Core practical 5: Investigate the oxidation of ethanol

Objective

- To oxidise ethanol and use heating under reflux and distillation as practical techniques

Safety

- Wear goggles.
- Ethanol is flammable.
- Acidified sodium dichromate is an oxidising agent. It is both corrosive and a carcinogen – wear chemical-resistant gloves.

Specification links

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

Procedure

fig A Reflux apparatus

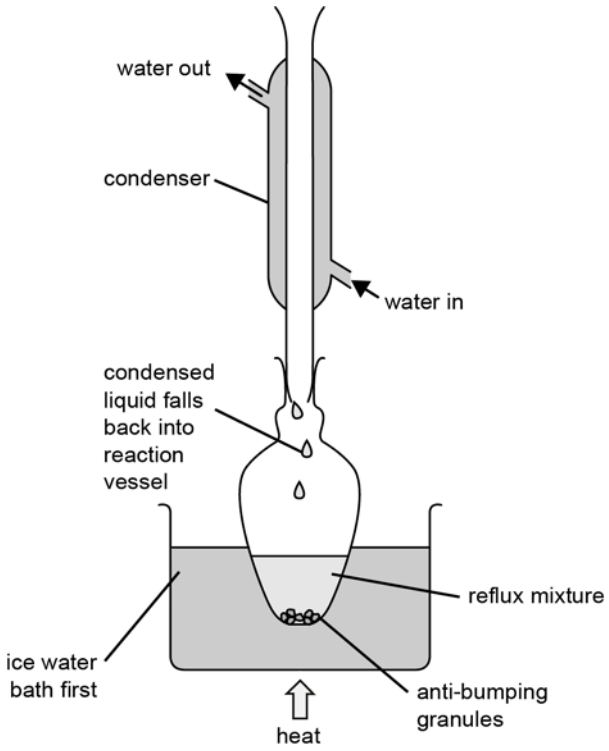
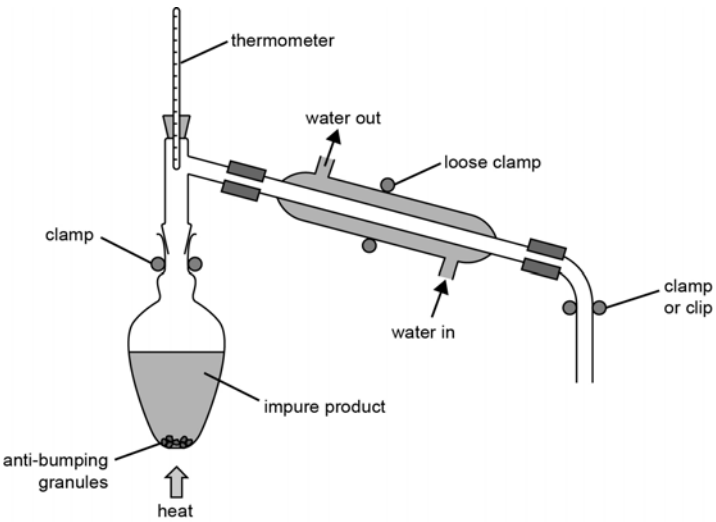


fig B Distillation apparatus



Notes on procedure

- This practical procedure is best carried out over two lessons if the students have not previously used *Quickfit*® apparatus.
- Alternatively, you may use an electric heating mantle to heat the flask containing the reflux mixture.

1. Carefully add 20 cm^3 of acidified sodium dichromate solution to a 50 ml pear-shaped flask. Cool the flask in an ice-water bath.
2. Set the flask up for reflux (see fig A) keeping it in the ice-water bath.
3. Place a few anti-bumping granules into the pear-shaped flask.
4. Measure out 1 cm^3 of ethanol.
5. Using a pipette, add the ethanol a few drops at a time down the reflux condenser. This must be done slowly. Allow for the reaction to subside after each addition before adding more.
6. When all of the ethanol has been added, remove the ice-water bath and allow to warm to room temperature (approximately 5 minutes).
7. Position the flask in a hot water bath using water from a kettle. Light a Bunsen burner and maintain a boiling water bath for 20 minutes. Allow the apparatus to cool.
8. Distil your product using the apparatus shown (see fig B). Collect 3-4cm^3 of clear, colourless liquid.

Answers to questions

1. $CH_3CH_2OH + 2[O] \rightarrow CH_3COOH + H_2O$
2. $2CH_3COOH + Mg \rightarrow Mg(CH_3COO)_2 + H_2$
3. $2CH_3COOH + CaCO_3 \rightarrow Ca(CH_3COO)_2 + H_2O + CO_2$
4. There is no change with acidified potassium dichromate as all the ethanol is oxidised. There is no change with Fehling's solution as oxidation goes to completion – any ethanol made is oxidised to ethanoic acid as it cannot leave the apparatus.

Sample data

Analysis of results

- pH of distillate = 3.5
- no change with acidified potassium dichromate solution
- effervescence observed when calcium carbonate added to distillate
- effervescence observed when magnesium added to distillate
- no change observed when distillate warmed with Fehling's solution

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- Wear chemical-resistant gloves.

All the maths you need

- Recognise and make use of appropriate units in calculations.
- Use ratios, fractions and percentages.
- Translate information between graphical, numerical and algebraic forms.
- Plot two variables from experimental or other data.

Equipment

- chemical-resistant gloves
- 20 cm^3 acidified sodium dichromate solution
- anti-bumping granules
- two 50 ml pear-shaped (or round-bottomed) *Quickfit*® flasks
- *Quickfit*® condenser
- ethanol
- dropping pipette
- two 10 ml measuring cylinders
- bench acidified potassium dichromate (labelled acidified potassium dichromate for analysis)
- apparatus for distillation with thermometer going up to at least 110 °C
- calcium carbonate powder
- spatula
- magnesium ribbon
- beakers
- 4 test tubes
- universal indicator paper
- Fehling's solution
- Bunsen burner
- ice-water bath

Diagram

fig A Reflux apparatus

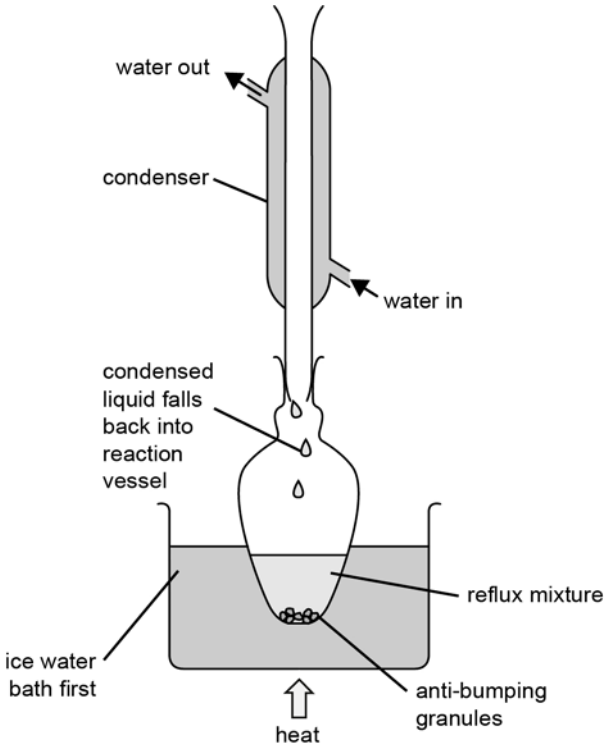
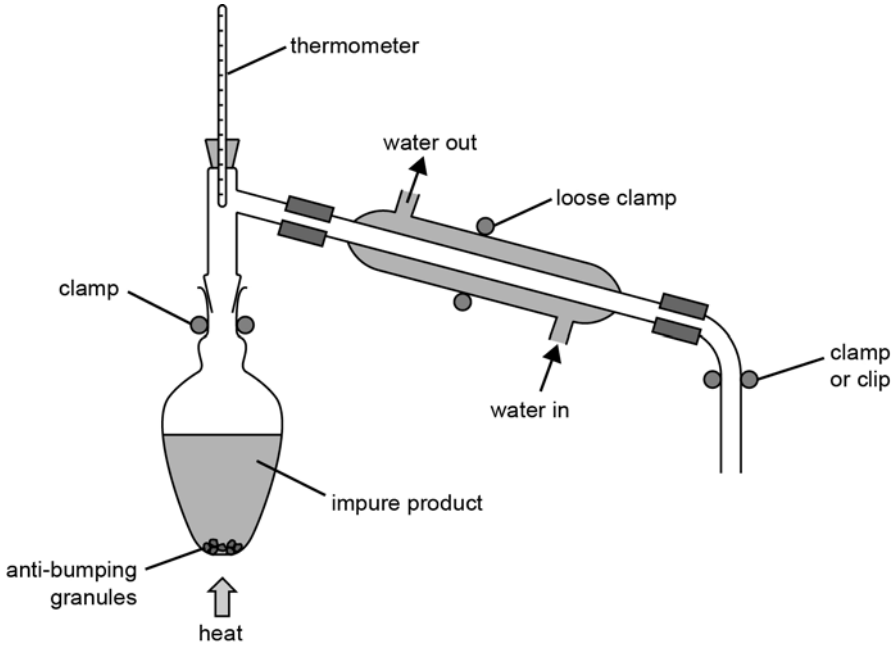


fig B Distillation apparatus



Procedure

1. Carefully add 20 cm^3 of acidified sodium dichromate solution to a 50 ml pear-shaped flask. Cool the flask in an ice-water bath.
2. Set the flask up for reflux (see fig A) keeping it in the ice-water bath.
3. Place a few anti-bumping granules into the pear-shaped flask.
4. Measure out 1 cm^3 of ethanol.

5. Using a pipette, add the ethanol a few drops at a time down the reflux condenser. This must be done slowly. Allow for the reaction to subside after each addition before adding more.
6. When all of the ethanol has been added, remove the ice-water bath and allow to warm to room temperature (approximately 5 minutes).
7. Position the flask in a hot water bath using water from a kettle. Light a Bunsen burner and maintain a boiling water bath for 20 minutes. Allow the apparatus to cool.
8. Distil your product using the apparatus shown (see fig B). Collect 3-4cm^3 of clear, colourless liquid.

Analysis of results

Split the distillate into four portions and perform the following tests on each portion:

- Measure the pH of the distillate using universal indicator paper.
- Add a few drops of acidified potassium dichromate solution to 1 cm^3 of the distillate. Warm the mixture in a 60 °C water bath.
- Add a quarter of a spatula of calcium carbonate powder to 1 cm^3 of the distillate.
- Add a 1 cm long length of magnesium ribbon to 1 cm^3 of the distillate.
- Add 1 cm^3 of Fehling's solution to 1 cm^3 of the distillate. Warm the mixture gently using a water bath.

Learning tips

- You should understand when distillation conditions and reflux conditions are used in the oxidation of alcohols.
- You should be able to write equations for the oxidation of primary and secondary alcohols.
- You should know that carboxylic acids are weak acids and that they show the typical reactions of acids. You should be able to write equations for these reactions.

Questions

1. Write an equation for the oxidation of ethanol to ethanoic acid. Use [O] to represent the oxidising agent.
2. Write an equation for the reaction of the distillate with magnesium.
3. Write an equation for the reaction of the distillate with calcium carbonate.
4. Explain the results of the tests involving acidified potassium dichromate and Fehling's solution.

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● To oxidise ethanol and use heating under reflux and distillation as practical techniques
Safety
● Wear goggles. ● Ethanol is flammable. ● Acidified sodium dichromate is an oxidising agent. It is both corrosive and a carcinogen – wear chemical-resistant gloves. ● Caution is required when preparing the oxidising agent. Preparation is best done in small batches rather than in bulk for the whole class.

Equipment per student/group	Notes on equipment
calcium carbonate powder	
spatula	
magnesium ribbon	1 cm per student/group
beakers	for water bath
4 test tubes	
universal indicator paper	
Fehling's solution	Make solution up by mixing equal parts of Fehling's A and Fehling's B. Use immediately.
Bunsen burner	
chemical-resistant gloves	
20 cm^3 acidified sodium dichromate dihydrate solution	Measure 100 cm^3 of dilute (3 M) sulfuric acid and cool in an ice-water bath. Once cool, add 19 g sodium dichromate dihydrate and stir to ensure all of the solid is dissolved. Keep mixture in an ice-water bath.
anti-bumping granules	If anti-bumping granules are not available, pumice or porous pot are suitable alternatives.
two 50 ml pear-shaped (or round-bottomed) *Quickfit*® flasks	
Quickfit® condenser	
ethanol	
dropping pipette	
two 10 ml measuring cylinders	
bench acidified potassium dichromate (labelled as acidified potassium dichromate for analysis)	Normal test reagent (made up as per the CLEAPSS instructions)

apparatus for distillation with thermometer going up to at least 110 °C	
ice-water bath	

Notes