Preparation of ethanoic acid and an investigation of some of its properties

Theory

A sodium dichromate solution acidified with sulfuric acid can be used to oxidise a primary alcohol to a carboxylic acid. Primary alcohols are first oxidised to the corresponding aldehyde by a mixture of these reagents. To ensure complete oxidation to the acid two conditions in particular are important:

1. Using more oxidising agent than alcohol (i.e. the ethanol is the limiting reagent).
2. Refluxing the reaction mixture for 20 to 30 minutes.

$$C_2H_5OH \xrightarrow{Na_2Cr_2O_7/H_2SO_4} CH_3COOH$$

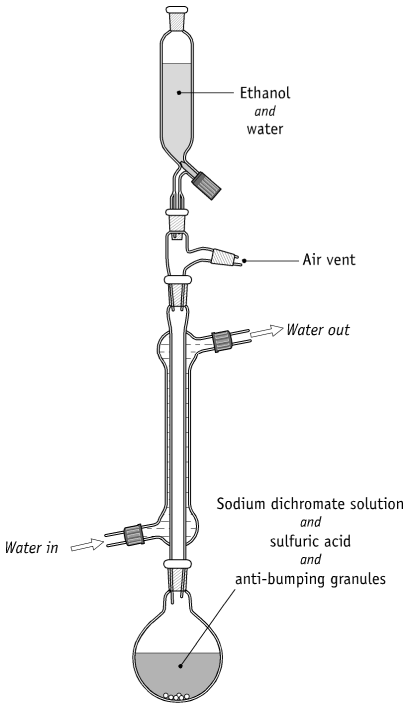
(The balanced equation for the reaction is:
$3C_2H_5OH + 2Cr_2O_7^{2-} + 16H^+ \rightarrow 3CH_3COOH + 4Cr^{3+} + 11\ H_2O$)

Procedure

NB: Wear your safety glasses.

Preparation

Put a few anti-bumping granules and 10 cm^3 of dilute sulfuric acid in the round bottomed or pear shaped flask.



In a fume cupboard, add in 9 g of sodium dichromate and dissolve by careful swirling. Use a small dry funnel to avoid crystals of dichromate being caught on the neck of the flask. Mop up any spilt drops as sodium dichromate solution is highly irritating to the skin and eyes, and may also cause damage to clothes and the work surface.

Slowly with swirling and cooling in an ice bath, add 6 cm^3 of concentrated sulfuric acid. Set up the apparatus as shown in Fig. 1 for reflux distillation with addition. If necessary, use a clamp to hold the water outlet in the sink.

Mix 2 cm^3 of ethanol and 10 cm^3 of deionised water in the dropping funnel. Add the solution from the dropping funnel **dropwise** down the condenser, while swirling the contents of the flask and cooling it if necessary to prevent too vigorous a reaction.

Remove the dropping funnel and still head from the top of the assembled apparatus. Boil the mixture gently using a water-bath for about half an hour.

Cool the apparatus; dismantle and rearrange for distillation as in Fig. 2. Direct heating without a water bath must be used, as the boiling point of the mixture will eventually exceed 100 ^0C.

Distil off about 15 cm^3. This is aqueous ethanoic acid.

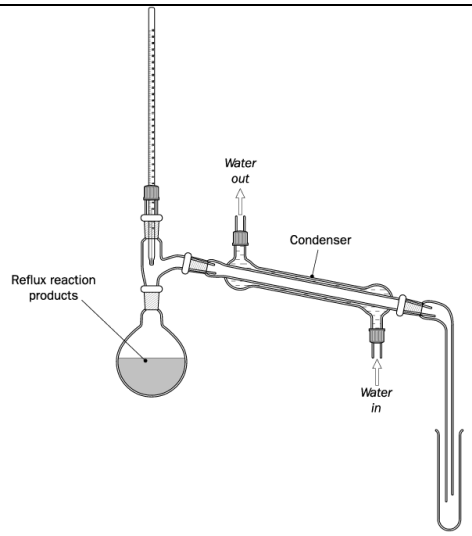


Fig 2

(b) Properties

Divide the distillate into three portions in test tubes.

1. **Odour:** Smell the distillate by carefully wafting some of the vapour towards your nose. Compare its smell to that of ethanol, and record your observations in a table of data copied into your practical report book from the table below.

2. **Test with universal indicator paper:** Dip some universal indicator paper in the distillate and record your observations.

3. **Test with magnesium:** Coil the 5 cm clean strip of magnesium loosely, drop it into one of the test tubes, and swirl. Record your observations.

4. **Test with sodium carbonate** Add 1 g of anhydrous sodium carbonate powder into the second test tube, and swirl. Record your observations.

5. **Esterification**

(i) With care add 2 drops of concentrated sulfuric acid to the third test tube.

(ii) Add 1 cm^3 of ethanol and warm gently. Carefully smell the reaction product. Record your observations.

Test	Observation	Deduction
Odour		
Universal indicator paper		
Magnesium strip		
Anhydrous sodium carbonate		
Ethanol with concentrated sulfuric acid		

Specimen results

Test	Observation	Deduction
Odour	Vinegar odour	Ethanoic acid present
Universal indicator paper	Changes colour from green to red	Solution is acidic
Magnesium strip	Effervescence	Hydrogen gas generated
Anhydrous sodium carbonate	Effervescence	Carbon dioxide gas generated
Ethanol with concentrated sulfuric acid	Sweet odour and oily droplets	Ethyl ethanoate produced

student questions

Explain the term reflux distillation.

Reflux distillation involves condensing the vapour from a boiling liquid in such a way as to return the condensed material to the reaction vessel. In this way a reaction may be carried out at quite a high temperature while preventing the loss of any of the reactants or products.

Explain why it was thought necessary in stage 5 of the preparation to add the alcohol-water mixture through the condenser.

When the alcohol water mixture meets the acidified dichromate solution a strongly exothermic reaction occurs. Without the presence of the condenser the reagents could spray out.

Name three possible impurities present in the final distillate.

The distillate may contain, along with the ethanoic acid, some water, ethanal and ethyl ethanoate.

What colour change happened in the reaction vessel during the reaction? Name the species responsible for each colour.

During the reaction, the orange dichromate ion ($Cr_2O_7^{2-}$) changes to the green chromium(III) ion (Cr^{3+}).

Assuming the density of ethanol is 0.80 g cm^{-3}, calculate the number of moles of ethanol used in the experiment.

Mass of ethanol = volume x density
= 2 cm^3 x 0.80 g cm^{-3}
= 1.6 g
Moles of ethanol = Mass / Molar mass
= 1.6 g / 46 g mol^{-1}
= **0.035 mol**

Calculate the number of moles of sodium dichromate used in this experiment, given that its formula is $Na_2Cr_2O_7.2H_2O$.

Moles of sodium dichromate = Mass / molar mass
= 9 g / 298 g mol^{-1}
= **0.03 mol**

Given that from the balanced equation 3 $C_2H_5OH \equiv 2Na_2Cr_2O_7$, show clearly that the ethanol is the limiting reactant.

2 mol $Na_2Cr_2O_7 \equiv$ 3mol C_2H_5OH
So 0.03 mol of dichromate would oxidise 1.5 x 0.03 mol of ethanol.
= 0.045 mol of ethanol
0.035 mol (of ethanol) < 0.045 mol
Ethanol is the limiting reactant.

Given that from the balanced equation 1 $C_2H_5OH \equiv 1\ CH_3COOH$, calculate the theoretical yield in grams of ethanoic acid.

1 mol $C_2H_5OH \equiv$1 mol CH_3COOH
0.035 mol $C_2H_5OH \equiv$ 0.035 mol CH_3COOH
Mass of ethanoic acid = moles x molar mass
0.035 mol x 60 g mol^{-1} = **2.1 g**

If the actual yield of ethanoic acid were required, further purification of the distillate would be necessary. Explain how this might be carried out.

Redistil the aqueous ethanoic acid collecting the fraction that boils between 116 ^{0}C and 118 ^{0}C.

Find the percentage yield of ethanoic acid in this experiment, given that the actual yield was 1.35 g.

The theoretical yield of ethanoic acid was 2.1 g.
% yield = Actual yield x 100 / Theoretical yield
= 1.35 g x 100 / 2.1 g
= 64%

What is the function of the anti-bumping granules?

The function of the anti-bumping granules is to maintain a smooth gentle boiling by preventing super-heating.