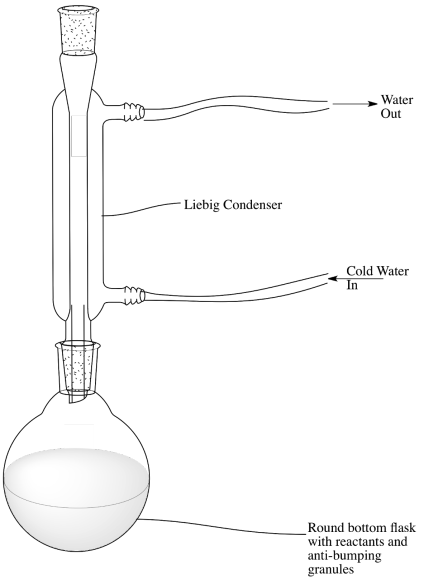
OCR (B) Chemistry A-Level

PAG 06: Synthesis of an organic solid

6.2 Preparation of benzoic acid

Equipment list (Part 1 and 2)

- Measuring cylinder (10 cm^3)
- Dropping pipettes
- Quickfit apparatus:
  - pear-shaped or round-bottom flask
  - Liebig condenser and tubing
- 250 cm^3 beaker
- Glass rod
- Anti-bumping granules
- Apparatus for filtration under reduced pressure (Buchner funnel and Buchner flask)
- Filter papers to fit the above
- Balance accurate to two decimal places
- Boiling tube
- Melting point apparatus
- Capillary tubes
- TLC plate
- Sample tube and lid
- Bunsen burner
- Distilled water
- Ethanol
- Methyl orange
- Methyl benzoate
- HCl (2 mol dm^{-3})
- NaOH (2 mol dm^{-3})



Method

Part 1: Preparation

1. Add 2.0 cm^3 of methyl benzoate into a pear-shaped flask. Add 10 cm^3 of the sodium hydroxide, 10 cm^3 of ethanol and a few anti-bumping granules. Attach to condenser.
2. Heat the flask **gently** for 5 minutes.
   Then boil under **reflux** for another 15 minutes.

Heating under reflux prevents the flask from boiling dry by preventing reagents from evaporating/escaping. It also ensures even heating.

3. Allow the flask to cool and then remove the solution by pouring into a beaker.
4. Rinse the flask with distilled water into the beaker so no product is lost.
5. Add ten drops of methyl orange to the solution and then acidify with the hydrochloric acid.

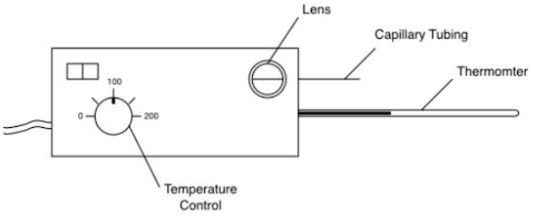
Solid benzoic acid crystallises.

6. Filter the mixture under reduced pressure to obtain an impure benzoic acid.

Part 2: Recrystallisation and melting point

1. Add the impure benzoic acid to a boiling tube.
2. Using a boiling tube in a water bath, purify the benzoic acid via recrystallisation using a **minimum** volume of boiling water.
3. Then cool the boiling tube and its contents. Filter the purified benzoic acid under **reduced pressure.**
   Place on a watch glass and allow to dry.
4. Weigh the **dry** product and record yield.
5. Determine the melting point of the pure benzoic acid.

To determine the melting point, place a small amount of product in a capillary tube and melt in your chosen apparatus, measure the temperature using a thermometer. An impure product will melt over a **range** of temperatures (usually lower than the pure product) and the pure product will melt at a either a single temperature or over a much smaller temperature range.



Errors (how yield is lost in this process)

- ❏ Crystals are lost when filtering.
  When filtering, make sure to wash the buchner funnel, flask and the vessel you are using to transport the product into the filtration system with solvent.
- ❏ Some product stays in solution after recrystallisation.
  Although you are using a **minimum** amount of solvent, you still need to ensure you are using a sufficient amount to dissolve all the crystals.
- ❏ Other side reactions occurring
  Purify more than once to get rid of these other products, however there is little you can do to prevent loss of yield due to this issue other than using purer starting reactants to begin with.

Part 3: Thin layer chromatography, TLC

Equipment list (Part 3)

- UV light or container with iodine as locating agent.
- Thin layer chromatography plate (silica-coated, fluorescent)
- Beaker (100 cm^3)
- Pencil

- Dropping pipette
- Melting point tubes
- Chromatography solvent
- Watch glass or other suitable cover for the beaker

Method

1. Prepare your TLC plate by drawing a pencil line approximately 1 cm from the bottom. Then mark three crosses along the line.
2. Pour the solvent into a beaker to a depth below the pencil line and cover the beaker e.g. with a watch glass.
3. Dissolve some of the methyl benzoate in ethanol.
4. Dip a fine dropping pipette into the solution, then dab it onto the first cross on the TLC plate. Allow the spot to dry. Repeat a few times.
5. Add dissolved samples of the impure and recrystallised benzoic acid to the other crosses.
6. Place the TLC plate in the beaker and cover with a watch glass. Let the solvent to rise up the plate. When the solvent has nearly reached the top, remove the plate from the beaker and mark the line of the solvent front with a pencil.
7. Let the plate dry in a fume cupboard.
8. Place the plate under a UV light and circle the locations of the substances using a pencil.
9. Calculate the R_f values for each visible substance.

Risk Assessment (all parts)

Hazard	Risk	Control
Methyl benzoate	Harmful if swallowed.	Wear safety glasses, don't place food or drink near the substance, wear gloves if handling.
2 mol dm^{-3} NaOH	Corrosive to skin, eyes etc.	Wear gloves, safety glasses and a lab coat. Keep away from the edge of surfaces and don't breathe in.
2 mol dm^{-3} HC*l*	Irritant to skin and eyes.	Wear gloves, safety glasses and a lab coat. Keep away from the edge of surfaces.
Ethanol	Flammable.	Wear safety glasses, handle with care and keep away from a naked flame.
Chromatography solvent	Highly flammable.	Wear safety glasses, handle with care and keep away from a naked flame.

Samples of the solid product from Parts 1 and 2	Irritant.	Wear safety glasses, handle with care and with gloves on.
Methyl orange indicator	Flammable and toxic	Handle with care, keep away from a naked flame. Don't consume. Keep away from food and drink and wear gloves when handling.