

NEATH PORT TALBOT COLLEGE
COLEG CASTELL NEDD PORT TALBOT

School of Maths & Science
Science Practical

Preparation and analysis of Aspirin

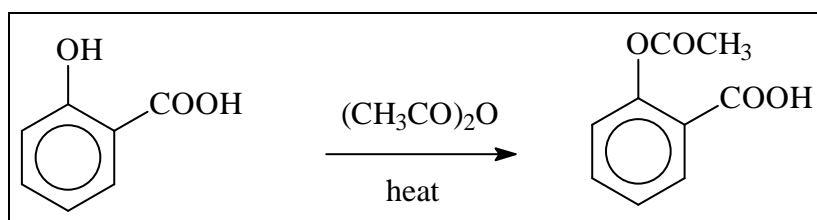
◆ **Aim**

At the end of this practical session you should be able to:

1. Follow a written procedure to prepare a sample of aspirin;
2. Use a melting point apparatus to determine the melting point of aspirin using;
3. Use an infrared spectrometer to record an infrared spectrum;
4. Write a report, in the appropriate style, on the preparation of the aspirin.

◆ **Introduction**

In this practical you carry out a laboratory technique to prepare aspirin. The first stage of the practical involves the conversion of 2-hydroxybenzoic acid to aspirin:



A detailed procedure is given on page 3 of the handout. Once you have prepared aspirin you will assess its purity by measuring its melting point and recording the infrared spectrum.

◆ Safety



Control Measures

- The wearing of **safety glasses, gloves** and a **laboratory coat at all times** will be sufficient to take account of most hazards and significant risks.
- Keep stoppers on bottles as much as is possible.
- Keep flammable liquids away from flames.
- All waste is to be placed in the labelled container immediately after use.
- You are reminded of the need of good laboratory practice in order to maintain a safe working environment.
- **Do not remove** ethanoic anhydride or conc. phosphoric acid from the fume cupboard

Hazards



(Highly) Flammable

Ethanol, ethanoic anhydride



Corrosive

Concentrated phosphoric acid,
ethanoic anhydride



Harmful/ Irritant

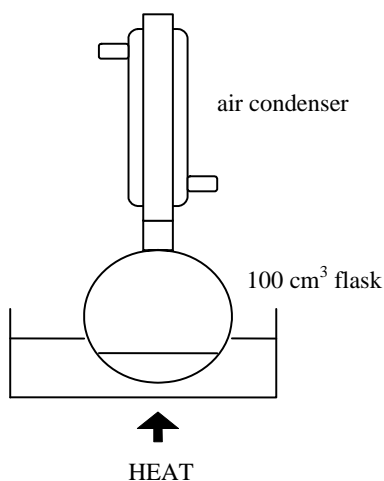
Ethanoic anhydride
(vapour irritating and harmful to eyes)

◆ Procedure

Part 1: Formation. (Wear gloves and safety glasses throughout procedure).

1. Place 3 g of dry 2-hydroxybenzenecarboxylic acid (**record actual mass used**) in a 100 cm³ round bottomed flask and add 7 cm³ of ethanoic anhydride followed by 3 drops of concentrated phosphoric acid (**CARE!**). (Transfer in fume cupboard only).
Fit an air condenser. (See fig 1).
2. Mix the contents of the flask by gentle swirling and then warm to about 50-60 °C on a water bath for 15 minutes.
3. Pour the contents of the flask into a 100 cm³ beaker and cool under running water, occasionally agitating the flask. A paste should form.
4. Add slowly 50 cm³ of distilled water, stir the mixture well with a glass rod and collect the precipitate that forms (your crude sample of aspirin) by suction filtration (Complete in fume cupboard due to irritating vapour).

Fig. 1



Part 2: Re-crystallisation.

5. Transfer the crude product to a clean 100 cm³ conical flask and add 5 cm³ of ethanol followed by 15 cm³ of distilled water.
6. Warm the mixture gently on the steam bath (60-70 °C), with occasional agitation until all the solid dissolves.
7. Cool the solution carefully under a tap. If crystals do not appear cool in ice.
8. Collect the crystals using suction filtration.
9. Dry the re-crystallised product at room temperature in a desiccator and record the mass of aspirin obtained.

Part 3: Analysis.

10. Determine the melting point and record an infrared spectrum of the re-crystallised product.

◆ Results

Mass of 2-hydroxybenzenecarboxylic acidg (record to 2 dp)

Mass of aspiring (record to 2 dp)

◆ Calculation

Since 1 mole of 2-hydroxybenzenecarboxylic acid should give 1 mole of aspirin (see equation on page 1) then:

- no of moles 2-hydroxybenzenecarboxylic acid used = expected moles of aspirin
- expected mass of aspirin can be obtained (*expected moles x molar mass aspirin*)
- **% yield** = $\frac{\text{actual mass of aspirin obtained}}{\text{expected mass of aspirin}} \times 100$

◆ Homework

Write a report on the preparation of aspirin.

Sections to report:

Title

Aim

Procedure

Results

Calculation

Note:

The report should contain the procedure as you actually did it. When you describe the procedure write it in the third person past tense. i.e. **Do not use 'I' or 'We'** in the procedure. The procedure should **NOT** be written in point form.

e.g. *Acceptable:* '3.12 g of 2-hydroxybenzenecarboxylic acid was dissolved placed in a round bottomed flask together with 7 cm³ of ethanoic anhydride

Not acceptable: 'I put about 3 g of 2-hydroxybenzenecarboxylic acid into a flask.....'