ChBE 203

Organic Chemistry Laboratory

Experiment 10

Dehydration of Methylcyclohexanols

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TABLE OF CONTENTS

1. PURPOSE

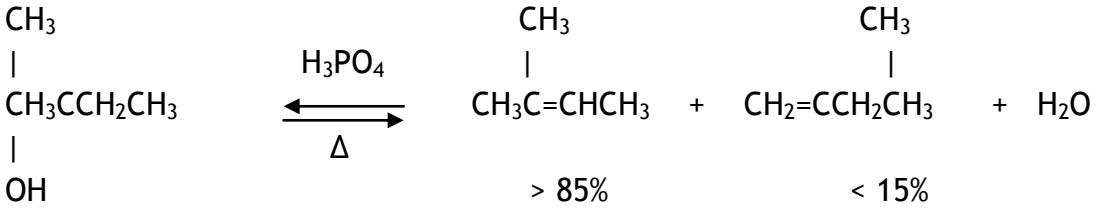
The purpose of this experiment is performing the dehydration reaction of methylcyclohexanol in the presence of phosphoric acid, by using fractional distillation method.

2. THEORY

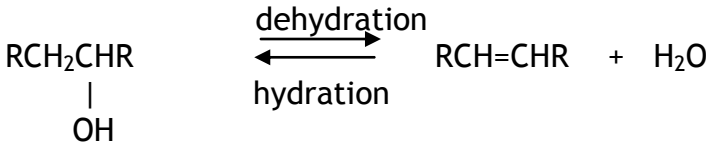
2.1 Cyclohexanol

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.

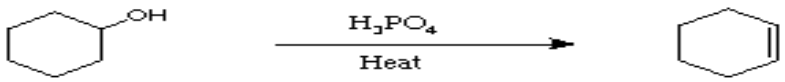
Dehydration of 2-methyl-2-butanol produces primarily 2-methyl-2-butene, a tri-substituted alkene, rather than 2-methyl-1-butene, a di-substituted alkene:



Alkenes can be hydrated (adds water) in the presence of an acid catalyst.
The hydration of an alkene is the reverse of the acid catalyzed dehydration of an alcohol:



Cyclohexanol is dehydrated to cyclohexene according to the following reaction:[1]



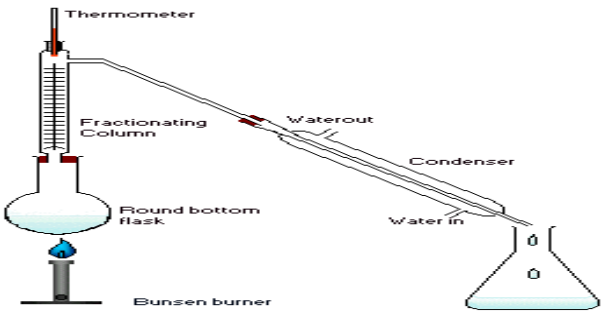
T_b = 160-161°C T_b = 83°C

2.2 Distillation

Distillation is a widely used method for separating mixtures based on differences in the conditions required to change the phase of components of the mixture. To separate a mixture of liquids, the liquid can be heated to force components, which have different boiling points, into the gas phase. The gas is then condensed back into liquid form and collected. Repeating the process on the collected liquid to improve the purity of the product is called double distillation. Although the term is most commonly applied to liquids, the reverse process can be used to separate gases by liquefying components using changes in temperature and/or pressure. Distillation is used for many commercial processes, such as production of gasoline, distilled water, xylene, alcohol, paraffin, kerosene, and many other liquids. Types of distillation include simple distillation (described here), fractional distillation (different volatile 'fractions' are collected as they are produced), and destructive distillation (usually, a material is heated so that it decomposes into compounds for collection).[2]

2.3 Fractional Distillation:

Fractional distillation is a method of obtaining the performance of a multiple simple distillations within a single pass process. This is achieved by running the vapor through a fractionating column. A fractionating column is a tube in which the vapor is forced to travel over cooling surfaces on which the less volatile parts of the vapor condense and then trickle back into the vaporizing vessel. Here the most volatile fractions of the vapor reach the top and escape, in turn to be liquified by the condenser which drains into a collection vessel. As this process continues the most volatile components are taken off first, temperatures then increase over time, while the more volatile components are taken off in sequence. [3]



3. APPARATUS

3.1 Equipment

- Graduated cylinder (50mL)

- Thermometer

- Thermometer adapter

- Pipette (5mL)

- Suction Bulb

- Erlenmeyer Flask (25 and 250mL)

- Fractionating Column

- Condenser

- Round Bottom Flask (500mL)

- Stand

- Clamps

- Boiling Chips

- Heater

- Separatory Funnel

3.2 Chemicals

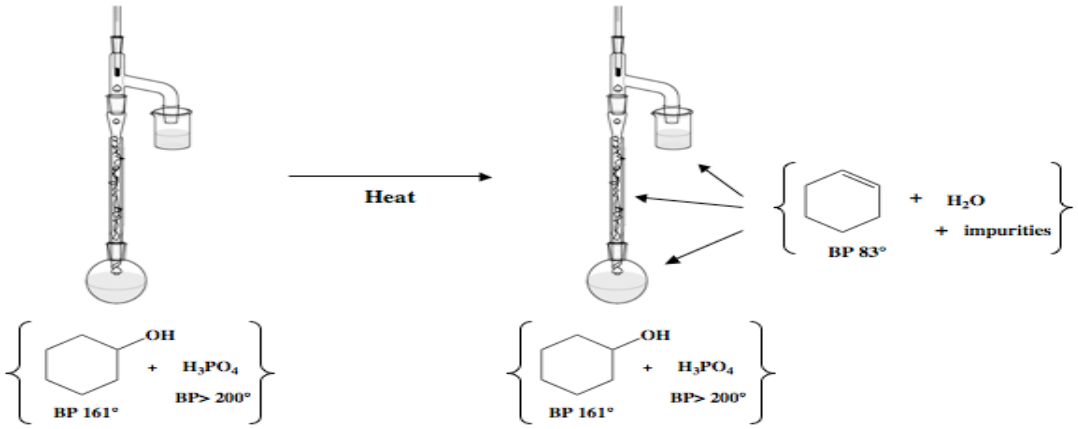
- Methylcyclohexanol

- Phosphoric Acid %85

- Saturated Sodium Bicarbonate Solution

4. PROCEDURE

17.13 g of methlycyclohexanol was measured and was added into the round bottom flask. 5mL of %85 phosphoric acid was added to the round bottom flask. Several boiling chips were put into the flask and it was shaken in circular motion for the materials to mix. Fractional distillation apparatus was set up. The receiving flask was placed in a beaker full of ice. The heater was turned on and as the mixture boiled and evaporated, required observation s were made. The contents of the receiving flask were poured into a separatory funnel. 10mL of saturated sodium bicarbonate solution was added into the funnel. Lower phase was discarded and the upper phase was transferred into a flask. The product was measured.



Cyclohexanol plus Phosphoric Acid are heated

As the reaction proceeds, cyclohexanol is being converted to cyclohexene and water (plus some impurities). The low-boiling cyclohexene and water boil at the high temperatures and distill up the column and into the collection vial. As with all distillations though some is left behind in the flask and in the column.

5. CALCULATIONS

-Calculation of the percentage of yield:

$$Percentage\ of\ yield = \frac{Mass\ of\ actual\ yield}{Mass\ of\ theoretical\ yield} \times 100\%$$

$$Percentage\ of\ yield = \frac{4.30g}{14.43g} \times 100\%$$

$$Percentage\ of\ yield = 29.80\%$$

6. RESULTS AND DISCUSSIONS

The experiment was done in a fractional distillation apparatus. As the alcohol and acid are heated, the reaction takes place and produces alkene and water. Fractional distillation apparatus, inherently prevents material loss. Because of the faulty set up of the distillation apparatus, most critical interval of the thermometer was unobservable during the reaction. 40-80^{o}C interval was blocked by the thermometer adapter. Because of this we were unable to observe the temperature where evaporation took place. Obtained yield was 4.3g (%29.8). By utilizing a strong acid, an alcohol can be dehydrated to become an alkene. By using 85% phosphoric acid and methylcyclohexanol, we performed such dehydration. The phosphoric acid is acts as a catalyst in this experiment, it speeds up the reaction and increases its yield. The evaporation of the mixture and movement of the gas inside the apparatus was observable. The gas was white and dense. As it proceeded down in the cooling part of the distillation apparatus, it became liquid and by the time it reached the receiving flask, it was completely liquid. The cooling process is very similar to the reflux apparatus's cooling mechanism. There is an outer shell, a water in and water out. Constant flow of cold water around the gas, cools it down and therefore it becomes a liquid. We used a small round bottom flask compared to the heater. This caused a slow rate of boiling due to relatively small contact surface. Because it took longer than expected to boil the mixture, we did not have the opportunity to perform the second distillation, which was to be done with a simple distillation apparatus. %85 phosphoric acid is a strong acid and needs to be handled with extra caution. Working without gloves should never be even considered but especially in this experiment, it can result in serious tissue damage.

REFERANCES

[1] http://www.chem.umass.edu/~samal/269/cyclohexene.pdf

[2] http://chemistry.about.com/cs/5/f/bldistillation.htm

[3] http://www.canadaconnects.ca/chemistry/10104/