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Beckmann Rearrangement

Practical 12 - Supervised synthesis and reaction-record practical

Aim

To prepare or assess caprolactam by Beckmann rearrangement and explain its reaction, isolation and characterisation.

Principle

Cyclohexanone oxime rearranges to epsilon-caprolactam under the selected activated-oxime conditions. Activation of the oxime leaving group enables migration of the group anti to it, giving a nitrilium intermediate that leads to an amide. A cyclic ketoxime gives ring expansion.

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. Review the authorised acid-activation process and its temperature controls with the supervisor. Execute only the approved scale and quench; a supplied reaction record may be used.
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 113.16 g/mol gives 1.1316 g theoretical product. A dry isolated mass of 0.8487 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

• Activation of the oxime leaving group enables migration of the group anti to it, giving a nitrilium intermediate that leads to an amide. A cyclic ketoxime gives ring expansion.

• Differentiate oxime and lactam using the selected IR and NMR evidence. Identical molecular formulae do not distinguish the isomers.

Result and Interpretation

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

• Differentiate oxime and lactam using the selected IR and NMR evidence. Identical molecular formulae do not distinguish the isomers.

Precautions

• Strong-acid activation and quenching require specific supervised controls; do not substitute reagents or scale.

• 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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