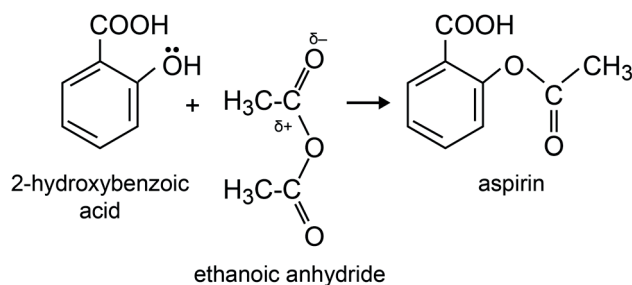


## Core practical 16: Synthesise aspirin from 2-hydroxybenzoic acid

Objectives	
<ul style="list-style-type: none"> <li>• To perform and explain the reactions of acid anhydrides</li> <li>• To synthesise aspirin from 2-hydroxybenzoic acid</li> </ul>	
Safety	Specification links
<ul style="list-style-type: none"> <li>• Perform the experiment in a well-ventilated room.</li> <li>• Wear a lab coat and use eye protection.</li> <li>• Wear heat-protective gloves when handling hot equipment.</li> <li>• Tie long hair back.</li> <li>• Ethanoic anhydride is corrosive.</li> <li>• Concentrated sulfuric acid is corrosive.</li> <li>• 2-hydroxybenzoic acid is harmful.</li> </ul>	<ul style="list-style-type: none"> <li>• Practical techniques 1, 2, 4, 7, 8, 11</li> <li>• CPAC 1a, 2a, 2b, 3a, 4a, 4b</li> </ul>
Procedure	Notes on procedure
<ol style="list-style-type: none"> <li>1. Weigh 2.0 g of 2-hydroxybenzoic acid and put it in a pear-shaped flask. Clamp the flask and suspend it in a beaker of water.</li> <li>2. Add 5.0 cm<sup>3</sup> of ethanoic anhydride to the 2-hydroxybenzoic acid. Add five drops of concentrated sulfuric acid to the mixture in the flask. Fix a condenser on the flask.</li> <li>3. In a fume cupboard, carefully warm the mixture in the water bath using a Bunsen burner. Gently swirl the mixture until all the solid has dissolved.</li> <li>4. Continue warming the mixture for another 10 minutes.</li> <li>5. Remove the flask from the hot water bath and add 10 cm<sup>3</sup> of crushed ice and some distilled/deionised water to break down any unreacted ethanoic anhydride.</li> <li>6. Stand the flask in a beaker of iced water until precipitation appears to be complete.</li> <li>7. Filter off the product using a Büchner funnel and suction apparatus.</li> <li>8. Wash the crystals with the minimum volume of iced water.</li> <li>9. Recrystallise the aspirin in the minimum volume of a mixture of ethanol to water (1 : 3).</li> <li>10. Filter and dry.</li> <li>11. Measure the mass of the pure, dry crystals.</li> <li>12. Measure the melting temperature of the product using melting temperature apparatus</li> </ol>	<ul style="list-style-type: none"> <li>• The procedure will have to be split over more than one lesson because the crystals need to dry before their melting temperature can be measured.</li> <li>• It may be necessary to demonstrate to students how to recrystallise an organic solid and how to measure a melting temperature.</li> </ul>

## Answers to questions

- The hydroxyl group
- 



- 2-hydroxybenzoic acid = 138; aspirin = 180
- 2.6 g
- This will vary if students use their own results:  $\frac{\text{their yield}}{2.6} \times 100$ ; using sample data gives 81%.
- Because of impurities in the sample; the crystals may not be dry.
- Unreacted 2-hydroxybenzoic acid.
- Students should record a melting temperature range rather than a single temperature. This is because impurities in the sample cause the solid to melt over a temperature range; rather than sharply at one temperature. The narrower the range and the closer the students' value to 136 °C, the purer the sample.

## Sample data

2.1 g of aspirin

Melting temperature range: 132–135 °C

Melting temperature: 136 °C

**Core practical 16: Synthesise aspirin from 2-hydroxybenzoic acid****Objectives**

- To perform and explain the reactions of acid anhydrides
- To synthesise aspirin from 2-hydroxybenzoic acid

**Safety**

- Perform the experiment in a well-ventilated room.
- Wear a lab coat and use eye protection.
- Wear heat-protective gloves when handling hot equipment.
- Tie long hair back.
- Ethanoic anhydride is corrosive.
- Concentrated sulfuric acid is corrosive.
- 2-hydroxybenzoic acid is harmful.

**All the maths you need**

- Substitute numerical values into algebraic equations using appropriate units for physical quantities.

**Equipment**

- |                                                |                                                                                    |
|------------------------------------------------|------------------------------------------------------------------------------------|
| • 10 cm <sup>3</sup> ethanoic anhydride        | • dropping pipette                                                                 |
| • 2 g 2-hydroxybenzoic acid                    | • ice                                                                              |
| • 1 cm <sup>3</sup> concentrated sulfuric acid | • Bunsen burner, tripod, gauze and safety mat                                      |
| • distilled/deionised water                    | • mass balance (2 d.p.) and weighing boat                                          |
| • two 10 cm <sup>3</sup> measuring cylinders   | • Büchner funnel, Büchner flask, water/suction pump and filter paper to fit funnel |
| • condenser                                    | • melting temperature apparatus and melting temperature tube                       |
| • pear-shaped flask                            |                                                                                    |
| • stand, clamp and boss                        |                                                                                    |
| • two 250 cm <sup>3</sup> beakers              |                                                                                    |

**Procedure**

1. Weigh 2.0 g of 2-hydroxybenzoic acid and put it in a pear-shaped flask. Clamp the flask and suspend it in a beaker of water.
2. Add 5.0 cm<sup>3</sup> of ethanoic anhydride to the 2-hydroxybenzoic acid. Add five drops of concentrated sulfuric acid to the mixture in the flask. Fix a condenser on the flask.
3. In a fume cupboard, carefully warm the mixture in the water bath using a Bunsen burner. Gently swirl the mixture until all the solid has dissolved.
4. Continue warming the mixture for another 10 minutes.
5. Remove the flask from the hot water bath and add 10 cm<sup>3</sup> of crushed ice and some distilled/deionised water to break down any unreacted ethanoic anhydride.
6. Stand the flask in a beaker of iced water until precipitation appears to be complete.
7. Filter off the product using a Büchner funnel and suction apparatus.
8. Wash the crystals with the minimum volume of iced water.
9. Recrystallise the aspirin in the minimum volume of a mixture of ethanol to water (1 : 3).
10. Filter and dry.
11. Measure the mass of the pure, dry crystals.
12. Measure the melting temperature of the product using melting temperature apparatus.

**Analysis of results**

- Record the mass of dry aspirin obtained and its melting temperature range.

**Learning tip**

- Percentage yield =  $\frac{\text{actual yield}}{\text{expected yield}} \times 100$

**Questions**

- Which functional group of the 2-hydroxybenzoic acid reacts with the ethanoic anhydride?
- Draw the structural formulae for the reactants and product involved in the formation of aspirin from 2-hydroxybenzoic acid.
- Calculate the relative molecular masses of 2-hydroxybenzoic acid and aspirin.
- Calculate the theoretical yield.
- Calculate the percentage yield.
- Why might the apparent yield be higher?
- What would you expect to be the main impurity in your sample?
- The actual melting temperature of aspirin is 136 °C. Is this similar to the value you recorded? Why do you think there might have been a difference?

## Core practical 16: Synthesise aspirin from 2-hydroxybenzoic acid

### Objectives

- To perform and explain the reactions of acid anhydrides
- To synthesise aspirin from 2-hydroxybenzoic acid

### Safety

- Use eye protection.
- Ethanoic anhydride is corrosive.
- Concentrated sulfuric acid is corrosive.
- 2-hydroxybenzoic acid is harmful.
- Consult CLEAPSS Hazcards<sup>®</sup> 39, 52 and 98A. Perform a risk assessment using up-to-date information before this practical is carried out.

### Equipment per student/group

### Notes on equipment

10 cm <sup>3</sup> ethanoic anhydride	Corrosive
2 g 2-hydroxybenzoic acid	Harmful
1 cm <sup>3</sup> concentrated sulfuric acid	Corrosive
distilled/deionised water	
two 10 cm <sup>3</sup> measuring cylinders	
condenser	
pear-shaped flask	This needs to be clamped (stand, clamp and boss).
stand, clamp and boss	
two 250 cm <sup>3</sup> beakers	
dropping pipette	
ice	Crushed ice would be ideal.
Bunsen burner, tripod, gauze and safety mat	
mass balance (2 d.p.) and weighing boats	
Büchner funnel, Büchner flask, water/suction pump and filter paper to fit funnel	
melting temperature apparatus and melting temperature tube	

### Notes