

OCR (A) Chemistry A-Level

PAG 5: Synthesis of an organic liquid



5.1 Synthesis of an haloalkane

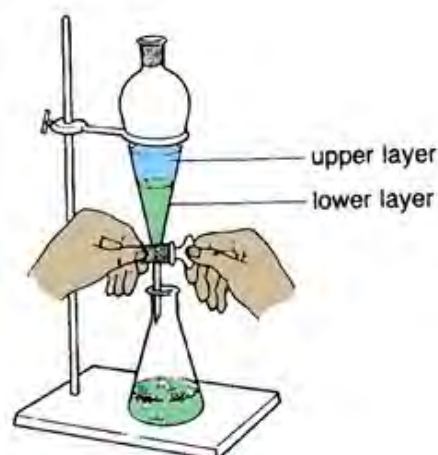
Method

Part 1 : Preparation

1. Into a 10 cm³ measuring cylinder pour about 6.5 cm³ of 2-methylpropan-2-ol.
2. Weigh the measuring cylinder with the contents inside.
3. Pour the 2-methylpropan-2-ol into a separating funnel of 50 cm³.
4. Weigh the empty measuring cylinder to deduce the mass of 2-methylpropan-2-ol.
5. Measure 20 cm³ of concentrated hydrochloric acid.
6. Gradually add the acid to separating funnel.
7. Place the stopper on the separating funnel and shake it vigorously, releasing the pressure when required, for 20 minutes.

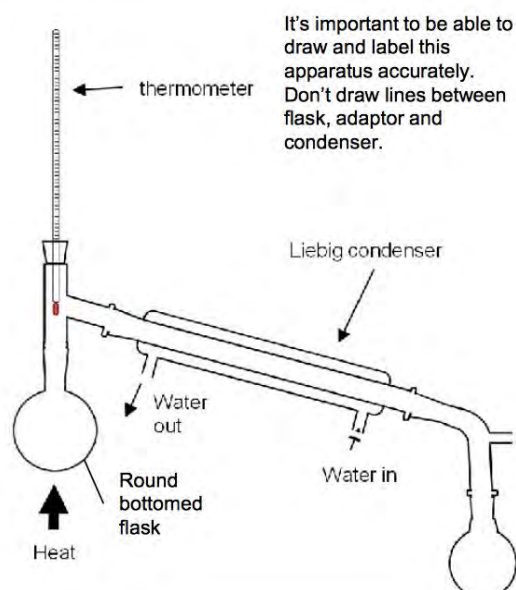
Part 2 : Separation

1. Let the mixture to separate.
2. Once separated, remove the stopper and open the tap to collect the bottom aqueous layer.
3. Close the tap and add 10 cm³ of 5% sodium hydrogencarbonate to the separating funnel.
4. Shake the mixture in the funnel gently and release the pressure as required.
5. Let the mixture to separate and remove the aqueous layer.
6. Continue adding hydrogen carbonate and repeat the steps until there is no pressure build up.
7. Collect the organic layer into a 100 cm³ conical flask.
8. Add anhydrous sodium sulfate slowly while swirling the conical flask, until the liquid is clear.



Part 3 : Distillation

1. Weigh an empty sample collecting tube.
2. Set up the apparatus as shown in the diagram.



3. Add the sample in the conical flask to the round bottom flask and add some anti bumping granules.
4. Using a small beaker, collect the liquid impurities that comes through the condenser.
5. When the temperature reaches 48°C collect the liquid using the weighted sample tube until there is no more liquid coming through the condenser.
6. Weigh the sample tube to calculate the mass of the product.

Note that:

- A water bath or electric heater should be used to heat the mixture if there are flammable substances present.
- Anti-bumping granules prevent large bubbles from forming and ensure that the liquid doesn't boil too vigorously as this would result in the mixture boiling over into the condenser and undesired impurities would contaminating the product.
- The condenser should be tilted slightly down, so any liquid can run into the collection flask.
- The bulb of the thermometer should be at the T junction connecting to the condenser to measure the correct boiling point.
- The water must enter at the lowest point and leave at the highest point to go against gravity as this ensures that water fills the condenser (prevents backflow of water), maximising heat transfer for condensation (more efficient cooling).
- The collection flask must not be sealed to the condenser, the system should not be air tight because as it is heated the air inside the system expands. If it is air tight then the air cannot escape and may cause the apparatus to crack.

Safety

- 2-methylpropan-2-ol - highly flammable liquid; harmful if inhaled.
- Concentrated hydrochloric acid - causes severe skin burns and eye damage:
 - Wear goggles and gloves when handling.
 - Should be handled within the fume cupboard.
- Wear eye protection throughout.

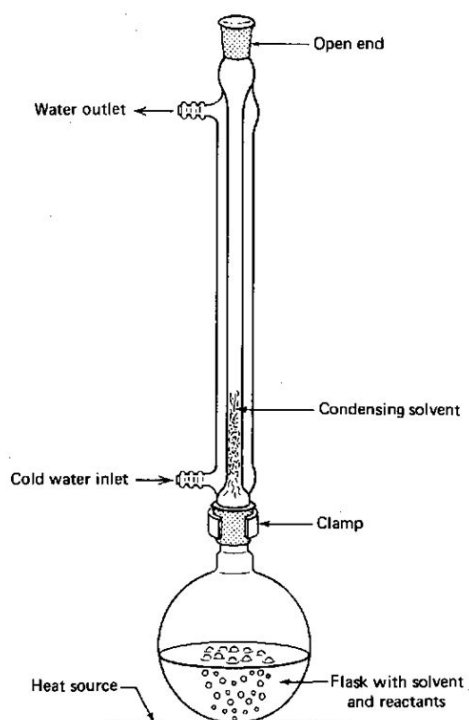
5.2 Preparation of cyclohexene



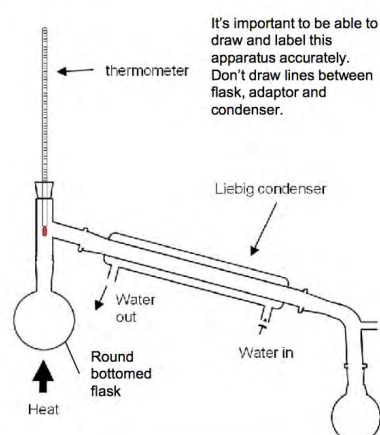
Method

Part 1 : Preparation

1. Pour 10 cm³ of cyclohexanol into a 50 cm³ pear-shaped or round bottom flask
2. Using a plastic graduated dropping pipette, carefully add approximately 4.0 cm³ of concentrated phosphoric acid to the flask.
3. Add a few anti bumping granules to the flask and then assemble the reflux apparatus as shown in the diagram.



4. Heat the flask gently at about 70°C for 15 -20 minutes ensuring that all the condensation happens in the lower half of the condenser.
5. Stop the heating and allow the apparatus to cool before assembling for distillation as shown in the diagram.
6. Slowly heat the flask, distilling over any liquid which boils between 70°C and 90°C.



Part 2 : Purification



1. Pour the distillate into a separating funnel and add equal amount of saturated sodium chloride solution.
2. Shake the mixture and allow the two layers to separate.
3. Carefully run off the lower layer into a beaker (for later disposal) and then transfer the upper layer, which contains the crude cyclohexene, into a small conical flask.
4. Add a few lumps of anhydrous calcium chloride or to the crude cyclohexene to remove water.
5. Stopper the flask, shake the contents and allow this to stand until the liquid becomes clear.
6. Decant the liquid into a clean, dry sample container that has been weighed.
7. Reweigh the container, calculate the mass of dry cyclohexene produced and determine the percentage yield of the product.
8. Test the distillate using bromine water, to confirm that it contains an alkene. (Goes colourless if present).

Further improvement

9. Redistill the cyclohexene collecting the distillate produced between 81°C and 85°C to obtain a further pure cyclohexene.

Note

- A water bath or electric heater should be used to heat the mixture if there are flammable substances present.
- Anti-bumping granules prevent large bubbles from forming and ensure that the liquid doesn't boil too vigorously as this would result in the mixture boiling over into the condenser and undesired impurities would contaminate the product.
- The condenser should be tilted slightly down, so any liquid can run into the collection flask.
- The bulb of the thermometer should be at the T junction connecting to the condenser to measure the correct boiling point.
- The water must enter at the lowest point and leave at the highest point to go against gravity as this ensures that water fills the condenser (prevents backflow of water), maximising heat transfer for condensation (more efficient cooling).
- The collection flask must not be sealed to the condenser, the system should not be air tight because as it is heated the air inside the system expands. If it is air tight then the air cannot escape and may cause the apparatus to crack.

Safety

- Cyclohexanol - harmful if inhaled or swallowed.
 - Ensure the lab is well ventilated.
- Concentrated phosphoric acid - causes severe skin burns and eye damage.
- Anhydrous calcium chloride - causes serious eye irritation.
 - Wear eye protections throughout the experiment.
- Cyclohexene - highly flammable and may be fatal if swallowed.
- Wear chemical resistant gloves.
- When using the separating funnel ensure the stopper is firmly held in place and the nozzle is pointing away from everyone else.

