Lab 3
Distillation

Reading: Zubrick, pages 144-145, 150-152, 155-170,179-183, and 305-320.

Pre-Lab: Look up the structures and boiling points of cyclohexane and toluene.

Introduction: The recrystallization technique you learned last lab is a method for purifying solids. This week we will learn how to perform a distillation, a method for purifying liquids. Distillation is a common wet-chemical technique for separating organic compounds based on differences in boiling points. Upon heating a mixture of organic compounds, the more volatile compounds (those with the lowest boiling point) will vaporize first (i.e. be converted to gases), leaving the higher-boiling compounds behind in the mixture. By isolating the vapor produced at different temperatures and condensing them (i.e., converting gases to liquids), you can effectively separate and purify compounds based on their boiling points.

While there are multiple factors that contribute to the efficiency of distillation, the first, most obvious is the boiling points of the compounds themselves. If one compound is significantly more volatile than another ($\Delta bp > 40^oC$), then the compounds can be effectively separated in one vaporization step, in a process called simple distillation. However, if the boiling points of the compounds are too similar, then the vapor produced at any given temperature will be a mixture of the two compounds. For example, a mixture that is 50:50 toluene:hexane will boil at 90^oC and produce a vapor composed of 70% cyclohexane and 30% toluene. In this case, a process called fractional distillation must be used. Fractional distillation uses a column that allows many small distillations to occur as the vapor ascends the fractional distillation column.

In this experiment you will separate a mixture of toluene and cyclohexane by simple and fractional distillation. You will determine the weight of each compound isolated and you will (in the subsequent GC lab) determine the relative efficiencies of separations by simple and fractional distillations.

Procedure:
Two pairs will work together. One pair will set up a simple distillation, the other will set up a fractional distillation.

After setting up your simple (or fractional) distillation apparatus, have it checked by your lab instructor. Next, measure out 50 mL of the cyclohexane-toluene mixture and add it to your boiling flask. Don't forget the boiling chips. Heat the mixture slowly to boiling. Adjust the heater so that the distillation rate is about 1 drop per second for simple or 10 drops per minute for fractional. Record the temperature at the first drop and every 2 mL after that. The first few drops should be collected in a waste beaker and discarded. For the simple distillation, collect 3 separate fractions: 0-15 mL, 15-30 mL and 30-45 mL in three 25ml Erlenmeyer flasks or beakers. For the fractional distillation, after the first few drops, the temperature should remain steady. This is your first fraction and you should collect it until the temperature starts going up. Collect your second fraction until the temperature becomes steady again. At this time, collect 5-10 ml for the third fraction. For both distillations, there should be about 5 mL of liquid left in the distillation

flask. NEVER RUN A DISTILLATION EXPERIMENT TO DRYNESS! After you have collected the last fraction, turn off and remove the heat from the distillation set-up and allow it to cool. Use your graduated cylinder to record the exact volume of your fractions and save a small amount for the GC lab next week.

The lab report for this experiment will be combined with the report for the GC experiment. Thus, no report is due next week.

Results:
Make a graph of temperature versus volume collected for both the simple and fractional distillations.

Safety Information:
Cyclohexane and toluene are organic solvents. Avoid skin contact, breathing, etc.

Waste Disposal:
The solution left over in your round bottom flask goes in the non-halogenated waste.