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Diploma in Pharmacy 1st Year

Pharmaceutical Chemistry

Experiment

To perform the assay of calcium gluconate by complexometric titration.

Aim:

To perform the assay of calcium gluconate by complexometric titration.

Reference :

‘ Dr. Gupta G.D. , Dr. Sharma Shailish , Kaur Baljeet ’ “Practical Manual of Pharmaceutical Chemistry” Published by Nirali Prakashan, Page no 37 - 40

Apparatus and Material Required :

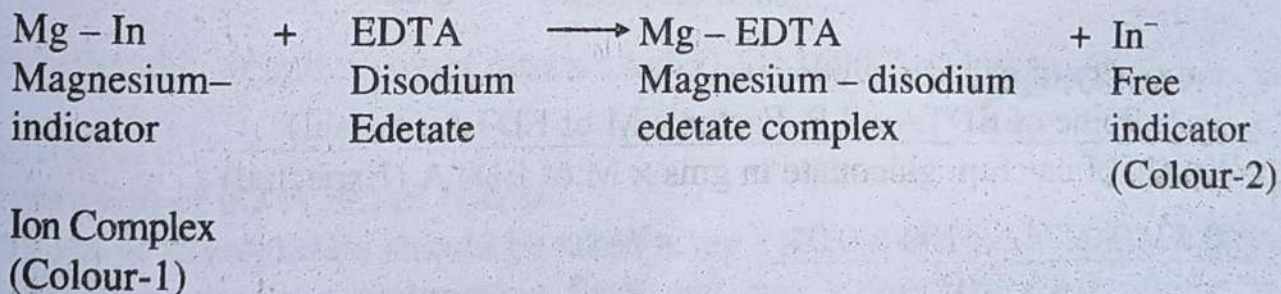
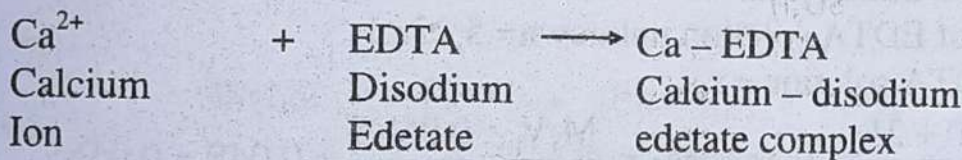
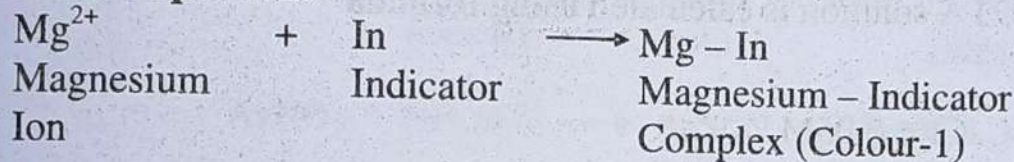
Burette, burette stand, conical flask, volumetric pipette, beaker, volumetric flask, funnel, glass rod, wash bottle, digital/analytical balance, ultrasonicator, calcium gluconate ($C_{12}H_{22}CaO_{14}$) disodium edetate (EDTA), ammonia (NH_3), calcium chloride ($CaCl_2$) ammonium chloride (NH_4Cl), magnesium sulphate ($MgSO_4$), hydrochloric acid (HCL), and solochrome black-T indicator or mordant black II.

Theory:

- The assay of calcium gluconate is based upon a replacement Complexometric titration. Magnesium forms a compound with a mordant black II indicator combination, indicating the first colour. Due to the fact that the magnesium-indicator complex is more stable than the calcium-indicator complex, calcium has no effect on it. Calcium and EDTA were produced when titrated against disodium edetate
- The drop of EDTA breaks the magnesium-indicator combination, allowing the free Indicator to establish a complex with the magnesium after all of the calcium has been ingested. Detecting the second colour at that time determines the endpoint.
- The reaction that is involved in this titration is as follows:

Chemical Equations

Chemical Equations



Procedure:

Procedure Preparation of 0.05M EDTA Solution

14.6gm of EDTA should be dissolved in 1000ml distilled water.

For Standardisation of EDTA Solution

- 1) 10 ml of 0.05M CaCl₂ solution should be pipette out into a conical flask.
- 2) 5ml of pH buffer solution should be added.\
- 3) It should be titrated against EDTA.
- 4) The titration should be repeated for the concordant values.
- 5) The values should be noted in a tabular form.

For Assay

- 1) 800g of calcium gluconate should be accurately weighed and dissolved in 150 ml of H₂O containing 5 ml of dil. HCl.
- 2) 5ml of M/20 MgSO₄ solution and 10 ml of pH 10 buffer solution should be added.
- 3) The contents of the flask should be titrated against M/20 EDTA solution using SBI indicator.
- 4) A blank titration should be performed and this value should be subtracted from sample titration.

The titration is performed with two more samples, and the percentage of calcium in the drug is calculated as follows:

Each 1cm³ of 0.05M EDTA=0.02242gm of calcium gluconate

Or. Each 1cm³ of 0.025M EDTA = 0.04484gm of calcium gluconate

Observation Table

S. No.	PVATC. of Calcium Gluconate (gms)	Burette Reading (ml)		Volume of EDTA Solution Rundown (ml)
		Initial	Final	
1	0.8	0	20	20
2	0.8	20	21	21

Calculation

The molarity of EDTA solution is calculated using formula

$$M_1V_1 = M_2V_2$$

Where,

$$M_1 = M \text{ of } CaCl_2 = 0.05M$$

$$V_1 = \text{volume of } CaCl_2 = 5ml$$

$$V_2 = \text{volume of EDTA solution unknown} = 5.25ml$$

$$M_2 = M \text{ of EDTA solution} = x$$

$$\text{Average reading} = \frac{20 \times 21}{2} = 20.5 = M_2 = \frac{M_1 V_1}{V_2} = \frac{0.05 \times 5}{5.25} = 0.048 = 0.05$$

$$\text{Per cent purity of calcium gluconate} = \frac{\text{Volume of EDTA} \times \text{IP. Factor M of EDTA (Actual)}}{\text{Weight of calcium gluconate in gms} \times \text{M of EDTA (Expected)}}$$

$$= \frac{20.5 \times 0.02242 \times 100 \times 0.04}{0.8 \times 0.05}$$

$$= \frac{1.838}{0.04} = 45.95\%$$

Result: The percentage purity of calcium gluconate was found to be 45.95%.

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