

Assay of Calcium Gluconate by Complexometric Titration

Experiment 10 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To standardise the stated titrant and calculate the content of the named compound from a prescribed titration.

Principle

EDTA complexes calcium in a 1:1 mole ratio under the prescribed conditions. The endpoint indicator responds to the availability of calcium at the selected pH. Calcium gluconate monohydrate and anhydrous calcium gluconate have different formula masses.

Reaction and Calculation Basis

- $\text{Ca}^{2+} + \text{EDTA} \rightarrow \text{calcium-EDTA complex}$; mole ratio 1:1.
- Monohydrate formula mass $\approx 448.39 \text{ g/mol}$. Mass in mg = $V \times C \times 448.39$.

Requirements

- Prescribed assay method, balance, burette, pipette, volumetric glassware and labelled test portion.
- Titrant: Approximately 0.05 mol/L EDTA.
- The primary standard or certified standard, prescribed medium and endpoint system named below.

Standardisation of the Titrant

1. Prepare calcium standard

Use a certified calcium standard or the prescribed calcium carbonate primary standard. For carbonate, dissolve using the specified acid treatment and handle carbon dioxide as directed.

2. Set conditions

Adjust to the specified complexometric pH and add the prescribed calcium endpoint indicator.

3. Titrate and calculate

Titrate with EDTA to the stated endpoint. $\text{CEDTA} = \text{moles calcium} \div \text{corrected EDTA volume in litres}$; obtain the required concordance.

Procedure

1. Confirm hydrate

Record the test-material formula and assay basis. Weigh and dissolve by the prescribed method.

2. Set pH

Establish the stated calcium-complexation conditions and add the specified indicator, such as the institution's prescribed murexide system.

3. Titrate

Use standardised EDTA and mix consistently. Recognise the defined indicator endpoint rather than an arbitrary faint colour.

Pharmaceutical Chemistry**4. Calculate**

Use one mole EDTA per mole calcium gluconate and the formula mass for the stated hydrate. Apply any dilution or blank correction.

Observations and Record

Record	Standardisation	Assay
Standard / test mass	___ g	___ g
Initial / final burette	___ / ___ mL	___ / ___ mL
Blank / corrected titre	___ / ___ mL	___ / ___ mL
Concentration / result	___ mol/L	___% on stated basis

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- Standardisation example: 0.10009 g pure CaCO_3 , 100.09 g/mol, gives 1.000 mmol calcium. A 20.00 mL EDTA titre gives 0.05000 mol/L.
- For 0.5000 g calcium gluconate monohydrate, corrected titre 22.30 mL at 0.05000 mol/L gives $22.30 \times 0.05000 \times 448.39 = 499.95$ mg.
- Assay $\approx 99.99\%$ on the monohydrate basis. Anhydrous material uses approximately 430.37 g/mol instead.

Result

Titrant concentration = ___ mol/L. Corrected titre = ___ mL. Calculated content = ___% on the ___ basis; prescribed criterion comparison: ___.

Precautions

- Use the prescribed pH and calcium-selective endpoint system.
- Other metal ions can interfere; the method's preparation and controls matter.
- Do not use the monohydrate factor for anhydrous material.
- Use the actual standardised concentration, correct for the prescribed blank and account for any aliquot or dilution. The monograph defines acceptance limits and the as-is, dried or hydrated basis.

Viva Questions and Answers**1. What is the calcium:EDTA ratio?**

1:1 in the stated complexometric calculation.

2. Why control pH?

Complex stability and indicator behaviour depend on it.

3. What is the 0.05 mol/L monohydrate factor?

Approximately 22.4195 mg per mL.

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