The microscale synthesis of aspirin

In this experiment you will be preparing 2-ethanoyloxybenzenecarboxylic acid (aspirin) from the reaction between 2-hydroxybenzoic acid (salicylic acid) and ethanoic anhydride. The use of chemical splash goggles is recommended.

The reaction is:

\[
\text{CO}_2\text{H} \quad \text{OH} + \quad \text{CH}_3\text{C}=\text{O} \quad \rightarrow \quad \text{CO}_2\text{H} \quad \text{O}-\text{C}-\text{CH}_3 + \text{CH}_3\text{CO}_2\text{H}
\]

\[\text{2-Hydroxybenzoic acid} \quad \text{(Salicylic acid)} \quad \text{Ethanoic anhydride} \quad \text{2-ethanoyloxybenzenecarboxylic acid} \quad \text{(Aspirin)}\]

Instructions

1. Half-fill a 50 cm\(^3\) beaker with deionised water, and heat to 70–80°C.
2. Weigh 0.23 g of 2-hydroxybenzoic acid (salicylic acid) into a test-tube.
3. Add 25 drops of ethanoic anhydride followed by one drop of 85% phosphoric acid.
4. Place in the water bath and leave for 15 min.
5. While still warm add 1.5 cm\(^3\) of deionised water (use the measuring cylinder) and cool to room temperature until crystallisation begins, then cool in an ice bath.
6. Filter through a small filter funnel and recrystallise in a test-tube using a mixture of 0.7 cm\(^3\) ethanol and 2 cm\(^3\) of deionised water.

Health & Safety

Students must wear suitable eye protection (Splash resistant goggles to BS EN166 3). This experiment should be done in a fume cupboard.

2-Hydroxybenzoic acid is harmful if swallowed or in contact with the skin and can cause eye damage.

Ethanoic anhydride is CORROSIVE, harmful if swallowed or inhaled and flammable.

Phosphoric acid (85%) is CORROSIVE.

Credits

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Health & safety checked May 2018

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