AQA Chemistry A-level

Required Practical 5

Distillation of a product from a reaction

Preparation of cyclohexene by the dehydration and distillation of cyclohexanol.

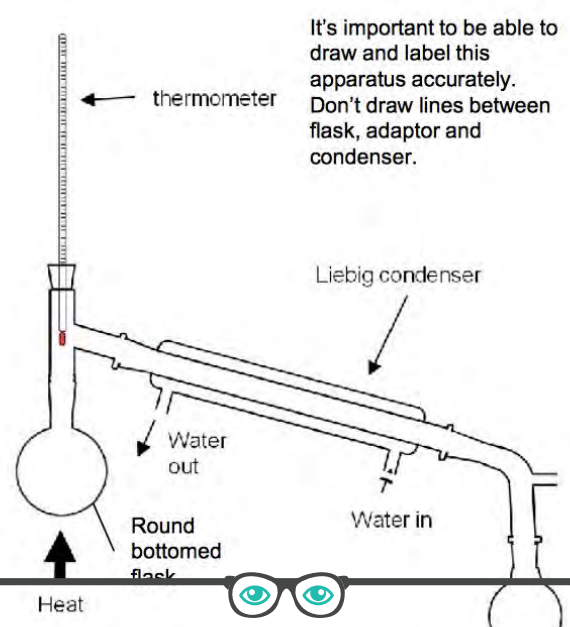
Method	Accuracy	Explanation
1. Pour 20 cm^3 of cyclohexanol into a 50 cm^3 pear-shaped flask that has been weighed. Reweigh the flask and record the mass of cyclohexanol.	• Record to appropriate precision for the balance used.	Weighing by difference.
2. Using a plastic graduated dropping pipette, carefully add approximately 8.0 cm^3 of concentrated phosphoric acid to the flask.	• 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 or 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.	• 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 a 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 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.

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.	• Keep the test tube cool to avoid 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.	• 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 cm3 of liquid distillate.		
9. Use Tollen's reagent to test the distillate for ethanal. (Should produce a silver mirror).		

Tollens' silver mirror test:

1. Add 5 drops of sodium hydroxide solution to 2 cm of silver nitrate solution in a new, clean test tube.
2. Add just enough dilute ammonia solution to dissolve the brown precipitate completely.
3. Using a beaker of hot water (50 - 60 °C), gently warm a 5 cm depth of this test reagent in a test tube.
4. Add 10 drops of the distillate containing ethanal to the warmed Tollens' reagent in the test tube.
5. Wait a few minutes and note any observations. (A silver mirror should form).

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.