

OCR (B) Chemistry A-Level

PAG 05: Synthesis of an organic liquid



5.2 Synthesis of cyclohexene

Equipment

General

- Thermometer
- 50 cm³ round bottom flask
- Laboratory tubing
- Glassware clips
- Liebig condenser
- Heat source
- Clamp stands and clamps.

Preparation

- Cyclohexanol
- Concentrated H₃PO₄
- Dropping pipette

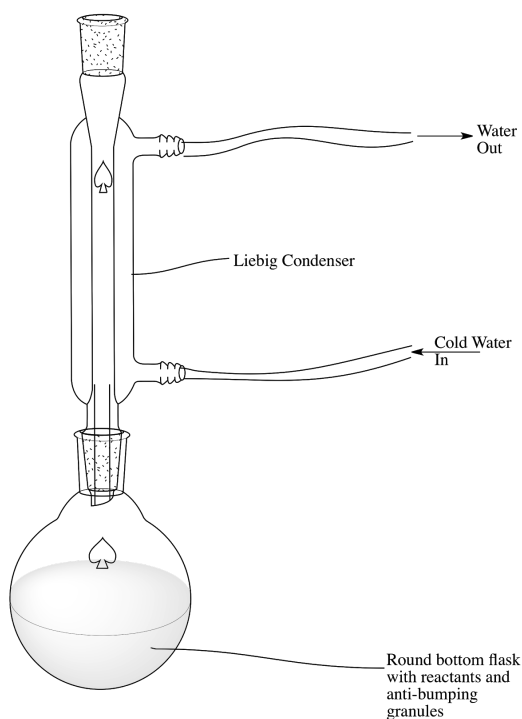
Purification

- T-Shaped still head
- 25 cm³ round bottom flask
- Anti-Bumping granules
- Separating funnel
- Saturated NaCl solution (Brine)
- 50 cm³ Conical flask

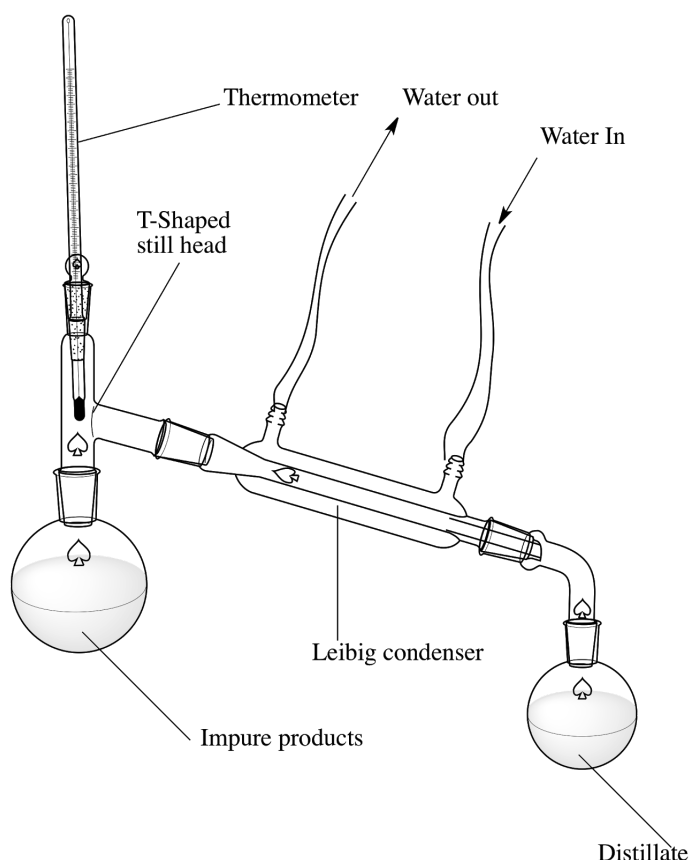
Method

Part 1 : Preparation

1. Pour 10 cm³ of cyclohexanol into the 50 cm³ round bottom flask.
2. Using a graduated dropping pipette, add 4 cm³ of concentrated phosphoric acid to the flask.
3. Set up the apparatus for reflux as shown.



4. Heat the flask carefully at 70 °C for 15-20 minutes ensuring that no vapours escape the condenser.
5. Turn off the heat and allow the flask to cool before setting up a distillation as shown below. Use clips to seal any joints between pieces of glassware except for between the collection flask and the condenser. Also make sure that the condenser is tilted towards the collection flask to allow the distillate to run into the flask.



6. Heat the flask containing the impure products, collecting only the distillate which boils at 70°C - 90°C

Part 2 : Purification

1. Pour the distillate into the separating funnel and add an equal volume of saturated NaCl solution.
2. Put a stopper in the funnel and, making sure the tap at the bottom is closed, shake it gently back and forth. Allow the two layers to separate.
3. Run the lower layer off into a beaker then transfer the upper layer into a conical flask.
4. Add anhydrous calcium chloride to the conical flask, put a stopper on the flask and shake it, allow it to stand until the liquid becomes clear.
5. (Optional) Redistill the product and collect the distillate that comes off at 81°C to 85°C.
6. Pour the final product into an already weighed sample tube.
7. Weigh the sample tube again, calculate the mass of dry cyclohexene produced and determine the percentage yield of the product.
8. Test the distillate with bromine water to check if an alkene is present, the bromine water will turn colourless in the presence of an alkene.



Errors

- The thermometer may not read the correct value of the vapours that pass into the condenser, resulting in unwanted impurities in the distillate.
Place the bulb of the thermometer at the junction in the still head, this will give an accurate reading of the temperature of vapours passing into the condenser.
- In the distillation some liquid may boil over into the collection flask, contaminating it.
Use anti-bumping granules to prevent violent boiling.

Risk Assessment

Hazard	Risk	Control
Cyclohexanol	Harmful if inhaled or swallowed.	Perform the experiment in a very well ventilated area or a fume cupboard.
Concentrated phosphoric acid	Very corrosive. Causes severe skin burns and eye damage.	Wear protective gloves and goggles.
Anhydrous calcium chloride	Causes serious eye irritation.	Wear goggles, wash hands before and after the experiment.
Cyclohexene	Highly flammable and possibly fatal if swallowed.	No open flames to be used throughout the experiment, round bottom flasks are to be heated using heating mantles or hot water/oil baths. Gloves must be worn and hands must be washed before and after the experiment.
Separating funnel	Gases could build up in the funnel during shaking, a spill may occur.	Ensure the stopper is held tightly in the funnel, point the nozzle away from people.

