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Ammonium Chloride by Acid-base Titration

Experiment 13 - 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

In a formol teaching assay, formaldehyde converts ammonium species to hexamethylenetetramine and releases an equivalent amount of acid, which is titrated with standard sodium hydroxide. The formaldehyde reagent is neutralised or blank-corrected as required. This is a named teaching route; use the institution's prescribed acid-base assay if different.

Reaction and Calculation Basis

- Teaching reaction: $4\ NH_4Cl + 6\ HCHO \rightarrow C_6H_{12}N_4 + 4\ HCl + 6\ H_2O$.
- NH_4Cl molar mass ≈ 53.49 g/mol. For corrected volume V in mL and NaOH concentration C in mol/L, mass NH_4Cl in mg = V × C × 53.49.

Requirements

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

Standardisation of the Titrant

1. Prepare the standard

Use certified potassium hydrogen phthalate, KHP, on its stated purity and drying basis. Accurately weigh and dissolve in the specified water amount.

2. Titrate

Titrate with NaOH using the prescribed phenolphthalein or potentiometric endpoint. Record blank correction and obtain the required concordant results.

3. Calculate

KHP reacts 1:1 with NaOH. C_{NaOH} = mass KHP × purity fraction ÷ (204.22 × NaOH volume in litres). Store and use the titrant as prescribed to limit carbon dioxide uptake.

Procedure

1. Check the method

Confirm that the laboratory uses the formol route and record the required reagent quantities, endpoint and test-portion basis.

2. Prepare the test

Accurately weigh and dissolve ammonium chloride in the prescribed water. The instructor provides the specified neutralised formaldehyde reagent.

3. Liberate titratable acid

Add the formaldehyde reagent and allow the prescribed reaction conditions. Run the specified matching blank.

4. Titrate

Titrate to the stated endpoint with standardised NaOH, recording initial and final readings.

5. Calculate

Use the blank-corrected volume and one mole NaOH per mole ammonium chloride under the valid reaction conditions.

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: 0.40844 g KHP (purity assumed 1.000) consumes 20.00 mL NaOH. C = (0.40844/204.22)/0.02000 = 0.1000 mol/L.
- For a 0.1000 g NH_4Cl test, corrected NaOH titre = 18.70 mL at 0.1000 mol/L. Mass found = 18.70 × 0.1000 × 53.49 = 100.03 mg.
- Assay ≈ 100.03/100.0 × 100 = 100.0%. A result near 100% is not automatically a pass without the prescribed criterion.

Result

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

Precautions

- Formaldehyde requires the laboratory's ventilation and handling controls.
- A reagent blank is important; acidic reagent contamination otherwise biases the assay.
- Do not use a simple direct NaOH endpoint for ammonium chloride without the prescribed reaction or validated alternative method.
- 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 add formaldehyde in this method?

It makes ammonium content measurable through the corresponding released acid.

2. Why standardise NaOH?

Its effective concentration can change, including through carbon dioxide absorption.

3. What is the equivalence at 0.1 mol/L?

1 mL corresponds to approximately 5.349 mg NH_4Cl in this 1:1 method.

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