

S343 Lab: Elimination Reactions

Assigned reading: Mohrig, "Techniques in Organic Chemistry Chapter 20: Gas Chromatography"

Lab goal: Determine product distributions for elimination reactions and relate to mechanism.

Procedure: You will work with a partner to accomplish the following two elimination reactions. Both partners must stay in the lab until the entire project is done, so consider proper teamwork principles to accomplish an overall efficient use of time.

Elimination of 2-methyl-2-butanol under acidic conditions: Place 2.4 grams of 2-methyl-2-butanol in a 25 mL round bottom flask with a stir bar. Carefully add 15 mL of 6 M sulfuric acid to the flask. Add an air condenser to the flask and complete the fractional distillation apparatus shown in Figure 12.17 (except the air condenser will replace the fractionation column.) Place the receiving flask in an ice water bath. Gently heat the reaction in a water bath at 85-90 °C. Collect the distillate that comes over below 45 °C. Make a GC sample as directed below.

Elimination of 2-chloro-2-methylbutane under basic conditions: Put 23 mL of basic 1-propanol solution in a 50 mL round bottom flask. (There are 3.0 g of KOH per 23 mL of the solution.) Slowly pour 3.0 mL of 2-chloro-2-methylbutane into the basic solution. Assemble a fractional distillation apparatus (Figure 12.17), but with a jacketed condenser as the fractionation column. Use a tared, 25 mL round bottom flask in ice as a collecting flask. Heat and stir the reaction for one hour in a 75-80 °C water bath, monitoring the temperature closely so that it does not exceed 80 °C during the reflux period. Add additional ice to the receiving flask as necessary. A white precipitate will form in the reaction flask. After one hour, increase the water bath temperature to 90-95 °C and distill the reaction mixture. Collect all the distillate boiling below 45 °C. Make a GC sample as directed below.

Gas Chromatography: Prepare a GC sample for the autosampler. (You will leave your sample to be analyzed by an automated GC sampler.)

1. Obtain a GC vial and cap.
2. Place 2-3 drops of your distillate in the vial, then add heptane to AT LEAST the 0.5 mL mark.
3. Snap on the cap, making sure it is on TIGHTLY!
4. Label the vial with a Sharpie with your three initials, the day (R or F) and the time (1:20 or 2:30) of your lab. For example: LCB, R 1:20
5. Place in the GC vial collection box.
6. Be sure to have your AI explain how you will receive the data.

GC parameters:

Column Type: HP-5 5% Phenylmethyl Siloxane

Oven: 65 °C, isothermal for 5 min

He flow: Flow rate 7.5 mL/min

Injector: Auto-injection, volume 1.0 uL

Heater 150 °C

FID Detector: Heater 250 °C

Observations and Results:

Record a main reaction data table and any observations and results that are appropriate.

What are the accepted boiling points for your expected compounds? Which compound will have a shorter retention time on the GC? (For nonpolar columns, the lower boiling substance will travel faster through the column. In this case, an authentic sample of the lower boiling compound had a retention time of around 1.3 minutes, and the higher boiling component had a retention time of around 1.4 minutes. These times may vary slightly on your run, but the order should be the same.)

Attach the GC trace, and make a table that includes retention time and integration.

Discussion comments: The following are some questions to keep in mind as you are writing your discussion.

1. What was the product distribution in the acid catalyzed reaction? Use data to quantify this product distribution.
2. What was the product distribution in the base catalyzed reaction? Use data to quantify this product distribution.
3. How do the data suggest that the two reactions go through different mechanisms? Propose mechanisms for each set of conditions (and make a figure using ChemDraw) and explain how the different mechanisms could lead to different product distributions.

Conclusion: Summarize the main conclusions of the lab.

Reference(s): Did you use any authentic data?

Lab 5 assignment: Turn in a hardcopy of your lab notebook (carbon copy or photocopy) and an experimental section

- Due at the beginning of lab next week
- 25 pts based on in-lab performance, completion of all sections, correctness of content, experimental section
- No Electronic submission
- Follow the format given in “Writing a Formal Report”
 - Past, passive
 - Separate entry for “Elimination under basic conditions” and “Elimination under acidic conditions”
 - Include data at the end of entry