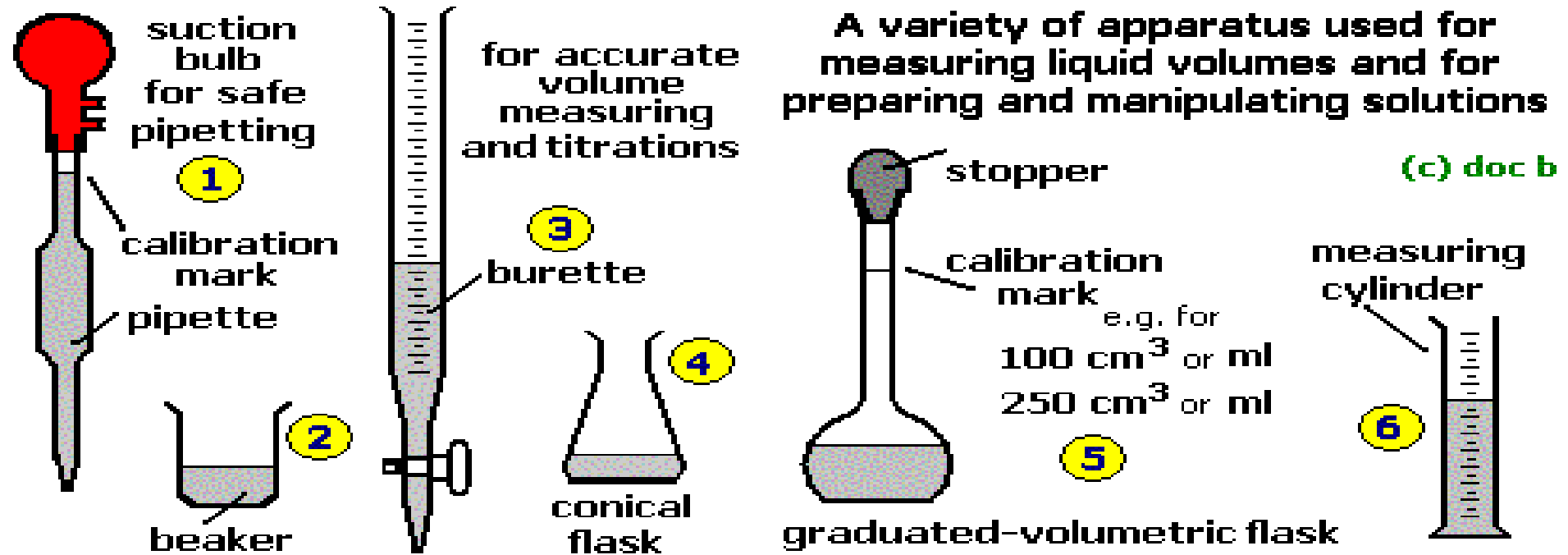
Complexometric Titration-Classification:



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Metal ion Indicators in Complexometric Titration:

List of complexometric indicator

<i>Indicator</i>	<i>Suitable for</i>
Eriochrome Black T or Solochrome Black T (Ethylenediaminetetraacetic acid)	Aluminum, cadmium, zinc, calcium and magnesium, and other rare earth metals
Thymolphthalein complexone	Calcium (Ca), strontium (Sr) and Barium (Ba)
Xylenol orange	Gallium, indium, scandium, and zinc/ cobalt titrations
Hematoxylin	Bismuth, copper and zirconium
Ammonium purpurate or Murexide	Calcium ions
Bromopyrogallol red	Chelometric titrations
Calcein (fluorescent metal indicator)	Calcium detection
Calmagite	Calcium and magnesium
Dithizone	Mercury and lead
Murexide	Calcium copper, nickel, cobalt, and thorium
Patton-Reeder Indicator (Calconcarboxylic acid)	Calcium with magnesium
Curcumin	Boron
Fast Sulphon Black	Copper

Demasking Reagent

“Demasking agent release the masked metal by increasing the backward reaction of masking”.



Zn-CN	Formaldehyde-acetic acid solution (HCHO)
Al-EDTA	Triethanolamine
Pb-EDTA	2,3-dimercaptopropanol

Masking Reagents:

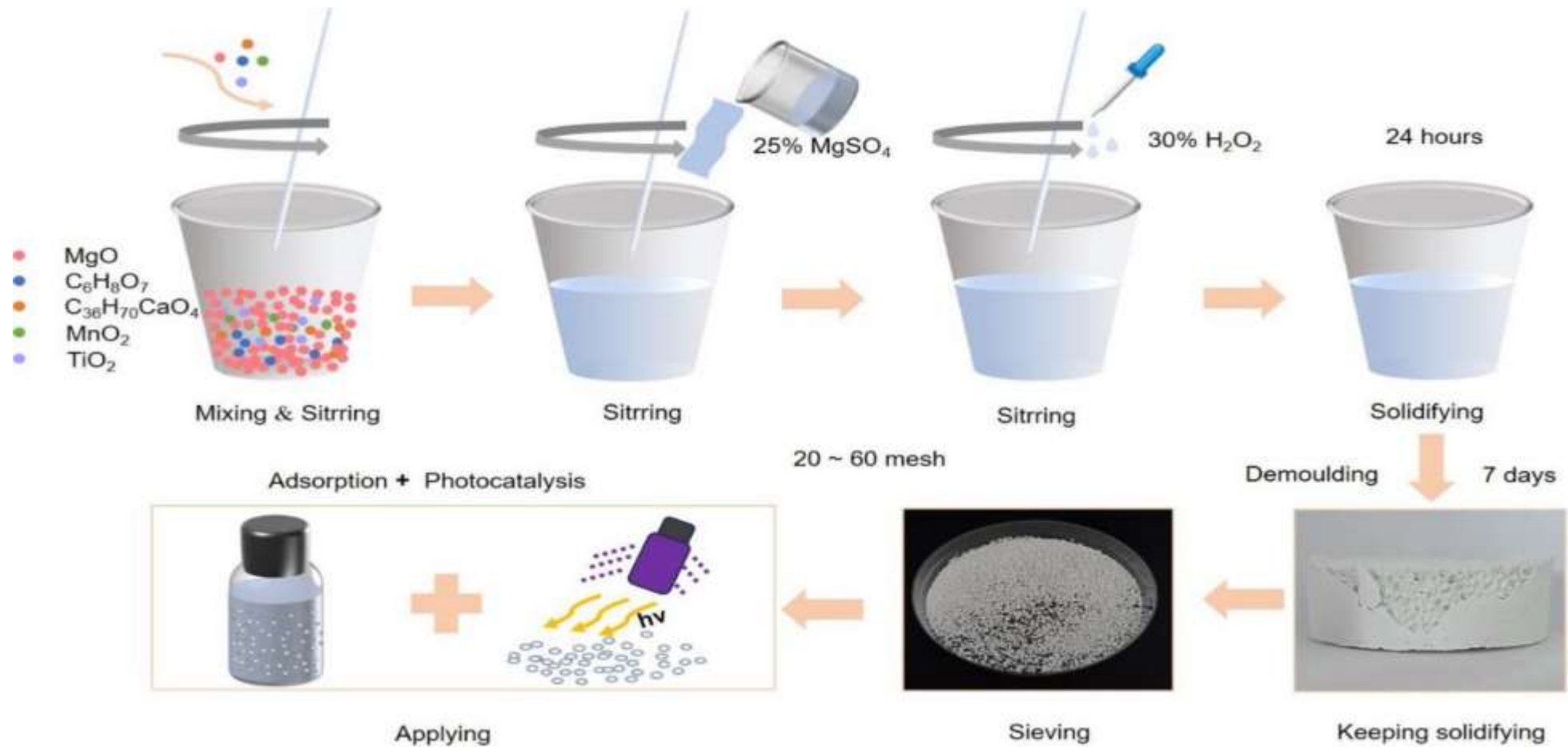
Masking by Precipitation

Many heavy metals can be separated in the form of insoluble precipitates

Precipitating agents	Metals
sulphate ✓	Pb and Ba ✓
oxalate ✓	Ca and Pb ✓
fluoride ✓	Ca, Mg and Pb ✓
Ferrocyanide ✓	Zn and Cu ✓
Sodium sulphide ✓	Co, Cu and Pb ✓
8-hydroxy quinoline	many heavy metals
Thioglycerol	Cu

Mrs. Sara Sirisha Koopa
Assistant Professor

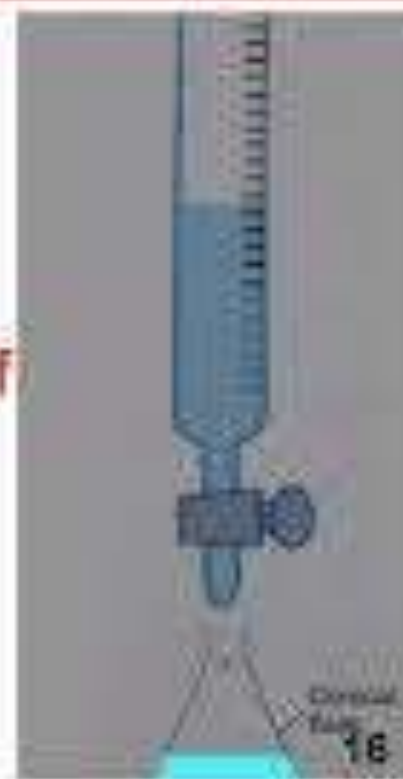
Estimation of Magnesium Sulphate:



Estimation of Magnesium Sulphate:

Titration for estimation of Magnesium Sulphate

- Weigh accurately about 0.3g magnesium sulphate and dissolve in 50 ml Of water.
- Add 10ml of strong ammonia-ammonium chloride solution & 0.1 g of mordant black II mixture as indicator,
- Titrate with Disodium edetate solution, Untill a blue colour is obtained,



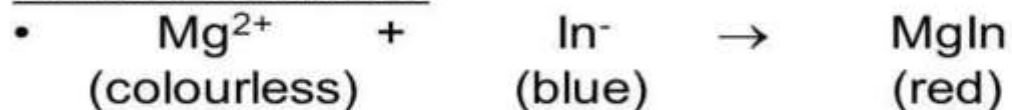
Estimation of Magnesium Sulphate:

Compounds changing colour when binding to metal ion.

K_f for Metal-In⁻ < K_f for Metal-EDTA.



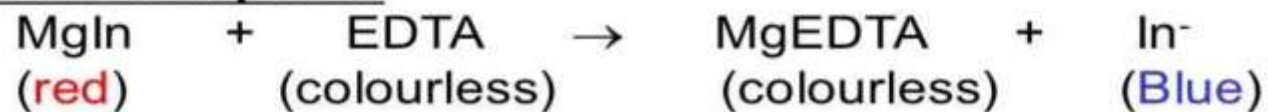
Before Titration:



During Titration: Before the end point



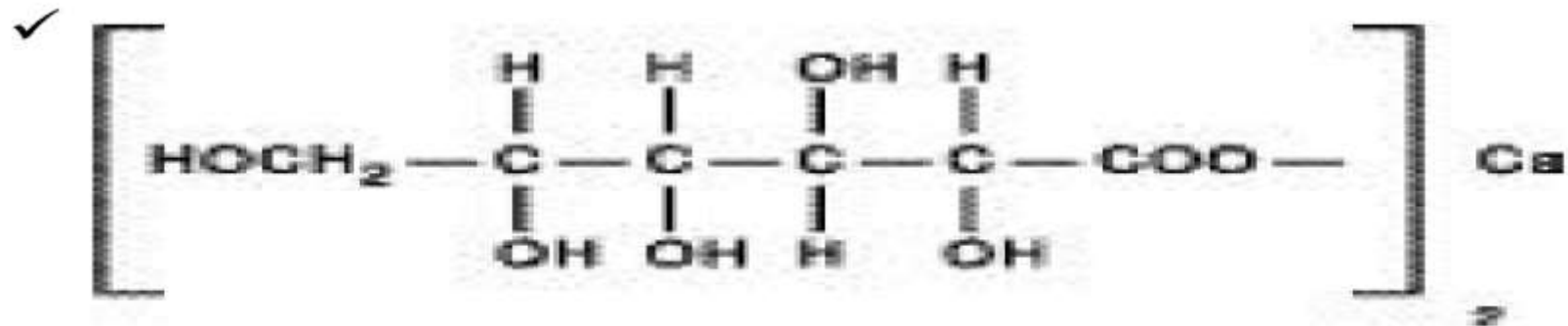
At the end point:



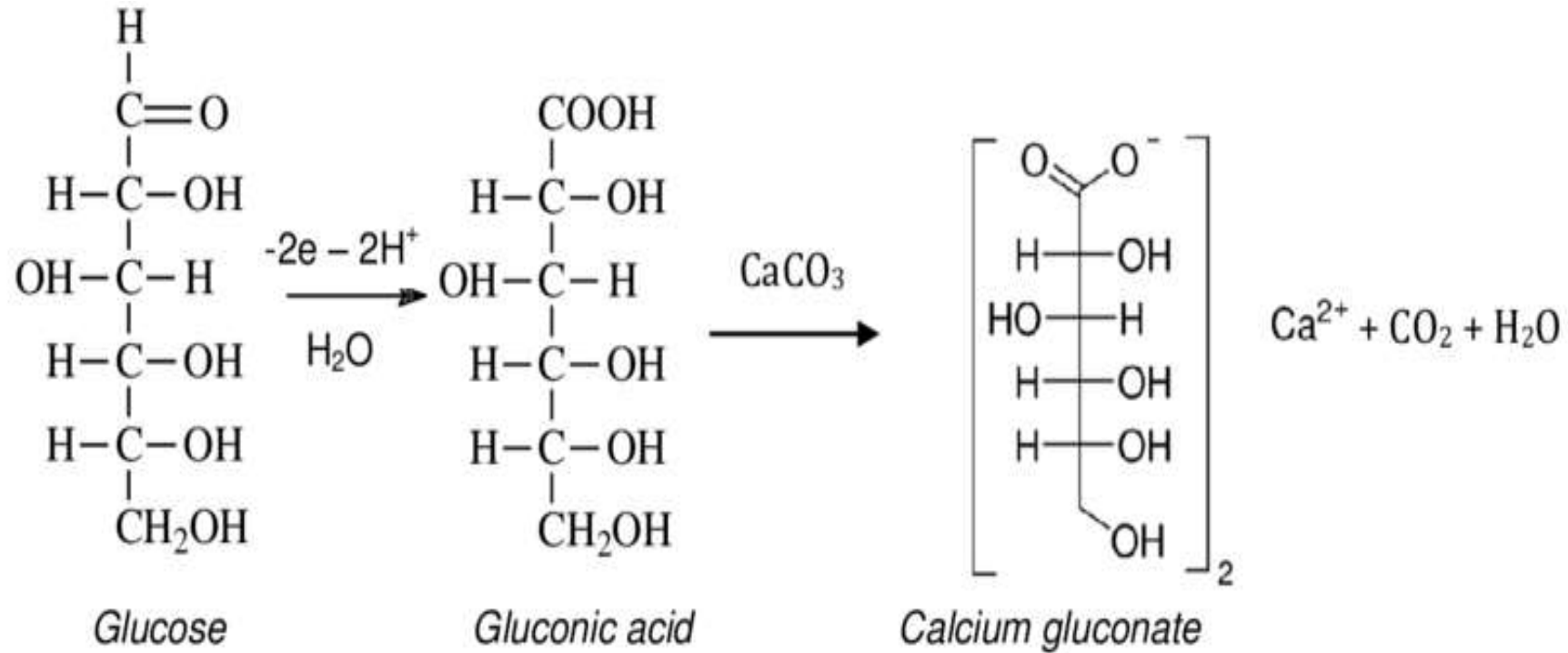
Estimation of Calcium gluconate:

CALCIUM GLUCONATE IP

- ✓ Formula: $\text{Ca}(\text{C}_6\text{H}_{11}\text{O}_7)_2 \cdot \text{H}_2\text{O}$
- ✓ Molecular weight: 448.40 g
- ✓ IP Limit: calcium gluconate contains not less than 99% not more than 103% of calcium gluconate



Estimation of Calciumgluconate:



Estimation of Calcium gluconate:

Calculations:

1 ml of 0.05 M EDTA = 0.02242 g of Calcium gluconate

15.3 ml of 0.0711 M EDTA = W g of Calcium gluconate

Cross multiplication

$$1 \times 0.05 \times W = 15.3 \times 0.0711 \times 0.02242$$

$$W \text{ g} = \frac{15.3 \times 0.0711 \times 0.02242}{0.05} = \frac{0.02438}{0.05} = 0.4876 \text{ g}$$

Estimation of Magnesium Sulfate

1. Principle

- ❑ Magnesium ions (Mg^{2+}) react with EDTA (ethylenediaminetetraacetic acid) in a 1:1 ratio to form a stable complex. At pH 10, Eriochrome Black T forms a wine-red complex with Mg^{2+} .
- ❑ As EDTA is added, it binds with Mg^{2+} , releasing the indicator, which changes color to blue at the endpoint.

2. Procedure

Standardization of EDTA solution- Follow the same procedure as given earlier by using 0.01M $CaCl_2$ solution.

Assay Procedure

- ❑ Pipette 25.0 mL of $MgSO_4$ solution into a conical flask. Add 5 mL of buffer solution (pH 10). Add 2–3 drops of Eriochrome Black T; the solution turns wine-red.
- ❑ Titrate with 0.01 M EDTA from a burette until the color changes from wine-red to clear blue.
- ❑ Record the volume of EDTA used. Repeat the titration to get concordant readings.

$$\text{The Percent purity of } MgSO_4 = \frac{\text{Vol. of EDTA} \times \text{I.P. factor} \times 100 \times \text{M of EDTA (Actual)}}{\text{Wt. of } MgSO_4 \text{ in grams} \times \text{M of EDTA (Expected)}}$$

1. Complexometric titrations are based on reaction between metal ions.
A. Acids B. Oxidizing agents C. Chelating agents D. Precipitants.
2. Most commonly used chelating agent is.
A. Oxalic acid B. Dimercaprol C. EDTA D. Sodium citrate.
3. Which is NOT a type of Complexometric titration?
A. Direct B. Back C. Replacement D. Redox.
4. Metal ion indicators form.
A. Insoluble precipitates B. Covalent bonds.
C. Weak Colored complexes D. Irreversible complexes.
5. Eriochrome Black T is used for estimation of.
A. Sodium B. Calcium & Magnesium C. Zinc D. Iron

Complexometric Titration – MCQ Assessment

6. Masking agents are used to.

A. Increase rate B. Remove indicator C. Prevent interference D. Change pH.

7. A masking agent for calcium ions is.

A. EDTA B. Sodium fluoride C. Ammonium chloride D. EBT

8. Demasking agents are used to.

A. Stabilize complexes B. Remove EDTA C. Release metal ions D. Change color.

9. End point in MgSO_4 estimation shows color change.

A. Red to blue B. Blue to red C. Wine red to blue D. Yellow to colorless.

10. Calcium gluconate is estimated using.

A. KMnO_4 B. EDTA C. HCl D. AgNO_3 .

Recommended Books:

1. A.H. Beckett & J.B. Stenlake's, Practical Pharmaceutical Chemistry Vol I & II, Stahlone, Press of University of London.
2. A.I. Vogel, Text Book of Quantitative Inorganic analysis.
3. P. Gundu Rao, Inorganic Pharmaceutical Chemistry.
4. Bentley and Driver's Textbook of Pharmaceutical Chemistry.
5. John H. Kennedy, Analytical chemistry principles.

