ASSESSED PRACTICAL – SKILL I

Name _____ **Date** _____

Introduction

For this exercise you are given full instructions for the practical procedure and these must be followed exactly. It is however up to you to consider suitable safety procedures and to organise your time appropriately. You should also carefully consider what is the most effective way of handling the materials and apparatus in order to obtain the maximum reliability.

The Oxidation of Ethanol

Ethanol is a primary alcohol and can be oxidised to either an aldehyde or a carboxylic acid.

$$CH_3CH_2OH + [O] \longrightarrow CH_3CHO + H_2O$$

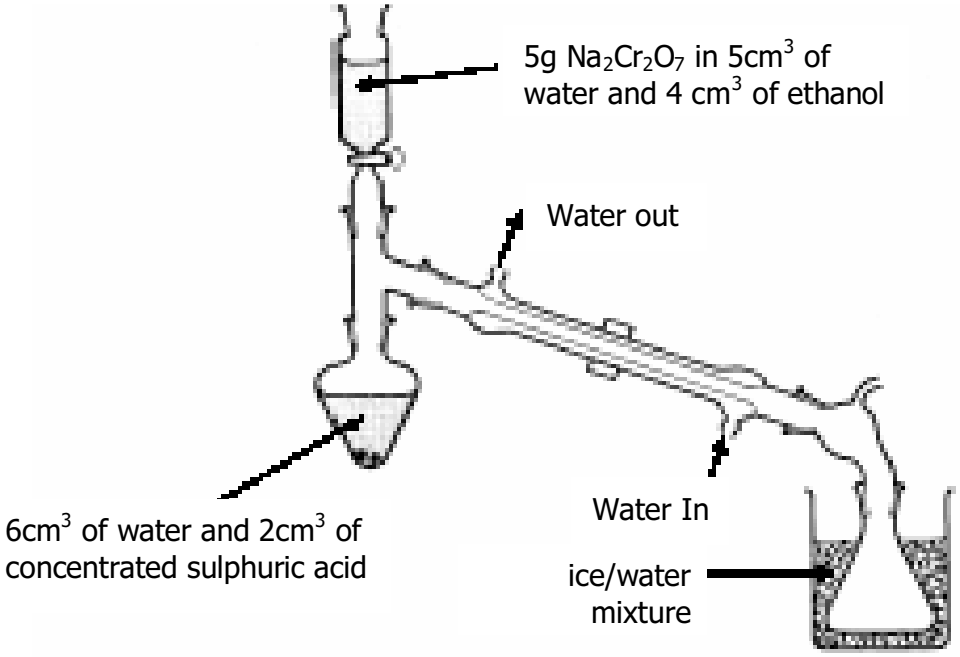
$$CH_3CH_2OH + 2[O] \longrightarrow CH_3COOH + H_2O$$

The purpose of this experiment is to oxidise ethanol and then to test the product to determine whether it has been oxidised to ethanal or oxidised to ethanoic acid.

Read through the procedure below before starting the experiment. You should comment on the hazard of the chemicals used in the experiment.

*M*ethod

To 6cm3 of water in the pear-shaped flask, add 2cm3 of concentrated sulphuric acid, and set up the apparatus as shown below, but with a stopper in place of the dropping funnel. Ensure that all of the glass joints are greased.



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During the experiment, you will need to record all your observations clearly in an appropriate format. Record these appropriately in the space below. You should also consider the hazards associated with the materials and techniques used within the experiment and record these in the space below.

1. Make up a solution containing 5g of sodium dichromate in 5cm^3 of water, add 4cm^3 of ethanol and pour the mixture into the dropping funnel.
2. Warm the acid in the pear shaped flask until it is almost boiling and turn off the electrothermal mantle.
3. Carefully remove the stopper and put the dropping funnel in position, as shown in the diagram.
4. Add the mixture containing the ethanol at such a rate as to maintain the boiling of the mixture in the pear shaped flask. Collect the distillate and write down all observations in the space below.
5. Carry out the following tests on the distillate and record your observations.

Test 1 *Test* for the presence of a carbonyl group to find out whether ethanal had been formed.

Put 5 cm^3 of 2,4-dintriophenylhydrazine in a test tube and cautiously add 5 drops of the distillate. Record your observations.

Test 2 *Test* for the presence of an acid group to find out whether ethanoic acid had been formed.

Put 5 drops of the distillate in a test tube and add 5 drops of universal indicator solution. Record your observations.

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Safety

Comment about the hazard of the chemicals in the context of this experiment.

- **Ethanol** – Ethanol irritates the eyes. Inhalation of high concentration of vapour may cause irritation of the eyes and respiratory tract. The substance may cause effects on the central nervous system. In contact with skin may produce dry skin. In order to prevent those, I decided to wear protective gloves and safety goggles during the experiment.
- **Sodium dichromate** - The substance is corrosive to the eyes, the skin and the respiratory tract. The substance burns skin, and produces eye redness, blurred vision, sore throat, cough, wheezing. For personal protection, I decided to wear protective gloves, protective clothing and safety goggles.
- **2, 4-dinitrophenylhydrazine** - is potentially explosive when dry and may decompose rapidly or explosively if heated or subject to shock. It is also very flammable. For personal protection I wore safety glasses.
- **Sulfuric Acid** - The substance is a strong oxidant and reacts violently with combustible and reducing materials. The substance is a strong acid, it reacts violently with bases and is corrosive to most common metals forming a flammable/explosive gas (hydrogen - see ICSC 0001). Reacts violently with water and organic materials with evolution of heat (see Notes). Upon heating, irritating or toxic fumes (or gases) (sulfur oxides) are formed. For protection I wore protective gloves and safety glasses.
- **Potassium Dichromate** - The substance is a strong oxidant and reacts with combustible and reducing materials. The solution in water is a weak acid. The substance is irritating to the skin and the respiratory tract. The substance is corrosive to the eyes. The substance may cause effects on the kidneys and liver, resulting in impaired functions. This was used in small amounts as an oxidising agent and it was not felt necessary to use it in the fire cupboard. For protection I wore protective gloves and safety goggles.
- **Universal Indicator Solution** - Highly Flammable, Harmful by inhalation, in contact with the skin and if swallowed, Irritating to eyes. In most universal indicator solutions the solvent is ethanol; various indicators are dissolved in this to give the necessary pH-dependent colour changes. Ethanol is very flammable, so I should treat solution as a fire hazard. For protection I should wear safety glasses when using the solution and I should keep bottles of indicator solution away from naked flames. Small amounts of indicator solution can be flushed down a sink unless local rules prohibit this. As ethanol is flammable, I should ensure that no solution remains in the sink where it might be a fire hazard.

  For personal protection protective gloves and safety goggles were worn during the experiment. Because of the chemicals used, a window was left open during the experiment in order to assure ventilation.

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Observations

Ethanol has been oxidised using potassium dichromate (VI) as an oxidising agent in dilute acid. Initially, ethanol has been oxidised to form an aldehyde, but if the oxidising agent is used in excess, further oxidation to the carboxylic acid occurs. Ethanol has been oxidised to give ethanoic acid, by heating under reflux potassium dichromate (VI).
Before starting the experiment I ensured that the desk was clean and that all the apparatus and all the glass joints were greased and cleaned. I checked the hazard cards and read carefully through each chemical notes I was going to use in the experiment. When setting up the apparatus, I decided to use clips in order to seal all the joints, for safety procedures and to obtain the maximum reliability. After carefully setting up the apparatus, I accurately measured 5 g of Sodium Dichromate using a balance and wearing protective gloves. I also measured 4 cm^3 of Ethanol and 2 cm^3 of concentrated Sulphuric Acid wearing protective gloves and safety goggles. As the oxidation of Ethanol is an exothermic reaction, during the experiment, I accurately measured the temperature, using an appropriate thermometer, in order to obtain a reliable product. The initial temperature was 21 ºC and it kept increasing until 95 ºC. At this temperature, I decided to turn off the heating and stop the experiment, as the boiling point of the water is 100 ºC and at this temperature I will get water instead of an aldehyde or a carboxylic acid. I added the mixture containing the Ethanol at such a rate as to maintain the boiling of the mixture in the pear shaped flask. During the distillation process I wore protective gloves and safety goggles, for personal protection.

Conclusion

Use your results to decide whether an aldehyde or a carboxylic acid had been formed during this experiment. Explain how you made your decision.

After collecting enough distillate to proceed with the two tests, I measured 5 cm^3 of 2, 4 – dinitrophenylhydrazine using an adequate measuring cylinder and then put it into a test tube. I cautiously added 5 drops of the distillate wearing protective gloves and safety goggles. I observed a bright orange or yellow precipitate which shows the presence of the carbon-oxygen double bond. Therefore an aldehyde has been formed.

For the second test, I put 5 drops of the distillate in the test tube and then added another 5 drops of universal indicator solution. Universal Indicator solution is a mixture of compounds that take on a variety of colors at different pHs. pH is a quantitative measure of the acidity or basicity of a solution. The more acidic a solution is, the lower its pH. The pH scale runs from 0 to 14. pHs less than 7 are acidic, pHs greater than 7 are basic and a pH of 7 is neutral, neither acidic nor basic. It ranges from red (a strong acid) to a deep violet (a strong alkali). Universal indicator turns neutral solutions green. I observed an orange precipitate and which had a pH of 3, demonstrating that an acid group has been formed. The second test shows that ethanoic acid has been formed, and therefore a carboxylic acid has been formed during the experiment.