CHEM 51LB EXPERIMENT 5
DEHYDRATION OF 1- AND 2-BUTANOL AND
DEHYDROBROMINATION OF 1- AND 2-BROMOBUTANE:
ANALYSIS OF GASEOUS PRODUCTS BY GAS CHROMATOGRAPHY

REACTIONS: Elimination
TECHNIQUES: Gas Chromatography

In this experiment, we will compare and contrast eliminations under acidic and basic conditions. We will conduct the acid-catalyzed dehydration of a primary and a secondary alcohol. We will also conduct the base-induced dehydrobromination of the isomeric halides. The stereo- and regio-chemistry the four reactions will be investigated using gas chromatography to analyze the product mixtures.

READING ASSIGNMENT:
- Elimination experiment background handout!
- This handout!
- Gas Chromatography in Making the Connections (2
- Supplementary info can be found in Janice Gorzynski Smith (3rd 
& 4th ed), Ch 8, Ch 9.8-9.9

PRE-LAB ASSIGNMENT:
- Complete all portions of pre-lab notebook work according to guidelines
- Rewrite the procedure in your own words!
- Sapling assignment

IMPORTANT SAFETY INFORMATION

All alkyl halides are harmful if inhaled, ingested, or absorbed through the skin. Wear gloves, and keep all chemicals/reactions in the hood at all times.

Potassium tert-butoxide is corrosive and moisture-sensitive. Avoid contact with skin, eyes, and clothing.

Sulfuric acid is corrosive and causes burns. Avoid contact with skin and eyes. Notify the instructor if any acid is spilled.

1-butanol and 2-butanol are flammable and toxic. Avoid contact with skin, eyes and clothing.

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EXPERIMENTAL:

You will begin in groups of four and then split into pairs. One pair will perform Part A of this experiment while the other pair will perform Part B. At the end of the experiment, you will swap data with the other pair in your group. **Do not leave the lab without all of the data you need.**

**PART A**

**A1. Dehydration of 2-Butanol**

Obtain an experiment kit from the stockroom.

In a reaction tube, place 2 drops of concentrated sulfuric acid. Add a boiling stone and connect the reaction tube to the gas collection assembly as shown in Fig. 4.1.

Assemble the gas collection apparatus shown in the figure 4.1 before the reactants are mixed. To position the gas collection tube, carry out the following steps:

1. Fit the collection tube with a septum stopper in one end.
2. Fill the collection tube with water. *What is the purpose of the water?*
3. Insert the end of the Teflon tubing that is coming out from the top of the septa into the collection tube all the way up to reach the stopper.
4. Place your index finger over the open end of the tube and invert it (water should have remained in the tube).
5. Place the tube, with the open end down, into a 250-mL beaker containing approximately 1/2 water.
6. Remove your finger (the column of water should remain in the tube.)
7. Connect the other end of the Teflon tubing to the reaction tube as shown in figure 4.1.
After the assembly is complete, use a syringe to add 0.2 mL of 2-butanol to the reaction tube and heat the mixture in a preheated sand bath. Generation of gaseous product can be observed by watching the rapid decrease of the water level in the collection tube. **Keep heating until no more bubbles escape from the bottom of the collection tube.** Remove the Teflon tubing from the gas collection tube, and then from the beaker, before removing the reaction tube from the heat, **all the while ensuring your collection tube stays below the beaker’s water level.** Why is it important that the collection tube stays below the water level? Use this sequence of steps in shutting down the reaction to prevent water from being sucked back into the hot reaction tube while it is cooling down. Label the beaker containing the collection tube kept in the water and submit it to your TA for the gas chromatographic analysis.

The butenes have been determined to elute from the non-polar column (SE30) the GC is equipped with in the following order: 1-butene, trans-2-butene, and cis-2-butene. If you did not know the order of elution for the gases, how could you determine it experimentally? Determine the relative amount (% composition) of the three components of gas products, assuming that the amount of each substance in the gaseous mixture is proportional to the area under its corresponding GC peak. Assume that these areas are equal to the peak height (mm) x the peak widths at a half-height (mm).

**A2. Dehydration of 1-Butanol**

Repeat the procedure for part A1 using 1-butanol instead of 2-butanol. Be sure to read chemical labels carefully and keep track of which alcohol you are adding!

**PART B**

**B1. Dehydrobromination of 1-Bromobutane**

Obtain an experimental kit from the stockroom.

Assemble the gas collection apparatus shown in the figure 4 before the reactants are mixed. To position the gas collection tube, carry out the following steps:

1. Fit the collection tube with a septum stopper in one end.
2. Fill the collection tube with water. **What is the purpose of the water?**
3. Insert the end of the Teflon tubing that is coming out from the top of the septa into the collection tube all the way up to reach the stopper.
4. Place your index finger over the open end of the tube and invert it (water should have remained in the tube).
5. Place the tube, with the open end down, into a 250-mL beaker containing approximately 1/2 water.
6. Remove your finger (the column of water should remain in the tube.)
7. Connect the other end of the Teflon tubing to the reaction tube as shown in figure 4.
In a 10 mL round-bottom flask equipped with a water-cooling condenser place 5 mL of 1M potassium \( t \)-butoxide (or 575 mg of potassium \( t \)-butoxide powder and 5 mL of \( t \)-butanol) using a pipette. Add a boiling stone to the tube, and connect the condenser to the gas collection assembly as shown in Fig. 4.

![Fig. 4 Assembly for the Generation of Gaseous Products](image)

Assemble the gas collection apparatus shown in Fig 4 before the reactants are mixed. After the assembly is complete, use a syringe to add 0.5 mL of 1-bromobutane to the reaction tube and heat the mixture in a preheated sand bath. Generation of gaseous product can be observed by watching the rapid decrease of the water level in the collection tube. **Keep heating until no more bubbles escape from the bottom of the collection tube.** Remove the Teflon tubing from the gas collection tube, and then from the beaker, before removing the reaction tube from the heat, all the while ensuring your collection tube stays below the beaker’s water level. Use this sequence of steps in shutting down the reaction to prevent water from being sucked back into the hot reaction tube while it is cooling down. Label the beaker containing the collection tube kept in the water and submit it for the gas chromatographic analysis.

**B2. Dehydrobromination of 2-Bromobutane**
Repeat the procedure for part B1 using 2-bromobutane instead of 1-bromobutane. Be sure to read chemical labels carefully and keep track of which alkyl halide you are adding!

The butenes have been determined to elute from the non-polar column (SE30) the GC is equipped with in the following order: 1-butene, trans-2-butene, and cis-2-butene. If you did not know the order of elution for the gases, how could you determine it experimentally? Determine the relative amount (% composition) of the three components of gas products, assuming that the amount of each substance in the gaseous mixture is proportional to the area under its corresponding GC peak. Assume that these areas are equal to the peak height (mm) x the peak widths at a half-height (mm).
NOTES FOR WRITING YOUR REPORT:

Write a complete lab report as described in the Report Guidelines for Students handout. Include all of your results. The following guidelines are merely recommendations of things to cover, not the limits.

Your theory section should include:
- An overview of E1 and E2 reactions and their mechanisms.
- A brief overview of the factors and conditions that favor each reaction.
- A hypothesis of the products that will be obtained for each reaction.

Your results section should include:
- A data table with the % composition of the products for all four reactions.
- Attach all GC print-outs attached to the end of your report.

Your discussion section should include:
- Explain WHY the four reactions give you different product compositions by analyzing each of the reactions: what set of reaction conditions relate to what kind of product distribution and what does that tell you about the operating mechanism and the factors that affect product distribution.
- If your GC plots did not match what you expected, provide an explanation as to what might have happened.