

CIE Chemistry A-Level
Practicals for Papers 3 and 5
Organic Synthesis Practicals



Synthesis of cyclohexene by the dehydration and distillation of cyclohexanol

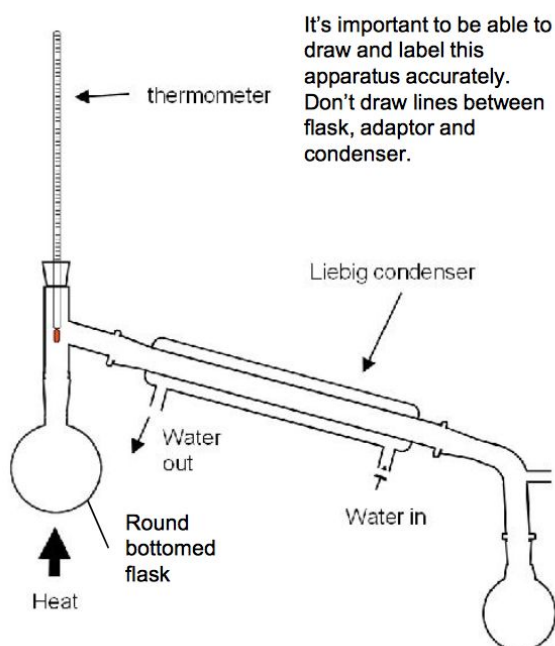
Method	Accuracy	Explanation
1. Pour 20 cm ³ of cyclohexanol into a 50 cm ³ pear-shaped flask that has been weighed. Reweigh the flask and record the mass of cyclohexanol.	<ul style="list-style-type: none"> Record to appropriate precision for the balance used. 	Weighing by difference.
2. Using a plastic graduated dropping pipette, carefully add approximately 8.0 cm ³ of concentrated phosphoric acid to the flask.	<ul style="list-style-type: none"> Shake continuously. 	
3. Add a few anti-bumping granules to the flask and assemble the semi-micro distillation apparatus, so that the contents of the flask may be distilled. Heat the flask gently , distilling over any liquid which boils below 100 °C.		The cyclohexene has a lower boiling point so can be separated in this way.
4. Pour the distillate into a separating funnel and add 50 cm of saturated sodium chloride solution. Shake the mixture and allow the two layers to separate.		Saturated sodium chloride solution acts as a drying agent.
5. 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.		This removes water, a little cyclohexanol and phosphoric acid.
6. Add a few lumps of anhydrous calcium chloride to the crude cyclohexene to remove water. Stopper the flask, shake the contents and allow this to stand until the liquid becomes clear.		
7. Decant the liquid into a clean, dry sample container that has been weighed.		
8. Reweigh the container, calculate the mass of dry cyclohexene produced and determine the percentage yield of your product.	<ul style="list-style-type: none"> Assume that all of the dry distillate is cyclohexene. 	
9. Test the distillate using Bromine water , to confirm that it contains an alkene. (Goes colourless if present).		



Common method taken from mark schemes:

1. **Acidify** the potassium manganate (VII) solution combining a volume of a regular laboratory concentration with an equal volume of dilute sulfuric acid.
2. Pour the mixture into a pear-shaped/round flask, with a still head containing a **thermometer**.
3. Attached to a condenser with an ice cooled collecting vessel.
4. Add a few **anti-bumping granules**.
5. Heat the flask **gently**.
6. **Collect** sample at **boiling point** of the desired product.
7. The **cooled** collection vessel is essential to **reduce evaporation** of the product.

Diagram:



Key to remember:

- 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.



Preparation of ethanal by the oxidation and distillation of ethanol

Method	Accuracy	Explanation
1. Make the oxidising agent by dissolving potassium dichromate (VI) in dilute sulfuric acid . The concentration of the potassium dichromate (VI) should be approximately 1 g in every 10 cm ³ of this dilute acid.		
2. Using a 25 cm ³ measuring cylinder, carefully measure out 12 cm ³ of the acidified potassium dichromate (VI) solution and pour this into a boiling tube.		
3. Cool the boiling tube in a beaker of cold water.	<ul style="list-style-type: none"> Keep the test tube cool. 	<ul style="list-style-type: none"> Avoids loss of the volatile ethanal.
4. Using a 10 cm ³ measuring cylinder, carefully measure out 2 cm ³ of ethanol.		
5. Using a teat pipette, slowly add the 2 cm ³ of ethanol dropwise, to the oxidising agent in the cooled boiling tube, shaking the tube gently to mix the contents.		
6. Add a few anti-bumping granules to the boiling tube and attach to it a bung fitted with a right-angled glass delivery tube .		Anti-bumping granules allow a more even heating of the mixture.
7. Clamp the boiling tube at about 450 in a beaker of water so that the delivery tube goes to a test tube which is immersed in cold water in a beaker.	<ul style="list-style-type: none"> Keep the test tube cool to avoid loss of the volatile ethanal. 	
8. Gently heat the beaker of water containing the ethanol to slowly distil off approximately 5 cm ³ of liquid distillate.		
9. Use Tollen's reagent to test the distillate for ethanal. (Should produce a silver mirror).		

Safety precautions:

- Make sure the Tollens' reagent is disposed of thoroughly by **rinsing** it away with plenty of water.
- Rinse any glassware that has contained the reagent with a little **dilute** sulfuric acid when finished.



Synthesis by 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 **lower density liquid** (organic product) **layer above**.

- Put the distillate of impure product into a separating funnel
- **Wash product** by either:
 - o Sodium hydrogen carbonate solution, shaking and releasing pressure from CO₂ produced - **Sodium hydrogen carbonate removes acidic impurities** by neutralisation (converts to H₂O, CO₂ and Na₂SO₄)
 - o 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

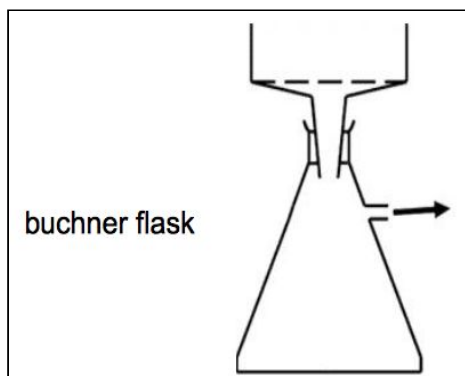
Anhydrous calcium chloride is a drying agent and soaks up any remaining water – the liquid will remain cloudy until all the water drops have been removed.

Recrystallization of crude synthesised product (to purify):

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.

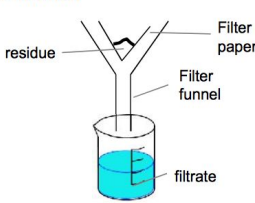


4. Suction filtrate with a Buchner flask to separate out crystals.		The water pump connected to the Buchner flask reduces the pressure and speeds up the filtration.
5 Wash the crystals with distilled water.		To remove soluble impurities .
6. Dry the crystals between absorbent papers.	To remove excess water.	Water would affect % yield.



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

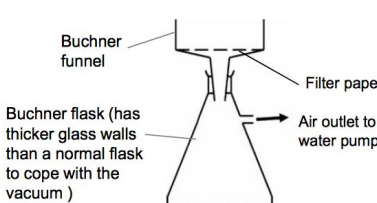


residue

Filter paper

Filter funnel

filtrate



Buchner flask (has thicker glass walls than a normal flask to cope with the vacuum)

Air outlet to water pump

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.

Loss of yield in this process:

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

