A-level Chemistry exemplar for required practical 10 – part a

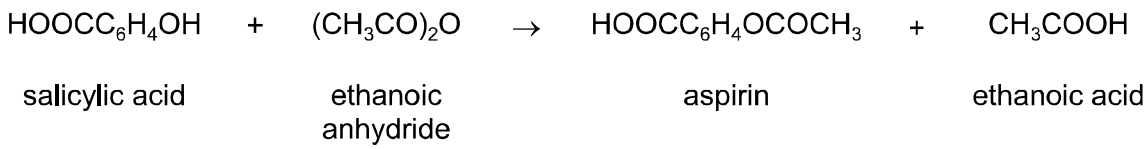
Preparation of an organic solid and a test of its purity:

To prepare a sample of aspirin.

Student sheet

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-hydroxybenzencarboxylate) 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.

Requirements

You are provided with the following:

Part 1

- salicylic (2-hydroxybenzenecarboxylic) acid
- 100 cm^3 conical flask
- 10 cm^3 measuring cylinder
- ethanoic anhydride
- concentrated sulfuric acid in a dropping bottle
- 400 cm^3 beaker
- tripod, gauze and Bunsen burner
- thermometer
- 250 cm^3 beaker
- reduced pressure filtration apparatus
- filter paper
- stirring rod
- deionised or distilled water in a wash bottle
- spatula.

Part 2

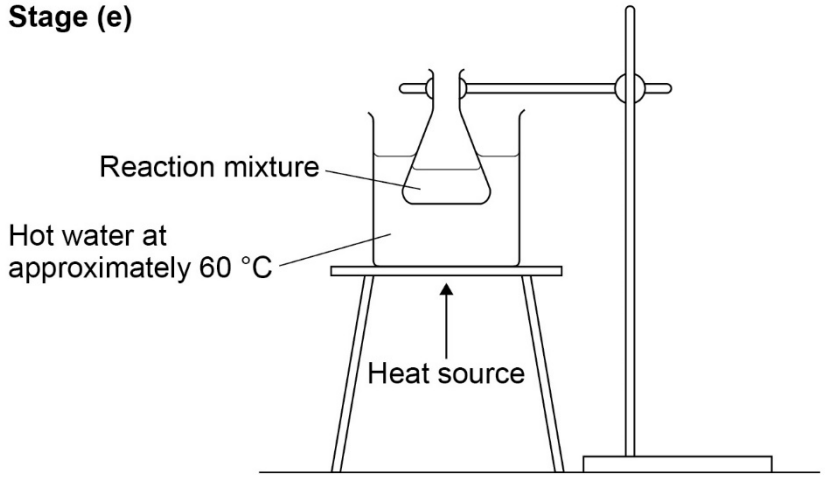
- 25 cm^3 measuring cylinder
- boiling tube
- ethanol
- thermometer
- deionised or distilled water in a wash bottle
- 250 cm^3 beaker
- 100 cm^3 conical flask
- stirring rod
- kettle
- access to a digital mass balance (reading to 2 decimal places).

Suggested method

Part 1 Preparation

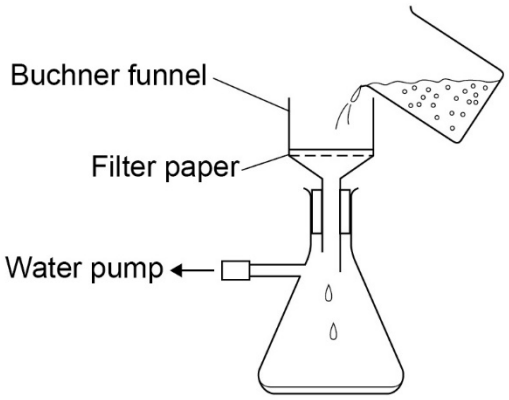
a) Weigh out approximately 6.00 g of salicylic acid directly into a 100 cm^3 conical flask.
b) Record the mass of salicylic acid used.
c) Using a 10 cm^3 measuring cylinder, add 10 cm^3 of ethanoic anhydride to the flask and swirl the contents.
d) Add 5 drops of concentrated sulfuric acid to the flask and swirl the mixture in the flask for a few minutes to ensure thorough mixing.
e) Warm the flask for about 20 minutes in a 400 cm^3 beaker of hot water at approximately 60 °C. The temperature in the flask should **not** be allowed to rise above 65 °C.

Stage (e)



f) Allow the flask to cool and pour its contents into 75 cm^3 of water in a beaker, stirring well to precipitate the solid.
g) Filter off the aspirin under reduced pressure, avoiding skin contact.

Stage (g)



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^3 measuring cylinder, measure out 15 cm^3 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.
NB 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. **Do not scrape the filter paper.**
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^3 of water in a 100 cm^3 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 scratch the insides of the flask with a glass stirring rod to obtain crystals. Cool the whole mixture in an ice bath.
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.

Analysing the effectiveness of this method of preparation of aspirin

a) Calculate the theoretical yield of aspirin which should be formed from 6.00 g of salicylic acid.
b) 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.
c) Calculate the atom economy for the preparation of aspirin by this method.
d) 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.

To test the purity of an organic solid by measuring its melting point

The purity of an organic solid can be determined in part by measuring its melting point and comparing the value with the known Data Book value of the melting point for that compound. A pure dry solid will melt at a precise temperature whereas an impure solid will melt over a **range** of temperatures which are **lower** than the melting point of the pure solid.

Melting point apparatus varies in type from the most simple using an oil bath to the more sophisticated electrothermal devices. In every case, the same general principle applies that the heating of a small quantity of the solid in a thin-walled melting point tube should be undertaken slowly and with care. When melting occurs, the solid should collapse into a liquid without any change in temperature and the way in which this occurs can give a clue to the purity of the solid. Repeat measurements should be taken with further samples of the organic solid to verify the reliability of the value obtained.

The method will not work if the solid decomposes on heating.

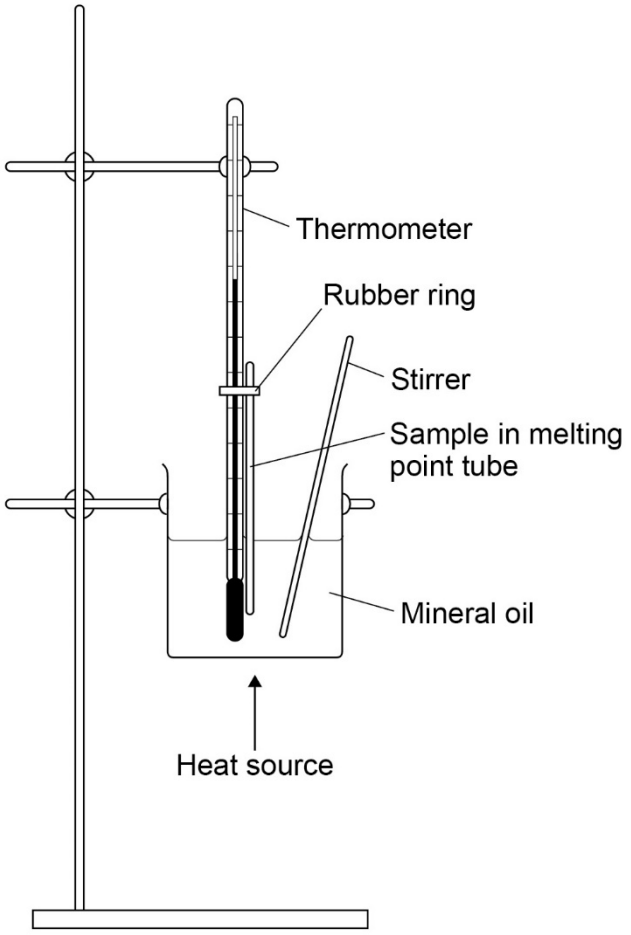
Requirements

You are provided with the following:

- pure benzenecarboxylic acid
- other pure organic solids as desired by the centre
- thermometer (0 °C to 250 °C range)
- melting point apparatus to include either:
  an electrothermal melting point apparatus **or** oil bath (Thiele tube or small beaker half-filled with mineral oil)
- tripod, gauze and Bunsen burner
- rubber ring to attach melting point tube to thermometer (if needed)
- melting point tubes
- watch glass
- spatula.

Suggested method

a) Powder a sample of the organic solid by crushing it gently with a spatula onto the surface of a filter paper.
b) Fill three melting point tubes with the organic solid to a depth of approximately 0.5 cm.
c) Set up the melting point apparatus provided and mount one of the melting point tubes ready for taking a measurement.



d) Heat the apparatus gently and observe the temperature at which the solid collapses into a liquid. The melting point will be in the range 100 °C to 200 °C.
e) Allow the melting point apparatus to cool and repeat the measurement of the melting point of the solid with the other two samples. If the first reading is taken as an approximate value, then the subsequent heating of the other two samples can be done much more slowly as this approximate value is approached.
f) On the basis of the three measurements that you have taken, record the melting point of the organic solid.
g) Ask your teacher for the Data Book value of the melting point for the solid that you have tested and compare this value with your own.

A-level Chemistry exemplar for required practical 10 – part b

Preparation of a pure organic liquid:

The preparation of ethyl ethanoate.

Student sheet

An ester is a chemical compound that is formed when an organic acid reacts with an alcohol. Esters frequently have distinctive odours and are naturally occurring flavour and fragrance chemicals in many fruits and plants. In this practical, the ester ethyl ethanoate is prepared and purified by distillation.

Requirements

Stage 1: Preparation of ethyl ethanoate

- 12 cm^3 glacial ethanoic acid
- 10 cm^3 ethanol
- 15 drops concentrated sulfuric acid
- anti-bumping granules
- disposable dropping pipette
- 10 cm^3 measuring cylinder
- 25 cm^3 measuring cylinder
- 250 cm^3 beaker
- 50 cm^3 pear-shaped flask
- condenser (with rubber tubing)
- clamp stand
- clamps and bosses
- heatproof mat
- Bunsen burner
- tripod and gauze.

Stage 2: Isolation of ethyl ethanoate

- 4.5 g sodium carbonate
- 15 cm^3 distilled or deionised water
- 100 cm^3 beaker
- 50 cm^3 beaker
- separating funnel and stopper
- anhydrous sodium sulfate
- boiling tube
- 50 cm^3 pear-shaped flask.

Stage 3: Purification of the ethyl ethanoate

- thermometer (0–100 °C) and adapter
- three-way adapter
- condenser (with rubber tubing)
- receiver adapter
- anti-bumping granules
- joint clips
- 250 cm^3 beaker
- clamp stand
- clamps and bosses
- heatproof mat
- Bunsen burner
- tripod and gauze
- access to a balance.

Suggested method

Stage 1: Preparation of ethyl ethanoate

a) Put a few anti-bumping granules in a 50 cm^3 pear-shaped flask.
b) In a fume-cupboard, add 10 cm^3 ethanol, 12 cm^3 glacial ethanoic acid and 15 drops of concentrated sulfuric acid to the flask.
c) Place a 250 cm^3 beaker containing some water on a tripod and gauze over a Bunsen burner.
d) Clamp the pear-shaped flask in the beaker of water so that the reaction mixture is below the water line.
e) Add a condenser so that it is set up for heating with reflux. Clamp the condenser. Do **not** insert a stopper.
f) Light the Bunsen burner to heat the hot water bath. Raise the temperature of the hot water until the mixture in the flask is gently boiling. Continue the gentle boil of the reaction mixture for about 15 minutes. Turn off the Bunsen burner and cool the mixture by removing the hot water bath.

Stage 2: Isolation of ethyl ethanoate

g) Prepare a saturated solution of sodium carbonate by combining 4.5 g of sodium carbonate with 15 cm^3 of distilled water in a 100 cm^3 beaker.
h) In a fume cupboard, transfer the reaction mixture from the pear-shaped flask to the beaker and stir.
i) Transfer the mixture to a separating funnel. Stopper it and turn it upside down gently and then open the stopcock to vent the system. Invert at least 15–20 times, opening the stopcock each time.
j) Allow the two layers to separate. Ethyl ethanoate is less dense than water, therefore the top layer is ethyl ethanoate.
k) Remove the stopper, open the stopcock and slowly drain off the waste aqueous layer into a 50 cm^3 waste beaker, then close the stopcock.
l) Transfer the remaining ethyl ethanoate into a dry boiling tube containing about 1 g of anhydrous sodium sulfate. Agitate the tube so that any water is absorbed into the anhydrous solid.
m) Decant the ethyl ethanoate into a clean, dry 50 cm^3 pear-shaped flask.

Stage 3: Purification of the ethyl ethanoate

n) Add a few anti-bumping granules to the pear-shaped flask.
o) Set up the apparatus for distillation.
p) Place the flask in the 250 cm^3 beaker and clamp it so the crude ethyl ethanoate is below the water line.
q) Weigh a clean, dry 100 cm^3 conical flask on an analytical balance. Record the mass in your Data Table.
r) Place the conical flask under the receiver.
s) Light the Bunsen burner and heat the flask in the hot water bath. Heat until the ethyl ethanoate is gently boiling.
t) As the ethyl ethanoate vapours start to carry over and condense, record the temperature of the vapours in a suitable table. Record this temperature at the beginning and end of the distillation.
u) Distil the ethyl ethanoate until no more distillate comes over. There should be some liquid remaining in the round-bottom flask. Never distil to dryness.
v) Turn off the Bunsen burner.
w) Reweigh the conical flask with the distilled ethyl ethanoate.

Stage 4: Yield calculation

Which is the limiting reagent - ethanoic acid or ethanol?

What is the percentage yield from the limiting reagent?