WJEC (Eduqas) Chemistry A-level

SP OA4c - Two-Step Synthesis

Methods and images taken from the WJEC practical handbook

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Aim

To **synthesise** 3-nitrobenzenecarboxylic acid via **nitration** of methyl benzenecarboxylate followed by **alkaline hydrolysis** of the ester functional group.

Apparatus and Chemicals

- Deionised water
- Access to a 3 decimal place mass balance (minimum 2 decimal place)
- Access to ice
- Spatula
- Weighing boat
- 2 x 100 cm^3 conical flask
- 25 cm^3 measuring cylinder
- Ice bath
- Glass Pasteur pipette with rubber teat
- Glass stirring rod
- 250 cm^3 beaker
- Buchner funnel
- Suction apparatus
- Filter paper
- 10 cm^3 measuring cylinder
- Hot water bath
- Round bottomed flask
- Reflux condenser
- Thermometer
- Sample vial
- Labels/suitable pen
- Anti-bumping granules
- Heating mantle / Bunsen burner with water bath
- Clamp stand
- Melting point apparatus / Thiele tube
- Capillary tube
- Concentrated H_2SO_4 solution
- $C_6H_5COOCH_3$ (methyl benzenecarboxylate)
- Concentrated HNO_3 solution
- CH_3OH (methanol)
- NaOH
- Concentrated HCl solution
- 0.1 mol dm^{-3} HCl solution

Safety Considerations

★ Concentrated H_2SO_4 - corrosive
★ $C_6H_5COOCH_3$ solution - flammable
★ Concentrated HNO_3 - corrosive
★ CH_3OH solution - flammable, toxic
★ NaOH - corrosive
★ Concentrated HCl solution - corrosive
★ 0.1 mol dm^{-3} HCl solution - irritant

Method

Part 1

1. Carefully add 20 cm^3 of concentrated H_2SO_4 solution to a conical flask.
2. Add 10.2 g of $C_6H_5COOCH_3$ to the conical flask.
3. Cool the reaction mixture to 0°C in an **ice bath**.
4. Slowly add, with stirring, 12.5 cm^3 of a 1:1 mixture of concentrated H_2SO_4 solution and concentrated HNO_3 solution. Monitor the temperature of the reaction with a thermometer as you perform this step and ensure that the temperature does not rise above 10 °C.
5. Pour the reaction mixture slowly **onto ice** in a beaker.
6. When the ice has melted, filter the mixture under **reduced pressure**.
7. Wash the solid once with **cold deionised water** and twice with cold CH_3OH.
8. **Recrystallise** the solid product from hot methanol using a **hot water bath**.
9. Dry the recrystallized product, record its mass and measure its melting point.
10. Use the mass of the product to calculate the **percentage yield**.

Part 2

1. To a **round bottomed flask** add 9.0 g of your product from part 1, 20 cm^3 of deionised water and 4.0 g of NaOH.
2. Attach a **condenser** to the round bottomed flask and heat the reaction mixture under reflux for 5-10 minutes.
3. Carefully pour the reaction mixture, with stirring, into 12.5 cm^3 of concentrated HCl solution. This step should be performed in a **fume cupboard**.
4. Cool the reaction mixture to room temperature using an ice bath.
5. Filter the mixture under **reduced pressure** and wash with cold deionised water.
6. **Recrystallise** the solid from hot 0.1 mol dm^{-3} HCl solution using a hot water bath.
7. Dry the recrystallized product, record its mass and measure its melting point.
8. Use the mass of the product to calculate the **percentage yield**.