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CHEM 345
10/20/2019

Preparation and Distillation of Cyclohexene

Reference: Handout; Chemistry Lessons: Elimination/dehydration reactions, le Chatelier's principle, distillations; Green Lessons: use of safer/milder reagents

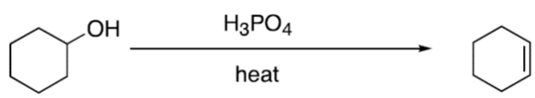
Purpose: To synthesize cyclohexene from cyclohexanol from an acid-catalyzed dehydration reaction

Table of Reagents:

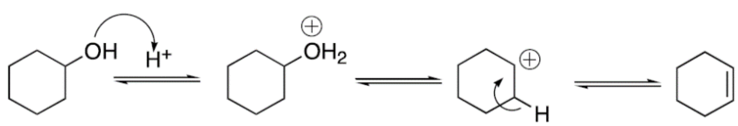
Reagents	MW	BP (°C)	MP (°C)	Density
Cyclohexanol	100.158 g/mol	161.8 °C	25.93 °C	0.9624 g/cm^3
Phosphoric Acid	97.994 g/mol	158 °C	42.35 °C	1.834 g/cm^3
Cyclohexene	82.143 g/mol	82.98 °C	703.5 °C	-

Balanced Chemical Equation:

- **Reaction**



- **Mechanism**

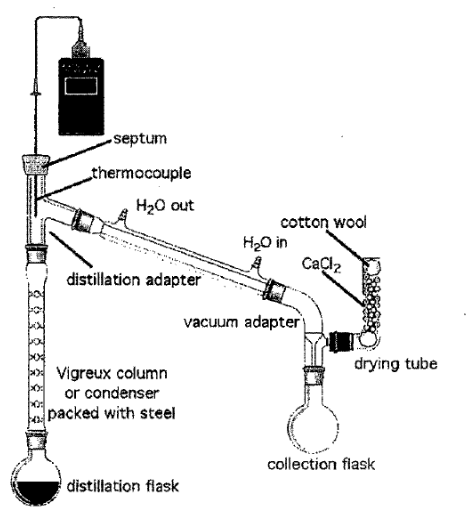


Safety:

- Cyclohexene → flammable and bad odor (place in ice bath below the collection flask)
- Phosphoric Acid → Corrosive (DO NOT TOUCH!)

Experimental Procedures	Data & Observations
1. Obtain a 50 mL round-bottom flask with boiling chips; add 0.074 moles of cyclohexanol and 1.75 mL of 86% H_3PO_4. Swirl to mix!	*Cyclohexanol Used:* 7.4232 g

2. Follow diagram below to set-up distillation
Make sure seal is good with the rubber septum or cyclohexene will escape!



Observations:

- Distillation Flask → boiling
- Collection Flask → foggy

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3. Heat reaction mixture at low for 5 min, then slowly increase temperature. Keep distilling until volume remaining in distillation flask is ~1 mL

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4. Transfer distillate to separatory funnel and wash with 5 mL of water. Separate the layers and transfer organic layer to dry Erlenmeyer flask. **Remove any water droplets, before adding drying agent ($NaSO_4$)***

Observations:

- Organic Layer → top
- Water → bottom

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5. Add Sodium Sulfate to flask and swirl gently. Let sit for 5 min. (If clumps happens, add more $NaSO_4$). Drying agent should be free-flowing and liquid should be clear if water has been removed.

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6. Decant/pipette liquid from drying agent into dry round bottom flask. (Distillation for next step)

- Appropriate size depends on yield; should be half full at beginning of distillation

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7. Simple distillation → add distillation adapter and condenser to the flask; Be sure thermometer is positioned properly. Distill the organic material → collect in the 80 °C – 90 °C range. Record boiling range observed.	
8. Determine mass of cyclohexene in weighted vial. If possible, record IR of sample. Seal and label vial with cyclohexene for next class	*<u>Mass of Cyclohexene:</u>* 1.0630 g

Post-lab Questions:

1. Start → 0.074 moles of Cyclohexanol used

$$0.074\ mol\ cyclohexanol \times \frac{1\ mol\ cyclohexene}{1\ mol\ cyclohexanol} = 0.074\ moles\ of\ cyclohexene$$

- The theoretical yield in obtaining cyclohexene should be equal to the initial amount used of cyclohexanol, since it's a 1:1 ratio in reactants to products (depicted in the chemical equation above).

2.

- Mass of Cyclohexene Product: 1.0630 g
- $\%\ Yield = \frac{Moles\ of\ Cyclohexene\ obtained}{Theoretical\ yield\ of\ Cyclohexene}$
    - $1.0630\ g\ of\ Cyclohexene\ \times \frac{1\ mol}{82.143\ g} = 0.0129\ mol\ of\ Cyclohexene$
    - $\%\ Yield = \frac{0.0129\ moles}{0.074\ moles} \times 100 = 17.43\ \%\ yield\ of\ Cyclohexene$

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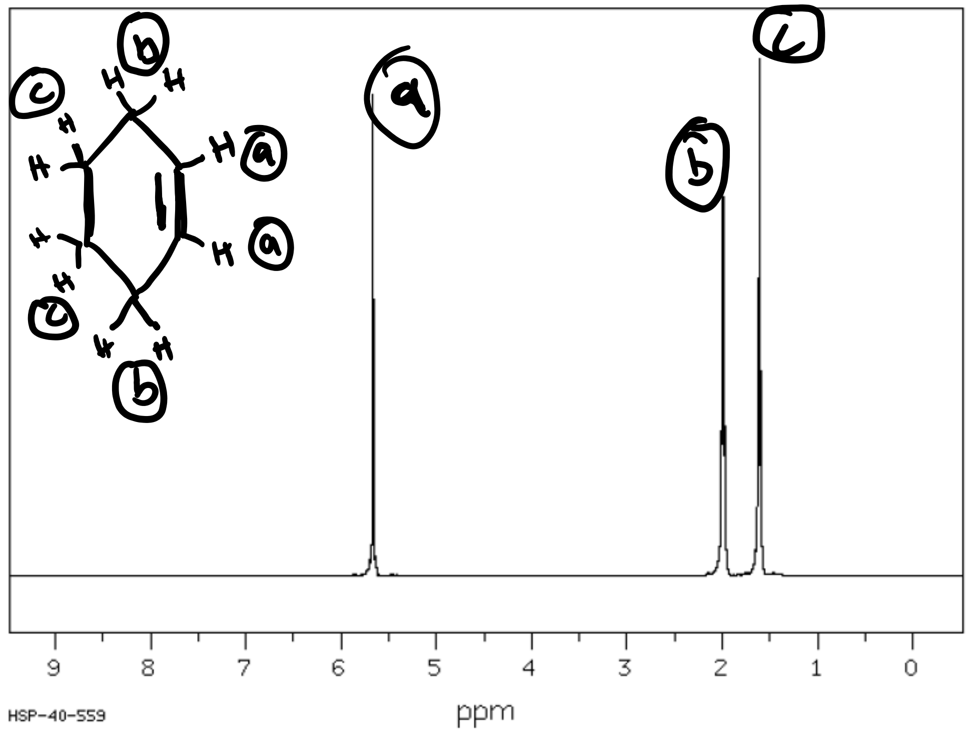
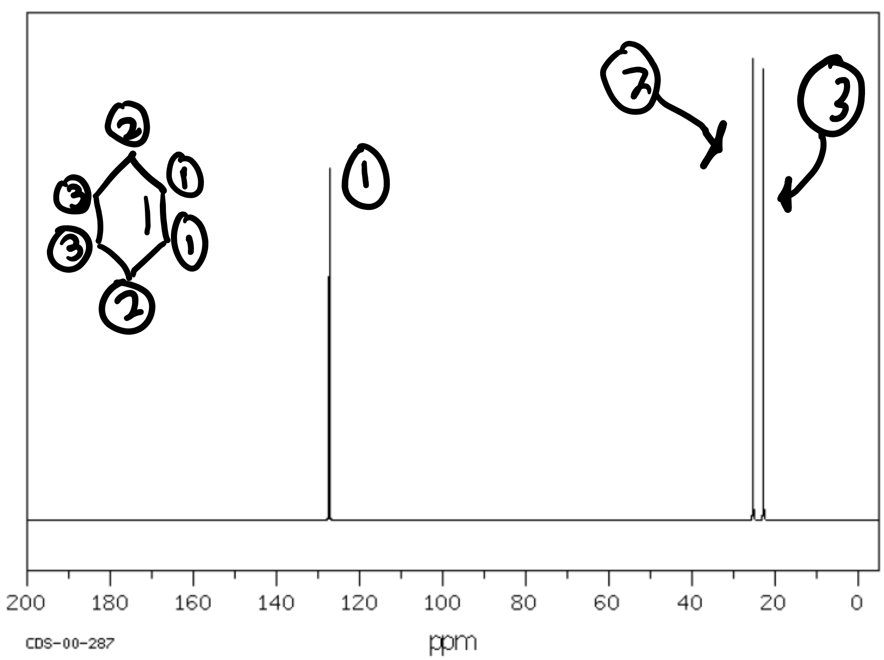
3. Spectroscopic Interpretation

a. Spectra of Cyclohexene

^1H NMR ($CDCl_3$)



^{13}C NMR ($CDCl_3$)



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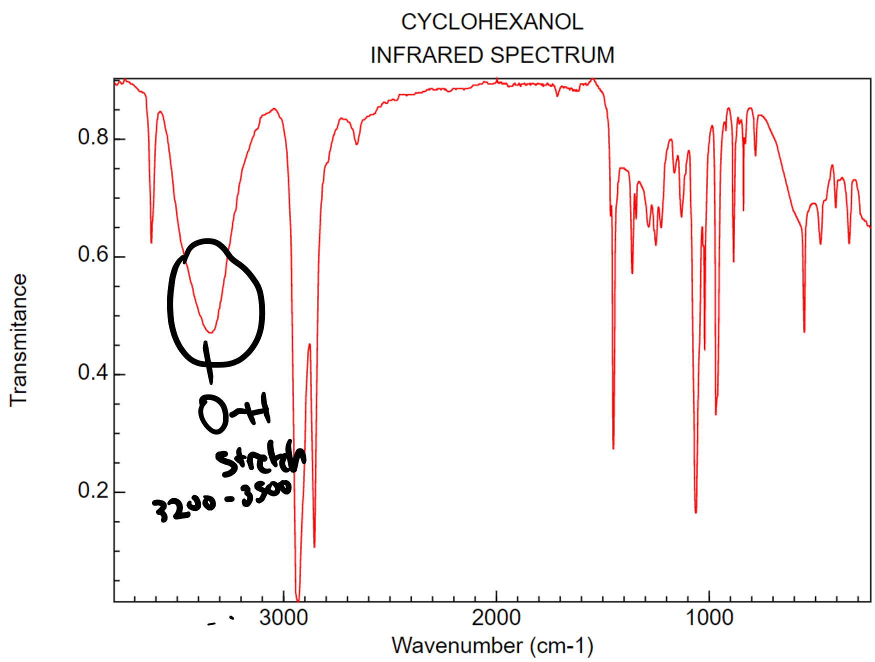
b.



c. Yes, we will be able to determine if our reaction was a success, since cyclohexanol has a broad O-H stretch around the 3200 – 3500 cm^{-1}, while cyclohexene does not have the O-H stretch. In addition, we will also be able to see the C=C, double bond stretch appearing around the 3010 – 3100 cm^{-1} on cyclohexene, which will be absent in cyclohexanol.

4. Complete the sentence

- Phospheric acid serves as a **catalyst** in this reaction.

5. In order to drive the reaction towards the products area, we must keep decreasing the amount of cyclohexene to the process going (if not will reach equilibrium). In addition, as stated in Le Chatlier's principle, if a disturbance is present, the system wants to shift against the disturbance in order to maintiain equilibrium. In our case, we could increase the amount of phospheric acid (catalyst) or increase the temperature to further push the reaction towards the products resulting in a better yield.

6.