

Assay of Ferrous Sulphate by Redox Titration

Experiment 9 - 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

Cerium(IV) oxidises Fe(II) to Fe(III) in the prescribed acidic medium. One mole of Ce(IV) accepts one electron from one mole of Fe(II). The hydrate formula and oxidation state of the test material must match the calculation.

Reaction and Calculation Basis

- $\text{Fe}^{2+} + \text{Ce}^{4+} \rightarrow \text{Fe}^{3+} + \text{Ce}^{3+}$.
- On the $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ basis: mass in mg = $V(\text{mL}) \times C(\text{mol/L}) \times 278.01$.

Requirements

- Prescribed assay method, balance, burette, pipette, volumetric glassware and labelled test portion.
- Titrant: Approximately 0.1 mol/L cerium(IV) titrant.
- The primary standard or certified standard, prescribed medium and endpoint system named below.

Standardisation of the Titrant

1. Choose the standard

Use a certified Fe(II) standard or prescribed ferrous ammonium sulphate standard on its stated purity and hydrate basis.

2. Prepare

Prepare the standard in the specified acid medium, limiting oxidation during preparation.

3. Titrate and calculate

Titrate using the prescribed redox indicator or potentiometric endpoint. $C_{\text{Ce(IV)}} = \text{moles Fe(II)} \div \text{corrected titrant volume in litres}$. Obtain the required concordant results.

Procedure

1. Identify the material

Record whether the test is $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ or another stated basis. Check appearance and oxidation concerns.

2. Dissolve promptly

Accurately weigh and dissolve in the prescribed acidic medium. Avoid unnecessary delay and exposure before titration.

3. Titrate

Use standardised Ce(IV) and the specified endpoint system. Record titre and any required blank.

Pharmaceutical Chemistry**4. Calculate**

For heptahydrate use 278.01 g/mol and the 1:1 mole ratio. Apply any monograph-specific basis 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.

- Illustrative standard: 0.39214 g pure ferrous ammonium sulphate hexahydrate, approximately 392.14 g/mol, supplies 1.000 mmol Fe(II). A 10.00 mL titre gives 0.1000 mol/L Ce(IV).
- For 0.5000 g FeSO₄·7H₂O, corrected titre 18.00 mL at 0.1000 mol/L gives $18.00 \times 0.1000 \times 278.01 = 500.42$ mg.
- Assay = $500.42/500.0 \times 100 \approx 100.1\%$ on the heptahydrate basis.

Result

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

Precautions

- Fe(II) oxidation before titration can lower the measured ferrous content.
- Use the prescribed acidic medium and endpoint; do not choose an indicator solely by colour preference.
- Do not use anhydrous FeSO₄ mass in a heptahydrate calculation.
- 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. Why is the mole ratio 1:1?**

Both changes involve one electron per reacting ion.

2. What oxidation change occurs?

Fe(II) becomes Fe(III), while Ce(IV) becomes Ce(III).

3. What is 1 mL of 0.1 mol/L equivalent to?

Approximately 27.801 mg FeSO₄·7H₂O.

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