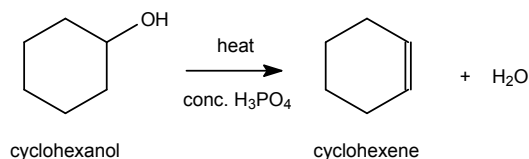
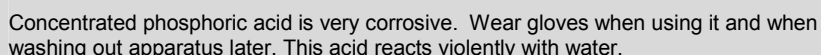




You are going to make cyclohexene by the dehydration of cyclohexanol. You will learn several new techniques including This experiment will give you a further opportunity to do a distillation and to learn some new purification techniques.

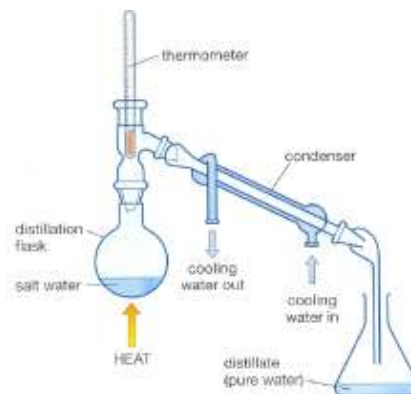


Cyclohexanol and cyclohexene are flammable. The bottle of cyclohexanol must be kept stoppered when not being used. The substances should be kept away from naked flames. Cyclohexanol is also harmful if inhaled or spilt on skin.



The fumes from this experiment are unpleasant. Use tubing to carry the fumes below bench level. The residues from each experiment should be washed down the fume cupboard sink with large amounts of water.

- 1) Place 10 cm³ of cyclohexanol into a pear shaped flask.
- 2) **In a fume cupboard**, holding the flask with a clamp and pointing the flask towards the back of the fume cupboard, carefully add 4 cm³ of phosphoric acid dropwise with swirling.
- 3) Set up the apparatus shown with your flask.
- 4) Heat the flask very slowly and gently, collecting the liquid that distils between 70-90°C. Do not let the flask boil dry.



- 5) The distillate will be a mixture of mainly cyclohexene and water, together with other impurities such as unreacted cyclohexanol. Pour this mixture into a separating funnel.
- 6) Add an equal volume of sodium chloride solution to help separate the organic and aqueous layers. Stopper the funnel and shake before allowing the layers to settle.
- 7) Run off the aqueous lower layer into a beaker, and then collect the organic layer in a small conical flask.
- 8) Add two or three small pieces of anhydrous calcium chloride, a drying agent, and stopper the flask. Shake for a few minutes until the liquid is clear.
- 9) Decant the alkene into a clean flask and redistil it, collecting the liquid distilling between 81-85°C.
- 10) Find the mass of your product.

11) Use some bromine water to check that the product is unsaturated.

1) Give the result of the test with bromine water and explain what the result means.

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- 2) Write an equation for the reaction between cyclohexene and bromine.

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- 3) Give the mass of cyclohexene produced.

- 4) Calculate the percentage yield of cyclohexene (the density of cyclohexanol = 1.0 g/cm^3).

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- 5) In what way did the use of the sodium chloride solution and the separating funnel assist to purify the cyclohexene?

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- 6) In what way did the use of the calcium chloride solution assist to purify the cyclohexene?

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- 7) In what way did the final distillation assist to purify the cyclohexene?

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- 8) Give the boiling point range of this final distillate.

- 9) What significance does the range of the boiling point have?

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- 10) What significance does the value of the boiling point range have?

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- 11) The actual boiling point of cyclohexene is 82.8°C . Comment on the purity of your cyclohexene given your answers to 8, 9 and 10.

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