

# WJEC (Wales) Chemistry A-level

## SP 4.8c - Two-Step Synthesis

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## SP 4.8c - Two-Step Synthesis

### 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<sup>3</sup> conical flask
- 25 cm<sup>3</sup> measuring cylinder
- Ice bath
- Glass Pasteur pipette with rubber teat
- Glass stirring rod
- 250 cm<sup>3</sup> beaker
- Buchner funnel
- Suction apparatus
- Filter paper
- 10 cm<sup>3</sup> 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<sub>2</sub>SO<sub>4</sub> solution
- C<sub>6</sub>H<sub>5</sub>COOCH<sub>3</sub> (methyl benzenecarboxylate)
- Concentrated HNO<sub>3</sub> solution
- CH<sub>3</sub>OH (methanol)
- NaOH
- Concentrated HCl solution
- 0.1 mol dm<sup>-3</sup> HCl solution

### Safety Considerations

- ★ Concentrated H<sub>2</sub>SO<sub>4</sub> - corrosive
- ★ C<sub>6</sub>H<sub>5</sub>COOCH<sub>3</sub> solution - flammable
- ★ Concentrated HNO<sub>3</sub> - corrosive
- ★ CH<sub>3</sub>OH solution - flammable, toxic
- ★ NaOH - corrosive
- ★ Concentrated HCl solution - corrosive
- ★ 0.1 mol dm<sup>-3</sup> HCl solution - irritant





## Method

### Part 1

1. Carefully add 20 cm<sup>3</sup> of concentrated H<sub>2</sub>SO<sub>4</sub> solution to a conical flask.
2. Add 10.2 g of C<sub>6</sub>H<sub>5</sub>COOCH<sub>3</sub> to the conical flask.
3. Cool the reaction mixture to 0°C in an **ice bath**.
4. Slowly add, with stirring, 12.5 cm<sup>3</sup> of a 1:1 mixture of concentrated H<sub>2</sub>SO<sub>4</sub> solution and concentrated HNO<sub>3</sub> 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<sub>3</sub>OH.
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<sup>3</sup> 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<sup>3</sup> 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<sup>-3</sup> 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**.

