CH 222 Summer 2022: **"Molar Mass Determination by Freezing Point Depression"** ***Lab*** *- Instructions*

Step One:

Print this lab! You will need a printed (hard copy) version of pages I-7-6 through I-7-7 to complete this lab. If you do not turn in a printed copy of the lab, there will be a 2-point deduction.

Step Two:

Watch the lab video for the "Freezing Point" lab, found here:

http://mhchem.org/y/7.htm

Step Three:

Complete pages I-7-6 through I-7-7 using the "Freezing Point" video and the actual lab instructions on pages I-7-2 through I-7-5. Include your name on page I-7-6! **Include a computer generated graph** of the data!

Step Four:

Complete the lab work and calculations on your own, then **turn it in at the beginning of lab to the instructor on Tuesday, July 19**. **Include a computer generated graph.** The graded lab will be returned to you the following week during recitation.

If you have any questions regarding this assignment, please email (mike.russell@mhcc.edu) the instructor! Good luck on this assignment!

Molar Mass Determination by Freezing Point Depression

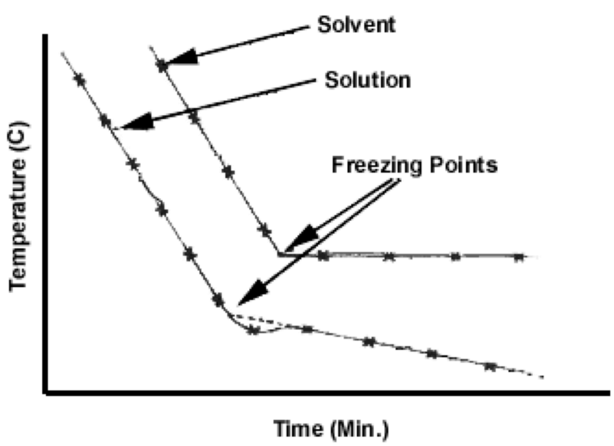
If a nonvolatile solute is added to a liquid, a number of physical properties of the pure substance change, including vapor pressure depression, freezing point depression, and boiling point elevation. These alterations are collectively known as **colligative properties** of solutions. The colligative properties of a solution change in proportion to the concentration of solute dissolved in solvent and depend only on the number of solute particles present in a given amount of solvent and not on the type of particles dissolved. Therefore, the concentration of the solute is most conveniently expressed in terms of **molality** (*m*) or **moles of solute / kg of solvent**.

The change in the freezing point (ΔT_f) in ˚C for a nonvolatile organic solvent can be determined using the following equation, where $\mathbf{k_f}$ is characteristic for the solvent used: $\mathbf{\Delta T_f = k_f \cdot \mathit{m}}$

We can determine the **molar mass** of the solute using this equation by measuring the change in the freezing point of the solution and solving the equation for molality. The calculated molality can be used to determine the moles of solute that in turn can be used to calculate the molar mass (grams / mole) of the solute.

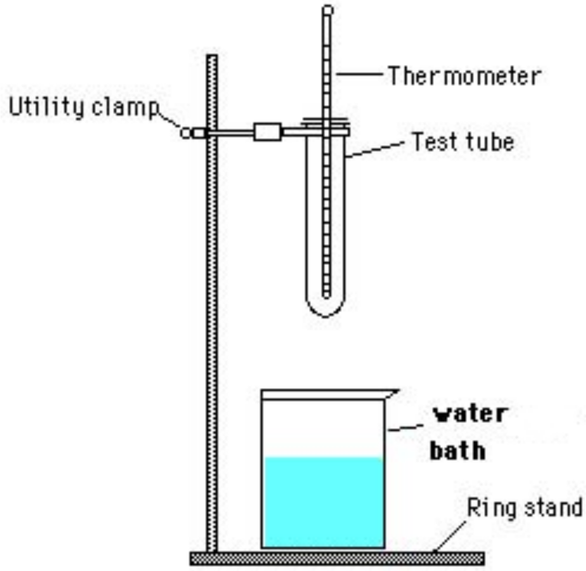
When a pure substance is heated to a liquid state and allowed to cool, initially the temperature will fall quite rapidly. As the substance approaches its **freezing point**, solid will begin to form and the temperature will begin to level. The freezing point of the pure liquid is the constant temperature observed while the liquid is solidifying.

The cooling behavior of a solution is somewhat different from that of a pure liquid. The temperature at which the solution begins to freeze is **lower** (i.e. *depressed*) than for the pure solvent. Additionally, there is a slow gradual fall in temperature as freezing proceeds. The **change in temperature, ΔT**, between the freezing point of the pure substance and the freezing point of the solution is used to calculate the molality of the solution.



In both the pure liquid and the solution, a **supercooling** effect may be seen. As the solid begins to form, the temperature may drop *below* the actual freezing point initially and then come back up to the freezing point temperature as the solid forms. Supercooling is usually not observed if adequate churning of the sample is provided. When determining the freezing point, the super-cooling effect should be ignored.

In this experiment, you will first determine the freezing point of a pure solvent, **lauric acid** ($C_{12}H_{24}O_2$). Next, you will use a known solute, **benzoic acid**, to depress the freezing point of the solvent and calculate the molar mass of the benzoic acid.



This picture should also include a hot plate under the beaker and a "swizzle stick" around the thermometer

<u>PROCEDURE</u>: Part A: Determining the Freezing Point for Lauric Acid

1. Set up ring stand and test tube clamp beside the hot plate

2. Fill a large beaker about ¾ full with tap water and heat on hot plate.

3. Mass 10.000-12.000 g of solid lauric acid ($C_{12}H_{24}O_2$) and record the mass. Add the lauric acid to a large test tube.

4. Place the test tube in the beaker and heat until the lauric acid is completely melted. Put a "swizzle stick" around a thermometer and place it into the liquid. Gently stir with the swizzle stick (not the thermometer!) to uniformity. Do not overheat - you will need to cool it in step 5 (perhaps read ahead?) You may need to readjust your thermometer so it remains in the solution.

5. Remove the test tube from the hot water and allow it to cool. Once the temperature reaches 50 °C, begin to record the temperature every 30 seconds until the temperature plateaus for 3 minutes or reaches 35 °C. (This should take 5-10 minutes). Mix ***gently*** to maintain uniformity (test tubes are glass! Careful!) The biggest source of error in this lab is the temperature so use care in reading. Make sure you record the temperature to the correct number of significant figures – if unsure, ask!

Note: Be sure that data is recorded in the lab data books for all lab partners (perhaps the note keeper could record in both note books as the other student mixes?)

PROCEDURE: **Part B: Determination of the Molar Mass for Benzoic Acid**

6. Mass 1.000 - 1.200 g of benzoic acid and record the mass. Add to the test tube with the lauric acid. Be careful not to lose any sample of lauric acid that may have solidified to the thermometer or swizzle stick.

7. Place test tube in hot water and melt the mixture, stirring to uniformity.

8. Remove the test tube from the hot water and allow it to cool. Once the temperature reaches 50 °C, begin to record the temperature every 30 seconds until the temperature plateaus for 3 minutes or reaches 35 °C. (This should take 5-10 minutes). Mix ***gently*** to maintain uniformity (test tubes are glass! Careful!)

9. Repeat steps 6 through 8, adding an additional 1.000 - 1.200 grams of benzoic acid to the solution. Do ***NOT*** make a new solution!

10. Time to clean up! To clean up, re-melt your solid and pour into waste container. You may need to add a tiny bit of hot water along the sides of the tube to help melt the solid. Wipe the thermometer and swizzle stick with a wee bit of acetone and/or methanol if needed.

CALCULATIONS: Determining the Molar Mass of the Benzoic Acid

Once you have completed your experiment, you will need to create a **graph in Excel** (or a similar program; no hand drawn graphs will be accepted.) Use **time** as the x-axis and **temperature** as the y-axis. Plot the data from your pure lauric acid experiment (Part A) and also the data from each of the benzoic acid solutions (Part B) **using the same graph**. Label the axes and use different colors and/or marking symbols to distinguish the trials.

Important: your graph should take up an entire page of paper (no small graphs, print in "landscape" mode), *and* the **y-axis should *not* start at zero** (i.e. if your data points end around 25 °C, have the minimum y-axis value be 20 °C, not zero °C (which is what these programs often default to when creating graphs.)

Determine the freezing point for each trial graphically; **draw a circle** on your graph representing the freezing point in each trial. Remember, the freezing points can be found at the intersection between the "steep" cooling curve and the "gradual" cooling curve.

The k_f value for lauric acid is 3.90 °C/m. Using the data from Part A and Part B, calculate the molar mass of benzoic acid in each trial. Show how you calculated these values in your lab report.

Average the two molar mass values and calculate the **parts per thousand**.

Using the Internet or a textbook, determine the structure of benzoic acid and **include a Lewis structure for benzoic acid in your lab report**. (Hand drawn Lewis structures are ok.)

Determine the actual molar mass of benzoic acid using the Lewis structure. **Calculate the percent error** of benzoic acid using your average molar mass value and the accepted molar mass value. *Recall:* **Percent error = absolute value{(actual - experimental)/ actual}*100%.**

POSTLAB QUESTIONS:

1. Determine the effect of the following on the final molar mass calculation. Give your reasoning. Tell if the molar mass will be **higher**, **lower** or **not change**.
    a. The thermometer you were using read temperatures consistently 1.2 ˚C higher than the real temperature.
    b. You added 3 g of unknown solute during your freezing point determination.
    c. In adding your unknown solute to your cyclohexane, you spilled some into your water bath.

2. A student performs a freezing point analysis. She determines that the freezing point of 21.00 g of stearic acid (where k_f = 4.89°C/m) is 68.20 ˚C. She adds 2.07 grams of an unknown compound to her sample and determines the freezing point to be 65.53 ˚C. She adds an additional 1.97 g of the unknown compound and determines the new freezing point to be 63.03 ˚C.
    a. Determine the molar mass of the unknown compound using the 2.07 g of sample.
    b. Determine the molar mass of the unknown compound using the *combined* samples (*hint:* 2.07 + 1.97 = 4.04 g total solute)
    c. Determine the average molar mass of the compound and the parts per thousand for the two trials.

Molar Mass Determination by Freezing Point Depression

YOUR NAME: ____

DATA: *Watch the video (***http://mhchem.org/y/7.htm***) for assistance with this lab.*

Graph the following data in Excel, Numbers, Sheets or a similar computer program (hand drawn graphs will not be accepted for credit.) Use **Time (minutes)** as your x-axis and **Temperature °C** as your y-axis.

- You will have two different data streams ("**PDB**" and "**Solution I**" which use a common set of axes; use a color or point marking system to differentiate "PDB" from "Solution I".
- Ensure that your temperature axis reflects an appropriate range of values for the data provided (i.e. don't start at zero!) **Attach the graph to this lab when turning it in to the instructor**.

Time (minutes)	PDB (deg C)	Solution I (deg C)
0.5	63.7	61.3
1.0	62.1	59.7
1.5	60.8	58.2
2.0	59.1	56.5
2.5	57.5	54.8
3.0	55.8	53.4
3.5	54.0	52.0
4.0	53.0	50.8
4.5	53.0	50.2
5.0	53.0	50.2
5.5	53.0	50.2
6.0	53.0	50.1
6.5	53.0	50.0
7.0	53.0	50.0
7.5	53.0	49.9
8.0	53.0	49.8
8.5	53.0	49.7

ANALYSIS:

- On your graph, determine the freezing point for the "PDB" and "Solution I" curves. Look for the spot where two slopes on the same line come together; this is the official freezing point. **Mark the graph at the freezing point**, and enter the actual numbers below.

Freezing Point of PDB (°C): ________________ **Freezing Point of "Solution I" (°C): ________________**

- The "PDB" for your graph stands for "para-dichlorobenzene". Draw the structure of PDB here.

- Using the information from the graph and the freezing points, **determine the molar mass of the solute** in "Solution I" if **2.35 g** of the unknown compound were dissolved in **30.46 g** of PDB. (**k_{fp}** for PDB = **7.10 °C/m**) *Show work!*