

Preparation of 5-Nitrosalicylic Acid

Practical 11 | Pharmaceutical Organic Chemistry II

Aim

To prepare 5-Nitrosalicylic Acid, and interpret the recorded observations.

Principle

- Electrophilic aromatic nitration introduces a nitro group. The directing effects of the hydroxy and carboxyl substituents must be considered when identifying the product.
- 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.
- 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

- Salicylic acid and the instructor-demonstrated nitration 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
Starting-material amounts	_____
Limiting reactant	_____
Reaction observations	_____
Dry product mass	_____
Identity and purity observations	_____

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. What is the limiting reactant?

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

2. Does a high yield establish purity?

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

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