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Umbelliferone by Pechmann Condensation

Practical 17 - Supervised synthesis and reaction-record practical

Aim

To prepare or assess umbelliferone by Pechmann-type condensation and explain its reaction, isolation and characterisation.

Principle

The adopted resorcinol and malic-acid-based method supplies the carbon framework for 7-hydroxycoumarin, umbelliferone.

Acid-promoted condensation and ring formation produce the coumarin lactone. The exact carbonyl precursor controls substitution: resorcinol with ethyl acetoacetate commonly gives 4-methylumbelliferone instead.

Materials and Requirements

- Approved microscale reaction SOP and reagent specifications
- Supervised hood, temperature control, glassware and method-specific work-up equipment
- TLC references, balance and appropriate melting-point or spectral methods
- 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 reaction and mechanism, identify stoichiometry, limiting reagent and possible side products. Enter the authorised quantities, addition order and temperature/time limits in the record.
3. Verify that the authorised method targets umbelliferone rather than its 4-methyl analogue. Review or execute the supervised condensation, controlled work-up and purification record.
4. Monitor progress by the prescribed TLC or other analytical method, retaining the starting-material comparison.
5. Isolate and purify by the authorised work-up, record dry mass and examine identity and purity by complementary methods.
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 / preparation	Conditions / observation	Analytical evidence	Mass / yield
Starting materials	Identity / quantities	Reference evidence	Calculate mol
Reaction progress	Actual time / temperature	TLC or assay	Record
Purified product	Appearance	Identity / purity	Dry mass

Calculations

- Theoretical product mass = limiting-reactant moles x stoichiometric product ratio x product molar mass
- Isolated yield (%) = dry isolated product mass / theoretical product mass x 100
- Rf = zone migration / solvent-front migration

Worked Example

Illustrative 1:1 conversion from 10.0 mmol limiting reagent to a product of molar mass 162.14 g/mol gives 1.6214 g theoretical product. A dry isolated mass of 1.2161 g corresponds to 75.0% yield.

Use the actual reaction stoichiometry, salt or solvate form and purity basis; this illustration is not an expected experimental result.

Mechanism and identity record

• Acid-promoted condensation and ring formation produce the coumarin lactone. The exact carbonyl precursor controls substitution: resorcinol with ethyl acetoacetate commonly gives 4-methylumbelliferone instead.

• Confirm the intended molecular mass and substitution pattern by suitable spectra; fluorescence alone does not establish identity.

Result and Interpretation

• Report isolated yield and the independent identity/purity observations.

• Confirm the intended molecular mass and substitution pattern by suitable spectra; fluorescence alone does not establish identity.

Precautions

• Strong-acid condensation and thermal work-up require trained supervision and a compound-specific SOP.

• Use the institution-approved SOP and trained supervision for reactive additions, heating and work-up.

• Do not infer purity from yield or a single TLC zone.

Viva Questions and Answers

What determines theoretical yield?

The limiting reagent and balanced reaction stoichiometry.

How is conversion different from isolated yield?

Conversion measures reactant consumption; isolated yield also reflects selectivity and recovery losses.

Why use complementary identity tests?

A single physical or chromatographic observation may not distinguish impurities or isomers.

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