Chemistry 238 Experiment 6: Distillation Winter 2008

You will work with a partner on this lab; decide which of you will do the *simple* distillation and which of you will do the *fractional* distillation of the unknown mixture.

• **Pre-lab:**

Read: Experiment 6 Part A only (pp. 51 – 56), Technique 14 (pp. 703 – 707, 713 – 714), Technique 15 (pp. 715 – 724)

Skim: Technique 22 (pp. 797 – 817)

Prepare for class on Tuesday, February 12: "Purpose," and "Materials and methods". Please include a sketch of the setup *you* used (not your partner's).

Under the "Materials" section, draw the structures of the five possible unknown compounds and state their boiling points.

The setup will include a temperature probe, data logger and laptop computer, not a thermometer, and your output will appear on screen as a time vs. temperaure graph. If you bring a USB flash drive, you should be able to save your data in some compatible format (like Excel). You may even be able to e-mail your results to your account, though the wireless connection requires a computer lab ID (not just your net ID).

Randy Engel suggests these modifications:

The fractional distillation will go more slowly than last year since we're using more stainless steel sponge. Pages 53 and 54 describe it well. They should turn the heat up initially especially with the fractional distillation and then back off when distillate begins to come over. Depending on their unknown and the amount of heat, the fractional distillation can take a while, perhaps even as long as an hour. It shouldn't take that long if they push it, but if you encourage them to push it too much, they may not get good separation. If they start with 7 mL, they should be able to get a sample at 4.5 mL, but make sure they use the 10 mL round-bottom flask. And if they run short, they can take the last few drops.

I think we had problems last year with samples evaporating overnight. The Teflon stoppers with O rings and caps on a 3 mL conical vial may give the best seal. And then I think it would be good to store them in the freezer. The directions say to take 2 drops – maybe we should have them save more, like 4.

Reserve the next few pages for "Procedure" and "Data" and "Results". Especially for the data section, you will taping in your gas chromatogram and your distillation graphs, so leave a few blank pages.

Data section: **Prior to lab**, make a data table to record the distillation temperature and volume of distillate.

Goal for Tuesday: complete the distillation, and make sure you have the two drops at 1.0 mL and 4.5 mL of distillate in sealed conical vials. Tentatively identify the two liquids in the unknown mixture (you may, of course, talk to your partner about the identities and compare data) by their boiling points.

Goal for Thursday: Perform the gas chromatograph (GC) on the conical vial samples.

• Post-lab:

Include a copy of the GC output, including peak integration, in the data section. Do this for both samples you run.

Plot the temperature–volume data for the distillation and include the plot in the calculations/analysis section.

• Lab Result Report: (Due Thursday, February 21 at the beginning of lab)

Photocopy the lab, all parts, including copies of the distillation graph and the GC output.

No conclusion section is needed.

Answer questions:

End of Technique 14 (pp. 714, 715): 1 ab, 3, 7

End of Technique 15 (p. 731): 1 abcd, 2

Abstract:

- attach this to the **front** of your report
- must be **word-processed** on a **separate sheet of paper**

Your name and your partner's name, North Seattle Community College
A COMPARISON OF SEPARATION BY SIMPLE AND FRACTIONAL DISTILLATION OF A _____/_____ MIXTURE

Using the format like in the previous abstract, write a 75 to 100 word abstract that starts by answering the question: "which distillation results in better separation of these two liquids?" Explain both theoretical and practical reasons why your answer is the way that

it is — remember to use both the distillation curve and the GC results in your explanation.