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Copper Sulphate by Iodometry

Experiment 15 - 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

Cu(II) reacts with iodide to precipitate CuI and release iodine. The iodine is titrated with sodium thiosulphate. Combining the reaction ratios gives one mole of thiosulphate per mole of copper. Starch is added near the endpoint rather than into a strongly iodine-rich mixture.

Reaction and Calculation Basis

- $2 \text{Cu}^{2+} + 4 \text{I}^- \rightarrow 2 \text{CuI} + \text{I}_2$.
- $\text{I}_2 + 2 \text{S}_2\text{O}_3^{2-} \rightarrow 2 \text{I}^- + \text{S}_4\text{O}_6^{2-}$.
- For $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, mass in mg = $V \times C \times 249.68$.

Requirements

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

Standardisation of the Titrant

1. Use an iodate standard

Accurately weigh certified potassium iodate on its stated purity basis and dissolve as prescribed.

2. Liberate iodine

Add the specified excess iodide and acid in the approved iodometric flask. Carry out the prescribed blank and protect the reaction from unnecessary light and delay.

3. Titrate

Titrate liberated iodine with thiosulphate; add starch near the endpoint. One mole iodate consumes six moles thiosulphate. $C_{\text{thio}} = 6 \times \text{moles iodate} \div \text{corrected volume in litres}$.

Procedure

1. Prepare the test

Accurately weigh $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and prepare in the monograph's medium. Control acidity and interferences as prescribed.

2. Release iodine

Add the stated excess iodide, mix and observe iodine colour with CuI precipitate.

3. Titrate promptly

Titrate with standardised thiosulphate until the prescribed near-endpoint stage, then add starch.

4. Complete the endpoint

Continue to disappearance of the iodine-starch colour. If the prescribed method includes a near-endpoint thiocyanate step to release adsorbed iodine, apply it only at that stage.

Pharmaceutical Inorganic and Analytical Chemistry**5. Calculate**

Use corrected titre and the pentahydrate molecular mass. Distinguish a redox endpoint from simple disappearance of precipitate.

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.

- Standard example: 0.07133 g KIO₃, approximately 214.00 g/mol, gives $6 \times 0.07133/214.00$ mol thiosulphate equivalents. A 20.00 mL titre gives approximately 0.1000 mol/L.
- A 0.5000 g CuSO₄·5H₂O portion with corrected titre 20.00 mL at 0.1000 mol/L gives $20.00 \times 0.1000 \times 249.68 = 499.36$ mg.
- Assay = $499.36/500.0 \times 100 \approx 99.87\%$.

Result

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

Precautions

- Prevent iodine loss by unnecessary delay or open-vessel exposure.
- Add starch at the prescribed near-endpoint stage.
- Follow the specified acidity and handling of iodine adsorbed by CuI.
- 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 iodometry indirect here?**

The copper reaction releases iodine, and the iodine is what is titrated.

2. Why is the copper:thiosulphate ratio 1:1?

Two copper ions release one iodine molecule, which consumes two thiosulphate ions.

3. What is the pentahydrate factor at 0.1 mol/L?

Approximately 24.968 mg CuSO₄·5H₂O per mL.

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