

Preparation and characterization of alkene



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ABSTRACT The purpose of this experiment is to prepare cyclohexene from cyclohexanol, and know the properties of alkene. The first part of this experiment is preparation of cyclohexene. Simple distillation set-up is assembled. (See Figure 1). Exactly 6. mL of cyclohexanol is weighed in a quick-fit one-necked pear shaped flask. 1. 50mL of 85% H₃PO₄ is measured using the 2mL pipet and added to the weighed cyclohexanol. Another 0. 25mL of concentrated H₂SO₄ is also added. The reagents are mixed using stirring rod. Glass beads were added after it was shake very well. The heat regulator was switched to 5 or 6 and allowed to distill at 130-140°C until 0. 50mL of the mixture was left in the pear-shaped flask. The hydrocarbon layer of the distillate (mixture of cyclohexene and water) was transferred to another test tube using dropper. The hydrocarbon layer was extracted twice with 2. mL of cold water. Anhydrous calcium chloride powder was added to the hydrocarbon layer (enough to still see the liquid) and stirred occasionally for 6minutes. The solid was allowed to settle down. The liquid part of the mixture was removes using pipet and transferred to the 25mL quick-fit round bottom flask. Simple distillation set-up is again assembled. The hydrocarbon was distilled and the distillate at 82-85°C was collected. The receiver was weighed in order to get the weigh of the distillate to be used for computing the percentage recovery. The second part of this experiment is properties of alkene.

The prepared cyclohexene was used as test compound. For flammability, 2 drops of the compound was placed in an evaporating dish and lit with match. For solubility, 2 drops of compound was placed in a micro test tube, and 0.50 mL of water was added. For, bromination, 2 drops of compound was placed in a micro test tube. 1M Br₂ in CCl₄ was added dropwise, and shaken until change is observed. For oxidation, 2 drops of compound was placed in a micro test tube. 1M KMnO₄ was added dropwise, shaken, and observed for 2 minutes. For sulfuric acid test, 2 drops of compound was placed in a micro test tube. 0.0 mL of concentrated H₂SO₄ was added, and observed if formation of heat is present. Keywords: albumin, casein, invertase, Bradford Assay, Warburg-Christian Assay, Benedict's reagent

INTRODUCTION

Alkenes are more reactive than alkanes due to the presence of a double bond. The carbon-carbon double bond consists of a strong bond and a weak p bond. The typical reactions of alkenes involve the breaking of this weaker p bond, viz. , and formation of two sigma (s) bonds. [pic] through ionic mechanism. However, some addition reactions proceed through free-radical mechanism. Higher alkenes contain a long chain of carbon.

That part of the chain that forms an alkane-like structure (consisting of C-C bonds), may undergo substitution reaction as also shown by alkanes. Some characteristic reactions shown by alkenes are described below: Combustion - Alkenes, like alkanes, are highly combustible. Alkenes burn with a luminous flame to give carbon dioxide and water. The flame becomes luminous because of the higher carbon content of alkenes than alkanes. Their combustion reactions are exothermic. [pic] Due to the luminosity of the flame, the lower alkenes may be used as illuminants. Addition reactions The

p electrons of the carbon-carbon-double bond are available to an electrophile (any species seeking electrons). Thus, the addition reactions shown by alkenes are in fact electrophilic addition reactions. [pic] [pic] addition product Some addition reactions proceed through free-radical mechanism. Addition of hydrogen -Alkenes add hydrogen in the presence of platinum or nickel catalyst, to form alkanes. The reaction termed as hydrogenation, is an exothermic reaction. $C_nH_{2n} + H_2 \rightarrow C_nH_{2n+2} + \text{heat}$ This is known as Sabatier-Senderens reduction. $CH_2=CH_2 + H_2 \rightarrow CH_3-CH_3 + 132.2 \text{ kJ}$ ethene ethane Addition of halogens Alkenes react with halogens to form dihaloalkanes. The order of reactivity is, chlorine > bromine > iodine. Simply mixing together the two reactants, usually in an inert solvent like carbon tetrachloride, best carries out the reaction. [pic] Alkene dihaloalkane [pic] ethene 1, 2-dibromoethane [pic] propene 1, 2-dibromopropane Addition of bromine is useful for the detection of the carbon-carbon double bond. When a 5% solution of bromine in carbon tetrachloride is added to an alkene, it gets decolorized. This indicates the presence of a double bond in the molecule. This test is called 'bromine test'. Addition of sulphuric acid In accordance to Markownikoff's rule alkenes readily add concentrated sulphuric acid to form alkyl hydrogen sulphates. For example, Ethene gives, [pic] ethane sulphuric acid ethyl hydrogen sulphate Propene gives, [pic] Isopropyl hydrogen sulphate An alkyl hydrogen sulphate on boiling with water gives the alcohol and sulphuric acid. Alcohols are prepared from alkenes obtained from the cracking of petroleum. For example, [pic] ethyl hydrogen sulphate ethanol MATERIALS AND METHODS The reagents used in this experiment are cyclohexanol, 1M Br₂ in CCl₄, 85% H₃PO₄, 1M KMnO₄, concentrated H₂SO₄, and anhydrous calcium chloride.

The apparatus used in this experiment are quick fit, 2mL pipet, test tubes, and ordinary pipet. The first part of this experiment is preparation of cyclohexene. Simple distillation set-up is assembled. (See Figure 1). Exactly 6.0 mL of cyclohexanol is weighed in a quick-fit one-necked pear shaped flask. 1.50 mL of 85% H_3PO_4 is measured using the 2mL pipet and added to the weighed cyclohexanol. Another 0.25 mL of concentrated H_2SO_4 is also added. The reagents are mixed using stirring rod. Glass beads were added after it was shake very well. The heat regulator was switched to 5 or 6 and allowed to distill at $130\text{--}140^\circ\text{C}$ until 0.50 mL of the mixture was left in the pear-shaped flask. The hydrocarbon layer of the distillate (mixture of cyclohexene and water) was transferred to another test tube using dropper. The hydrocarbon layer was extracted twice with 2.0 mL of cold water. Anhydrous calcium chloride powder was added to the hydrocarbon layer (enough to still see the liquid) and stirred occasionally for 6 minutes. The solid was allowed to settle down. The liquid part of the mixture was removed using pipet and transferred to the 25mL quick-fit round bottom flask.

Simple distillation set-up is again assembled. The hydrocarbon was distilled and the distillate at $82\text{--}85^\circ\text{C}$ was collected. The receiver was weighed in order to get the weight of the distillate to be used for computing the percentage recovery. The second part of this experiment is properties of alkene. The prepared cyclohexene was used as test compound. For flammability, 2 drops of the compound was placed in an evaporating dish and lit with match. For solubility, 2 drops of compound was placed in a micro test tube, and 0.50 mL of water was added. For, bromination, 2 drops of compound was placed in a micro test tube. M Br_2 in CCl_4 was added

dropwise, and shaken until change is observed. For oxidation, 2 drops of compound was placed in a micro test tube. 1M KMnO_4 was added dropwise, shaken, and observed for 2 minutes. For sulfuric acid test, 2 drops of compound was placed in a micro test tube. 0.5 mL of concentrated H_2SO_4 was added, and observed if formation of heat is present.

RESULTS AND DISCUSSION

I. Preparation of Cyclohexene In the presence of a strong acid, an alcohol can be dehydrated to form an alkene. The acid used in this experiment is 85% phosphoric acid and the alcohol is cyclohexanol.

The phosphoric acid is a catalyst and as such increases the rate of reaction but does not affect the overall stoichiometry. It can be seen from the balanced reaction that 1 mole of alcohol produces 1 mole of alkene. The theoretical yield of alkene in moles is therefore equal to the number of moles of alcohol used.

Boiling Point of	Theoretical yield	Cyclohexene	of Cyclohexene	Weight of

Cyclohexanol,	Mole of Cyclohexene	g	Actual yield of	% yield

Cyclohexene Write out the balanced equation to calculate the theoretical yield. For this reaction, this is very simple. One reactant produces one product (water is also a product but we are only interested in the cyclohexene here) in a 1: 1 ratio.

Note that the phosphoric acid is a catalyst and is not involved in the yield calculation. [pic] One molecule of cyclohexanol should produce one molecule of cyclohexene. One mole (mol) of cyclohexanol should produce one mole of cyclohexene. If 2.05 g of cyclohexanol is used (use the actual amount used in your experiment) convert this to moles by dividing by the molecular

weight of cyclohexanol (MW = 100. 2 g/mol). 2. 05 g cyclohexanol / 100. 2 g/mol = 0. 0205 mol (or 20. 5 mmol) Because 1 mol of cyclohexanol should produce 1 mol of cyclohexene, 0. 0205 mol of cyclohexanol should produce 0. 0205 mol of cyclohexene.

Convert this number of moles of cyclohexene to grams of cyclohexene by multiplying by the MW of cyclohexene (82. 1 g/mol). 0. 0205 mol x 82. 1 g/mol = 1. 68 g cyclohexene In other words, 2. 05 g of cyclohexanol should produce 1. 68 g of cyclohexene. The percent yield is the percentage of the theoretical yield that you actually obtain after isolating product at the end of the procedure. Let's say that after the final fractional distillation of the cyclohexene, 1. 22 g was collected. The percent yield then would be percent yield = (actual / theoretical) x 100 = (1. 22 g / 1. 68 g) x 100 = 73 % This assumes that the 1. 2 g that was obtained was 100% pure. Let's say that when the gas chromatographic analysis was done the sample was found to be 89% cyclohexene and 11% toluene. This means that the 1. 22 g of liquid isolated in the distillation was not pure cyclohexene. The actual amount of cyclohexene collected then was 1. 22 g x 0. 89 = 1. 09 g (small print: an assumption is being made: that the GC detector response is the same for cyclohexene and toluene. We will accept this assumption for our purposes. In fact it is a good assumption in this case). The percent yield then would be (1. 09 g actual / 1. 68 g theoretical) x 100 = 65%.

II. Properties of Alkene | | |

TESTS | OBSERVATION | | | | Flammability | | | | | Solubility | | | | |

Bromination | | | | | Oxidation | | | | | Sulfuric Acid | | REACTIONS INVOLVED

| | | | Flammability Test | | | |[pic] | | Bromination | | | |[pic] | | Oxidation | | | |

Sulfuric Acid Test | Combustion of Alkenes -The alkenes are highly flammable

and burn readily in air, forming carbon dioxide and water. Addition of Water - Water can add across the double bond of the alkenes to form aliphatic alcohols. This hydration reaction is catalysed under a number of different conditions. Bromine reacts with an alkene to form a colorless dibromide. Thus, if a bromine solution is added dropwise to an alkene, the solution will decolorize until all of the alkene has reacted. Alkanes on the other hand do not react with bromine in this way, so the very first drop of bromine solution will cause a reddish-orange color to persist. Alkanes do react with bromine by a free radical mechanism initiated by light, but this reaction is much slower.) Potassium permanganate reacts with alkenes to produce a colorless diol and a finely-divided brown precipitate of manganese dioxide. Under acidic conditions the diol can further react to form a colorless dicarboxylic acid. Thus, when permanganate is added dropwise to an alkene, the purple color disappears and a brownish suspension forms until all of the alkene has reacted. Permanganate does not react with alkanes, so the first drop of permanganate added to an alkane immediately results in a purple solution. Reaction with Sulphuric Acid Similarly, fuming sulphuric acid absorbs cyclohexene at room temperature to form -----, with much evolution of heat. CONCLUSIONS AND RECOMMENDATIONS The purpose of this experiment is to prepare cyclohexene from cyclohexanol, and know the properties of alkene. Cyclohexanol was properly prepared from cyclohexene. There are two general methods for the preparation of alkenes. Both methods involve the dehydration of the appropriate aliphatic alcohol (i. e. the removal of a molecule of water from an alcohol). dehydration of the alcohol using concentrated sulphuric acid as the dehydrating agent, or by passing the vapour of the alcohol over hot alumina.

The properties of alkene were known by undergoing the test compound into different test. It is recommended that the reaction is carried out in a fractional distillation apparatus. As the alcohol and acid are heated, alkene and water are produced and co-distill into a collection vial. As in any distillation, unless precautions are taken, some of the product will be lost as hold-up in the apparatus. Hold-up would result in a reduced yield of product. To overcome this problem and to ensure that a maximum amount of product is distilled, a higher boiling " chaser" solvent is added to the distillation flask and the distillation is continued until the temperature rises well over the BP of cyclohexene. REFERENCES