Experiment 1

**MOLAR MASS DETERMINATION BY FREEZING POINT DEPRESSION**

Whenever a substance is dissolved in a solvent, the vapor pressure of the solvent is lowered. As a result of the decrease in the vapor pressure, the boiling point, freezing point, and osmotic pressure of the solvent are changed. The magnitude of these changes depends on the number of solute particles dissolved in a given mass of solvent. At low solute concentrations, the changes in the vapor pressure, boiling point, freezing point, and osmotic pressure of a solution are all proportional to the amount of solute that is dissolved in the solvent. These four properties of solutions are collectively known as **colligative properties**.

The colligative properties of a solution depend only on the number of solute particles present in a given amount of solvent and are independent of the nature of the particles dissolved. If the solute is a nonvolatile substance, the vapor pressure and the freezing point of the solution is lower than that of the pure solvent and the boiling point is higher. Some common uses of colligative properties are the addition of "antifreeze" to automobile radiators to lower the freezing point of water and the sprinkling of salt on icy sidewalks to melt the ice by lowering its freezing point.

Colligative properties are useful for determining molar masses of unknown compounds and the degree of dissociation in solution of known compounds. For low concentrations of a nonvolatile solute, the freezing-point depression of a solvent is given by the relationship:

$$\Delta T_f = K_f m$$  \hspace{1cm} (1)

where $\Delta T_f = T_f (\text{solvent}) - T_f (\text{solution})$, $T_f$ is the freezing temperature, $K_f$ is the molal freezing point depression constant for the solvent (which is simply a proportionality constant characteristic of the solvent used), and $m$ is the molality of the solution. Molality is a unit of concentration that is defined as:

$$m = \frac{\text{mol(solute)}}{\text{kg(solvent)}}$$ \hspace{1cm} (2)

In order to determine the molar mass of an unknown compound by measuring the freezing point depression of a solvent, the molal freezing point depression constant for the solvent must be known. The value of $K_f$ is determined by dissolving a measured amount of a known compound into a given amount of solvent and then measuring the depression of the freezing point. The value of $K_f$ is equal to the number of Celsius degrees that the freezing point of the solvent is lowered when 1 mole of solute particles is present in 1000 g of solvent (i.e., the difference in the freezing point of the pure solvent and a 1 molal solution). **NOTE:** It is important when determining the value of $K_f$ for a solvent that the solute does not dissociate when it dissolves so that the number of particles of solute in solution is equal to the number of molecules added. The freezing points and molal freezing point depression constants of some common solvents are listed in Table 1.
To experimentally determine the molar mass of an unknown substance by the freezing point depression method, a solution is prepared by adding a measured mass of sample (i.e., the solute) into a known amount of solvent. From the observed freezing point depression (ΔT), the molal concentration of the unknown substance in the solution can be calculated rearranging Equation (1):

\[
m = \frac{\Delta T}{K_f}.
\]  

Rearranging the definition of molality [Equation (2)] and using the mass of solvent (in kg), the number of moles of unknown substance can be determined from the relationship:

\[
\text{mol (solute)} = m \times \text{kg (solvent)}.
\]  

The molar mass (MM) of a compound is defined as the number of grams per mole. Therefore, from the definition of molar mass, the molar mass of the unknown sample can be determined by dividing the number of grams of solute by the number of moles of solute:

\[
\text{MM} = \frac{\text{g (solute)}}{\text{moles(solute)}}.
\]
Example: A solution containing 15.00 g of glucose in 100.0 g of water is found to freeze at -1.53°C. What is the molar mass of glucose?  

**NOTE:** The solvent is water since it is present in excess.

**Answer:**

From Table 1, \( K_f \) for water is 1.86°C m\(^{-1}\) and the freezing point of water is 0.00°C. From the data given:

\[
\Delta T_f = T_f (\text{solvent}) - T_f (\text{solution}) = 0.00 - (-1.53) = 1.53°C.
\]

From the freezing point depression and the molal freezing point depression constant, the molality of the solute can be determined from Equation (3):

\[
\text{molality} = \frac{1.53°C}{1.86 °C \text{ m}^{-1}} = 0.823\text{m}.
\]

Using the molality and the number of kilograms of solvent, the number of moles of solute can be determined using Equation (4):

\[
\text{mol} = 0.823\frac{\text{mol}}{\text{kg}} \times 0.1000\text{kg} = 0.0823\text{mol}.
\]

**NOTE:** In order for the units to cancel, the mass of solvent, which was given in grams, must be converted into kilograms (100.0 g = 0.1000 kg).

From the number of grams of solute and the number of moles of solute, the molar mass can be determined from Equation (5):

\[
\text{MM} = \frac{15.00\text{g}}{0.0823\text{mol}} = 182\text{ g mol}^{-1}.
\]

The true molar mass of glucose (\( \text{C}_6\text{H}_{12}\text{O}_6 \)), determined from its formula, is 180.15 g mol\(^{-1}\). The percent error, which is a comparison of a measured value with its actual or true value, is given by the relationship:

\[
\text{percent error} = \frac{|\text{actual} - \text{experimental}|}{\text{actual}} \times 100\%.
\]

In this case, the percent error is:

\[
\text{percent error} = \frac{180.15 - 182}{180.15} \times 100\% = 1\%.
\]

**NOTE:** Only one significant figure is justified in the final answer.
In this experiment, the molar mass of an unknown compound will be determined by the freezing point depression method using cyclohexane ($\text{C}_6\text{H}_{12}$) as the solvent. Cyclohexane is used as the solvent because it has a convenient melting point (6.47°C) and a relatively large value for $K_f$ (20.0). All other things being equal, the larger the value of $K_f$, the larger will be the change in the freezing point for the same molality of solute. Thus the larger the value of $K_f$, the more sensitive the solvent is for determining molar masses by the freezing point depression method.

Because most thermometers are not precisely calibrated, it cannot be assumed that a thermometer would indicate the correct freezing point of cyclohexane. Thus the freezing point of cyclohexane, as measured with each thermometer, must first be determined. The freezing point will be obtained by studying the rate at which liquid cyclohexane cools in an ice-water bath. This procedure is necessary because the temperature at which a liquid freezes is often difficult to determine by simple visual observation, due to supercooling and because the solidification of solutions takes place over a range of temperatures. Therefore, temperature-time graphs called cooling curves, as shown in Figure 1, will be constructed.

![Figure 1. Cooling curve for a pure substance.](image)

Because the melting point of cyclohexane is 6.47°C, it is a liquid at room temperature. If a test tube containing cyclohexane is cooled by immersing it in an ice-water bath at 0°C, the temperature of the sample will vary with time, as shown in Figure 1. Initially, the temperature will fall quite rapidly. When the freezing point is reached, solid cyclohexane will begin to form, and the temperature will remain constant until the sample completely solidifies. The freezing point of a pure liquid is the constant temperature observed while the liquid is freezing to a solid.
The cooling behavior of a solution is somewhat different from that of a pure liquid and is shown in Figure 2. The temperature at which the solution begins to freeze is lower than for the pure solvent. In addition, there is a slow gradual fall in temperature as freezing proceeds. Clearly, the drop in temperature as the solution freezes makes determining the 'freezing point' somewhat ambiguous. However, the 'best' value for the freezing point of a solution is obtained by drawing two straight lines connecting the points on the cooling curve. The first line connects points where the solution is all liquid (i.e., the "liquid cooling" line). The second line connects points where solid and liquid coexist (i.e., the "liquid freezing" line). The temperature at which the two lines intersect is the freezing point of the solution. Extrapolation is necessary because when a solution freezes, the solid that forms has a different composition from the liquid. Usually, the solid is pure or nearly pure solvent, and therefore the remaining liquid becomes increasingly concentrated in solute as solidification progresses. The increase in solute concentration continues to lower the freezing point during solidification and produces the sloping "liquid-freezing" line in the cooling curve that follows the start of solidification. The extrapolation technique allows the easy and accurate determination of the 'initial' freezing point of a solution.

![Cooling Curve Diagram](image)

Figure 2. Cooling curve for a solution containing a small amount of solute.

With both pure liquids and solutions, the temperature may fall below the freezing point without the formation of solid and then suddenly rise back up to it as solid forms. This effect is called **supercooling**, and is illustrated in the cooling curve for the solution shown in Figure 2. When drawing the "liquid-freezing" line, ignore any points where supercooling is observed. Clearly, supercooling produces an ambiguity in the determination of
the freezing point and should be minimized. Gentle stirring of the solution as it cools will reduce the amount of supercooling. However, in order to establish the proper straight line in the solid-liquid region, it is necessary to record the temperature until the trend with time is smooth and definitely established.
EXPERIMENTAL PROCEDURE

A. Determination of the Freezing Point of Cyclohexane

**CAUTION:** Vapors of organic compounds are always potentially hazardous. Avoid breathing cyclohexane vapor unnecessarily. Cyclohexane is flammable, be sure to avoid any ignition source. There should be no flames in lab!

Obtain a sample of an unknown solid from the instructor. Be sure to record the unknown number on the preliminary data sheet. Check out a digital thermometer from the stockroom. **NOTE:** The digital thermometers used in this experiment are both fragile and expensive (approximately $20.00), so handle them with care. Obtain from the cart in the front of the laboratory a #4 2-hole rubber stopper (with Teflon sleeves), a universal clamp, a wide-mouth test tube, a stirrer made of stainless steel, a short piece of tygon tubing, and a ruler.

Record the mass of the clean, dry wide-mouth test tube placed inside a 250 mL beaker, as shown in Figure 3, to a precision of ±0.001 g on the preliminary data sheet. As demonstrated by the instructor, using the repipet, deliver 20.0 mL of cyclohexane into the wide-mouth test tube. Place the test tube containing the cyclohexane back into the 250 mL beaker and obtain the mass to the same precision as before. Record the mass of the test tube containing the cyclohexane in the 250 mL beaker on the preliminary data sheet. The difference between these two masses is the mass of the cyclohexane. **[NOTE:]** Since the density of cyclohexane is 0.7781 g mL⁻¹, the mass of cyclohexane should be ~15.5 g.

![Figure 3. Procedure for obtaining the mass of the cyclohexane.](image-url)
Pack a 400 mL beaker with crushed ice. Add enough water to bring the level of the ice-water slurry to about 350 mL. Place the 400 mL beaker into a 600 mL beaker. The 600 mL beaker serves to trap air and help insulate the ice-water bath so that the ice will not melt as rapidly. Insert the short piece of tygon tubing onto the thermometer and slide it up as far as it will go and then insert the thermometer into the center hole of the two-hole rubber stopper. The tygon tubing should position the thermometer so that it will nearly reach the bottom when inserted into the wide-mouth test tube. Insert the stirrer through the outside hole of the rubber stopper (from the bottom) and assemble the apparatus as shown in Figure 4. **NOTE:** Ring stands are in the large cabinets in the lab benches. The tip of the thermometer should be well covered by cyclohexane. Attach the wide-mouth test tube to the ring stand using a universal clamp. The test tube should be clamped so that the level of the cyclohexane is below the level of the ice-water bath in the beaker. Be sure that the loop of the stirrer circles around the thermometer and slides easily.

![Figure 4. Freezing point depression set-up.](image)

Stir the liquid slowly but continuously to minimize supercooling and maintain a uniform temperature. Start recording the temperature of the cyclohexane to the nearest 0.1°C at about 10°C and record the temperature every 15 seconds on the Preliminary Data Sheet. When the freezing point of cyclohexane is reached, crystals will begin to form and the temperature should remain constant. The number of crystals will increase in the liquid as
cooling proceeds until the solution may solidify to the point where it can no longer be stirred. Keeping recording data until the temperature remains constant (±0.1°C for 4 minutes). When plotted, the cooling curve should resemble the cooling curve for a pure substance shown in Figure 1. The horizontal portion of the graph indicates the freezing point of the cyclohexane as measured by your thermometer.

B. Determination of the Freezing Point of a Cyclohexane Solution

Obtain the mass of your unknown in its vial to a precision of ±0.001 g. Pour one-half of the unknown sample (approximately 0.25 g) into the test tube containing the cyclohexane used in part (A). Reweigh the sample vial to obtain the mass of the solute by difference. Record the masses on the preliminary data sheet.

Allow the test tube to warm until the temperature is above 10°C. Be certain that none of the unknown sticks to the inside of the test tube. If some of the unknown does stick to the inside of the test tube wall above the solvent, mix the unknown into the solvent by gently tilting and rotating the test tube. Be sure that all of the unknown solute has dissolved into the cyclohexane and mix the solution thoroughly.

Pour off some of the water in the ice-water bath and add additional ice to make a thick slurry. Readjust the level in the ice-water bath to approximately 350 mL. Insert the test tube into the ice-water bath as before. Stir the liquid slowly but continuously to minimize supercooling and maintain a uniform temperature. Start recording the temperature of the solution to the nearest 0.1°C at about 9°C and record the temperature every 15 seconds on the Preliminary Data Sheet. When the freezing point of the solution is reached, crystals of cyclohexane will begin to form. However, the temperature will continue to slowly drop as the cyclohexane freezes out of the solution. Keep recording data for at least 6 minutes or until the temperature trend remains constant. The number of crystals in the solution will increase as cooling proceeds until it may solidify to the point where the solution can no longer be stirred. When plotted, the cooling curve should resemble the cooling curve for a solution containing a small amount of solute shown in Figure 2. The intersection of the "liquid cooling" line with the "liquid freezing" line is the "initial" freezing point of the solution as measured by your thermometer. NOTE: If supercooling occurs, the temperature may rise slightly shortly after the appearance of crystals of cyclohexane. If supercooling occurs, those points should be ignored in drawing the "liquid cooling" and "liquid freezing" lines to determine the freezing point.

Add half of the remaining unknown to the solution in the test tube and record the mass of the vial on the Preliminary Data Sheet. NOTE: The total mass of unknown in the solution is now approximately 0.35 g. Allow the solution to warm to 10°C and repeat the entire procedure described above, except start recording the temperature of the solution to the nearest 0.1°C every 15 seconds at about 8°C.

Add the rest of the unknown to the solution in the test tube and record the mass of the vial on the Preliminary Data Sheet. NOTE: The total mass of unknown in the solution is now approximately 0.45 g. Repeat the procedure as before except start recording the temperature of the solution at about 7°C.

NOTE: The vapor pressure of cyclohexane is not too large--especially when it is cold. As a result, the mass of cyclohexane may be assumed to be the same for all four experiments.
To dispose of the cyclohexane-unknown solution, allow the solution to warm to room temperature and pour the liquid into the disposal jars provided in the hoods for that purpose. Do **NOT** pour the solution into the sink! To remove the solid, which solidifies on the test tube and the thermometer, use a 5 mL portion of acetone to dissolve solids remaining in the test tube or on the thermometer. Pour the acetone rinsings into the same cyclohexane-unknown waste container. As before, do **NOT** pour the acetone rinse into the sink! If necessary, repeat the rinsing procedure with up to 2 additional portions of acetone. Do not use more than a total of 15 mL of acetone. If any solid still remains on the sides of the test tube, wash with Alconox solution and vigorous scrubbing and then rinse with hot water. The rinsings with water may be disposed of in the sink.

Return the equipment to the cart upon completion of the experiment and return the ring stand to the large cabinet in the lab bench. Also, be sure to turn the thermometer off (to help extend the life of the battery) and return it to the stockroom. Return the vial, which contained the unknown to the instructor.

**Before leaving the laboratory, transfer all of the necessary information from the preliminary data sheet onto the laboratory report and enter the data into the computer. Be sure to turn in the preliminary data sheet with the laboratory report.**

**NOTES:**

1. If the wide-mouth test tube has any residual solids on the inside, it must be cleaned and dried before being used in this experiment. The test tube may be cleaned by rinsing it with a 5 mL portion of acetone to dissolve solids remaining in the test tube. Pour the acetone rinsings into the disposal jars provided in the hoods for that purpose. Do **NOT** pour the acetone rinse into the sink! If necessary, repeat the rinsing procedure with up to 2 additional portions of acetone. Do not use more than a total of 15 mL of acetone. If any solid still remains on the sides of the test tube, wash with Alconox solution and vigorous scrubbing with a test tube brush and then rinse with hot water. The rinsings with water may be disposed of in the sink. The test tube must be **dry** before being used in this experiment. If water was used to clean the test tube, rinse the test tube with a final 5 mL rinse of acetone and, as before, pour the acetone containing rinse into the waste container and then dry the test tube by placing it over the dryers in the hood as demonstrated by your instructor. If it was not necessary to use water to clean the test tube, the test tube may be dried after the final acetone rinse as described above.

2. Plot your data while waiting for the solution to warm and the unknown to dissolve.
A student wishes to determine the freezing point of a solution made by dissolving 2.137 g of camphor in 28.101 g of paradichlorobenzene (PDB). The following temperature-time data were obtained:

Temperature (°C)    58.5  57.0  55.3  53.9  52.6  51.4  50.0  48.9  49.1
Time (min)          0.0   0.5   1.0   1.5   2.0   2.5   3.0   3.5   4.0

Temperature (°C)    49.2  49.1  49.0  48.9  48.8  48.7  48.6  48.5
Time (min)          4.5   5.0   5.5   6.0   6.5   7.0   7.5   8.0

1. Plot the temperature-time data on the graph paper below.

2. From the graph, estimate the freezing point of the solution.  
   \( FP = \) ______ °C
3. From other data, the freezing point of PDB was found to be 53.1°C. What is the freezing point depression, \( \Delta T \)? (Show all work!)

\[
\Delta T = \underline{\text{___________ }} \ ^\circ \text{C}
\]

4. Using Equation (1), what is the molality of the camphor?

\[
m = \underline{\text{___________ }} \text{ mol kg}^{-1}
\]

5. From the molality of the solution [Question (4)] and the number of kilograms of solvent (PDB) present, using Equation (2), determine how many moles of camphor were present.

\[
mol = \underline{\text{___________ }}
\]

6. From the definition of molar mass (i.e., MM = grams per mole), knowing the number of grams and moles of camphor, calculate its molar mass.

\[
MM = \underline{\text{___________ }} \text{ g mol}^{-1}
\]

7. If the molecular formula of camphor is \( \text{C}_{10}\text{H}_{16}\text{O}\), what is the percent error in the determination of its molar mass?

\[
\text{percent error} = \underline{\text{___________ }} \%
\]

8. Students prepared two cyclohexane solutions having the same mass of solute. However, Student 1 used 13.354 g of cyclohexane, Student 2 used 15.339 g. Which student will observe the larger change in the freezing point? Explain.

9. Would you expect the unknowns used in this experiment most likely to be molecular or ionic compounds? Explain.

10. Pure cyclohexane and the cyclohexane solutions may supercool at their freezing points. Define supercooling and explain how we attempt to minimize supercooling in this experiment.
PRELIMINARY DATA SHEET: FREEZING POINT DEPRESSION

DATA

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Plot the temperature-time data for pure cyclohexane and for each of the three solutions. To avoid overlapping the graphs, add 6 minutes to all observed times in making the graph of the cooling curve for pure cyclohexane. Add 4 minutes to all times for Solution 1. Add 2 minutes to all times for Solution 2. Use times as observed for Solution 3.

Estimated freezing points from graphs:

Pure Cyclohexane = ________ °C  Solution 1 = ________ °C  Solution 2 = ________ °C  Solution 3 = ________ °C
LABORATORY REPORT:  FREEZING POINT DEPRESSION

DATA

Unknown Number __________
Mass of wide-mouth test tube [in 250 mL beaker] (g) __________
Mass of wide-mouth test tube plus cyclohexane [in 250 mL beaker] (g) __________
Mass of vial plus unknown (g) __________
Mass of vial less Sample 1 (g) __________
Mass of vial less Sample 2 (g) __________
Mass of vial less Sample 3 (g) __________
Estimated freezing points from graphs:
Cyclohexane = ________°C  Solution 1 = ________°C  Solution 2 = ________°C  Solution 3 = ________°C

CALCULATIONS

(Show all work!)

<table>
<thead>
<tr>
<th>Solution 1</th>
<th>Solution 2</th>
<th>Solution 3</th>
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<tbody>
<tr>
<td>Mass of unknown (g) (total amount in solution)</td>
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<td>__________</td>
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<tr>
<td>Mass of cyclohexane (g)</td>
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<td>Freezing point depression, ΔT_f (°C) (total depression)</td>
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<td>Molality of solute (mol kg(^{-1}))</td>
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<td>Molar mass of unknown (g mol(^{-1}))</td>
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<td>Average molar mass (g mol(^{-1}))</td>
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QUESTIONS

1. How would the following errors affect the calculated value of the molar mass (high, low, or no effect), if you were not aware that they had occurred? Explain your answers.
   a. A small amount of unknown adheres to the inside of test tube and did not get dissolved in the cyclohexane.
   b. Some solvent is lost by volatilization.
   c. The thermometer used in this experiment was miscalibrated to read 0.5°C lower than the actual temperature over its entire scale.
No experiment you perform, however brilliantly conceived and executed, will satisfy more than 5% of the people concerned.