CHEM 51LB: EXPERIMENT 2 PART 2
PURIFICATION AND CHARACTERIZATION OF SUBSTITUTION PRODUCT

REACTIONS: None
TECHNIQUES: recrystallization, melting point, thin layer chromatography

This is the second part of a two-part experiment. Previously, you reacted one of three possible nucleophiles with benzyl bromide in the presence of sodium hydroxide to produce a solid product. In this lab period you will purify the product using recrystallization and identify it and, therefore, your unknown nucleophile via melting point and thin layer chromatography (TLC). Additionally, you will analyze the saved aliquot of your filtrate for the presence of reactants, product, or products of side reactions. A $^1$H NMR spectrum of your product will be provided after the lab period.

READING ASSIGNMENT:

- Technique 14: Melting Points and Melting Ranges in Techniques in Organic Chemistry 3rd Ed. pgs 175-183.
- Supplementary information in Janice Gorzynski Smith (3rd ed or 4th ed), Chapter 7 & 8.

PRE-LAB ASSIGNMENT:

- Follow the instructions in the “Chem 51L Notebook Guidelines” document located on the course website to prepare your pre-lab in your ELN.
- Complete the online Sapling pre-lab questions.

CAUTION

Ethanol, hexanes, and ethyl acetate are volatile, toxic and flammable. Use in the fume hood.

EXPERIMENTAL NOTES:

Immediately set aside ~20 mg of your crude product for later.

Recrystallization:
Heat approximately 20 mL of ethanol and 20 mL of water in separate flasks or beakers, but on the same hot plate. Adjust the temperature of the hot plate so that the ethanol is hot but not boiling. Recrystallize your crude product from ethanol/water by placing your product into an appropriately sized Erlenmeyer flask and slowly adding portions of the hot ethanol just until the solid is completely dissolved. You will need to stir or swirl the flask containing your product solution and keep it warm during this process. Why is it important to keep the solution warm? Be careful not to add too much solvent! What will happen if you add too much solvent? What can you do if you add too much solvent? Add water dropwise and while swirling your recrystallization flask just until a slight cloudiness persists. What does the cloudiness being present mean? Add hot ethanol again
dropwise until the cloudiness disappears.  *Why does the cloudiness disappear?* Allow the solution to cool to room temperature and then place the flask into an ice bath to complete recrystallization. *Why don’t we just put the solution directly into the ice bath without cooling to room temperature first?* Cool a small beaker of water in ice while you wait for crystallization to occur. (Note that this is the general procedure for recrystallizing from mixed solvent when the exact solvent ratios needed are not known. You will be expected to remember this general procedure in the future.)

Vacuum filter the solid and rinse with ice-cold water.  *Why is it important to rinse the crystals? Why is it important that the water is ice-cold?* Allow the solid to dry in the filter funnel while pulling air through the sample for at least 15 minutes. Record the mass of the dried product.

**Melting Point:**
Obtain a melting range for the recrystallized product and your crude product you set aside earlier (if you did not do so in the previous lab period or if the previous crude melting point was very low). *Why are we interested in the melting point of both crude and recrystallized solid?* Perform mixed melting point experiments using your pure product with the standards provided to confirm the identity of what you isolated. *What does a mixed melting point experiment tell us?* If the melting range of your recrystallized product is low, try drying it again for a longer period of time.

**TLC (Product):**
Make a TLC sample of your recrystallized solid by adding ~10 mg of solid to a small beaker, flask or test tube and dissolving the solid in ~1 mL of acetone or ethyl acetate. Use this sample to compare your product to the TLC standards provided. This will help to confirm the identity of your product.

To help you understand TLC, you will run TLC plates of your product in three different eluting solvents: hexanes, 9:1 hexanes/ethyl acetate, and 1:1 hexanes/ethyl acetate. You will need to mix the eluting solvents yourself. Make 10 mL of each solvent mixture. *How will you make the eluting solvent mixtures? Which solvent system is most polar? Least polar?* Follow the general procedure outlined in your Techniques book for spotting, developing, and visualizing TLC plates. Note that your solid does not have to be completely dry for a TLC sample. If you found that your melting point of your recrystallized solid was low and decided to dry it again, take out enough to make a TLC sample and move forward with this portion of the experiment while the remaining solid is drying.

**TLC (Filtrate):  **May be done at any point during lab period. Use your time wisely!**
Remember that mass is neither created nor destroyed, so any reactants not converted to product, any product that stayed in solution, and any other products formed will be present in your filtrate. Use TLC to analyze your filtrate for the presence of these compounds. Standard TLC solutions will be provided for: all three possible nucleophiles, all three possible benzyl ether products, benzyl bromide, and benzyl alcohol. Elute your TLC plates with a 9:1 hexanes/ethyl acetate solution.

**Product Collection:**
Any product remaining after your analyses are complete will be collected. Place the remaining product in a vial and label the vial with the following information.

1. Names of all students in your group
2. Unknown #
3. Structure of product
4. Melting range of recrystallized product
Give the labeled vial containing product to your TA. Unused portions of your filtrate aliquot should be poured into the appropriate waste container.

**Data Analysis:**
Before you leave the lab for the day, use the data you collect to
1. determine the identity of your product
2. determine the identity of your unknown nucleophile
3. identify the contents of the filtrate to determine whether any additional substitution products formed
4. identify the effect of eluting solvent mixture polarity on TLC.
5. identify which of the three eluting solvents for TLC was the best for your product.

Before you leave the lab for the day, use the results of your data analysis to identify other students with the same unknown as yours and compare results.

After the lab period, your TA will combine like products and take a 1H NMR spectrum of this combined sample. You will receive your spectrum later in the week. Use this spectrum provided by your TA to confirm the identity of your product.

**LAB WRITE-UP:**
Complete the Experiment 2 Report Scaffold worksheet provided on the class website. Be sure to read and follow the instructions provided on the worksheet!