Virtual Chemlab – Grignard Addition

Name _____

Introduction

In this assignment, you will investigate the addition of a Grignard reagent to ketones, aldehydes, and carbon dioxide.

Now that you have some experience with this program, you will have the freedom to devise your own experiments. I will give you the parameters that you must investigate, and the results that you need to obtain. You can then decide, with your partner, what experiments will best fulfill these requirements. It's ok to play around a bit, just to see what will happen, but you will spend less time if you have some kind of logical plan. This is an excellent opportunity to use the scientific method, where you hold all variables constant except one and see what the effect of changing that variable will do. Then move on to another variable.

Before you come to lab, please review the reactions of Grignard reagents in chapter 12 of the lecture notes so that you will be able to correctly anticipate their behavior. (We have looked at the addition of Grignards to aldehydes and ketones, but we haven't done carbon dioxide yet – you will have to use the results of the program – or a little logic – to determine what will happen in this case.) Also, please review the "General Instruction for Running the Virtual Chemlab Program," and bring a copy with you to the lab. This tells you how to perform the operations like starting a reaction, washing, taking an IR, and so on. Also, please do the on-line questions before coming to lab.

Procedure

Login to your account. Locate the program under "academic software." Login to the organic chemistry lab using the word "chemlab." Go to the stockroom, click on the clipboard, and choose "Grignard Addition."

Look at the criteria listed below, and formulate a plan for how you will fill these requirements.

1) Investigate the effects of using water, ethanol, or ether as the solvent, as well as running the reaction neat.

2) Note the effect of running the reactions at different temperatures.

3) Try each of the three work-up solutions and observe the results with both types of products.

4) Experiment with all possible combinations of starting materials. There are 9 products possible; note that CO_2 gives a different kind of product than acetone or benzaldehyde.

5) Look at TLC data for each reaction to determine in what cases it is useful, what it can tell you.

6) Look at the IR spectra of all of the products, and note any important differences.

7) Obtain a boiling point for each product you make. Determine which of the compounds also have measurable melting points.

Begin doing experiments. Keep a record of what you do and what the results are on the "Virtual Chemlab Obervations" sheets. You must run at least 10 reactions. Take turns with your partner at the controls. You may turn in one assignment with both names on it.

The basic steps of each experiment are as follows:

1. Choose the reagents and solvent that you want to use. Place them in the reaction flask. (No reagents from the bench will be necessary in these experiments.)

2. Decide what temperature you will use (reflux, room temp, or ice), and set up the reaction accordingly.

3. Start the reaction, and use the help screen in the corner to observe what happens. Advance the time using the clock (I recommend 10 minute increments, unless the reaction goes too fast). Use TLC to monitor the reaction. Note how much time it takes to complete the reaction. If the reaction is unsuccessful, stop here.

4. Stop the reaction by putting it in the sep funnel. Choose a washing solution, and place it in the flask. Observe what goes into each layer. Remove the layer you want by dragging it to the desktop.

5. Characterize the product by taking an IR, NMR, and melting or boiling point.

6. Clean up the reaction by double clicking on the waste container.

Hints:

Legitimate issues: At least one of the products is water soluble. This means that it will not go into the ether layer during washing. When you drag off the water layer, the water will not be evaporated. You will have to distill the water off first, remove it from the collection flask, and then distill the product.

Possible program gliches: One of the combinations doesn't give a product under any conditions, without any explanation I can think of. Don't stress about it. Also, in one case everything seems to disappear when you put in into the sep funnel. It showed up again when I added a washing solvent.

When you have finished, answer the questions below. Turn in the observations sheets, and the question pages.

Questions (answer on these pages)

1. If the Grignard reagents used in this lab weren't available from a bottle, how could you make them? Write out a sample reaction.

2. What products are formed when water is used as the solvent in these reactions? What about ethanol? (Hint: the answer isn't "no products" - something does happen!)

3. How did the reaction times differ when the reactions were run at room temperature, reflux, ice temperature, and neat? Give examples.

4. Give the correct names and boiling points of each of the six alcohols that can be formed using these reactions (note – I do not guarantee that the program named them correctly!).

5. Give the structures and boiling points (if available) of the three carboxylic acids that could (in theory) be formed.

6. What are the important IR bands in the following products? Give the frequency and what bond it corresponds to.

2-cyclohexyl-2-propanol:

1-phenyl-1-propanol:

cyclohexanecarboxylic acid:

7. Which runs higher on a TLC plate, benzaldehyde or diphenylmethanol? Why?

8. Which runs higher on a TLC plate, benzaldehyde or cyclohexanecarboxylic acid? Why?

9. Draw the mechanism by which 1-phenyl-1-propanol can be formed.

10. By comparing it to the reaction to form 1-phenyl-1-propanol, deduce the mechanism by which propanoic acid can be formed, and draw it below.

11. What is the initial product formed in the reaction flask when a Grignard reacts with an aldehyde or ketone? Which work-up solutions are effective in converting this to the final product?

12. What is the initial product formed in the reaction flask when a Grignard reacts with carbon dioxide? Which work-up solutions are effective in converting this to the final product?

13. Why do different work-up solutions work with alkoxides than with carboxylates?

14. Which of the products were water soluble? Why?

15. In which cases was TLC useful in determining when the reaction was complete? Why wasn't it effective in the other cases?