Chemistry 150 Name(s):

Exercise 6: Experimental design for the freezing point depression lab

Due date: before the lab Wednesday, February 21, 2007 – only one exercise need be turned in per partnership.

Make a copy of this to use during the lab, or at least copy the relevant parts into your purpose, materials and methods (procedure) sections.

Objective: Given a research objective, design a series of experiments to carry out the objective.

Introduction: In lab on Wednesday, you and your partner will use some basic experimental equipment to determine the molar mass of an unknown substance based on how various amounts of it depress the freezing point of a solvent it dissolves in. The lab writeup is available on the course website but there is no step-by-step procedure. In research problems, scientists and engineers do not have a "cookbook" to which they can turn to figure out how to obtain a desired number. You are going to approach this problem in the same way those scientists and engineers would.

1. To begin writing an experimental protocol (procedure), you need to clearly articulate your goal. For this experiment, yours is:

Unlike many research problems, you have some theory to fall back on. In this experiment, the freezing point depression equation is known:

$$\Delta T_f = K_f \underline{m}$$

2. Define the quantities and their units in the equation above:

3. Determine which quantity is related to the unknown. In some cases, that's easy because one of the quantities in the equation is the unknown. But in this case, it is not the case. I recommend looking at the definitions of the quantities and their units to decide which factor might be most useful.

4. In order to accomplish the desired number in question 1, then, what other equation or equations do you need to connect that to the quantity in question 3?

5. So does this other equation require you to make another measurement in order to complete the calculation? If so, what quantity is it and what equipment will you need?

6. Going back to the original equation (question 2), how are you going to obtain the quantities not covered in question 3? Acceptable answers include specific pieces of equipment, or "looking up the number in a reference".

7. If any of the quantities in question 6 are to be measured, how many trials will you need? This is **not** an assessment of the variability (standard deviation) of the measurement – that will come later. Especially if a "delta" is in the equation, how many trials will you need to determine that difference?

8. Many experiments require a "baseline" measurement. Do this experiment need one? What quantity will be the "baseline"?

9. In this experiment, is it good to measure the baseline first or later? What practical consideration(s) is (are) there for your answer?

10. Set up a data table below that you will eventually copy into your Part 4 section for this lab in your lab notebook. Be sure to use the first row in the table as a heading list.

Unknown number ________

11. Now, for the practical matters specific to this lab. How do you measure a freezing point? What instruments will you need? Hint: the particular instrument shown in the photo of the lab writeup is not the device you will be using – you may recall back to the first two labs this quarter and a device that might be more useful than the one in the photo.

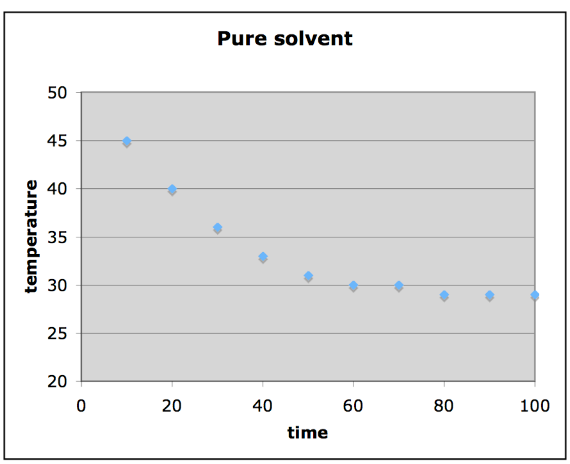
12. Even though the data table generation may be automated, set up another data table you will need in order to organize the data from the freezing point measurement:

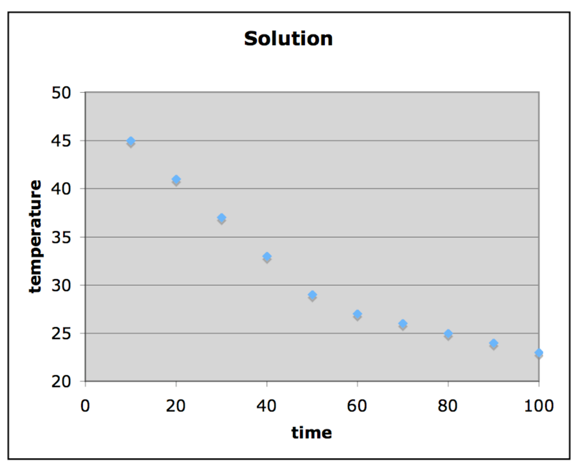
13. How can you determine a freezing point from the data above? What would be a better way to display this data, so that the freezing point could be more easily determined?

For the solutions of unknown solutes in the tert-butanol, here is what a colleague says about the procedure:

"For pure solvent, the curve should flatten out to a plateau. Extrapolate this plateau to the temperature axis to get the freezing point. The cooling curve of the solution will not have a plateau but will continue to drop at a constant rate. To get the freezing point from this curve, extrapolate the flat regions (more or less constant slope portions) of the curve until they intersect. From this intersection point, draw a horizontal line across to the temperature axis. Where it crosses is the freezing point of the solution. You will probably see what is known as supercooling (or cooling below the freezing point). This will appear as a dip in the curve just before it flattens out. Ignore these points when drawing your extrapolation lines."

14. Below are two such cooling "curves". As best as you can figure what he's saying, apply his procedure to the set of points in each graph to determine the freezing point of each. Draw an arrow to the temperature axis that indicates the freezing point temperature.





15. Practical concerns round out the design portion of the experiment.

a. How do you get the tert-butanol, which freezes around room temperature, to melt? What equipment/materials do you need? How do you contain the tert-butanol in the first place?

b. How do you get the tert-butanol to freeze relatively quickly, since you have only a couple hours to finish the whole experiment? What equipment/materials do you need?

c. How do you know that the temperature probe is reading the “true” temperature of the tert-butanol? In other words, how do you get rid of any temperature gradients inside the test tube?

d. How do you know when you have enough data? In the past, we’ve chosen the criterion that two trials whose final value differs by 5% was sufficient to end the experiment. However, 5% is arbitary; let’s try a different criterion.

In the equation on the first page, what type of equation is it (linear, logarithmic, square, etc.)?

So if you were to plot the independent variable in the equation versus the dependent variable, what **correlation coefficient** would you expect?

But the two variables do not have a simple dependent/independent relationship. In fact, is it possible to measure both variables independently (in other words, is there some distance between cause and effect?)?

Therefore, will the correlation coefficient necessarily be what you expected?

But you need at least five graph points to calculate a reasonable correlation coefficient. You might have time in the lab to do three measurements? How do you easily acquire more?