Name: Anna Li Report Submission Date: 3/14/20

Experiment Title: **Cyclohexene Synthesis and Gas Chromatography**

Section Day and Time Friday PM: If Make-Up, lab performed on date:

Teaching Assistant: Sarah Amorin

Lab Technique: Leave this section blank in your report. But, make sure to tell Gradescope it is on page one of your lab report. Your TA will assign the technique points when they grade the post-lab. Do not delete this text.

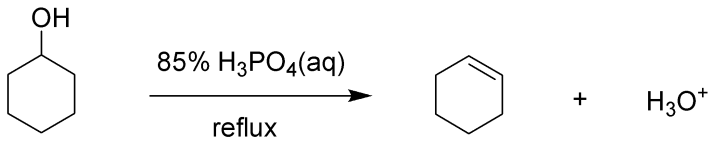
Purpose: The purpose of this experiment is to synthesize cyclohexene from cyclohexanol with an acid-catalyzed dehydration reaction. IR analysis, gas chromatography, and chemical tests with bromine in dichloromethane and potassium permanganate will be used to assess the identity and purity of the cyclohexene product.

Pre-Lab: Leave this section blank in your post-lab report. But, make sure to tell Gradescope it is on the appropriate page. *Your TA should have graded the prelab before you began the experiment*. They will assign the awarded points for the prelab with the report. Do not delete this text.

In-Lab Observations and Recordings:

- Experiment performed on 3/5/2020
- 2.002 g cyclohexanol used
    - Extra 1.0 mL of 85% phosphoric acid added (1.5 mL total)
    - Temperature range of distillation: 63 °C - ??? (did not reach)
    - Rinsed distillation column with water to retrieve cyclohexene product
    - Final product only consisted of ~4 drops and was not weighed, % yield very low
        - 2.002 g/100.158 g = 0.0200 mol cyclohexanol = 0.0200 mol cyclohexene theoretical yield = 1.64 g cyclohexene theoretical yield
        - [mass of 4 drops] = experimental yield of cyclohexene
        - [experimental yield of cyclohexene]/1.64 g x 100 = % yield
- Gas chromatography was not performed due to broken machine
- Chemical tests
    - Cyclohexene product
        - bromine in dichloromethane: solution became clear
        - potassium permanganate: precipitate formed, creating a cloudy suspension. Solution lost purple color
    - Cyclohexane control
        - bromine in dichloromethane: no color change
        - potassium permanganate: no color change, and no precipitate

Reaction Scheme:



Experimental Procedure:

Cyclohexanol (2.002 g) was added to a round-bottomed flask with enough 85% phosphoric acid (1.5 mL) to catalyze the reaction at a reasonable speed. The solution was heated until it became a dark yellow color. Then, it underwent fractional distillation without changing of collection vials. During the reaction work-up, the product was moved into a test tube and washed with water (~1 mL). The lower aqueous layer that resulted was removed to waste. 1 M sodium hydroxide (~1 mL) was added and thoroughly mixed in. The lower aqueous layer was removed to waste. Next, the test tube contents were washed with brine (~1-2 mL). The lower aqueous layer was again removed to waste. The organic layer was then transferred to a dry vial. Several spheres of calcium chloride were added and the vial was left to sit undisturbed for 5 minutes until water was fully removed from the solution. Finally, the product was analyzed with IR and tested with bromine in dichloromethane and potassium permanganate alongside a cyclohexane control.

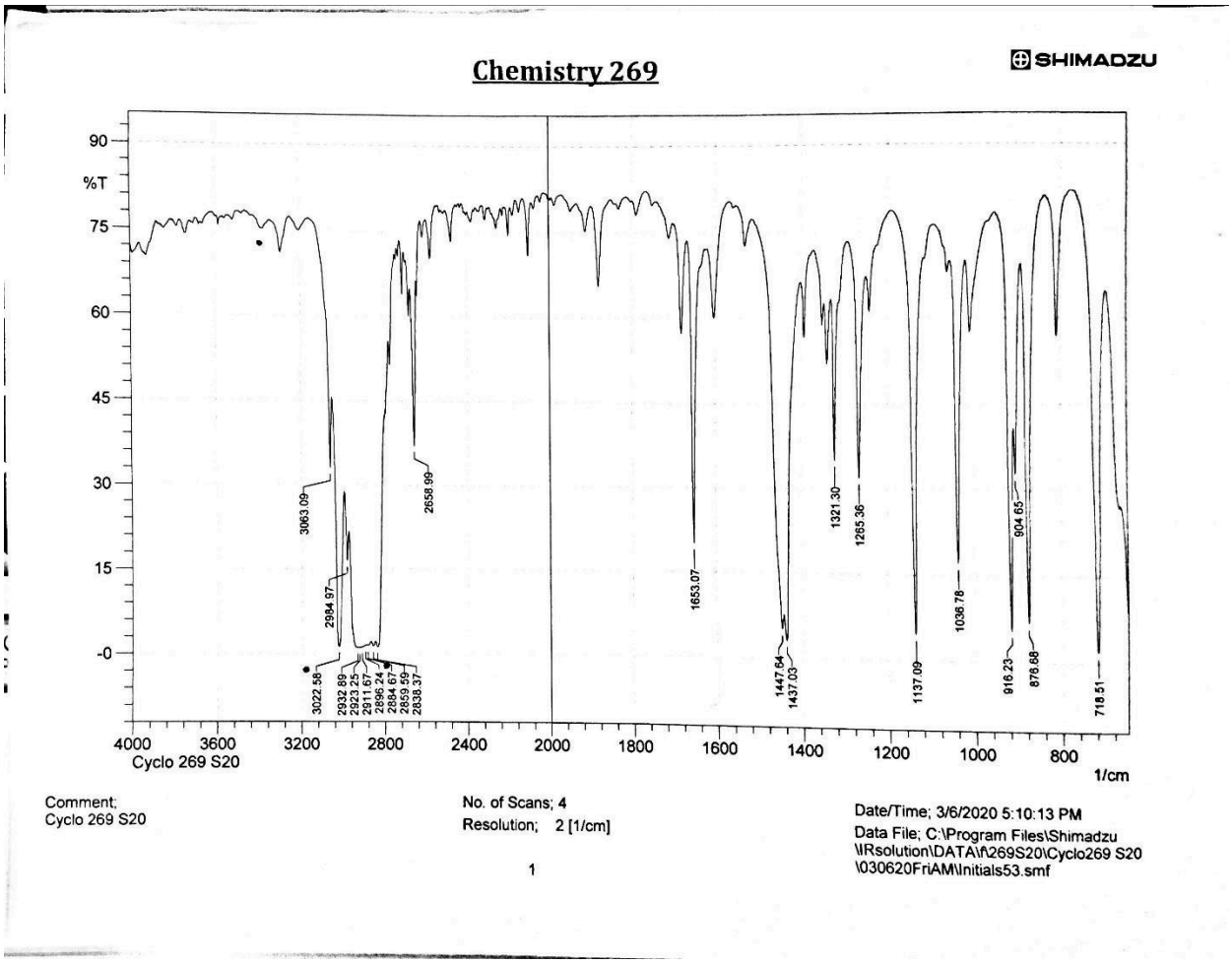
Results

Table 1. Cyclohexene

Reaction Product	Melting Point	Boiling Point	Percent Yield (%)	Character-ization Methods	Starting mass (cyclo-hexanol, g)	Theoretical yield (g)	Final mass	Distill-ation Range
Cyclo-hexene	Not obtained	Not obtained	Very low, Mass of 4 drops/1.642	IR, chem. tests with bromine in dichloro-methane and potassium perman-ganate	2.002	1.64	~4 drops	63 °C - ???

Table 2. Chemical Test Observations

Chemical	Bromine in Dichloromethane	Potassium Permanganate
Cyclohexene	Solution became clear	Precipitate formed, solution became clear
Cyclohexane	No change observed	No change observed



Discussion:

The purpose of this experiment was to synthesize cyclohexene from cyclohexanol through an acid-catalyzed reaction using phosphoric acid. Afterward, the cyclohexene product was characterized through IR analysis and chemical tests with bromine in dichloromethane and potassium permanganate. Gas chromatography could not be performed due to machine malfunctions.

The cyclohexene started distilling at 63 °C and, had the experiment continued, would have plateaued at about 71 °C. This is lower than the boiling points of both water and cyclohexene---100 °C and 83 °C, respectively---the two products of the acid-catalyzed reaction. This was likely caused by the formation of a positive azeotrope of the two substances that co-distilled at a lower

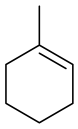
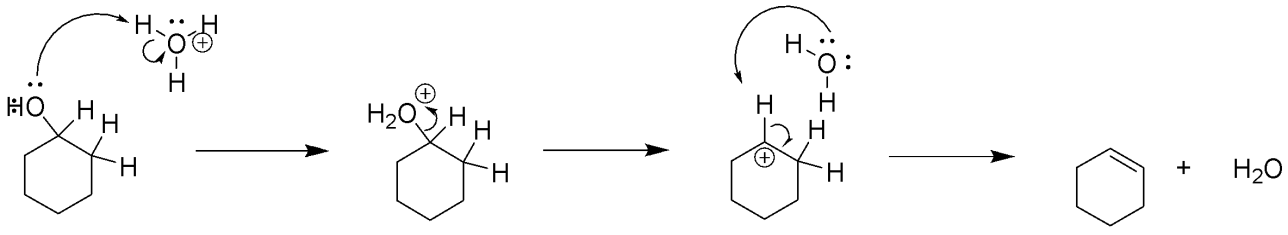
temperature than they would have separately. Thus, the boiling points of water and cyclohexene could not be obtained in this experiment.

IR analysis showed no peaks at around 3400 cm^{-1} which are where those associated with the presence of alcohol groups and water would appear, signifying that the sample should be relatively pure and free from cyclohexanol, an alcohol, and water. The presence of cyclohexene was confirmed by the presence of a significant peak at 3023 cm^{-1} which suggests the presence of the sp^2 hybridized carbons that should be present in cyclohexene's double bond. Furthermore, the peak from about 2800 – 2900 cm^{-1} reflects the presence of carbon-carbon single bonds, which is also expected from a sample of cyclohexene.

The chemical tests confirmed the presence of cyclohexene in the product as well. Bromine reacts with alkenes like cyclohexene to form a colorless dibromide and this color loss was exactly what was observed. Potassium permanganate, meanwhile, forms a colorless diol and a brown precipitate of manganese dioxide upon contact with an alkene. This loss of color was clearly observed, and though the precipitate was too fine to determine its color, it clearly formed a cloudy suspension. The precipitate might have been easier to see if more cyclohexene had been added. The cyclohexane control did not react visibly with either of these two chemicals.

The percent yield of cyclohexene was very low because distillation never plateaued, resulting in very few drops. This may have resulted from a faulty hot plate that could not raise the temperature of the solution enough to distill at a reasonable rate. Due to time constraints and a clearly unusual percent yield, the final mass was not taken. The experimental final mass and percent yield would have been uninformative about the efficiency of the procedure itself, because the problem probably originated with the heating apparatus.

Postlab Questions:

1. 
2. 

3. Raising the column temperature would reduce the retention time.
4. The peak for cyclohexene would increase in area while those of other components would decrease in area if more cyclohexene were added, changing the ratio of chemicals in the sample.