AQA Chemistry A-level

Required Practical 10

Preparation of a pure organic solid, test of its purity, and preparation of a pure organic liquid

Reflux

Reflux: continuous boiling and condensing of a reaction mixture.
Used because: this allows an organic reaction mixture to be heated without losing any reactants or products.

Anti-bumping granules (added to the flask in both distillation and reflux): to prevent vigorous or uneven boiling, by making small bubbles form instead of large bubbles.

Purifying an organic liquid:

Separating funnel

Purpose: to separate into two layers: higher density liquid (typically aqueous) is the bottom layer, and organic product layer above.

- Put the distillate of impure product into a separating funnel
- Wash product by either:
    - Sodium hydrogencarbonate solution, shaking and releasing pressure from CO_2 produced - Sodium hydrogen carbonate removes acidic impurities by neutralisation (converts to H_2O, CO_2 and Na_2SO_4)
    - Saturated sodium chloride solution – helps separate the organic layer from the aqueous layer
- Allow layers to separate in funnel, and then run and discard the aqueous layer
- Run the organic liquid into a clean, dry conical flask and add 3 spatula loads of drying agent (anhydrous sodium sulphate – drying agent should be insoluble in the organic liquid and not react with the organic liquid) to dry the organic liquid
- Carefully decant the liquid into the distillation flask
- Distil to collect pure product

Anhydrous calcium chloride is a drying agent and soaks up any remaining water – the liquid will remain cloudy until all the water drops have been removed.

Melting point:

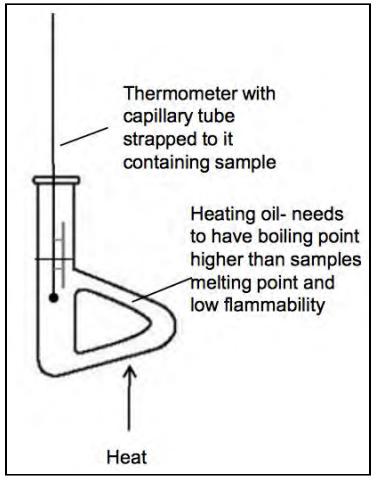
If the sample is very pure then the melting point will be sharp (same value as quoted in data books).

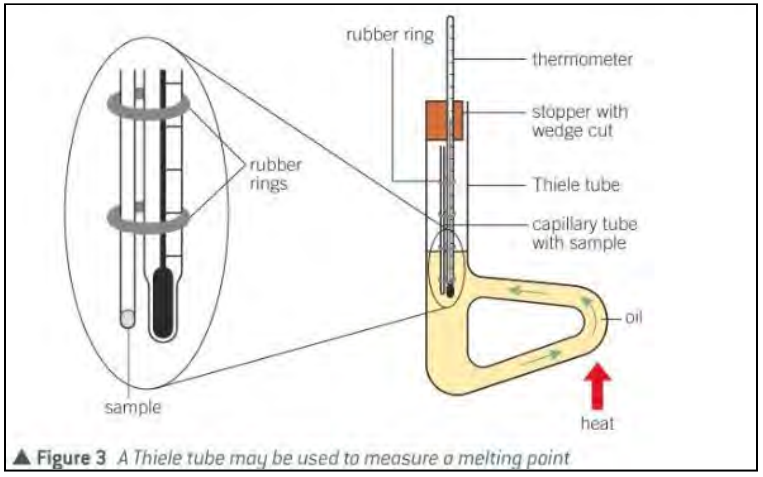
If impurities are present (and this can include solvent from the recrystallisation process) the melting point will be lowered and the sample will melt over a range of several degrees.

Can be measured in an electronic melting point machine or by using a practical set up where the capillary tube is strapped to a thermometer immersed in some heating oil. In both cases a small amount of the salt is put into a capillary tube. The tube is heated up and is heated slowly when near the melting point.

Compare experimentally determined melting point value with one quoted in a data source to determine purity.

Error may occur if the temperature on the thermometer is not the same as the temperature in the actual sample tube.





▲ **Figure 3** *A Thiele tube may be used to measure a melting point*

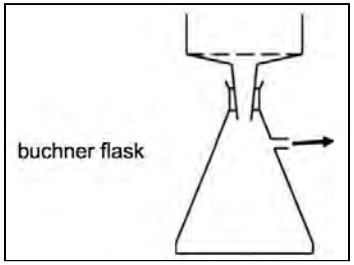
Measuring boiling point:

To determine purity of a liquid

- This can be done in a distillation set up or by simply boiling a tube of the sample in an heating oil bath.
- Pressure should be noted- changing pressure can change the boiling point of a liquid.
- Not the most accurate method of identifying a substance as several substances may have the same boiling point.
- To get a correct measure of boiling point the thermometer should be above the level of the surface of the boiling liquid and be measuring the temperature of the saturated vapour.

Recrystallisation:

Method	Accuracy	Explanation
1. Dissolve the impure compound in a minimum volume of hot (near boiling) solvent.	An appropriate solvent is one which will dissolve both compound and impurities, when hot, and one in which the compound itself does not dissolve well when cold. The minimum volume is used to obtain saturated solution and to enable crystallisation on cooling.	So the desired compound is pure in crystals formed.
2. Hot filter solution through (fluted) filter paper quickly.		This step will remove any insoluble impurities and heat will prevent crystals reforming during filtration
3. Cool the filtered solution by inserting beaker in ice.	Cool slowly to increase yield by ensuring all compound crystallizes. Ice will increase the yield of crystals	Crystals will reform but soluble impurities will remain in solution form because they are present in small quantities so solution is not saturated.
4. Suction filtrate with a Buchner flask to separate out crystals.		The water pump connected to the Buchner flask reduces the pressure and speeds up the filtration.
5 Wash the crystals with distilled water.		To remove soluble impurities.
6. Dry the crystals between absorbent paper.	To remove excess water.	Water would affect % yield.

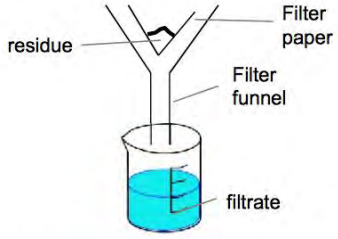


Loss of yield in this process:

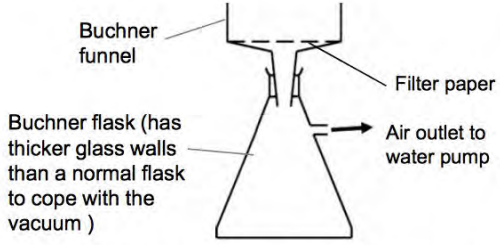
- Crystals lost when filtering or washing
- Some product stays in solution after recrystallization
- Other side reactions occurring

When making an insoluble salt, normally the salt would be removed by **filtration,** washed with **distilled water to remove soluble impurities** and then **dried on filter paper**

Filtration



This is gravitational filtration. Use if small amounts of solid are formed.



This is vacuum filtration. The apparatus is connected to a water pump which will produce a vacuum. Use if larger amounts of solid are formed.

For both types of filtration apparatus AQA expect filter paper to be drawn on the diagram