Preparing and purifying an organic liquid

OCR A H032/H432 · AS content

Show your working where needed. Check the worked answers when you finish.

Name _____ Date _____

1. Choose an apparatus arrangement

A reaction must be heated for 30 minutes and the starting liquid is volatile. Explain how an upright condenser lets the student keep the mixture hot for that time without losing much starting liquid.

2. Check a thermometer reading

A student sets up simple distillation but puts the thermometer bulb into the boiling liquid. Where should the bulb sit? Explain why the new position gives a more useful reading for identifying the fraction.

3. Check the water connections

A water-cooled condenser is fitted for distillation. Which connector takes in cold water and which lets it out? Explain the direction.

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4. Decide which layer to collect

A mixture forms two immiscible layers. The organic layer has density 0.81 g cm^{-3}, and the aqueous layer has density 1.04 g cm^{-3}. The product is in the organic layer. Explain which layer to keep and how to collect it without mixing the layers.

5. Explain the use of a solid

After separating the bulk aqueous layer, a student adds anhydrous magnesium sulfate to the organic layer and later removes the solid. Explain why the salt is used.

6. Interpret the collected fraction

At the same pressure, a pure reference liquid boils at 81 °C. A student collects a fraction from 72 °C to 86 °C. What does this broad range suggest, and what follow-up would improve the evidence?

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7. Calculate a preparation yield

A student prepares 1-bromobutane from 7.40 g of butan-1-ol with other reagents in excess and isolates 9.59 g of 1-bromobutane. Use the 1:1 mole ratio and M_r values 74.0 and 137.0 to calculate the percentage yield.

8. Check a recorded yield

The reaction can give a maximum of 4.60 g pure organic product. A student records collecting 5.08 g of liquid. Calculate the apparent percentage yield. Give one plausible reason for the result and a suitable check.

9. Extra challenge · Recover a liquid after reflux

Butan-1-ol is heated with sodium bromide and sulfuric acid under reflux. After cooling, the crude distillate contains 1-bromobutane, water and some unreacted butan-1-ol. At the same pressure, pure 1-bromobutane boils at 101 °C and butan-1-ol at 118 °C. The product-rich layer has density 1.25 g cm^{-3} and the aqueous layer 1.03 g cm^{-3}.

Plan how to separate, dry and redistil the product. Identify the layer to keep, explain each step and give one way to check the purity of the collected product.

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Worked answers

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1. Choose an apparatus arrangement

Vapours from the heated mixture cool in the condenser. They condense and flow back into the flask, so the mixture can be heated while reducing loss of volatile material.

2. Check a thermometer reading

Put the bulb at the entrance to the side arm so it sits in the vapour passing towards the condenser. It measures the temperature of the vapour being collected.

3. Check the water connections

Water enters at the lower connector and leaves at the upper one. Feeding water in from the bottom keeps the jacket full and allows air to escape through the upper outlet.

4. Decide which layer to collect

The organic layer is on top because it is less dense. Use a separating funnel. Run off the lower aqueous layer through the tap, then collect the organic layer separately.

5. Explain the use of a solid

Anhydrous magnesium sulfate takes up traces of water by forming hydrated solid. Removing the solid leaves the organic liquid with less water.

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Worked answers

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6. Interpret the collected fraction

A wide range is consistent with more than one volatile substance in the fraction. Redistil and collect the narrow fraction around 81 °C at the same pressure.

Check a second piece of evidence, for example an IR spectrum against a suitable reference. A narrow boiling range alone cannot establish identity or purity.

7. Calculate a preparation yield

7.40 ÷ 74.0 = 0.100 mol butan-1-ol, so 0.100 mol is the maximum amount of 1-bromobutane. Its maximum mass is 0.100 × 137.0 = 13.7 g. Yield = 9.59 ÷ 13.7 × 100 = 70.0%.

8. Check a recorded yield

5.08 ÷ 4.60 × 100 = 110% to three significant figures. The mass cannot all be pure product.

The sample may contain water. Dry it with an appropriate anhydrous salt, remove the salt and reweigh in a tared container. Residual solvent or an incorrect tare are other possibilities.

9. Extra challenge · Recover a liquid after reflux

Use a separating funnel. The product-rich layer is below the aqueous layer in this mixture, so drain the lower layer into a clean vessel. Add a suitable anhydrous drying agent, such as magnesium sulfate, then filter or decant the dried liquid from the solid.

Redistil the liquid and collect the narrow fraction around 101 °C at the given pressure. The higher-boiling butan-1-ol is less volatile. Compare the boiling range with the reference. An IR check for an alcohol O-H band can give additional evidence of the remaining alcohol or water.

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