CHEM–333: Lab Experiment 3:
Distillation and Gas Chromatography:

Prelab-Assignment: read Chapters 5 and 6.

> *Distillation is one of the most powerful techniques for purifying volatile organic compounds. Distillation is used to isolate many of life's essentials such as gasoline from oil or brandy from wine! This week's experiment will show you how to use distillation to purify a compound as well as analyze your products by HPLC.*

In this lab your TA will divide the class into two groups, half the class will perform a simple distillation, (Chapter 5; Experiment B) while the other half will perform a fractional distillation. Arrange to share your results with a student from the "other" group so you can compare and evaluate the efficiency of the fractional distillation versus simple distillation by gas chromatography. Come prepared work quickly to make sure that everyone has a chance to run their GC samples by the end of the lab.

Read the information at the beginning of Chapter 5 as well as Experiment A-C. Don't worry too much about the mathematics of Raoult's Law; you will see it again in physical chemistry. Pay particular attention to the vocabulary used in this family of purification techniques. Read all of Chapter 6 for its discussion of gas chromatography.

For your distillations (**remember to add boiling stones**) you will use 30 mL of an equal-volume mixture of cyclohexane and octane. Use an electric heating mantle (flask heater), and a 100 mL flask to ensure good heat transfer from the (oversized) mantle. Have your T.A. check your apparatus for proper connections and water flow **before** you begin heating (and don't forget a boiling chip). **Don't** rinse out your fractionating column; whatever you wash with will show up in your GC!

In order to measure the volume of distillate, set up test tubes in a test tube rack. Record the vapor temperature when the first drop hits the receiver (this is the "initial" boiling point) and for every 2 mL's collected (as in Experiment A).

Collect three fractions, or "cuts," and put them into small capped vials. Collect the first cut until the boiling point begins to rise rapidly above that of cyclohexane. (Collect the fraction in your graduated cylinder, then quickly transfer it to the vial so you can continue your temperature/volume measurements.) The second cut should contain the mixture obtained during the rapid temperature increase. The third cut should be started when the boiling point levels out near the boiling point of octane. You can stop heating after the distillate has demonstrated a constant boiling point. **Do not boil the distillation pot to dryness.** Seal the vials tightly to prevent evaporation before you run your GC's.

Dispose of your three fractions and the residue from your distillation pot in the appropriate waste containers provided. Be sure to return all shared glassware to the correct drawers.
Your T.A. will do at least one of your GC injections for you. Inject 3 μl of each sample, and record in your notebook (or directly on your GC trace) the operating parameters of the GC (injection volume, column type, oven temperature, flow rate, and detector settings).

In your notebook, plot the temperature/volume curve for your distillation (see Figure 5.2). Calculate the percent composition of your three GC runs based on peak area, assuming equal response to the thermal conductivity detector. Tape your GC traces into your notebook.