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# Comparison of Aspirin Synthetic Routes

Practical 4 - Synthetic-route comparison practical

## Aim

To compare alternative synthetic routes using quality, material, safety and process evidence.

## 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.

### Route comparison worksheet

• Draw two justified routes to the same product form and compare steps, overall yield, selectivity, impurity control, reagents, solvent and isolation demands.

• Calculate atom economy from balanced equations and process mass intensity from actual input masses. Define treatment of water and recovered solvent. A shorter route is not automatically safer or cleaner.

• For example, total input 20 g and isolated product 2 g give process mass intensity 10 g/g on the stated accounting basis.

### 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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