# WJEC (Wales) Chemistry A-level

## SP 4.8c - Two-Step Synthesis

Flashcards

Give the two processes required to produce 3-nitrobenzenecarboxylic acid from methyl benzenecarboxylate

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1. **Nitration** of methyl benzenecarboxylate to produce methyl 3-nitrobenzenecarboxylate

2. **Alkaline hydrolysis** of methyl 3-nitrobenzenecarboxylate to produce 3-nitrobenzenecarboxylic acid.

What reagents are required for the nitration of methyl benzenecarboxylate?

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Concentrated sulfuric acid ($H_2SO_4$)

Concentrated nitric acid ($HNO_3$)

# Name the mechanism for the nitration of methyl benzenecarboxylate

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Electrophilic substitution

# What apparatus is required to carry out nitration of methyl benzenecarboxylate?

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- Conical flask
- Ice bath
- Thermometer
- Beaker
- Digital mass balance
- Weighing boat
- 25 $cm^3$ measuring cylinder
- Buchner funnel and suction apparatus
- Hot water bath

# Outline the procedure to obtain methyl 3-nitrobenzenecarboxylate from methyl benzenecarboxylate

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1. Add 20 $cm^3$ of concentrated sulfuric acid and 10.2 g of methyl benzenecarboxylate to a conical flask.
2. Cool the reaction mixture to 0°C in an ice bath.
3. Slowly add 12.5 $cm^3$ of a 1:1 mixture of concentrated sulfuric acid and concentrated nitric acid. Ensure the temperature does not rise above 10 °C.
4. Pour the reaction mixture slowly onto ice in a beaker. Once the ice has melted, filter the mixture under reduced pressure.
5. Wash the solid once with cold deionised water and twice with cold methanol.
6. Recrystallise the solid product using hot methanol and a hot water bath.
7. Dry the product and record its mass.

During nitration, why must the reaction mixture be kept below 10ºC?

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If the solution goes above 10ºC, you will end up with more substituted nitro groups on the aromatic compound.

What is the purpose of washing the mixture with deionised water?

What is the purpose of washing the mixture with deionised water?

The deionised water will remove any water soluble impurities.

Give the equations for the reactions which take place between $H_2SO_4$ and $HNO_3$ to produce the electrophile $NO_2^+$

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$$H_2SO_4 + HNO_3 \rightarrow HSO_4^- + H_2NO_3^+$$

$$H_2NO_3^+ \rightarrow NO_2^+ + H_2O$$

During nitration, which chemical behaves as a catalyst?

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Sulfuric acid ($H_2SO_4$)

Sulfuric acid does not get used up since the $H^+$ ion removed from the benzene ring reacts with the $HSO_4^-$ ion to reproduce sulfuric acid.

# What is alkaline hydrolysis?

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Alkaline hydrolysis involves refluxing an ester with a dilute alkali to produce a carboxylate ion and an alcohol.

What apparatus is required for the alkaline hydrolysis of methyl 3-nitrobenzenecarboxylate to produce 3-nitrobenzenecarboxylic acid?

# What apparatus is required for the alkaline hydrolysis of methyl 3-nitrobenzenecarboxylate to produce 3-nitrobenzenecarboxylic acid?

- Digital mass balance
- Weighing boat
- Round bottom flask
- 25 $cm^3$ measuring cylinder
- Condenser
- Glass rod
- Buchner funnel and suction apparatus
- Filter paper
- Hot water bath
- Conical flask

# Outline the experimental procedure for the alkaline hydrolysis of methyl 3-nitrobenzenecarboxylate

# Outline the experimental procedure for the alkaline hydrolysis of methyl 3-nitrobenzenecarboxylate

1. Add 9.0 g methyl 3-nitrobenzenecarboxylate, 20 $cm^3$ of deionised water and 4.0 g of NaOH to a round bottom flask.
2. Attach a condenser and heat the reaction mixture under reflux for 5-10 minutes.
3. Whilst stirring, pour the reaction mixture into 12.5 $cm^3$ of concentrated HCl.
4. Cool the reaction mixture to room temperature using an ice bath.
5. Filter the mixture under reduced pressure. Wash the residue with cold deionised water.
6. Recrystallise the solid from hot 0.1 mol $dm^{-3}$ HCl using a hot water bath.
7. Dry the product and record its mass.

# Why is HCl added to the reaction mixture after alkaline hydrolysis?

# Why is HCl added to the reaction mixture after alkaline hydrolysis?

The HCl provides the $H^+$ ion to protonate the carboxylate ion. The 3-nitrobenzenecarboxylate ion is protonated to produce the required 3-nitrobenzenecarboxylic acid.

# What are the advantages of filtering under reduced pressure?

# What are the advantages of filtering under reduced pressure?

- Filtering under reduced pressure is much faster than standard filtration.
- Reduced pressure filtration is more efficient at removing residual liquid compared to standard filtration. This obtains a purer solid.

During recrystallisation, why is it necessary to add only a minimal amount of warm solvent to the solid derivative?

# During recrystallisation, why is it necessary to add only a minimal amount of warm solvent to the solid derivative?

A minimal amount of warm solvent is used to achieve a large yield of the required solid on recrystallisation. A larger yield is gained since using a minimal amount reduces the amount of solid lost by retention in the solvent.

# How do you use a Buchner funnel to filter under reduced pressure?

# How do you use a Buchner funnel to filter under reduced pressure?

The Buchner funnel is lined with filter paper and connected to vacuum suction apparatus by a rubber tube.

The substance is poured onto the filter paper and the liquid is sucked into the flask below. The solid will remain on the filter paper.

# How do you calculate percentage yield?

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$$\text{Percentage yield} = \frac{\text{Actual mass of product}}{\text{Theoretical mass of product}} \times 100$$