Vírtual Chemlab – Introductíon to Runníng Chemícal Reactíons

Introduction

This lab uses the Virtual Chemlab program to introduce you to the process of running reactions in the organic chemistry laboratory. Lab will be held in a computer classroom, where each student will be able to run the program.

You will be running an esterification reaction, in which a carboxylic acid and an alcohol are reacted in the presence of sulfuric acid to form an ester. You will be able to try several sets of starting materials, as well as different temperatures and work-up procedures. You will record your observations on a sheet which I will give you, rather than in your lab notebook.

Please read "Running a Reaction," "Introduction to Virtual Chemlab" and "General Instructions for Running the Virtual Chemlab Program" before coming to lab. Read all of the instructions in this lab.

<u>Procedure</u>

Trial 1: Run the reaction according to the following instructions.

Set up the reaction:

- Go to the stock room and choose "Esterification" on the clipboard of reactions.
- Drag a flask to the cork ring. Add 2-phenylacetic acid, then ethanol from the bottles on the shelf, and diethyl ether from the solvent jar.
- Go back to the lab. Add sulfuric acid from the reagent bottles on the bench.
- In order to run the reaction at the reflux temperature of the solvent, add the heater, the reflux condenser, and the nitrogen supply (in this order).
- Pull down the help screen and verify the chemicals in the flask. Write down the names and structures of all chemicals in the reaction.
- Take a TLC and observe what you see.

Run the reaction:

- Start the reaction by clicking on the stir button.
- Turn the clock forward 10 minutes. Take a TLC and interpret the results.
- Keep turning the clock forward 10 minutes and taking a TLC until the results indicate that the reaction is done.

• Observe in the help window and blackboard the structure of the product that was formed, and record the time it took for the reaction to come to completion. Explain what could have happened to the starting materials to create this product.

Isolate the product:

- Add the reaction to the sep funnel (ether is automatically added).
- Add a layer of 0.1 M NaOH solution (it is automatically shaken and allowed to separate). Observe what ends up in each layer.
- Remove the organic layer (it is automatically dried and rotovapped).

Purify the product and obtain the boiling point:

- Put the flask back on the stir plate. Add the distillation apparatus from the drawer, then add the nitrogen tube. Start the distillation by clicking on the stir button.
- Set the clock forward 20 minutes. Observe the boiling point.
- Set the clock forward 1 hour. Remove the product from the collection flask.

Characterize the product:

- Drag a salt plate from the IR machine to the product and observe the IR. What bands are present that correspond to the product?
- Take an NMR by dragging a sample tube from the NMR machine to the product and observe the NMR. Match up the bands to the expected peaks on the sample.

Trial 2: Try the reaction again, this time using room temperature instead of reflux. Watch the reaction by TLC to determine how long it takes to finish. Compare this to the reflux temperature. When the reaction is finished, record your observations, and discard the reaction (you don't need to isolate, purify, or characterize, since these results will be the same).

Trial 3: Run the reaction again, this time using a different alcohol. Since you will now have a different product, complete all of the steps. What differences do you see in the boiling point, IR, and NMR of this product and the original one? Can you explain them?

Trial 4: Run the reaction again, this time using a different carboxylic acid. Since you will now have a different product, complete all of the steps. What differences do you see in the boiling point, IR, and NMR of this product and the original one? Can you explain them?

Trial 5: Think of something different that you'd like to try - a different starting material, a different solvent, a different reaction temperature, etc. Run the reaction and report the results.

Questions

1) Explain the purpose of the following steps in a reaction:

- a) isolating the product
- b) purifying the product
- c) characterizing the product

2) Based on the results of the esterification reactions you ran, predict the products of the following reactions (refer to the structures given in the starting material bottles):

a) 2-phenylacetic acid + isopropanol + sulfuric acid

- b) butanoic acid + ethanol + sulfuric acid
- c) acetic acid + 3-methyl-1-butanol + sulfuric acid

3) What affect does changing the temperature of the reaction have on the results?

4) Explain how TLC is helpful in determining if a reaction is finished. Would this still work if no starting materials were visible? Would it work if no products were visible?

5) Why is 0.1 M NaOH used as the washing solution? (What does it remove that just water would not?)

6) What would the IR of the esterification reaction look like if you stopped the reaction too soon? What peaks would tell you that some of the starting materials were still present?

7) Why is TLC a better way to monitor the progress of a reaction than IR?

8) What did all of the NMR's have in common?

9) Which boiling point would you predict to be higher, ethyl 2-phenylacetate or methyl 2-phenylacetate, and why? Do the results match your prediction?