

WJEC (Wales) Chemistry A-level

SP 4.8b - Synthesis of an Organic Solid Product

Flashcards

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What reagents are required to make aspirin
(2-acetoxybenzenecarboxylic acid)?



What reagents are required to make aspirin (2-acetoxybenzenecarboxylic acid)?

- Salicylic acid (2-hydroxybenzenecarboxylic acid)
- Ethanoic anhydride



What are the three parts of the process to produce a pure organic solid?



What are the three parts of the process to produce a pure organic solid?

1. Synthesis of the compound
2. Filtration (vacuum filtration)
3. Purification (recrystallisation)



What apparatus is required to synthesise aspirin from 2-hydroxybenzenecarboxylic acid and ethanoic anhydride?



What apparatus is required to synthesise aspirin from 2-hydroxybenzenecarboxylic acid and ethanoic anhydride?

- Digital mass balance
- 25 cm³ pear shaped flask
- 10 cm³ measuring cylinder
- Hot water bath
- Ice bath
- Glass stirring rod
- Buchner funnel, suction apparatus
- Watch glass
- Sample vial
- Filter paper
- Melting point apparatus
- Capillary tube



Outline the experimental procedure to produce the impure aspirin from 2-hydroxybenzenecarboxylic acid and ethanoic anhydride



Outline the experimental procedure to produce the impure aspirin from 2-hydroxybenzenecarboxylic acid and ethanoic anhydride

1. Weigh out 1.0 g of 2-hydroxybenzenecarboxylic acid and transfer to a pear shaped flask.
2. Add 2 cm³ of (CH₃CO)₂O and 8 drops of concentrated H₃PO₄.
3. Connect the reflux condenser and place in a fume hood.
4. Warm the mixture in a hot water bath until all of the solid dissolves. Warm for 5 more minutes.
5. Add 5 cm³ of deionised water to the solution and stand the flask in a bath of iced water until precipitation is complete.
6. Filter the mixture under reduced pressure to obtain the impure derivative. Wash with a little cold water.



Why is the mixture heated under reflux?



Why is the mixture heated under reflux?

Heating under reflux prevents any vapours escaping from the reaction mixture. It also ensures the flask does not boil dry.



Why is the mixture placed in an ice bath after reaction?



Why is the mixture placed in an ice bath after reaction?

The cold ice will cause the solid to crystallise since solubility is reduced in cold conditions.



Why is H_3PO_4 added to the reaction mixture?



Why is H_3PO_4 added to the reaction mixture?

H_3PO_4 (phosphoric acid) is a catalyst which speeds up the rate of reaction by providing an alternative reaction pathway with a lower activation energy.



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.



Outline the experimental procedure to purify the crude aspirin and determine its melting point



Outline the experimental procedure to purify the crude aspirin and determine its melting point

1. Using a boiling tube in a water bath, dissolve the impure product in the minimum amount of warm ethanol.
2. Add 5 cm³ of warm water drop-wise. If the solution becomes cloudy, heat until it becomes clear again.
3. Place the boiling tube into ice water for 15 minutes or until the crystals stop forming.
4. Use a Buchner funnel to filter the purified derivative under reduced pressure.
5. Dry the purified product using filter paper.
6. Collect your sample in a dry sample vial.
7. Calculate the mass of the product. Calculate percentage yield of the product.
8. Use a capillary tube to measure the melting point of the product.



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



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

A minimal amount of warm ethanol 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 lost by retention in the ethanol.



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.



Explain how the melting point of a sample relates to the purity of the compound



Explain how the melting point of a sample relates to the purity of the compound

Pure substances will have a sharp melting point whereas a substance with impurities will melt over a range of temperatures.



How do you determine the melting point of a solid?



How do you determine the melting point of a solid?

1. A small sample of the solid is packed into a capillary tube.
2. The tube is placed into melting apparatus which houses a thermometer. The thermometer reading should be taken at the point at which the solid melts.
3. Record the range of temperatures over which the solid was observed to melt.



How do you calculate percentage yield?



How do you calculate percentage yield?

Percentage yield =

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

