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| Chemical Experiments |
| Oxidation of alcohols |



This reaction can be applied to curriculum for excellence.

CfE Higher Chemistry

 Nature’s Chemistry

 Chemistry of Cooking (Aldehydes), Oxidation of food

 Chemistry in Society

 Getting the most from reactants, Oxidising or reducing agents

CfE Advanced Higher Chemistry

 Organic Chemistry

 The preparation, properties and reactions of alcohols

## Introduction

In this activity, a primary alcohol (ethanol) is oxidised to an aldehyde (ethanal) using a solution of acidified sodium dichromate Vl a powerful oxidising agent.

Qualitative tests on the organic product are also described.

### You will need

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| Beaker with ice/water (perhaps 2) | Boiling tube |
| Sulphuric acid (2 mol l-1 ) | **Sodium** dichromate |
| Ethanol (IMS is OK) | Small funnel |
| Apparatus for distillation | Receiving flask (or test tube) with stopper |
| Access to a balance | Measuring cylinder, to measure 25cm3 |
| Dropping / Pasteur pipette(s) | Thermometer |

### Carrying out the reaction

1. Measure 20 cm3 of 2 mol l-1 sulphuric acid into a boiling tube.
2. Place the tube in a beaker filled ⅔ – ¾ with an ice-water mixture.
3. Add 1.7 g of sodium dichromate Vl-2-water (or 1.5 g of the anhydrous salt) to the boiling tube containing the acid. Stir until the solid has completely dissolved.
4. Check that the acidified dichromate Vl solution has cooled to below 10°C before proceeding further.
5. Use a Pasteur pipette to draw up 1 cm3 of ethanol.
6. Add 2-3 drops of ethanol to the cold acidified sodium dichromate Vl solution and stir/agitate the mixture.
7. Continue adding drops of ethanol (with stirring) to the boiling tube – still in the ice bath. This should be done slowly enough to ensure the temperature of the reaction mixture stays below 20°.

*On addition of drops of ethanol, the reaction mixture will change colour (from orange to green) and the temperature rise as this is an exothermic reaction). Allow the temperature to drop a little each time - before adding further drops of ethanol.*

### Distillation

*It is* ***possible*** *to carry out the distillation using a Bunsen burner but great care needs to be taken and we think it preferable to use an alternative heat source as ethanal is extremely flammable.*

1. Set up your apparatus for distillation as shown in the diagram (overleaf) using a 50 cm3 pear-shaped or round-bottomed flask.
2. Add a few anti-bumping granules to the flask.
3. Transfer the cold reaction mixture to the flask, carefully, using a small funnel (to avoid contamination of the neck of the flask).



1. Leave the reaction mixture in the flask for a few minutes to warm to room temperature before proceeding. This should be quite rapid as the exothermic alcohol - dichromate reaction is proceeding.
2. Switch on your heat source and gently heat the mixture in the flask, maintaining a gentle simmer, until 4 cm3 of clear colourless liquid has distilled over and collected in the receiving flask (or test tube).

*As ethanal is very volatile, and the vapour is irritating and carcinogenic, collect the distillate in a flask or test tube in and ice or cold-water bath*

1. Stop heating, remove the distillate and stopper the container.
2. Stopper the test tube.

## Safety

Wear indirect-vent goggles (BS EN166 3)

Use **sodium** dichromate Vl not potassium dichromate. This is essential as the potassium salt is much less soluble and the presence of solid dichromate Vl in the reaction mixture may lead to excessive 'bumping' and cause hot, corrosive liquid to splash out of the flask, possibly quite forcefully.

(Older methods using concentrated acid and carrying out the reaction in one step rather than cooling the initial reaction mixture makes such an eruption more likely)

## Disposal

After completing the distillation, allow the apparatus to cool. This can be speeded up by cooling the reaction flask in a beaker of cold water for 30 seconds or so.
Disconnect the flask and pour the green contents into a bowl of bucket of water (5 litres or so) - in a fume cupboard to contain the fumes.
The green chromium III mixture is of relatively low hazard and once added to water, can be safely washed to waste with plenty of fresh water.

## The chemistry

The dichromate ion is a powerful oxidiser but not quite to the same degree as potassium manganate VII and so it only oxidises the ethanol ‘part-way’ to the aldehyde rather than fully to the carboxylic acid.

The equation for the reaction (state symbols not included) is

3C2H5OH + 8H+ + Cr2O7 2- 3CH3CHO + 2Cr3+ + 7H2O
 orange green

While oxidising the ethanol to ethanal, the dichromate is reduced from orange Cr 6+ to green Cr 3+.

Stoichiometry

20 cm of 2 mol l-1 sulphuric acid = 0.04 moles of H2SO4 (= 0.08 moles of H+)

1 cm3 of ethanol has a mass of 0.79 g = 0.0171 moles.

1.7 g of sodium dichromate-2-water (gfm 297.99) = 0.0057 moles.
So the ethanol : dichromate ratio is 3 : 1(as required in the equation) with excess H+ ions (actually nearly 15 rather than the 8 required).

The distillate is not pure ethanal, rather a mixture of ethanal, ethanol and some acetals but there is sufficient aldehyde for any qualitative tests to give positive results.

Purifying the ethanal **is** possible but it is complex and time-consuming.

## Extensions

**Other alcohols**

This preparation can also be carried out with propan-l-ol to form propanal. The reaction equation and quantities of reagents will need to be adjusted appropriately and you will need to check the hazards and adjust the risk assessment if required.

**Tests**

It is possible to test the distillate for the presence of a carbonyl group using Fehling’s solution or Tollen’s reagent (the silver mirror test).