

## Making Aspirin

**Aim:** My aim is to synthesise an organic compound and determine its melting point.

### Background information:

Aspirin (2-(acetyloxy) benzoic acid) is a drug originating from the family of salicylates, used as an analgesic: drugs used against minor pains and aches, or antipyretic: drugs used against fevers. It also has an anticoagulant effect and is used in long lasting small-doses to prevent heart attacks. These doses block the formation of thromboxane in platelets. It eliminates platelet aggregation and thins the blood, thereby reducing the risk of a heart attack. Its molecular structure is shown below:



Aspirin produced for the purpose of blood thinning can be bought in 75 mg or 81 mg tablets. High doses are usually given instantly after a severe heart attack. These doses may also eliminate the synthesis of prothrombin, thus acting as another type of anti-coagulant.

However, an overdose can have fatal consequences. Initially, stronger doses cause ulcers and stomach bleeding. This has an affect menstruating women where the bleeding increases. Aspirin is no longer employed to control flu-like symptoms.

### Prediction:

I predict that we will produce 60% of aspirin in the experiment.

### Fair Test:

- Make sure all the chemicals are put in equal proportions for an effective yield outcome.

### Apparatus:

- 2g of 2-hydroxybenzoic acid (salicylic acid)
- 4cm<sup>3</sup> of ethanoic anhydride
- 85% Phosphoric acid

- 50cm<sup>3</sup> pear shaped flask
- Reflux condenser
- Weighing machine
- Bunsen burner
- Beaker
- Water
- Spatula
- Melting point apparatus
- Ice bath
- Pipette
- Capillary tubes to hold the aspirin powder to investigate its melting point.
- Measuring cylinder
- Clamp
- Goggles
- Ice with 250cm<sup>3</sup> beaker of crushed ice

### Safety:

- Proceed with caution when handling the Bunsen burner as the flames are dangerous and can burn
- Remove any obstructions. Tuck stools under work benches and store bags on a side table or under the table
- Take care when using the acid, they may be corrosive, irritants or flammable
- Reflux condenser maybe hot because of the heating process

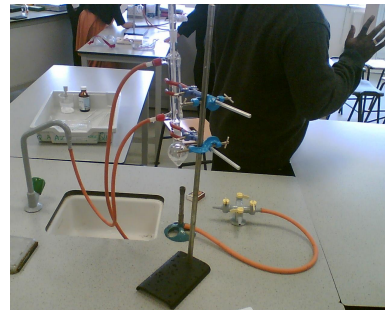
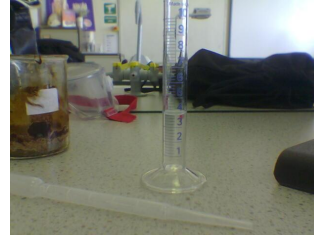
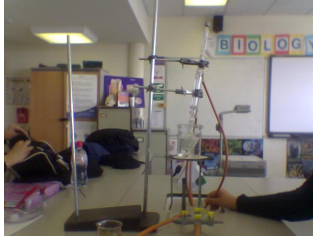
### Method:

1. Gather all the necessary equipment
2. Add 2g of 2-hydrobenzoic acid into a weighing bottle and weigh it. Add 4 cm<sup>3</sup> ethanoic anhydride into the pear shaped flask.
3. Add five drops of the 85% phosphoric acid into the mixture using a pipette and measuring cylinder. Whirl the mixture
4. Secure the flask onto the reflux condenser and heat the mixture on a bunsen burner for approximately 4 – 5 minutes
5. Add 2 cm<sup>3</sup> of water from the condenser to
6. Pour the mixture into 40 cm<sup>3</sup> cold water into a 100 cm<sup>3</sup> beaker to initiate crystallisation.
7. Place the mixture in a small beaker into an ice bath to complete the crystallisation process.
8. Collect the product using suction filtration. Wash it with distilled water.

### Images:



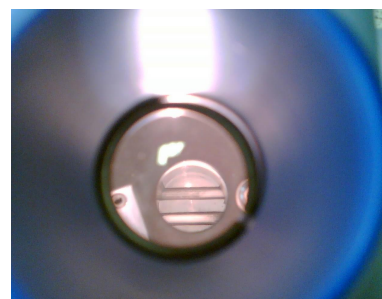
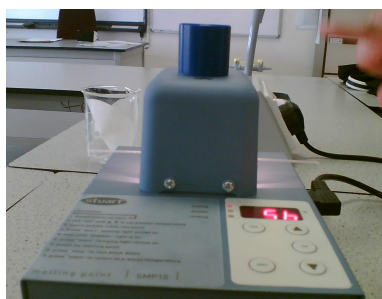
β The images display what apparatus was utilised during the experiment. The chemicals are shown in the first two images, the third β shows the assembled reflux apparatus, and the third shows the measuring cylinder and pipette used for the phosphoric acid. à



The first image among these two shows how the bunsen burner was used in the experiment to heat up the mixture. The clamp held the reflux apparatus in two places instead of one to ensure the stability of the apparatus and prevention from breaking. The second shows the disassembly of the apparatus.



The above pictures show how the experiment was unsuccessful the first time round. The crystals were burnt because of over heating and developed a brown fungus, therefore we had to repeat the experiment and were successful.



The first image shows the equipment utilised to measure the melting point of aspirin. The second shows what we saw through the eyepiece when the aspirin was still in its powder state. The powder took a considerable amount of time to melt, as the melting point was very high.

### Results:

When the crystals were obtained, we had to recrystallize the product. I dissolved the crystals in a small amount of a hot solvent and filtered it in a pre-heated funnel, which allowed the filtrate to crystallise. After this, I used vacuum distillation to dry the products and allowed the crystals to dry in a desiccator. By going through this process, we obtained 3.3g of aspirin. Below are the results obtained from melting the aspirin:

	Temperature	Change in state
As mentioned, we obtained 3.3g of aspirin. If a 100% of aspirin results in obtaining 5.3g of the product, I can use this information to calculate the percentage of 3.3g:	50°C	No change
	60°C	No change
	70°C	No change
	80°C	No change
	90°C	No change
	100°C	No change
	110°C	No change
	120°C	No change
	125°C	Aspirin has almost fully melted
	130°C	Aspirin melts
	140°C	Continues melting and reduces in content
	150°C	Aspirin at 150°C and continues to melt, but with little substance left.

$$5.3\text{g} = 100\%$$

$$1 \text{ g} = 18.8\%$$

Therefore:  $3.3\text{g} \times 18.8\% = 62.04\%$

### Evaluation

I learnt from this experiment that the melting point of aspirin is between 125 °C and 130 °C. This was ideal as the stated melting point is approximately 130 °C, thereby proving that the experiment was successful.

The experiment was moderately straight forward but we had initially faced difficulties. At first the experiment didn't go well as the aspirin had become brown and contained fungus on it instead of appearing white.

So we repeated the experiment and instead of using a bunsen burner, we placed the pear-shaped flask into boiling water and allowed the crystallisation process to take place.

The apparatus was moderately straight forward to use, however I did face some difficulty in assembling the reflux apparatus. The rubber tubing was difficult to secure on to it and the clamp at first wouldn't hold the ensemble together. However, once I overcame these difficulties, I was able to continue the experiment smoothly.

I didn't enjoy this experiment as much as I had anticipated, mainly due to the fact that the first attempt was unsuccessful. Although I enjoyed the melting process as it showed an immediate change of state rapidly from a powder form to liquid.

During the experiment, I observed that the water bath procedure produced more accurate results. Aspirin has a high melting point and after being removed from the melting apparatus, it solidifies but not in its original powder state.

The calculations show that the experiment wasn't entirely correct. I predicted that we would obtain 60% of the aspirin as working in school laboratory conditions wouldn't produce the full amount. However, according to the calculations we obtained 3.3g i.e. 62.04% of the aspirin. The results were probably slightly inaccurate because of human error and as mentioned, working in a laboratory condition where the supply for the products is limited for safety reasons.