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Microwave-Assisted Esterification Study

Practical 20 - Controlled heating comparison practical

Aim

To compare an approved microwave-assisted reaction with a matched conventional-heating method.

Principle

A multistep sequence connects the identity, amount and purity of an isolated intermediate to the next reaction. Overall isolated yield is the product of the fractional stage yields when based on consistent stoichiometry.

Materials and Requirements

- Approved two-step synthesis and supervised reaction records
- Intermediate and product references
- TLC, melting-point or other physical tests and appropriate spectra
- Approved method or teaching-data pack and a dated record sheet

Procedure

1. Define the preparation or dataset, study question and method version. Record identities, units and the criteria to be used before examining the outcome.
2. Draw the named sequence, atom changes, balanced stoichiometry and expected intermediates.
3. Prepare a stage-by-stage table of actual amounts, limiting reagents, authorised conditions and analytical checkpoints.
4. For each stage use the approved reaction and work-up, retain progress evidence and isolate a characterised intermediate before advancing.
5. Characterise the final product, reconcile masses and calculate both individual and overall yields with their purity basis.
6. Retain the original observations and calculations. State deviations, missing information and the limits of the conclusion; do not replace measured values with expected results.

Observations and Record

Enter actual measurements or the supplied teaching data. Record the method, material identity, conditions and units.

Stage	Starting amount / limiting reagent	Product identity / purity	Dry yield
Step 1	Record mol	Characterise intermediate	Calculate
Step 2	Record mol	Characterise final product	Calculate
Overall	Original basis	Traceability	Calculate

Calculations

- Overall fractional yield = stage-1 fractional yield x stage-2 fractional yield for a consistent sequence

Worked Example

Illustrative stage yields 80.0% and 75.0% give an overall yield of 0.80 x 0.75 = 60.0%.

A two-step 60% yield is not obtained by averaging the stage percentages.

Microwave comparison

• Use a purpose-built laboratory microwave with the approved vessel, pressure/temperature limits, fill and solvent compatibility. A domestic microwave is not appropriate.
• Select a named condensation or esterification model from the authorised method. Compare conversion, isolated yield, identity, time and measured energy on equal scales.
• Match chemical and thermal conditions as far as possible; shorter clock time alone does not prove a nonthermal effect or a greener process.

Selected comparison

• approved esterification model
• Use matched starting quantities and report the actual microwave equipment programme alongside the conventional-heating record.

Result and Interpretation

• Present the reaction scheme, mechanisms, raw observations, identity evidence and stage/overall yield calculations.

Precautions

• Apply the specific authorised hazards and quench procedure at each stage.
• Do not carry an unidentified intermediate into the next reaction.
• Do not label an untested teaching product for administration.

Viva Questions and Answers

Why isolate an intermediate?

It supports identity, purity and accurate input to the next step.

How are overall yields combined?

Multiply fractional yields on consistent stoichiometric bases.

Why can high conversion give low final yield?

Selectivity, purification and transfer losses may reduce recovery.

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