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Recrystallisation of Acetanilide

Experiment 15 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To purify acetanilide by recrystallisation and calculate mass recovery.

Principle

A useful crystallisation solvent dissolves much more of the compound when hot than when cold. Hot insoluble impurities can be removed before cooling; soluble impurities should largely remain in the mother liquor. Recovery and purity are separate outcomes.

Requirements

- Assigned impure acetanilide, water and the instructor's approved microscale method.
- Flask, controlled heater/bath, appropriate hot-filtration apparatus, vacuum filtration, drying setup and melting-point instrument.

Procedure

1. Weigh

Record the mass of the impure solid and prepare clean labelled vessels.

2. Dissolve

Add the selected solvent in small increments while heating under the assigned method until the solid just dissolves. Its low cold-water solubility makes a hot-water/cooling comparison useful; warm filtration apparatus helps avoid premature crystallisation.

3. Clarify

If hot insoluble matter is present, use the teacher-approved warmed filtration setup. Activated carbon is used only when justified and in a small assigned quantity.

4. Crystallise

Allow the clear solution to cool slowly undisturbed, then use the prescribed final cooling step. Record crystal formation instead of assuming full recovery.

5. Collect

Filter the crystals, wash with a small amount of cold appropriate solvent and dry to the specified condition.

6. Assess

Weigh the dry crystals, calculate recovery and measure melting range. Record whether the range narrows compared with the starting material; excessive solvent can reduce recovery.

Observations and Record

Record	Entry
Starting mass / solvent volume	_____
Dry recovered mass / recovery	_____
Before and after melting ranges	_____
Expected comparison	Typical parent region: 113-116°C, depending on method and standard

Worked Example

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

- Starting mass 1.00 g and dry recovered mass 0.72 g gives recovery = 72.0%. This is not a reaction yield or a direct purity assay.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use instructor-assigned quantities and prepared test reagents in the designated laboratory; wear eye protection and use the required fume hood.
- Do not taste unknowns, smell containers directly, burn unknown halogenated material or heat sealed vessels.
- Use fresh separate portions for independent tests. A single colour reaction does not establish molecular identity.
- Keep a vapour escape path and use the approved heating source; never heat a sealed crystallisation vessel.

Viva Questions and Answers

1. Why minimise hot solvent?

Excess solvent retains more compound after cooling and can reduce recovery.

2. Why wash with cold solvent?

It removes adhering mother liquor while limiting dissolution of the crystals.

3. Does high recovery prove purity?

No. Impurities can also increase the recovered mass.

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