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Sodium Chloride by Modified Volhard Method

Experiment 19 - 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

A known excess of silver nitrate precipitates chloride as AgCl. The remaining silver is back-titrated with thiocyanate in the prescribed acidic medium. Once silver is consumed, excess thiocyanate gives a ferric-thiocyanate colour. The modification prevents AgCl from interfering with the back-titration.

Reaction and Calculation Basis

- $\text{Ag}^+ + \text{Cl}^- \rightarrow \text{AgCl}$; $\text{Ag}^+ + \text{SCN}^- \rightarrow \text{AgSCN}$.
- If a matched blank is valid, $\text{NaCl mass in mg} = (V_{\text{blank}} - V_{\text{test}}) \times C_{\text{SCN}} \times 58.44$.
- General direct calculation: $\text{NaCl mass in mg} = (C_{\text{Ag}}V_{\text{Ag}} - C_{\text{SCN}}V_{\text{SCN}}) \times 58.44$, for concentrations in mol/L and volumes in mL.

Requirements

- Prescribed assay method, balance, burette, pipette, volumetric glassware and labelled test portion.
- Titrant: Standard silver nitrate and approximately 0.1 mol/L ammonium thiocyanate.
- The primary standard or certified standard, prescribed medium and endpoint system named below.

Standardisation of the Titrant

1. Standardise silver nitrate

Use certified sodium chloride on its specified preparation basis. Titrate the known chloride amount with AgNO₃ by the laboratory's validated endpoint, such as potentiometric detection. $C_{\text{Ag}} = \text{moles NaCl} \div \text{corrected volume in litres}$.

2. Standardise thiocyanate

Take a measured standard AgNO₃ aliquot, establish the prescribed acidic conditions and ferric indicator, then titrate with thiocyanate to the defined endpoint.

3. Calculate

The Ag:SCN ratio is 1:1. $C_{\text{SCN}} = C_{\text{Ag}} \times V_{\text{Ag}} \div \text{corrected } V_{\text{SCN}}$ when the volumes share the same units. Obtain the required concordant results.

Pharmaceutical Inorganic and Analytical Chemistry**Procedure****1. Prepare the test**

Accurately weigh and dissolve NaCl. Add the prescribed nitric-acid medium and a precisely measured excess of standard AgNO₃.

2. Manage the precipitate

Apply the laboratory's approved modified-Volhard strategy to prevent AgCl consuming thiocyanate. Where the validated teaching route separates AgCl, filter quantitatively and retain the prescribed filtrate and washings.

3. Back-titrate

Add the prescribed ferric indicator and titrate excess silver with standard thiocyanate to the persistent defined reddish complex colour.

4. Run the blank

Use the same silver addition and relevant reagent conditions without chloride, following the method's blank procedure.

5. Calculate

Use either silver added minus residual silver, or the correctly matched blank-test titre difference. Keep both titrant concentrations when they differ.

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.

- AgNO₃ standardisation: 0.14610 g pure NaCl / 58.44 = 2.500 mmol; a 25.00 mL titre gives 0.1000 mol/L AgNO₃.
- If 10.00 mL of 0.1000 mol/L AgNO₃ consumes 10.00 mL thiocyanate, CSCN = 0.1000 mol/L.
- Matched-blank example: V_{blank} = 25.00 mL and V_{test} = 8.00 mL at CSCN = 0.1000 mol/L. NaCl found = (25.00 - 8.00) × 0.1000 × 58.44 = 99.35 mg.
- For a 0.1000 g test portion, assay = 99.35%. Direct check: C_{AgVAg} - CSCNV_{test} = 2.50 - 0.80 = 1.70 mmol chloride.

Pharmaceutical Inorganic and Analytical Chemistry

Result

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

Precautions

- Follow the approved precipitate-protection or separation method; a casual endpoint reading in unprotected AgCl can be biased.
- Do not introduce an unapproved organic coating reagent.
- Keep silver-containing waste in the designated collection system.
- 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 this a back-titration?

Chloride consumes part of a known silver excess, and the remaining silver is measured.

2. Why remove or protect AgCl?

AgCl can react with thiocyanate and cause extra titrant consumption.

3. What forms the endpoint colour?

A ferric-thiocyanate complex after the available silver has been consumed.

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