Virtual Chemlab - Hydroboration-oxidation

Introduction

For our next experience with the Virtual Chemlab program, we will be working together in a computer classroom in the Smith Computer Center. We will be running the hydroboration-oxidation reaction, the same one we did in lab last week. We will be experimenting with different solvents, temperatures, starting materials, and so on.

Before coming to lab, please review "Introduction to Virtual Chemlab" and "General Instructions on Running the Virtual Chemlab Program," and make sure you remember the basics of running the program.

Rather than using your regular lab notebook, we will be using a set of "Virtual Chemlab Observations" pages to record this lab. These pages will be provided for you when you come to lab.

Procedure

Trial 1:

- We will run the first trial together. Please perform the experiment according to the instructions below, and fill out the observation page with all of the information for this trial.

  reaction: hydroboration-oxidation
  starting material: 1-hexene
  solvent: diethyl ether (Et_2O)
  reagent: BH_3-THF/H_2O_2
  temperature: room temp
  washing solvent: water

Trials 2 and 3 (comparing reaction temperatures):

- What effect do you think raising or lowering the temperature of the reaction will have? Write your prediction below.

- Repeat the first reaction, but instead of running it at room temperature, run it at reflux (see the general instructions for how to do this). Record the results, making note of any differences - was your prediction accurate? (You don't have to take an IR or distill this one, since we already did that with this product.) Then run the same reaction using an ice bath and record the results.

Trials 4 and 5 (comparing washing solvents):

- What difference will it make in the work-up if you use the NaOH solution or the HCl solution instead of just water? Make a prediction below.

- Repeat the reaction two more times, trying each of the work-up solutions. You only need to go to that point. Record the results.

Trials 6, 7, and 8 (comparing reaction solvents):

- Will water and ethanol work the same as ether as the solvent? Could the reaction be run without a solvent? Make some predictions below.

- Repeat the reaction with ethanol, with water, and without a solvent. Watch carefully to determine what is happening. Think about what you learned about this reaction in class - you should be able to justify your results.

Trials 9, 10, and 11 (comparing starting materials):

- Predict the products of the other starting materials in the space below, taking note of regioselectivity. Then take a guess about whether their boiling points will be higher or lower than the first product we obtained.

- Now perform the reaction with each of the other starting materials, using whatever combination of solvent, temperature, and work-up solution you think best, based on your previous results. Purify each by distillation and record the boiling point of each compound. If you obtain a mixture of products, separate them by distillation and record both boiling points.

Trial 12 (stopping midway through):

- Choose a starting material, solvent, and temperature which should be effective in producing a product. Predict what will happen if you stop the reaction before it is finished. What will happen during work-up? What will the TLC look like? What will happen during distillation?

- Now try it. Compare the results to what you predicted, recording your results.

Trial 13 (no help window):

- You should now have the ability to predict the results of any combination of conditions. Choose a set of conditions that you haven't yet tried, and predict below what you will be able to observe without the help window.

- **Turn off the help window** and try your reaction. Compare the results with what you thought you would observe.

Questions

1. What is the regioselectivity of this reaction? Give two products which demonstrate this selectivity.

2. Which starting material gave more than one alcohol product? Why did we obtain this result?

3. Are rearrangements possible in this reaction? Which starting material has the potential to rearrange and can therefore show us whether rearrangements will occur or not?

4. Why didn't the reaction work using ethanol or water as the solvent? Hint: What happened instead of the reaction that we wanted?

5. Which spot ran higher in TLC, the product or the starting material? Why?

6. Predict the contents of the sep funnel during work-up in each of the following situations:

a) 1-methylcyclohexene reacted with BH_3/THF/H_2O_2 using ethanol as the solvent

ether layer:

water layer:

b) 2-methyl-1-butene reacted with BH_3/THF/H_2O_2 using ether as the solvent, but only running the reaction for 2 hours at room temperature

ether layer:

water layer:

7. How can you use the help screen to know when a reaction is complete?

8. If there were no help screen, how could you use TLC tell you if the reaction were complete? Draw a TLC as it would look at the beginning, the middle, and the end of a reaction of 1-methylcyclohexene.

9. If the starting material is not visible by TLC, can you still tell from the TLC whether or not the reaction is complete? Why or why not?

10. What conclusion should you make if the IR contains bands at:

3400 and 1100 cm^{-1}

3100 and 1600 cm^{-1}

3400, 3100, 1600, and 1100 cm^{-1}