

Preparation of 7-Hydroxy-4-Methylcoumarin

Practical 02 | Medicinal Chemistry III

Aim

To prepare 7-Hydroxy-4-Methylcoumarin, and interpret the recorded observations.

Principle

- A Pechmann-type condensation of a suitable phenol with a beta-ketoester can construct the coumarin ring. Substituent positions must be checked on the final structure.
- A chemical transformation changes selected functional groups while retaining or constructing the required molecular skeleton.
- Product identity and purity require analytical evidence; appearance alone cannot establish either.
- Trace ring construction and identify the lactone carbonyl, 7-hydroxy group and 4-methyl group.
- Reaction record: draw starting material and product structures, mark the functional-group change, and retain the assigned stoichiometry.
- Characterization record: compare the assigned melting range, TLC or spectral evidence. A narrow melting interval can support purity but cannot alone establish complete identity.

Materials and Apparatus

- Assigned phenolic precursor, beta-ketoester and approved condensation system.
- Assigned starting materials and instructor-selected reagents.
- Balance, reaction glassware, stirring/heating arrangement, condenser where required, filtration equipment and identity/purity test apparatus.

Method

1. Write the assigned reaction scheme and balance the transformation before calculating reagent requirements.
2. Identify the limiting reactant and document the approved reaction scale, solvent and conditions.
3. Observe the instructor-directed reaction sequence and record changes without substituting reagents or conditions.
4. Complete the prescribed work-up to separate the product from reaction liquids and by-products.
5. Purify the product by the assigned method, dry it appropriately and determine the recovered mass.
6. Record the assigned identity and purity observations and compare them with the expected product.

Observation Record

Complete this record using actual laboratory findings or the provided teaching data.

Observation / parameter	Record / finding
Assigned route	_____
Limiting-reactant calculation	_____
Process observations	_____
Dry recovery	_____
Identity/purity evidence	_____

Calculations and Interpretation

- Theoretical product mass = limiting-reactant amount (mol) $\times$ product stoichiometric ratio $\times$ product molar mass.
- Percentage yield = dry product mass / theoretical product mass $\times$ 100.

Worked Illustration

Illustration: a theoretical product mass of 2.00 g and dry recovery of 1.50 g give 75.0% yield.

The values in this illustration are for calculation practice.

Result

Record the actual finding, calculated value with units where relevant, and a conclusion supported by the observations.

Finding: _____

Conclusion: _____

Precautions

- Use the approved reaction conditions and suitable ventilation.
- Keep product, starting material and waste streams separately labelled.
- Calculate yield from dry product and the limiting reactant; never use wet mass as pure-product mass.

Viva Questions

1. Why inspect the final molecular structure?

The intended connectivity and substituent positions distinguish the product from related intermediates or isomers.

2. What is the limiting reactant?

The reactant whose available amount permits the smallest theoretical amount of product.

3. Does a high yield establish purity?

No. Residual solvent, impurities and unreacted material can increase recovered mass.

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