

# OCR (B) Chemistry A-Level

PAG 06 - Synthesis of an organic solid

Preparation of methyl 3-nitrobenzoate

**(A level only)**

Flashcards



What two reactants are used to synthesis methyl 3-nitrobenzoate?



What two reactants are used to synthesis methyl 3-nitrobenzoate?

Methyl benzoate

Nitric acid



What is the purpose of concentrated sulfuric acid in the synthesis methyl 3-nitrobenzoate?



What is the purpose of concentrated sulfuric acid in the synthesis methyl 3-nitrobenzoate?

Catalyst (speeds up the rate of reaction)



In the preparation stage of methyl 3-nitrobenzoate, what should the temperature be kept below? Why?



In the preparation stage of methyl 3-nitrobenzoate, what should the temperature be kept below? Why?

Below  $10^{\circ}\text{C}$ , to maximise yield



Describe the process of filtration under reduced pressure



## Describe the process of filtration under reduced pressure

- Place a piece of filter paper in a Büchner funnel.
- Place the funnel in a Büchner flask. Connect the side arm of the flask to a water pump.
- Connect the water pump to a tap and turn the tap on to create the vacuum. Pour the solution into the funnel.
- Once the solution has drained through, rinse the solid on the filter paper with a small volume of solvent.



# Describe the process of recrystallisation



## Describe the process of recrystallisation

- Dissolve the solid in the minimum volume of hot solvent
- Filter to remove insoluble impurities
- Cool the filtrate
- Filter to remove soluble impurities (the product remains on the filter paper)



Why is ethanol used for the  
recrystallisation of methyl  
3-nitrobenzoate?



Why is ethanol used for the recrystallisation of methyl 3-nitrobenzoate?

- Methyl 3-nitrobenzoate is soluble in ethanol but not water
- Ethanol is a cheap and relatively safe solvent



During recrystallisation, why is the product dissolved in a minimum amount of solvent?



During recrystallisation, why is the product dissolved in a minimum amount of solvent?

If there is a large volume of solvent when the mixture is cooled (and solubility decreased), some of the product may remain in solution.



How does recrystallisation improve the purity of a product?



# How does recrystallisation improve the purity of a product?

The desired product is dissolved in an appropriate solvent that the impurities are insoluble in. Subsequent filtration leaves the insoluble impurities on the filter paper while the solution flows through.



Why is the solution cooled in ice immediately after recrystallisation?



Why is the solution cooled in ice immediately after recrystallisation?

To decrease the solubility of the product so that it crystallises. Lower temperatures increase the size of crystals formed.



Why must the crystals be completely dry before measuring their mass to calculate the percentage yield?



Why must the crystals be completely dry before measuring their mass to calculate the percentage yield?

Excess solvent on the crystals could falsely increase the yield.



# How can percentage yield be calculated?



# How can percentage yield be calculated?

$(\text{Maximum theoretical yield} \div \text{actual yield}) \times 100$



Give 3 reasons why the yield may be lower than 100%



Give 3 reasons why the yield may be lower than 100%

- Product may be lost when filtering/ transferring between apparatus
- Some product may stay in solution after recrystallisation
- Other side reactions may occur



What technique could be used to investigate the purity of the product before and after recrystallisation?



What technique could be used to investigate the purity of the product before and after recrystallisation?

Thin layer chromatography - comparison of  $R_f$  values from the product before and after recrystallisation to a pure lab sample.



What piece of apparatus is used to transfer a small amount of each liquid onto the chromatography plate?



What piece of apparatus is used to transfer a small amount of each liquid onto the chromatography plate?

Capillary tube



During chromatography, why should the beaker containing the solvent and the TLC plate be covered with a clear watch glass?



During chromatography, why should the beaker containing the solvent and the TLC plate be covered with a clear watch glass?

To prevent the evaporation of the solvent

The watch glass should be clear to allow the plate to be monitored



In chromatography, what is the solvent front?



In chromatography, what is the solvent front?

The furthest distance travelled by the solvent up the TLC plate



Why should the baseline be drawn in pencil on a TLC plate?



Why should the baseline be drawn in pencil on a TLC plate?

Pencil is insoluble in solvent so it won't travel up the plate and obscure the results



When using chromatography to assess the purity of a sample of methyl 3-nitrobenzoate, what is used to make the spots visible?



When using chromatography to assess the purity of a sample of methyl 3-nitrobenzoate, what is used to make the spots visible?

Iodine crystals



# How do you calculate an Rf value?



How do you calculate an Rf value?

$$R_f = \frac{\text{distance travelled by the substance}}{\text{distance travelled by the solvent}}$$



Without using chromatography, how can the purity of a sample be assessed?



Without using chromatography, how can the purity of a sample be assessed?

Use melting point apparatus

Pure substances have a specific melting point whereas impure substances will melt over a larger range of temperatures. The smaller the range, the purer the sample.



What safety precautions should be taken during this practical?



# What safety precautions should be taken during this practical?

- Methyl 3-nitrobenzoate is an irritant so wear safety goggles and avoid contact with skin
- Iodine crystals are harmful if in contact with skin or when inhaled so avoid contact with skin and keep away from your face
- Methyl benzoate is an irritant so wear safety goggles and avoid contact with skin
- Ethanol is highly flammable so keep away from naked flames

