Experiment 6

Preparation of Amylene

1

Preparation of Alkenes by Dehydration of alcohols

A dehydration reaction is reversible and usually has a small equilibrium constant.

This means that the equilibrium favors the reactants and not the products. In fact, the reverse reaction is a method often used to synthesize alcohols.

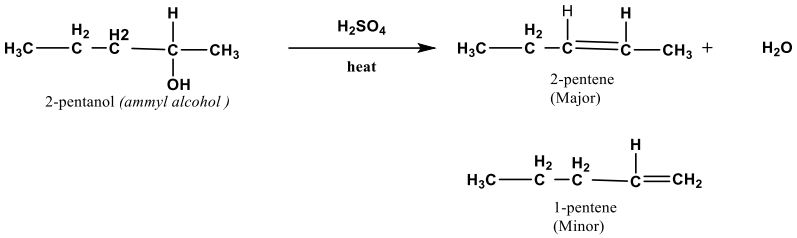
To improve the yields of a dehydration reaction:
The reaction mixture is heated in the presence of a catalytic quantity of acid.

The lower boiling product is distilled off as it forms, (either the water or the alkene).

2

Preparation of Alkenes by Dehydration of alcohols

In today`s experiment, **2-pentanol** is synthesized by dehydration of **Amyl Alcohol** .



Alcohols undergoes dehydration by an **E1 mechanism**.

The key intermediate in the mechanism is a alkyl cation, which can undergo substitution as well as elimination.

3

Preparation of Alkenes by Dehydration of alcohols

To prepare an alkene in good yield, it is necessary to **suppress the substitution reaction**.

In this experiment, the substitution reaction is suppressed by:

(1) the use of **strong acids** with anions that are relatively poor nucleophiles

(2) a high reaction **temperature**, which favors elimination.

(3) **distillation** of alkene from the reaction mixture as it is formed.

4

Preparation of Alkenes by Dehydration of alcohols

Other strong acids:

HBr, have nucleophilic anions, and thus yield more substitution than elimination products.

Concentrated sulfuric acid alone causes both oxidation and polymerization of the product alkene.

Fewer side reactions occur when concentrated **phosphoric acid** is used as the dehydrating agent, but the rate of the alkene formation is slow.

5

Procedure- Part A: preparation of amylene

Prepare a **1:1 sulfuric acid mixture** by adding cautiously, in small portions, 13.5 ml. of concentrated sulfuric acid to 13.5 ml. of cold water in a 100-ml. round bottomed flask. Cool the flask by swirling it gently in an ice bath or a stream of cold water between each addition.

Then add 27 ml. (22 g., 0.25 mole) of amyl alcohol with cooling and shaking. Mount the flask over a **steam bath** (Note 1) on a ring stand and attach it to an efficient condenser arranged for distillation. Fit the condenser with a receiving adapter which leads through a cotton plug into a 100-ml. Erlenmeyer **receiving flask packed in ice** (Note 2).

Heat the flask strongly with steam until alkene is no longer obtained in the distillate (Note 3).

6

Procedure- Part A: preparation of amylene

Transfer the cooled product to a **small separatory funnel** and add 15 ml of cold 10% sodium hydroxide solution. Invert the funnel, open the stopcock to release the pressure, then close the stopcock and shake vigorously, stopping occasionally to release the pressure.

Tap off and discard the lower aqueous layer and pour the alkene through the mouth of the separatory funnel into a small, *dry* Erlenmeyer flask. Add about 1 g of anhydrous calcium chloride and allow the flask to stand with cooling and occasional shaking.

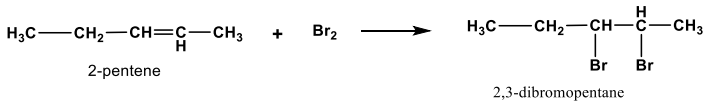
When the hydrocarbon is dry, as indicated by absence of turbidity, weigh the product and calculate the percentage yield and carry out the unsaturation tests.

When the hydrocarbon is dry, as indicated by absence of turbidity, transfer it into a small distilling flask fitted with a thermometer, and a *dry* condenser attached as before to vacuum adapter. Distill over a water bath collect the fraction boiling at 34°-41° in a small tarred collecting bottle cooled in an ice bath. Pure 2-penene is reported to boil at 36.5° at 760 mm Hg. Record the data and calculate the percentage yield.

7

Procedure- Part B : Test for unsaturation

1) **Bromine test:**
   Place 0.5 ml of **2-pentene** into a test tube . Add dropwise a 2% solution of Br_2/CCl_4 .
   Record your observations.



2-pentene

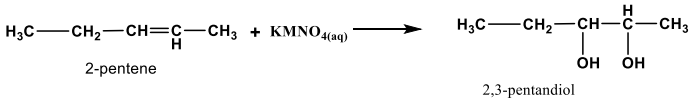
2,3-dibromopentane

8

Procedure- Part B : Test for unsaturation

2) Permanganate test:

Place 0.5 ml of **2-Pentene** into a test tube .
Add dropwise a 0.5% solution of potassium permanganate ($KMnO_4$) .
Shake the tube and record your observations.



2-pentene

2,3-pentandiol

3) Repeat the above tests using **amyl alcohol** instead of **2-pentene**

9

Property of t-amyl alcohol		Property 2-pentene	
Molar mass	88.09g/mol	Molar mass	70.14g/mol
Density	0.805g/mL	Density	0.662g/mL
Boiling point	119.3 degree Celsius	Boiling point	36.5 degree Celsius
Solubility in water	Partially soluble	Solubility in water	Insoluble

10

Distillation-Reminder