Changes to Exp 14:

The main changes are:

1) No longer using an unknown. This means skip all of Part B in the procedure.

2) You will be measuring the boiling point in the lab for your known compound. You will be using a sand bath. You will also look up the normal boiling point and use both in your data table and the graph. In the Discussion you will need to discuss whether your b.p. makes sense compared to the normal b.p. and why.

Page 5:

Paragraph 2: strike the last sentence about the unknown.

Last paragraph:

In part C (still labeled part C, even though Part B has been cut) of the experiment the boiling point of the known sample will be determined. The setup for the boiling point determination is different than shown in Figure 14.4. The key to a successful boiling point determination is to keep everything on a small scale. A 4-mL test tube containing 2 mL of the sample (about half filled) is used. A capillary tube is broken to a length of approximately 3.5 cm (about in half) and placed, open end down, in the liquid. A digital thermometer is inserted in the test tube. The test tube is clamped and placed in a sand bath. As the temperature increases, a fine stream of bubbles will come from the capillary tube as vapor from the unknown liquid forces air from the capillary tube. After a few seconds, the capillary tube will contain only vapor from the unknown. The liquid will boil and the temperature should stabilize as well. Once this happens the test tube is removed from the sand bath and the liquid starts to cool. When the vapor pressure falls below atmospheric pressure, liquid will start to enter the capillary tube. The temperature, at which vapor pressure equals atmospheric pressure, is the boiling point.

Page 6:

Ignore Figure 14.4. The TA will show you how to set things up for the boiling point determination.

Page 7:

Paragraph 2 (below eqn 6):

Graph ln P_s vs. 1/T for each of the three known compounds on the same graph. Include the boiling points (experimentally determined and literature value from the CRC or other source, cite your source) for each compound on the graph and label them. The vapor pressure for the normal boiling points taken from the literature is 760.0 mm Hg. For the experimentally determined boiling point, the vapor pressure is the barometric pressure in the lab. Each student must independently generate their graph. There will be only one graph for the known compounds.

There is a mistake for the unit for ΔH_{vap} toward the bottom of the page above the last paragraph. It should be kJ/mol (kJ*mol⁻¹).
Last paragraph:

In your discussion, report the values of $\Delta H_{\text{vap}}$ for the three known compounds and the boiling points. Discuss the trend in $\Delta H_{\text{vap}}$ for the three knowns. Your discussion should include how the trend in the $\Delta H_{\text{vap}}$ values for the knowns corresponds to the attractive forces present in the compounds (i.e., did you get the correct trend based on the attractive forces? What are those forces?). Also, discuss whether the values are reasonable (based on what you've learned in class and the textbook). Discuss whether the boiling points obtained in lab are reasonable and make sense compared to the normal boiling points from the literature. Are there any data points from the syringe data which are out of place (think of where they should be in relation to the boiling point).

Procedure:

There is NO Part B (unknown).

Part C: Determine the boiling point of the known compound.

13. Place a 4-mL test tube in utility ( buret) clamp so the top of the test tube is the same height as the top prong (use the end as a handle). Place the end in a beaker for stability. Place 2 mL of your known liquid in the test tube (about half full). Break off a capillary tube 4 cm from the closed end. Place the capillary, open end down, in the test tube with the sample. The end should be slightly above the liquid. Place the digital lollipop thermometer in the test tube with sample.

14. Holding the end of the clamp, place the test tube in a sand bath in the side hoods. Watch the sample closely. You are looking for bubbles coming from the open end of the capillary and for the temperature to stabilize. Point the top of the test tube away from your face (toward the rear of the hood). Don’t worry if some of the sample splatters out of the tube. When you have observed a steady stream of bubbles and the liquid starts to boil and the temperature stabilizes remove the test tube from the sand bath using the clamp as a handle. It will not take very long for this to happen (probably less than 30 seconds) so watch things closely.

15. When liquid starts to enter the capillary tube, record the temperature. This is the boiling point of the liquid. Again, pay close attention as this will take only 10-20 seconds.

16. Repeat the boiling point determination. If both values agree fairly closely you are done (average them for the reported boiling point). If not, repeat a third time and average the two values which are in closer agreement.

Points to Consider:

In this lab you are studying properties of compounds ($\Delta H_{\text{vap}}$ and boiling points) which are affected by attractive forces.

- Make Excel tables for the data and use as your report sheets in your report. Add a line for the normal boiling point above the existing line for boiling point (use the existing line for your experimentally determined b.p.). For the on-line data entry you’ll be using the site used previously to enter the data for the unknown. You can input only one b.p. so use the boiling point and pressure you determined in lab.

- Include the sample calculations.
• Include your graph for the three known compounds. The curve for each compound should have 7 data points, the five T-V data points and the two boiling points (experimentally determined and the normal b.p.). You should have only the one graph with the data for the 3 known compounds.

• What do your graphs show with respect to the $\Delta H_{vap}$ values for the knowns? What trends are evident and do they make sense in terms of the attractive forces present in the molecules? What are these attractive forces (be specific)? Are the $\Delta H_{vap}$ values reasonable based on what you've learned in lecture and the textbook? Do the boiling points show the same trends as the $\Delta H_{vap}$ and follow the correct trends based on the attractive forces?

• Does the measured boiling point seem reasonable compared to the normal boiling point from the literature? Should it be the same, higher or lower based on what you learned about boiling points? If it’s not what was expected, what might be the cause of any discrepancy? Do any of the other data points (from the five T-V data points) seem to be out of place with respect to the boiling points?

• What errors are present? As always, make sure to include at least two inherent errors and how they can affect the results and be corrected.