## The preparation of aspirin – teacher notes

## Introduction

## In this activity you use ethanoic anhydride to convert 2-hydroxybenzoic acid into aspirin.

## Diagram Description automatically generated

The reaction takes place easily in acidic solution but the product is formed as part of a mixture containing several other compounds. The product is formed in Stage 1 below and then separated from impurities in Stage 2.

Note that ethanoic anhydride reacts readily with water so all the apparatus must be dry.

## Apparatus

* Access to a fume cupboard
* Pear shaped flask, 25 cm3
* Hot water bath
* Measuring cylinder, 10 cm3
* Bath of iced water
* Glass stirring rod
* Buchner funnel and suction apparatus
* Watch glass

## Chemicals

* 2-Hydroxybenzoic acid, 1 g
* Ethanoic anhydride, 2 cm3
* Eight drops of concentrated phosphoric acid

## Health, safety and technical notes

* Read our standard health and safety guidance here <https://rsc.li/3AlERrN>
* Wear eye protection.
* Sulfuric acid can be used in place of phosphoric acid but may give lower yields.
* Some teachers have reported problems which were due to using ethanoic anhydride that had already been hydrolised to ethanoic acid. Add a drop to water to ensure it is still reactive.
* If no precipitate appears, scratch the inside of the beaker with a glass rod or add a seed crystal of aspirin.
* As much as 40% of the mass of product after filtering may be water. Overnight drying is preferable to oven drying.
* Students should obtain about 0.9 g of crude product from 1.0 g of 2-hydroxybenzoic acid.

Relative molecular masses are:

* 2-hydroxybenzoic acid: 138
* ethanoic anhydride: 102
* aspirin: 180

## Further investigations

Vary the reaction conditions to investigate the effect on percentage yield of:

* type of acid catalyst;
* concentration or volume of acid used;
* time of heating/cooling; and
* relative amounts of reagents

Use thin-layer chromatography (tlc) to investigate the purity of the product, using commercial aspirin as a reference. Ensure that this aspirin sample is not ‘soluble aspirin’ (sodium or calcium salt).

## Test for impurities

* Three test-tubes together with means of labelling them;
* A few crystals of the following substances:
  1. Phenol (NB phenol is a toxic substance; avoid spillage and wash hands after use)
  2. 2-Hydroxybenzoic acid (salicylic acid)
  3. Crude product from Activity 3
  4. Pure aspirin
* One per cent iron(III) chloride solution.

## Results

|  |  |
| --- | --- |
| Phenol + Fe3+ (aq) | Purple solution |
| 2-Hydroxybenzoic acid + Fe3+(aq) | Purple solution |
| Crude product + Fe3+(aq) | May have a purple tinge due to unreacted 2-hydroxybenzoic acid |
| Pure product + Fe3+(aq) | Very pale yellow |

## Answers

1. The crude product may contain 2-hydroxybenzoic acid, as well as water or ethanoic acid as impurities. 2-Hydroxybenzoic acid can be formed either from incomplete reaction or from hydrolysis of the product during its isolation.
2. ![Diagram, radar chart

   Description automatically generated]()

The OH group attached to the benzene ring produces a purple colour with Fe3+(aq) ions. The OH group in aspirin is part of the carboxylic acid group and does not react in the same way.