Preparation of ethanal and some of its properties

Theory

A sodium dichromate solution acidified with sulfuric acid can be used to oxidise a primary alcohol to an aldehyde. Further unwanted oxidation of the ethanol to ethanoic acid can be minimised by:

(a) using more alcohol than oxidising agent (i.e. the sodium dichromate is the limiting reagent)

(b) distilling off the ethanal as soon as it is made.

$$C_2H_5OH \xrightarrow{Na_2Cr_2O_7/H_2SO_4} CH_3CHO$$

(The balanced equation for the reaction is:
$3C_2H_5OH + Cr_2O_7^{2-} + 8H^+ \rightarrow 3CH_3CHO + 2Cr^{3+} + 7H_2O$)

Procedure

NB: Wear your safety glasses.

(a) Preparation

Set up the apparatus as shown in Fig.1 for distillation with addition. Put a few anti-bumping granules and 12 cm^3 of water in the round bottomed or pear shaped flask. Slowly, with swirling and cooling under a cold water tap, add 4 cm^3 of concentrated sulfuric acid.

Using a fume cupboard, dissolve 10 g of sodium dichromate in 10 cm^3 of water in a clean small beaker and then add 10 cm^3 of ethanol. Place this solution in the dropping funnel, using a small plastic funnel. Rinse the beaker out and mop up any spilt drops, as sodium dichromate is highly irritating to the skin and eyes.

Surround the conical flask, in which the ethanal distillate is to be collected, with ice/water in a large beaker. Heat the dilute acid until it boils and turn off the Bunsen burner or heating mantle.

Slowly (**a steady drip approach is recommended**) add the alcohol mixture from the dropping funnel so as to maintain a gentle boiling. **(Warning: If the mixture is added too rapidly, the distillate will be contaminated with chromium ions carried over by spurting.)** The ethanal distils off as it is formed.

Redistil the impure ethanal and collect the fraction boiling between 20 ^0C and 23 ^0C.

Properties

Oxidation by acidified potassium manganate(VII)

Using graduated disposable pipettes, place in a test-tube 2 cm^3 of ethanal, 1 $cm^3$of potassium manganate(VII) solution and 4 cm^3 of dilute sulfuric acid. Warm the test-tube in a water-bath and shake gently. Observe and record any colour change in a table of data copied into your practical report book (see the table below).

Oxidation by Fehling's solution

Using a graduated disposable pipette, place in a test-tube 1 cm^3 of Fehling's solution no. 1.

Using a separate pipette, add 1 cm^3 of Fehling's solution no. 2 – swirl the contents so that the blue precipitate initially formed will dissolve. Add 1 cm^3 of ethanal, heat gently and shake. Observe any change and record your observations.

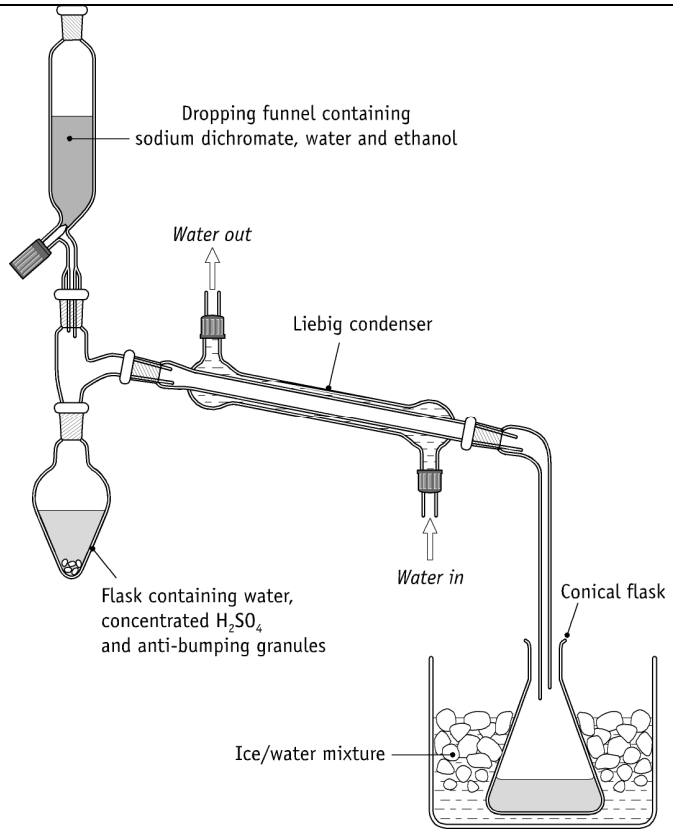

Fig.1

Oxidation by Tollen's reagent

Using a graduated disposable pipette, place in a **clean** (new if possible) test-tube 3 cm^3 of silver nitrate solution and 1 cm^3 of sodium hydroxide solution. Add aqueous ammonia solution drop by drop, with shaking, until the precipitate formed in stage 1 is just dissolved. Add two or three drops of ethanal, mix by shaking and warm in a water bath. Observe any change and record your observations, Rinse out the test-tube with dilute nitric acid and then water.

Test	**Observation**	**Deduction**
Acidified potassium manganate(VII)		
Fehling's solution		
Tollen's reagent		

Specimen results

Test	**Observation**	**Deduction**
Acidified potassium manganate(VII)	Solution decolourised	Manganate(VII) reduced to manganese(II) - ethanal oxidised
Fehling's solution	Red/brown precipitate formed	Copper(II) reduced to copper(I) oxide - ethanal oxidised
Tollen's reagent	Silver mirror formed	Silver(I) reduced to silver metal - ethanal oxidised

student questions

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+})

Why is it necessary to turn off the Bunsen burner or heating element before the reaction is started?
The reaction would become too vigorous, and some unreacted ethanol and other material is likely to be forced over into the conical flask. Once the mixture in the reaction vessel has been brought to the boil, further heating is not required as the oxidation of ethanol is an exothermic reaction, and gentle boiling can be maintained by regulating the flow from the dropping funnel.

What is likely to happen if excess sodium dichromate was used by mistake?
More ethanoic acid will be produced reducing the yield of ethanal

Why is ethanal collected in a vessel embedded in a beaker of ice-water?
Ethanal boils at 21 ^{0}C and must be kept cold in order to prevent losses through evaporation.

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
10 cm^3 x 0.80 g cm^{-3} = 8 g
Moles of ethanol = Mass / Molar mass
= 8 g / 46
= 0.17 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
= 10/298 mol
= 0.034 mol

Given that 3 moles C_2H_5OH require 1 mole $Na_2Cr_2O_7.2H_2O$ for complete reaction, show clearly that sodium dichromate is the limiting reactant.
$3C_2H_5OH \equiv 1\ Na_2Cr_2O_7.2H_2O$
So 0.034 mol of dichromate will only oxidise 3 x 0.034 mol of ethanol = 0.102 mol
The amount of ethanol actually present (0.17mol) is greater than 0.102 mol. Sodium dichromate is therefore the limiting reactant.

What use is made of the Fehling's solution test in the Leaving Certificate Biology course?
Fehling's solution is used to test for reducing sugars, i.e. saccharide molecules which contain an aldehyde group (-CHO).