

OCR (A) Chemistry A-level

PAG 5: Synthesis of an Organic Liquid

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5.1 Synthesis of a Haloalkane

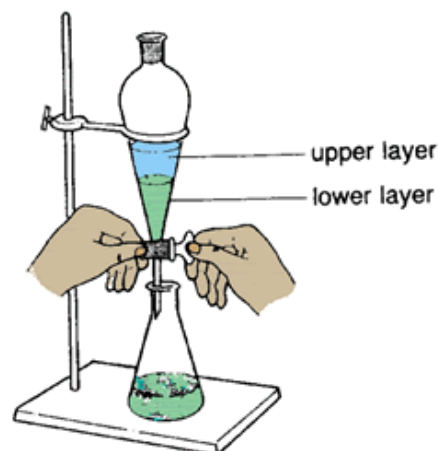
Method

Part 1: Preparation

1. Pour about 6.5 cm³ of 2-methylpropan-2-ol into a 10 cm³ measuring cylinder then weigh the measuring cylinder and its contents.
2. Pour the 2-methylpropan-2-ol into a 50 cm³ separating funnel.
3. Reweigh the empty measuring cylinder to deduce the mass of 2-methylpropan-2-ol used in the reaction.
4. Measure 20 cm³ of concentrated hydrochloric acid and gradually add the acid to the separating funnel.
5. Place the stopper on the separating funnel and shake it vigorously for 20 minutes, releasing the pressure when required.

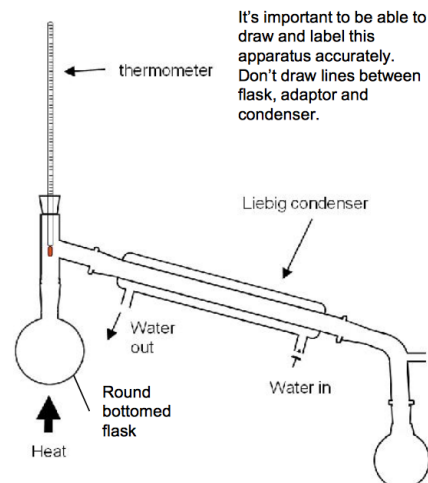
Part 2: Separation

1. Allow the mixture to separate.
2. Once separated, remove the stopper and open the tap to collect the bottom aqueous layer.
3. Once all the aqueous layer has been removed, close the tap and add 10 cm³ of 5% sodium hydrogencarbonate to the organic layer in the separating funnel.
4. Shake the mixture in the funnel gently, releasing the pressure when required.
5. Allow the mixture to separate and remove the bottom aqueous layer.
6. Repeat steps 3 to 5 until there is no pressure build up in the separating funnel.
7. Collect the organic layer into a 100 cm³ conical flask.
8. Add anhydrous magnesium sulfate slowly while swirling the conical flask, until the magnesium sulfate stops clumping.
9. Filter the solution through a filter paper into a clean round-bottom flask.



Part 3: Distillation

1. Weigh an empty sample tube.
2. Set up the apparatus as shown in the diagram.
3. Add the organic sample from the conical flask with anti-bumping granules to the round bottom flask and connect it to the apparatus.
4. Collect the liquid impurities that come through the condenser below 48°C in a small beaker.



- When the temperature reaches 48°C, collect the liquid from the condenser in the pre-weighed sample tube until no more liquid comes through the condenser.
- Weigh the sample tube and calculate the mass of the product.

Key Points

- 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. If anti-bumping granules weren't used, the mixture would boil over into the condenser meaning impurities would contaminate the product.
- The condenser should be tilted down slightly to allow any liquid to run into the collection flask.
- The bulb of the thermometer should be at the T junction connecting to the condenser to correctly measure the boiling point.
- The water must enter at the lowest point and leave at the highest point of the condenser to ensure that it is filled with water. This will maximise heat transfer for condensation so that cooling is more efficient.
- The collection flask must not be sealed to the condenser as this would make the system airtight. As heating occurs, air inside the system expands so air must be able to escape to avoid cracking the apparatus.

Safety

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

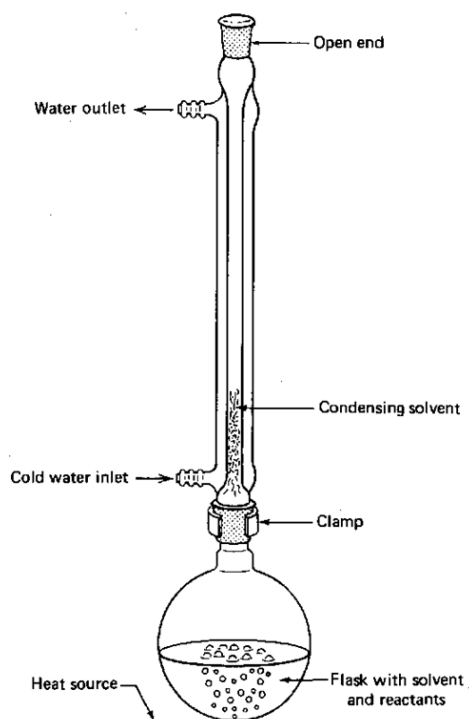
5.2 Preparation of Cyclohexene

Method

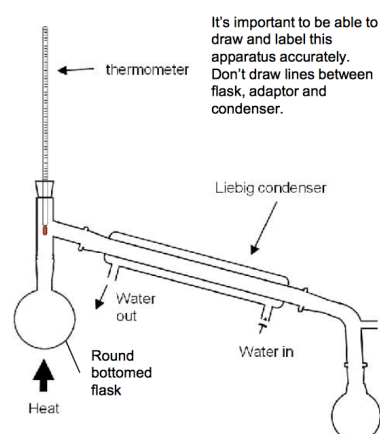
Part 1: Preparation

- Pour 10 cm³ of cyclohexanol into a 50 cm³ pear-shaped or round-bottomed flask
- Using a plastic graduated dropping pipette, carefully add approximately 4.0 cm³ of concentrated phosphoric acid to the flask.
- 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, collecting any liquid which boils between 70°C and 90°C .



Part 2: Purification

1. Pour the distillate into a separating funnel and add an equal amount of saturated sodium chloride solution.
2. Shake the separating funnel then allow the two layers to separate.
3. Carefully run off the lower aqueous layer into a beaker. Collect the upper layer, containing the crude cyclohexene, in a small conical flask.
4. Add a few lumps of anhydrous calcium chloride to the crude cyclohexene to remove any traces of water.
5. Stopper the flask, shake the contents and allow this to stand until the solid settles.
6. Weigh a sample container then decant the liquid into this container.
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. If an alkene is present, the bromine water will be decolourised.



Further improvement

9. Redistill the cyclohexene and collect the distillate produced between 81°C and 85°C to obtain a purer sample of cyclohexene.

Key Points

- 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. If anti-bumping granules weren't used, the mixture would boil over into the condenser meaning impurities would contaminate the product.
- The condenser should be tilted down slightly to allow any liquid to run into the collection flask.
- The bulb of the thermometer should be at the T junction connecting to the condenser to correctly measure the boiling point.
- The water must enter at the lowest point and leave at the highest point of the condenser to ensure that it is filled with water. This will maximise heat transfer for condensation so that cooling is more efficient.
- The collection flask must not be sealed to the condenser as this would make the system air tight. As heating occurs, air inside the system expands so air must be able to escape to avoid cracking the apparatus.

Safety

- Cyclohexanol – causes skin irritation; harmful if swallowed; harmful if inhaled.
- Phosphoric acid - causes severe skin burns and eye damage.
- Anhydrous calcium chloride - causes serious eye irritation.
- Cyclohexene - highly flammable; may be fatal if swallowed; toxic in contact with skin.
- Wear a lab coat, safety goggles and gloves.
- When using the separating funnel ensure the stopper is firmly held in place and the nozzle is pointing away from everyone else.
- Work in a fume cupboard.

