Experiment 6: Distillation

Read pp 173-183, 188-192 and 203-205, Chapter 12, in *LTOC*, and refer to pages 74-75, Technique 6.2, to learn about heating mantles. You will be given a mixture of two common solvents that you will have to distill using both simple and fractional distillation techniques. Study the figures on pages 181 and 191 carefully. You can watch the relevant videos, 12.1–12.4, Simple Distillation, Parts 1-3 and Fractional Distillation, at http://www.macmillanlearning.com/Catalog/studentresources/mohrig4e .

Your instructor will assign a 1:1 mixture to you. Record the names of the liquids that are contained in your mixture. **Outline** the following procedure:

Assemble the apparatus for a simple distillation (see Figure 12.7 on p. 181). You should refer to the sample set-up provided in the dispensing fume hood, especially noting the placement of clamps. Note that the position of the thermometer bulb should be right *below* the opening of the three-way distillation head. Position a thermowell under the distilling flask on a ring clamp and plug it into the thermowell control outlet under the fume hood sash. Should the heating become too vigorous, the thermowell can be lowered by lowering the ring clamp. Do **not** grease the joints when setting up the distillation apparatus as it may contaminate the liquids you are distilling. Attach the vacuum adapter to the condenser using a Keck clamp.

Water should flow from the bottom of the condenser to the top. You will connect your condenser to a "process chilled water" (PCW) unit that circulates cold water through a loop. Using Tygon tubing, connect the inlet of the condenser nearest the receiver to the nozzle labeled PCWS (S = supply) and the outlet nearest the thermometer to the nozzle labeled PCWR (R = return). Turn the knob labeled PCWR to open the "return" path first. Wait a couple seconds, then slowly turn the knob labeled PCWS to open the "supply" path. You should see water flow through the tubing and into the condenser. If you open PCWS first or turn the pressure from PCWS too high, the tubing will pop off.

You will use a graduated cylinder as the receiving vessel. Place a 25 mL graduated cylinder in a beaker of ice and position it such that the lip of the cylinder is as close as possible to the end of the vacuum adapter. Use wooden blocks, which are available in the facilities area, to raise the graduated cylinder, if necessary. If the tip of the adapter is too far above the graduated cylinder, drops of the distillate will partially evaporate as they fall (due to the airflow in the fume hood).

Measure **40 mL** of your assigned mixture into the 100 mL round bottom distilling flask. Add two or three boiling chips to promote even boiling. Make sure all connections in the apparatus are tight, then turn the thermowell control knob (be sure the outlet is switched on) to a setting between 60 and 80. Adjust the heat until the distillate drops at a regular rate of about one drop per second. Record the temperature and volume of the distillate at 5 mL intervals. After 25 mL of distillate has been collected, quickly replace the 25 mL graduated cylinder with a 10 mL graduated cylinder in ice. Continue to take volume and temperature readings until another 10 mL of distillate has been collected, at which point you may lower the thermowell to stop the distillation. Do *not* throw away the distillate or the liquid that remains in the round bottom flask.

When you have completed the simple distillation, you will proceed to the fractional distillation of the same mixture. Wait until the round bottom flask has cooled, then pour the distillate contained in the graduated cylinders into it. Add two new boiling chips and assemble the apparatus for fractional distillation (see Figure 12.17 on p. 191).

Refer to the sample set-up in the dispensing hood. The fractional distillation must be carried out *slowly*. Turn on the thermowell to heat the mixture to boiling. Once boiling starts, turn down the power. You should see a ring of condensate which *gradually* rises up the length of the fractionating column, such that the column eventually acquires a uniform temperature gradient. Increase the heat slightly if the ring of condensate stops rising. The vapor-condensate mixture should reach the top of the column only after several minutes. Once condensation of the vapors occurs, it should continue steadily at a rate not greater than one drop per second. Increase the heat slightly if the flow rate decreases significantly. Again, collect the distillate in the graduated cylinders, but do not place them in ice. Record the temperature and volume of the distillate at 5 mL intervals until the temperature starts to rise. When you see the temperature increase, record the temperature and volume of distillate at intervals of 0.5 or 1 mL until the temperature no longer continues to increase. At this point, record the temperature and volume of the distillate at 5 mL intervals until about 35 mL of total distillate has been collected. Do *not* distill to dryness.

After the fractional distillation is completed, pour the contents of the graduated cylinders and the round bottom flask into the **Laboratory Byproducts** jar. Rinse the fractionating column with acetone before you return it to the special equipment bin.

When turning off the process chilled water, close the PCWS outlet first, then close the PCWR outlet. Unclamp the condenser and pull off the tubing while holding it over the sink. It is likely that water will spurt out of the outlets with some force, so remove anything from the fume hood that you do not want to get wet before doing this.

Obtain data from a student who distilled the other mixture.

Name_____ Date _____

T. A. _____ Lab period _____

Results and Calculations (to be handed in at the next lab period)

The two mixtures are:

methanol and 1-propanol
methanol and 1-butanol

Plot the distillation temperature as a function of the volume of distillate for both the simple and fractional distillations for both mixtures. Attach these four plots. Use a plotting program, such as Excel.

Based on the shapes of the plots of simple versus fractional distillation, what conclusions can you draw regarding the efficiency of separation of the liquids for each method?

Compare the two simple distillation plots. Are their shapes different? If so, account for this difference. Are the shapes of the two fractional distillation plots different? Comment.