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Multi-Step Preparation and Characterisation of Aspirin

Practical 17 - Multistep synthesis practical

Aim

To plan and document a two-step synthesis with intermediate and final-product characterisation.

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.

Selected sequence

- Methyl salicylate to salicylic acid to aspirin
- Ester hydrolysis gives salicylic acid, followed by phenolic acetylation. Assess residual salicylic acid and hydrolytic instability.
- Final neutral-product molar mass for the model: 180.16 g/mol. Confirm the actual salt or solvate form.

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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