Lab-10 Dehydration of Cyclohexanol

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1 Introduction

This experiment uses an acid catalyzed dehydration to convert cyclohexanol to cyclohexene. Dehydration is a type of elimination reaction where an OH^- group is protonated to form an H_20 molecule which detaches from its original carbon bond forming a carbocation. A hydrogen proton from a carbon adjacent to the carbocation is also eliminated from the reactant. The remaining electrons form the π bond of the alkene product as result of the elimination reaction. The reaction is conducted at the boiling point of the product in a distillation apparatus. The product is separated from the reactants by vaporization, condensed in a west condenser, and collected into a separate flask. The separation by vaporization works because the boiling point of the cyclohexene product is lower than that of the reactants so only the product and water will vaporize from the solution.

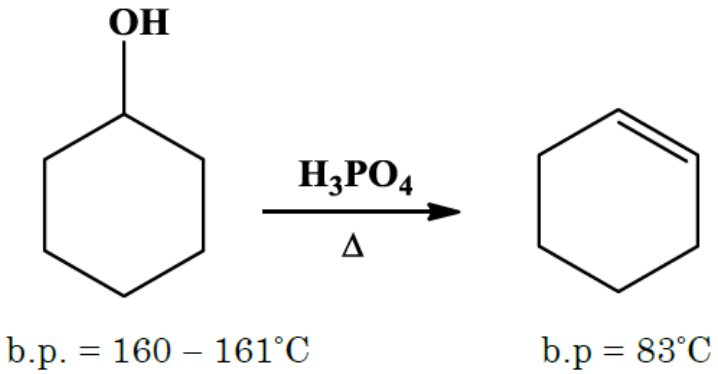

Figure 1: Summary of the conversion cyclohexanol to cyclohexene by dehydration

2 Materials

$10mL$graduated cylinder

$6.0mL$ H_3PO_4 85%

$6.0mL$ Na_2CO_3 10%

$3.00g$ $CaCl_2$ drying agent

2 Erlenmeyer flask ($50mL$)

2 round bottom flasks ($50mL$)

1 ring stand & clamps

heat/stir plate

heating mantle

variac

cotton ball

filter funnel

thermometer

distillation apparatus

$20mL$ cyclohexanol ($d = 0.96g/mL$)

3 Procedure

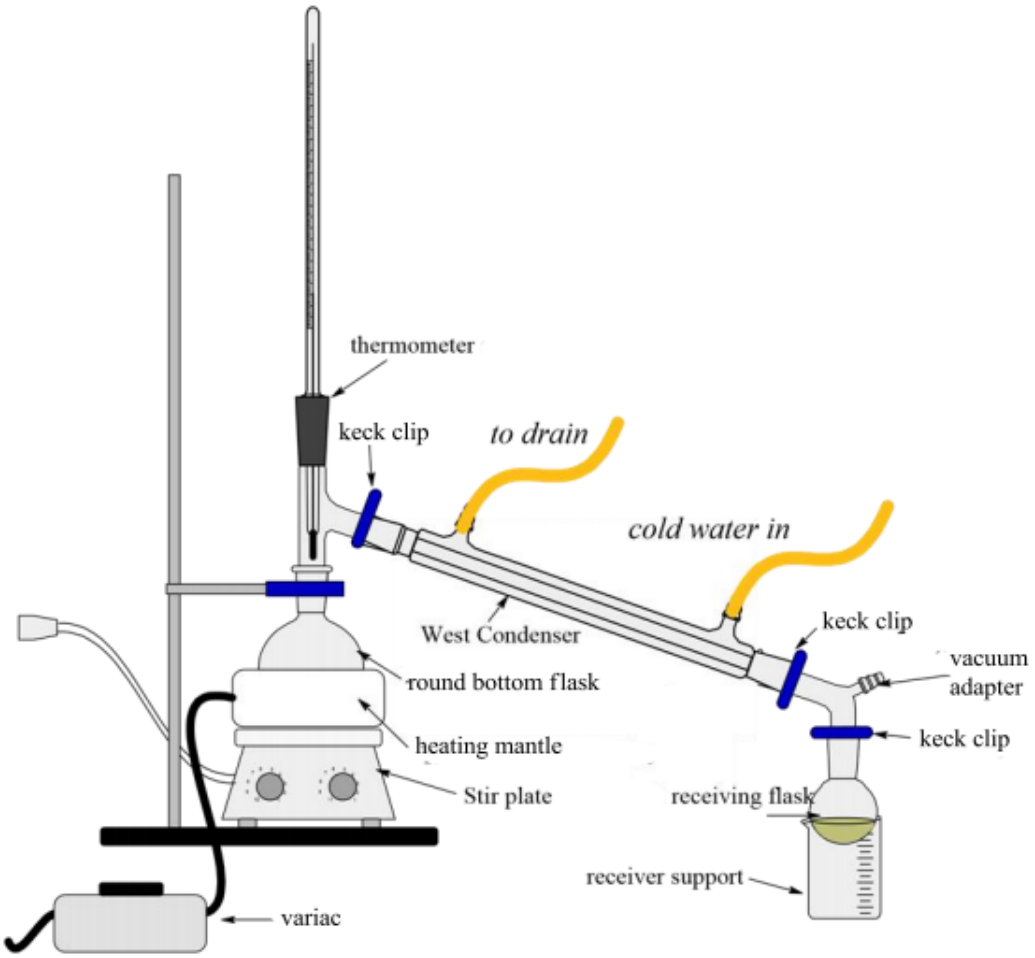

Figure 2: .

A distillation apparatus was set up according to the example in *figure* 2 prior to the experiment. All glassware was examined for cracks and leaks. $20mL$ cyclohexene, $6.0mL$ H_3PO_4 85%, and a magnetic stir bar were added to a $50mL$ round-bottom flask. The flask was then mounted to the distillation apparatus. The variac was set to 85% and the heat/stir plate knob was turned to initiate the mixing of the solution by the magnetic stir bar. The solution was mixed at 100° C until there was no more condensation being collected in the $50mL$ receiving flask. The collected product was transferred to a separatory funnel. $6.0mL$ Na_2CO_3 10% was added to the separatory funnel. The mixture was shaken and the aqueous layer was drained. H_2O was added to the remaining liquid in the separatory funnel. The mixture was shaken and the aqueous layer drained. The remaining Organic layer was then collected into a pre-weighed $50mL$ Erlenmeyer flask. $3.00g$ $CaCl_2$ was added to the $50mL$ Erlenmeyer flask as a drying agent. The product was left to dry until the next lab period. Once dried the $CaCl_2$ was removed from the product by gravity filtration. The product was weight and its purity was verified by gas chromatography.

4 Calculations/Results

Experimental Data

Mass cyclohexanol:

$$20.0mL \text{ cyclohexanol} * \frac{0.96\text{g cyclohexanol}}{1mL \text{ cyclohexanol}} = 19.2g \text{ cyclohexanol}$$

Mass $50mL$ Erlenmeyer flask:

Initial (empty) weight = $37.2434g$
Final weight (with cyclohexene) = $45.9004g$
Actual yield cyclohexene = $M_f - M_i = 8.6570g$

Overall reaction:

$$C_6H_{12}O \xrightleftharpoons[\triangle]{H_3PO} C_6H_{10}$$

Theoretical yield:

$$19.2g \text{ cyclohexanol} * \frac{1\text{mol cyclohexanol}}{100.161g \text{ cyclohexanol}} * \frac{1\text{mol cyclohexene}}{1\text{mol cyclohexnol}} * \frac{82.143\text{g cyclohexene}}{1\text{mol cyclohexene}} = 15.7\text{g cyclohexene}$$

Percent yield:

$$\text{Percent yield} = \frac{\text{Actual yield}}{\text{Theoretical yield}} * 100 = 55.1\%$$

Retention time

Distance (d) from origin to peak crest = 44.5 mm
Retention time = $d * \frac{1 \text{ sec}}{2mm} = 22.3 \ sec$

Gas Chromatography Graph

Figure 3 on *page* 4 displays the GC graph of the product that was taken in lab. The molecule exhibited one sharp peak indicating that the product was a pure sample.

IR Spectrum

Figure 4, on *page* 4 shows an example IR spectrum of pure cyclohexane. The molecule is characterized by an alkene (Sp^2 C-H) stretch just above $3000cm^{-1}$, a weak alkene (C=C) stretch around $1650cm^{-1}$, alkane (Sp^3 C-H) stretches between 2850-$3000cm^{-1}$, alkane (-C-H) bending between 1350-$1480cm^{-1}$, and out-of-plane (C-H) bending near the extremes of the 650-$1000cm^{-1}$ range.

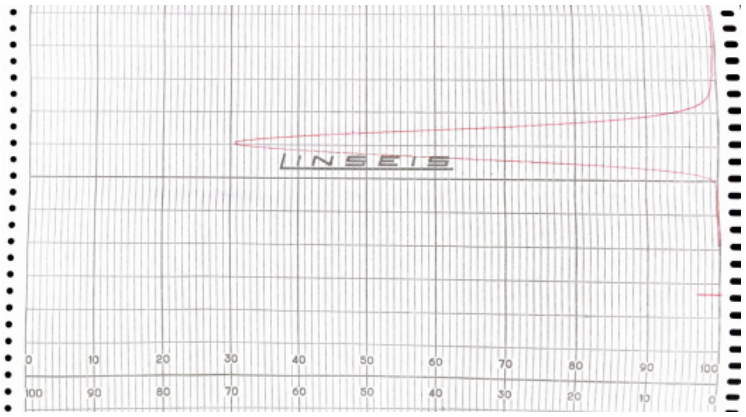

Figure 3:

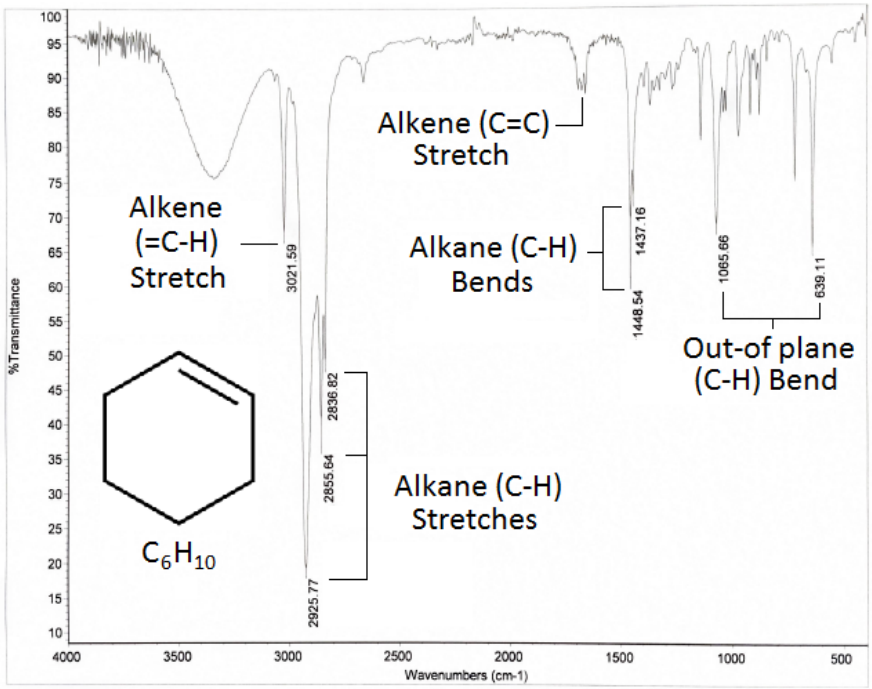

Figure 4:

5 Conclusion

The cyclohexanol was successfully converted to cyclohexene by an acid catalyzed dehydration reaction the collected product yielded 55.1% of the expected amount. A major reason for such a low yield is because the experiment was cut short due to time limitations. A higher yield could have been accomplished by allowing the experiment to continue until no more product was being collected in the receiving flask. The purity of the product was measured by gas chromatography to be 100%. This is evident by the single sharp spike on the GC graph.

Conclusion Responses

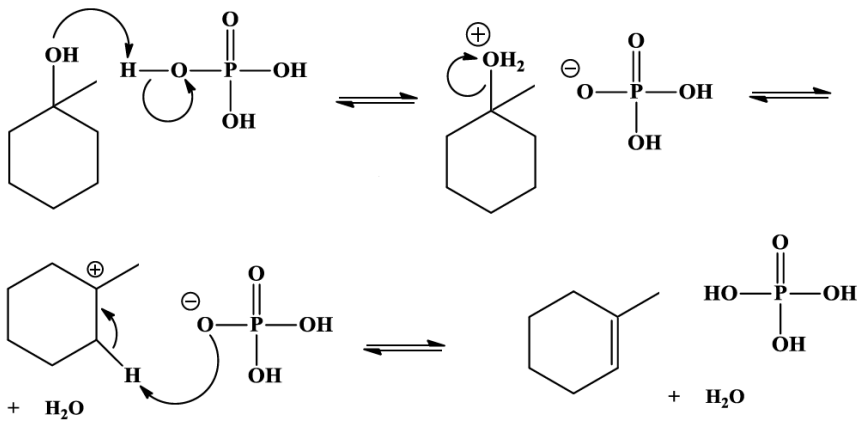

Figure 5: Mechinism of the conversion of 1-methyl-1-cyclohexanol to 1-methyl-1-cyclohexene

- The reaction mechinism, shown in *figure* 5, shows the reaction was catalyzed by H_3PO_4 85%. The first step of the reaction includes protonization of the OH group on the 1-methyl-1-cyclohexanol molecule by H_3PO_4, which converts the OH group to H_2O, a good leaving group. The H_2O then leaves the molecule creating a carbocation. An elimination reaction then takes place when H_2PO_4 is protonated by accepting the hydrogen proton from the β carbon adjacent to the carbocation. A π bond formation of an alkene results between the α carbocation and β carbon that donated the hydrogen proton, characterizing the 1-methyl-1-cyclohexene product.
- In general a major disadvantage of using concentrated sulfuric acid rather than H_3PO_4 85% is that it is a stronger acid which is 15% more concentrated, making it a more hazardous chemical to handle and work with. There is potential for toxic gasses and increased risk of severe chemical burns. With respect to the dehydration of alcohols, the concentrated sulfuric acid will react with and protonate the alkene product resulting in unwanted side reactions as well.
- Keeping the receiving flask on ice during the preparation of cyclohexene will minimize the cyclohexene vapors escaping the flask.
- If the actual yield of $0.138g$ cyclohexene (C_6H_{10})was obtained from $0.240g$ cyclohexanol ($C_6H_{12}O$) what is the percent yield?

  Actual yield:

  $0.138g$ cyclohexene (C_6H_{10})

  Theoretical yield:

$$0.240g\ C_6H_{12}O * \frac{1\text{mol } C_6H_{12}O}{100.161g\ C_6H_{12}O} * \frac{1\text{mol } C_6H_{10}}{1\text{mol } C_6H_{12}O} * \frac{82.143\text{g } C_6H_{10}}{1\text{mol } C_6H_{10}} = 0.197\text{g } C_6H_{10}$$

$$\text{Percent yield} = \frac{\text{Actual yield}}{\text{Theoretical yield}} * 100 = 70.1\%$$