

## Teacher Resource Bank

GCE Chemistry

PSA16: A2 Organic Chemistry

- Prepare a solid organic product



## Technical Sheet

### To prepare a sample of aspirin

Whenever possible, students should work individually.

If it is essential to work in a pair or in a small group, because of the availability of apparatus, supervisors must be satisfied that they are able to assess the contribution from each student to the practical activity.

**This PSA is likely to require two practical sessions.**

#### Requirements

##### **Part 1**

- salicylic acid
- 100 cm<sup>3</sup> conical flask
- 10 cm<sup>3</sup> measuring cylinder
- ethanoic anhydride
- concentrated sulfuric acid in a dropping bottle
- 400 cm<sup>3</sup> beaker
- Tripod, gauze and Bunsen burner
- Thermometer (-10 °C to 110 °C)
- 250 cm<sup>3</sup> beaker
- Reduced pressure filtration apparatus
- Filter paper
- Glass stirring rod
- Deionised or distilled water in a wash bottle
- Spatula

##### **Part 2**

- 25 cm<sup>3</sup> measuring cylinder
- Boiling tube
- ethanol
- Thermometer (-10 °C to 110 °C)
- Deionised or distilled water in a wash bottle
- 250 cm<sup>3</sup> beaker
- 100 cm<sup>3</sup> conical flask
- Glass stirring rod
- A kettle

Salicylic acid is unpleasant to work with and there is a hazard associated with skin contact, which should be avoided.

**Centres are expected to carry out and be responsible for their own safety risk assessments.**



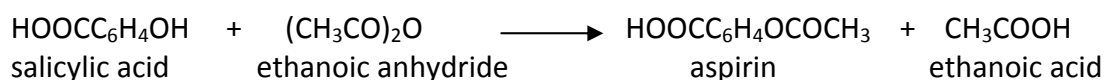
## Student Sheet

**The aim of this experiment is to prepare a sample of aspirin**

### Introduction

Aspirin is prepared by the acylation of salicylic acid (2-hydroxybenzenecarboxylic acid) using ethanoic anhydride as the acylating agent.

The reaction can be represented as follows.



Aspirin (2-ethanoylhydroxybenzenecarboxylic acid) is an antipyretic drug (reduces fever by lowering body temperature) and an analgesic (relieves pain).

Aspirin does not react in the acidic conditions in the stomach, but is hydrolysed in the alkaline conditions found in the intestines to produce ethanoate ions and salicylate (2-hydroxybenzenecarboxylate) ions. Salicylates lower the body temperature of feverish patients and have a mild analgesic effect relieving headaches and other pain. The toxic dose is relatively high, but symptoms of poisoning can occur with quite small quantities.

**It is the responsibility of the student to carry out and be responsible for their own safety risk assessment before carrying out this experiment. Wear safety glasses at all times. Assume that all of the reagents and liquids are toxic, corrosive and flammable.**

### Experiment

#### Part 1 The preparation

- Weigh out approximately 6.00 g of salicylic acid directly into a 100 cm<sup>3</sup> conical flask.
- Record the mass of salicylic acid used.
- Using a 10 cm<sup>3</sup> measuring cylinder, add 10 cm<sup>3</sup> of ethanoic anhydride to the flask and swirl the contents.
- Add 5 drops of concentrated sulfuric acid to the flask and swirl the mixture in the flask for a few minutes to ensure thorough mixing.
- Warm the flask for twenty minutes in a 400 cm<sup>3</sup> beaker of hot water at approximately 60 °C. The temperature in the flask should not be allowed to rise above 65 °C.



- f) Allow the flask to cool and pour its contents into 75 cm<sup>3</sup> of water in a beaker, stirring well to precipitate the solid.
- g) Filter off the aspirin under reduced pressure, avoiding skin contact.
- h) Collect the crude aspirin on a double thickness of filter paper and allow it to dry.

**Part 2   Purification**

- a) Using a 25 cm<sup>3</sup> measuring cylinder, measure out 15 cm<sup>3</sup> of ethanol into a boiling tube.
- b) Prepare a beaker half-filled with hot water at a temperature of approximately 75 °C. The safest way to do this is to use a kettle of boiling water and add water from the kettle to cold water in the beaker until the temperature is at approximately 75 °C  
N.B. The boiling point of ethanol is 78 °C and the temperature of the water in the beaker should not be allowed to go above this.
- c) Use a spatula to add the crude aspirin to the boiling tube and place the tube in the beaker of hot water.
- d) Stir the contents of the boiling tube until all of the aspirin dissolves into the ethanol.
- e) Pour the hot solution containing dissolved aspirin into approximately 40 cm<sup>3</sup> of water in a 100 cm<sup>3</sup> conical flask. If a solid separates at this stage, gently warm the contents of the flask in the water bath until solution is complete. You should avoid prolonged heating, since this will decompose the aspirin.
- f) Allow the conical flask to cool slowly and white needles of aspirin should separate.
- g) If no crystals have formed after the solution has cooled to room temperature, you may need to use an ice bath and to scratch the insides of the flask with a glass stirring rod to obtain crystals.
- h) Filter off the purified solid under reduced pressure and allow it to dry on filter paper.
- i) Record the mass of the dry purified solid.

## A2 Organic Chemistry

**PSA16    Prepare a solid organic compound**



### **Analysing the effectiveness of this method of preparation of aspirin**

The ability to process the data is NOT part of the PSA but this is a useful task to complete.

Your teacher can help you with this part of the work.

- Calculate the theoretical yield of aspirin which should be formed from 6.00 g of salicylic acid.
- Calculate the percentage yield of aspirin from your experiment and comment on the reasons for the losses that have occurred during the preparation and the purification of the solid.
- Calculate the atom economy for the preparation of aspirin by this method.
- Consider the reasons why the alternative preparative method which uses ethanoyl chloride rather than ethanoic anhydride, is not favoured by industry even though this alternative method has a higher atom economy.



## Teacher Notes and Marking Guidance

The specific marking guidance in the specification is as follows

**2 marks:** All areas of the task are carried out competently.

The quantities of reagents are appropriate for the preparation.

The apparatus set-up for the preparation is safe and appropriate.

The experiment is carried out safely and produces an appropriate quantity and quality of product.

**1 mark:** One of the areas of the task is performed poorly.

The quantities of reagents are inappropriate for the preparation **OR**

The apparatus set-up for each experiment is unsafe or inappropriate **OR**

The experiment is carried out with insufficient care or the yield is poor.

**0 marks:** At least two of the areas of the task are performed poorly.

The quantities of reagents are inappropriate for the preparation.

The apparatus set-up for each experiment is unsafe or inappropriate.

The experiment is carried out with insufficient care or the yield is poor.

### Guidance for Teachers

Teachers are expected to exercise professional judgement in assessing the competence of their candidates in following the instructions.

Candidates should have been given guidance in the correct use of equipment and this guidance **can continue during the practical session** for which this PSA forms a part.

If, however, the guidance required is fundamental or frequent, then the student should **not** be awarded 2 marks.

Most judgements of 2 marks, 1 mark or 0 marks will depend on whether the candidates are able to measure out the quantities of reagents with due care and attention to the hazards associated with each and whether the heating is carried out safely. The method yields good quality crystals of aspirin and this can be inspected visually.

The yield from the experiment is unlikely to be more than 2 g of product after handling losses.

It is important to remember when marking these practical exercises that PSA is about student competence and that for a student to score full marks on this exercise **perfection is neither expected nor required**.