

Edexcel International Chemistry <u>A-level</u>

Practical 16

The Preparation of Aspirin









In this experiment, a sample of acetylsalicylic acid is prepared by acetylation of 2-hydroxybenzoic acid with ethanoic anhydride. It is then purified by recrystallisation and the melting point of the product is recorded as a test for its purity.

Method

- 1. Weigh out 2g of 2-hydroxybenzoic acid and transfer to a pear shaped flask.
- 2. Add 5 cm³ of ethanoic anhydride and 8 drops of concentrated sulfuric acid solution.
- 3. Warm the mixture in a hot water bath under reflux for 5 minutes so the 2-hydroxybenzoic acid dissolves. Gently swirl the flask while heating the mixture.
- 4. Add 10 cm³ of ice to the solution and stand the flask in a bath of ice until precipitation is complete.
- 5. Filter the mixture under reduced pressure using a Büchner funnel and wash the crystals with a small amount of icy water.
- 6. Dissolve the impure product in the minimum amount of warm ethanol.
- 7. Add warm water.
- 8. Place the boiling tube into ice for 15 minutes.
- 9. Again, filter the purified derivative under reduced pressure.
- 10. Dry the purified product using filter paper.
- 11. Collect your sample in a dry, pre-weighed vial and calculate the mass of the product.

 Use this information to calculate the percentage yield of the product.
- 12. Measure the melting point of the product to test its purity the melting point of pure aspirin is 135 °C, the closer the melting point is to this value, the purer the product.

Key points

• When washing the product with ice cold water, add as little as possible to minimise product loss.

Safety

• Ethanoic anhydride and concentrated sulfuric acid are corrosive, so use gloves.





