Rowan College at Burlington County

CHE 241

Lab 7: Competing Nucleophiles

Watch me explain what is expected of you here.

Information you may need to complete this lab

(You've had this before on the previous 2 labs, but it's still info you will need for this lab)

What is GC? Here's a video

Here's the accompanying slideshow

This lab is more straightforward than the previous two labs. Here, you are going to determine which nucleophile dominates in the reaction based on GC data. If you were doing this lab in class, you would follow this procedure:

A. Solvent-Nucleophile Medium

1 Place 50 grams of ice in a 250 ml erlenmeyer flask and carefully add 38 ml of concentrated sulfuric acid. Set this mixture aside to cool. Weigh out 0.18 moles of ammonium chloride and 0.18 moles of ammonium bromide into a 250 ml beaker2. Crush any lumps of these reagents to a powder and transfer these solids to a 500 ml erlenmeyer flask using a powder funnel. Cautiously add the sulfuric acid solution to the ammonium salts, a little at a time. Swirl the flask to induce the salts to dissolve. Heat the mixture on a steam bath to achieve total solution. When solution has been achieved, allow the liquid to cool for no more than one minute3 and then pour 35 ml of the sulfuric acid-ammonium salt mixture into a 125 ml separatory funnel4 (for the t-butyl alcohol reaction) and the remainder into a 500 ml round bottom flask for reflux (for the n-butyl alcohol reaction). A small portion of the salts in the separatory funnel or the round bottom flask or both may precipitate out as the solution cools. Do not worry about these at this point; they will redissolve during the reactions.

B. SN Reaction - 1-Butanol.

Connect the 500 ml round bottom flask with the sulfuric acid-ammonium salts mixture to a condenser and add a boiling chip to the flask (use white boiling chips). Add 5 ml (0.055 moles) of 1-butanol through the condenser carefully. Start the water circulating in the condenser and then reflux gently5 for 75 minutes with a heating mantle. After reflux, cool the reaction flask in an ice-water bath before dissassembling the reflux apparatus6. The cooled mixture is transferred to a 125 ml separatory funnel taking care to leave behind any solid precipitate. Allow the layers to separate and drain the aqueous layer. Add 10 ml of water to the organic layer(s), shake and separate6. Wash the organic layer with 10 ml of sodium hydrogen carbonate solution, separate and drain the organic layer into a 50 ml beaker containing about 0.5 grams of anhydrous sodium sulfate. When the solution is clear, decant the product solution into a 50 ml erlenmeyer flask containing a few beads of anhydrous calcium chloride7. Stopper the flask, label and turn in to the Instructional Assistant or Instructor for Gas Chromatograph Analysis.

C. SN reaction - tert.-butyl alcohol

Measure 5 ml (0.052 moles) of t-butyl alcohol in a warm graduated cylinder and add this to the separatory funnel containing 35 ml of the warm sulfuric acid-ammonium salt solution. Stopper the separatory funnel and invert to release pressure8. After releasing pressure, turn the separatory funnel upright, swirl the funnel a couple of times and then invert it to release the mixing pressure. Repeat this until the pressures are substantially equalized; then invert the funnel and shake it vigorously with occasional venting for 2 minutes9. After shaking, allow the layers to separate for no more than 1 minute. Drain off most of the aqueous layer, wait 30 seconds, drain off the rest of the aqueous layer formed and a little of the organic layer into a beaker. This is to make certain that there is no water in the organic layer. Drain the remainder of the organic layer into a 50 ml beaker containing about 1 g of solid sodium hydrogen carbonate. As soon as the bubbling stops and a clear liquid is obtained, decant it into a clean, dry 50 ml erlenmeyer flask containing a few beads of anhydrous

calcium chloride7. Stopper the erlenmeyer flask, label and turn in to the Instructional Assistant or Instructor for Gas Chromatograph Analysis.

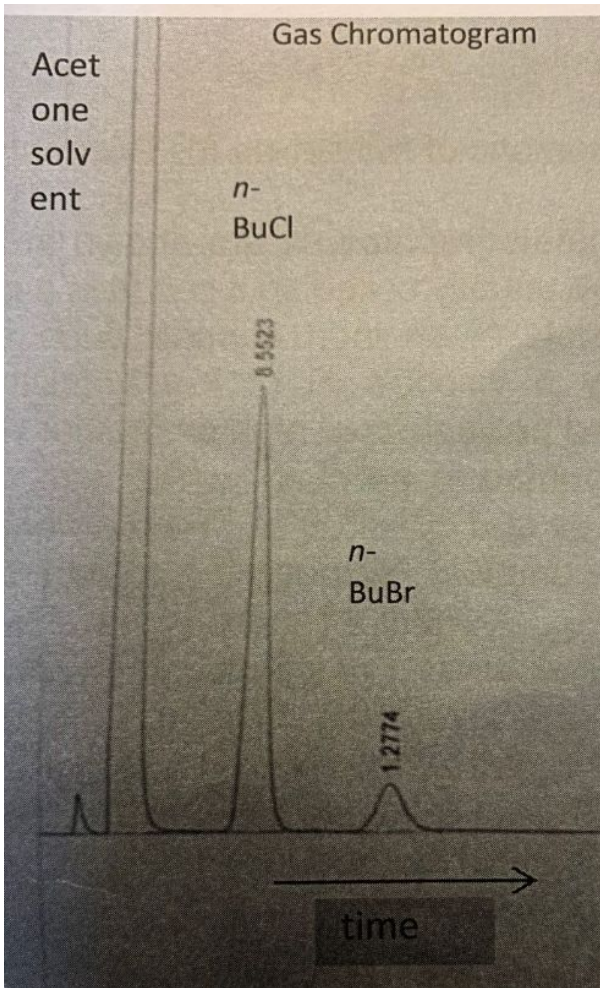
D. Gas Chromatography:

With the help of the Instructor or Instructional Assistant, obtain a gas chromtogram of the products from the reaction of n-butyl alcohol and t-butyl alcohol.

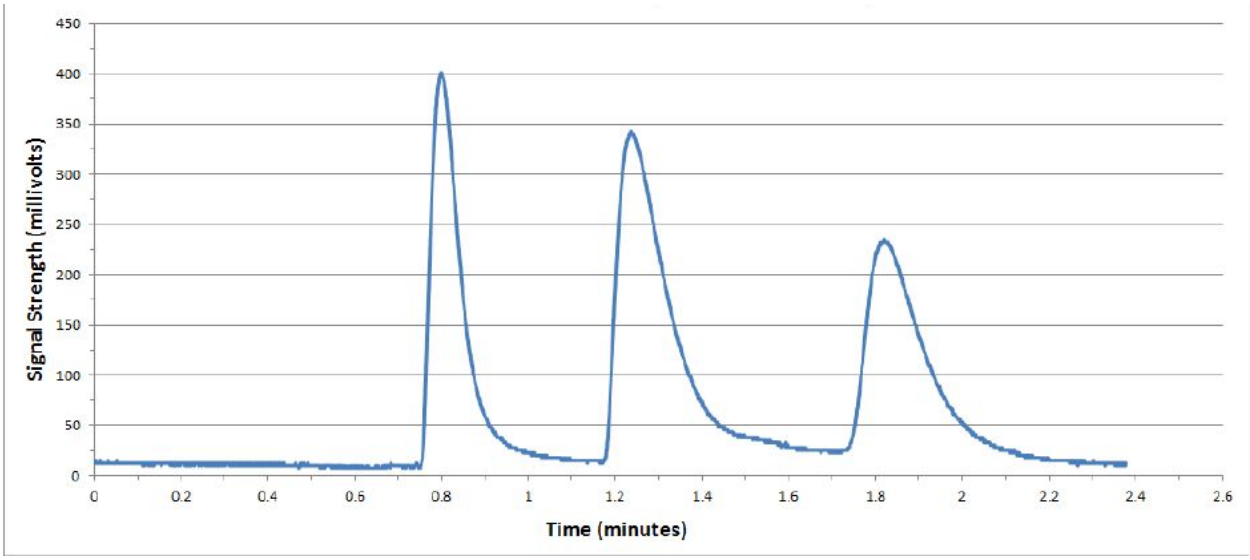
THE FOLLOWING POINTS MAY BE HELPFUL IN INTERPRETING YOUR RESULTS:

1. The retention time of similar compounds are usually in the order of increasing boiling points. The sensitivity of a thermal conductivity detector, such as used in the instrument employed for this experiment, is very nearly the same for comparable compounds.

You completed this lab and got these two GCs as data:



This GC is the results of part B. It shows a peak for the solvent (which is NOT important) and then a big peak for n-butylchloride and a smaller peak n-butylbromide.



This is GC for part C. It shows a solvent peak at around 0.8 min, which is not important to the results. The peak at 1.25 min that is identified as t-butylchloride and a peak at 1.85 min that is identified as t-butylbromide. While these two peak are slightly different sizes, they have roughly the same area.

For the write-up:

Comment specifically on the following:

a. Explain which halide predominated in each reaction and describe how you know.

b. Discuss the relative quantities of the halides in each reaction, and how this makes sense based on the reaction mechanism.

c. The relative rates of nucleophilic attack by the bromide ions and chloride ions on the two alcohols at these temperatures based on the relative areas on the GCs.

d. The relative reactivity of the two alcohols, based on the reaction conditions and the mechanism.

Write up your results formally, in paragraph form, making sure to address all the points above, and then submit it to me, once completed, by email to dwalsh@rcbc.edu