Core practical 6: Investigating chlorination of 2-methylpropan-2-ol

Objective

- To produce and purify a sample of 2-chloro-2-methylpropane

Safety

- Wear goggles and gloves.
- 2-methylpropan-2-ol is flammable and harmful.
- Concentrated hydrochloric acid is corrosive and the hydrogen chloride fumes it produces are toxic and corrosive. Carry out this practical in a fume cupboard or well-ventilated room.
- 2-chloro-2-methylpropane is flammable.
- Calcium chloride is an irritant.

Specification links

- Practical techniques 2, 4, 7, 9, 11
- CPAC 1a, 2a, 2b, 3a, 3b, 3c

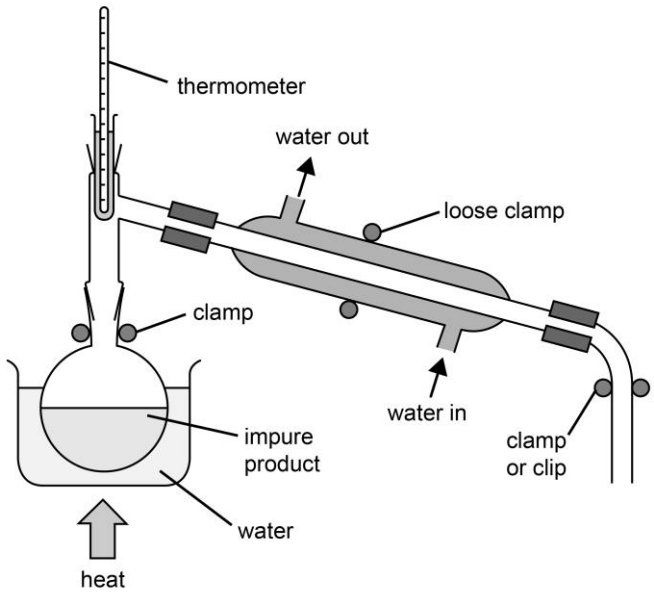
Procedure

1. Pour 10 cm^3 of 2-methylpropan-2-ol and 35 cm^3 of concentrated hydrochloric acid into a large conical flask. Swirl the contents of the flask very gently.
2. Place the bung in the mouth of the flask. Gently swirl again, then remove the bung to release the pressure.
3. Continue swirling the mixture with the bung fitted and then releasing the pressure for around 20 minutes. There should be two layers in the flask. The upper layer is the crude product.
4. Add approximately 6 g of powdered anhydrous calcium chloride to the flask and swirl until it has dissolved. This ensures that any unreacted alcohol is in the lower aqueous layer.
5. Transfer the reaction mixture to a separating funnel. Allow the mixture to settle into the two layers. Run off and discard the lower layer. Retain the upper organic layer in the separating funnel.
6. Add approximately 20 cm^3 of sodium hydrogencarbonate solution to the separating funnel. Swirl the funnel and remove the bung to release the pressure, caused by the production of carbon dioxide, at frequent intervals. Run off and discard the lower aqueous layer.
7. Repeat the washing with sodium hydrogencarbonate, shake the separating funnel and release the carbon dioxide gas produced, at frequent intervals.
8. Run off and discard the lower layer. Ensure none of the aqueous layer remains in the tap.
9. Run off the organic layer into a small conical flask. Add a spatula full of anhydrous sodium sulfate. Place the bung in the flask and swirl the contents to mix. Leave the mixture until the liquid looks completely clear, swirling occasionally.
10. Decant the organic liquid into a 50 cm^3 pear-shaped (or round-bottomed) flask.

Notes on procedure

- This practical procedure is probably best carried out over two lessons with the crude product being left to dry between lessons.

11. Set the flask up for distillation, as shown in the diagram below.



12. Collect the fraction boiling between 50 °C and 52 °C.
13. Place your pure product in a labelled sample tube.

Answers to questions

1. $(CH_3)_3COH + HCl \rightarrow (CH_3)_3CCl + H_2O$
2. (unreacted) HCl

   $HCl + NaHCO_3 \rightarrow NaCl + CO_2 + H_2O$
3. 2-methylpropan-2-ol can form hydrogen bonds, 2-chloro-2-methylpropane cannot.

   2-methylpropan-2-ol is able to form hydrogen bonds with water and so it is soluble in water.

   2-chloro-2-methylpropane cannot form hydrogen bonds with water and so is insoluble in water.

   Intermolecular hydrogen bonds in 2-methylpropan-2-ol are stronger than the dipole-dipole attractions and London forces between molecules in 2-chloro-2-methylpropane so more energy is required to separate the molecules.

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- Calcium chloride is an irritant.

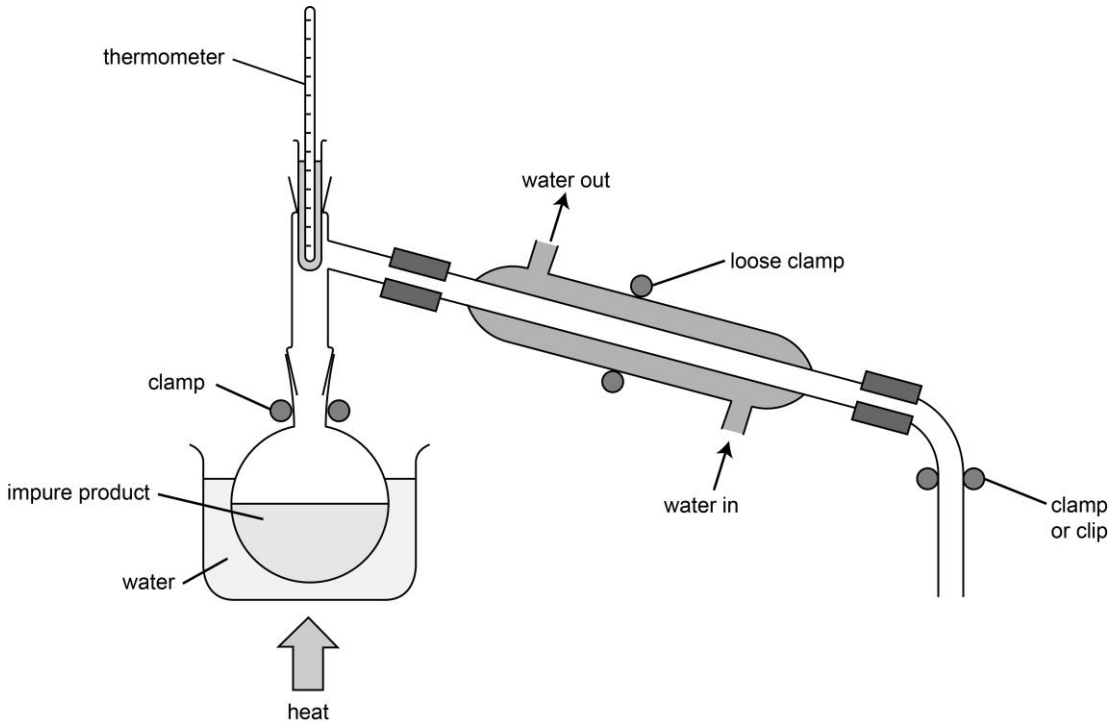
All the maths you need

- Use ratios to construct and balance equations.
- Use percentages to calculate percentage yield.

Equipment

- large 250 cm^3 conical flask with bung
- 100 cm^3 (or larger) separating funnel with bung
- 250 cm^3 beaker for liquid run out of separating funnel
- filter funnel to fit separating funnel
- apparatus for distillation with 50 cm^3 pear-shaped (or round-bottomed) flask and thermometer able to read up to 100 °C
- 25 cm^3 and 100 cm^3 measuring cylinders
- 2-methylpropan-2-ol
- sodium hydrogencarbonate solution
- 6 g powdered anhydrous calcium chloride
- anhydrous sodium sulfate
- small conical flasks with bungs
- sample tube
- silver nitrate solution
- dilute sodium hydroxide solution
- dilute nitric acid
- 70 cm^3 concentrated hydrochloric acid
- test tubes
- spatulas
- 5 cm^3 ethanol
- Bunsen burner

Diagram



Procedure

1. Pour 10 cm^3 of 2-methylpropan-2-ol and 35 cm^3 of concentrated hydrochloric acid into a large conical flask. Swirl the contents of the flask very gently.
2. Place the bung in the mouth of the flask. Gently swirl again, then remove the bung to release the pressure.
3. Continue swirling the mixture with the bung fitted and then releasing the pressure for around 20 minutes. There should be two layers in the flask. The upper layer is the crude product.
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9. Run off the organic layer into a small conical flask. Add a spatula full of anhydrous sodium sulfate. Place the bung in the flask and swirl the contents to mix. Leave the mixture until the liquid looks completely clear, swirling occasionally.
10. Decant the organic liquid into a 50 cm^3 pear-shaped (or round-bottomed) flask.
11. Set the flask up for distillation, as shown in the diagram above.
12. Collect the fraction boiling between 50 °C and 52 °C.
13. Place your pure product into a labelled sample tube.

Analysis of results

Perform the following test on the distillate:

- Place a few drops of the distillate into a test tube. Add 5 cm^3 of ethanol and 1 cm^3 of aqueous sodium hydroxide to the test tube. Warm the mixture in a water bath. Add excess nitric acid to the mixture followed by a few drops of silver nitrate solution.

Learning tips

- The –OH group in an alcohol can be replaced by a halogen. PCl_5 can be used to make a chloroalkane, HBr (which is made in situ) can be used to make a bromoalkane, and red phosphorus with iodine can be used to make an iodoalkane.
- The purity of a substance can be checked by measuring its boiling temperature.

Questions

1. Write an equation for the reaction of 2-methylpropan-2-ol with concentrated hydrochloric acid.
2. What is removed from the crude product when it is shaken with sodium hydrogencarbonate solution? Write an equation for any reaction that occurs.
3. 2-methylpropan-2-ol has a boiling temperature of 82 °C and is soluble in water. 2-chloro-2-methylpropane has a boiling temperature of 51 °C and is insoluble in water. Explain these differences.

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Objective

- To produce and purify a sample of 2-chloro-2-methylpropane

Safety

- Wear eye protection.
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- Concentrated hydrochloric acid is corrosive and the hydrogen chloride fumes it produces are toxic and corrosive.
- 2-chloro-2-methylpropane is flammable.
- Calcium chloride is an irritant.

Equipment per student/group	Notes on equipment
large 250 cm^3 conical flask with bung	
100 cm^3 (or larger) separating funnel with bung	
250 cm^3 beaker for liquid run out of separating funnel	
filter funnel to fit separating funnel	
apparatus for distillation with 50 cm^3 pear-shaped (or round-bottomed) flask and thermometer able to read up to 100 °C	
25 cm^3 and 100 cm^3 measuring cylinders	
2-methylpropan-2-ol	10 cm^3 per student
concentrated hydrochloric acid	35 cm^3 per student
sodium hydrogencarbonate solution	0.1 mol dm^{-3}
powdered anhydrous calcium chloride	6 g per student
anhydrous sodium sulfate	1 g
small conical flasks with bungs	No bigger than 100 cm^3
test tubes	
sample tube	
silver nitrate solution	Normal concentration of ion tests
dilute sodium hydroxide solution	
dilute nitric acid	
spatulas	
ethanol	5 cm^3 per student
Bunsen burner	
kettle	To prepare the water bath

Notes