

WJEC (Wales) Chemistry A-level

SP 4.8a - Synthesis of a Liquid Organic Product

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SP 4.8a - Synthesis of a Liquid Organic Product

Aim

To **synthesise** 2-chloro-2-methylpropane using 2-methylpropan-2-ol and concentrated hydrochloric acid and **purify** the product using a **separating funnel** followed by **distillation**.

Apparatus and Chemicals

- Deionised water
- Access to 2 decimal place mass balance
- 100 cm³ round bottom flask
- 100 cm³ conical flask
- 100 cm³ conical flask with stopper
- 10 cm³ measuring cylinder
- 50 cm³ measuring cylinder
- 50 cm³ separating funnel
- Ice bath
- Reflux condenser
- Thermometer
- Sample vial
- Labels/suitable pen
- Anti-bumping granules
- Heating mantle / Bunsen burner with water bath
- Clamp stand
- Filter funnel with cotton wool
- (CH₃)₃COH (2-methylpropan-2-ol)
- Concentrated HCl solution
- Granular anhydrous CaCl₂
- Saturated NaHCO₃ solution
- Anhydrous MgSO₄ powder

Safety Considerations

- ★ (CH₃)₃COH - flammable
- ★ Concentrated HCl solution - corrosive
- ★ CaCl₂ - irritant





Method

1. Add 30 cm³ of concentrated HCl solution to a stoppered conical flask. This step should be performed in a **fume cupboard**.
2. Cool the HCl solution in the **ice bath**.
3. When the HCl solution has cooled add 2.5 g anhydrous CaCl₂ to the solution.
4. Add 10 cm³ of (CH₃)₃COH to the reaction mixture and mix thoroughly.
5. Remove the mixture from the ice bath and allow it to **slowly warm up** to room temperature. Note that a gaseous side product can be formed at this stage and so the **stopper should be released periodically** to alleviate any build-up of pressure.
6. Transfer the reaction mixture to the separating funnel.
7. Stopper the funnel and **invert** it a few times to thoroughly mix the reaction mixture. You may need to alleviate the pressure in the funnel by holding it upside down and opening the tap.
8. Run off and discard the **aqueous layer**. If you are unsure which layer is the aqueous layer, add deionised water to the funnel and see which layer increases in volume.
9. Add 10 cm³ of saturated NaHCO₃ solution to the **organic layer** and mix thoroughly in the separating funnel. CO₂ gas is formed in this step so it is important to alleviate the pressure in the funnel periodically.
10. Run off and discard the aqueous layer.
11. Repeat steps 9 and 10 to wash the product for the second time.
12. Repeat steps 9 and 10 using 15 cm³ of deionised water to wash the product for a third time.
13. Transfer the organic product to a conical flask, add anhydrous MgSO₄ and **swirl vigorously**. Keep adding MgSO₄ until it no longer clumps together.
14. Transfer the organic product to the **round bottomed flask** through a filter funnel with a plug of cotton wool to remove the MgSO₄.
15. Set up the **distillation** apparatus.
16. Distil off the (CH₃)₃CCl (2-chloro-2-methylpropane) produced. Collect it in a clean, dry, pre-weighed 100 cm³ conical flask.
17. Record the temperature at which the liquid product is collected.
18. Calculate the mass of the product and use this information to calculate the product yield (note that the density of (CH₃)₃COH is 0.775 g cm⁻³).

