

Experiment 20

Freezing Point Depression

Pre-Lab Assignment

Before coming to lab:

- Read the lab thoroughly.
- Answer the pre-lab questions that appear at the end of this lab exercise.

Purpose

The freezing point of pure t-butanol will be measured via the Vernier temperature sensor. With this information, a small amount of unknown compound will be added to utilize the colligative property of freezing point depression for solutions to determine the compound's molecular weight.

Background

Impure mixtures or solutions will exhibit lower freezing points than their corresponding pure solvents. The degree of depression of the freezing point is dependent on the amount, rather than the identity, of the impurity introduced. This is called a colligative property. In everyday life, these colligative properties are used by adding salt or other compounds to sidewalks and streets to make the water only freeze at temperatures below 0°C. Rock salt is often added to an ice-water mixture at home in an ice cream freezer to depress the temperature of the mixture in the outer bucket of the freezer to -10°C or cooler.

The freezing point depression (ΔT_f), or the degrees by which the freezing point of the solution is lower than the freezing point of the pure solvent, can be calculated by Eqn. 1.

$$\Delta T_f = K_f * m \qquad \text{Eqn. 1.}$$

Here, K_f is the molal freezing point depression constant that is characteristic of the solvent. For water, $K_f = 1.86^\circ\text{C}/m$. For cyclohexane, $K_f = 20.2^\circ\text{C}/m$. The molality (m) of the solution is a unit of concentration defined by Eqn. 2.

$$\text{molality (m)} = \frac{\text{moles of solute}}{\text{kg of solvent}} \qquad \text{Eqn. 2}$$

Measuring the freezing point depression of a solution can be used to calculate that solution's concentration in molality. Since the molecular weight of a compound is grams/mole, knowing the kilograms of solvent used will yield the moles of solute, whose weight in grams can be measured beforehand. This provides a method for identifying the molecular weight of unknowns.

Example Problem: Determining the Molecular Weight of an Unknown

When 2.35 g of unknown was dissolved into 11.8 g of water, the observed freezing point of the solution was -2.15°C . Determine the molecular weight of the unknown.

Step 1: Solve for the molality of the solution using Eqn. 1

$$(0^{\circ}\text{C} - (-2.15^{\circ}\text{C})) = (1.86^{\circ}\text{C}/\text{m})(m)$$
$$m = \frac{2.15^{\circ}\text{C}}{1.86^{\circ}\text{C}/\text{m}} = 1.16 \text{ m}$$

Step 2: Rearrange molality to solve for moles of solute

$$11.8 \text{ g water} \times \frac{1 \text{ kg}}{1000 \text{ g}} = 0.0118 \text{ kg water}$$

$$1.16 \text{ m} = \frac{\text{mols solute}}{0.0118 \text{ kg}}$$

$$\text{mols solute} = (1.16 \text{ mols}/\text{kg})(0.0118 \text{ kg}) = 0.0137 \text{ mols solute}$$

Step 3: Calculate the molecular weight

$$\frac{2.35 \text{ g unknown}}{0.0137 \text{ mols unknown}} = 172 \text{ g/mol}$$

Supercooling is a phenomenon that occurs when a liquid cools below its freezing point, but does not turn into solid immediately. This usually happens when the temperature is decreased very quickly so that crystals do not have a chance to form. After a short time the liquid will "correct" itself and begin to solidify, increasing in temperature back to its freezing point as it does.

Remember that when substances undergo phase changes, the temperature remains constant. Only after the entire sample changes phase will the temperature then continue to change.

Procedure

Part I: Freezing Point of Cyclohexane

1. Obtain a clean, dry large test tube. Add approximately 8 mL of cyclohexane.
2. Obtain a temperature sensor and a lab computer or LabQuest 2 Interface.
3. If using a computer, make sure Graphical Analysis is on. Connect the sensor using the USB port. The screen should automatically show the sensor's current temperature.
4. Press Sensors, then Data Collection. Leave the mode as "Time Based". Change the rate to 2 samples per second and the duration to 30 minutes.
5. Put the temperature sensor inside the test tube in Step 1.
6. Obtain two Styrofoam cups. Nest them together and fill the inner cup about three-fourths of the way full with ice. Add approximately 50 mL of tap water.
7. On the computer or interface, press the Green Triangle (Start) button. Press the Graph icon in the top right hand corner.
8. Insert the test tube with the temperature sensor into the ice-salt mixture in the Styrofoam cups. Stir the mixture gently by moving in circles and up and down with the temperature sensor inside the tube while you record.
9. As the tube cools, the curve on the graph will continue to decrease until it eventually shows a definite change in slope. If the solution supercools, then the curve on the graph will decrease past the freezing point, then increase back to the freezing point. It is at and after the compound's freezing point that the slope change should be noted. Continue collecting data until the straight light created after the slope change is at least half as long as the previous cooling line, or until the slope of the cooling curve changes again and the sample is entirely frozen. Press the Red Square (Stop) button.
10. On the window with the graph, press and drag the grey box around the data that represents the "cooling" portion of the curve, where the slope was steadily decreasing. Go to Analyze, then Curve Fit. Change the Fit Equation in the drop-down menu to Linear. Click OK. The slope (m) and y-intercept (b) are shown on the right of the graph (Fig. 1). Copy these values into a trendline equation on your data sheet as "cooling".

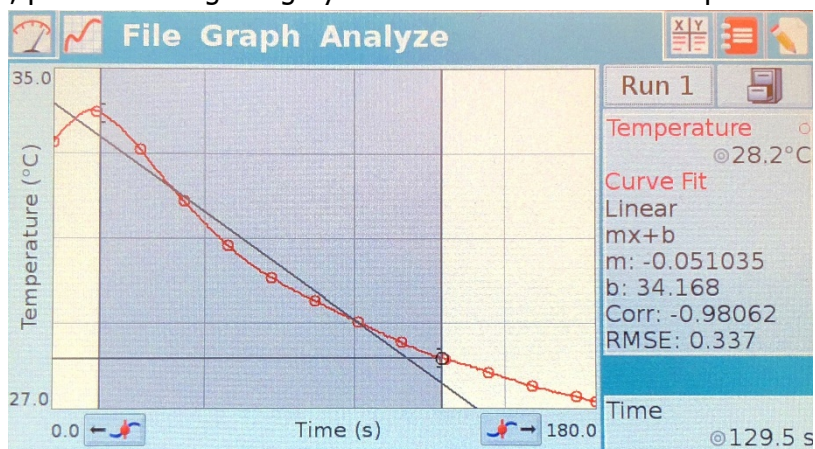


Fig. 1: Selecting "cooling" data to calculate a trendline

11. Repeat the process in Step 9, but click and drag the box around the data that represents the "frozen" portion of the curve, after any supercooling and the slope change was observed. Copy this equation onto your data sheet as "frozen".

12. Export the graph onto a flash drive and print to include with your post-lab.

13. The freezing point of the sample is the temperature (y-axis) at which the two lines, cooling and frozen, meet. Set the equations equal to one another and solve for x (time). Plug this x value into either trendline to solve for y (temperature). Report this freezing point on your data sheet.

Part II: Determination of the Molecular Weight of the Unknown

1. Obtain a clean, dry large test tube. Weigh it empty and record this mass on your data sheet.
2. Add approximately 8 mL of cyclohexane to the test tube and weigh it again. Record this mass on your data sheet.
3. Using your plastic dropper, add 0.8-1.0 g of your unknown liquid to the test tube and cyclohexane. Make sure to drop the unknown into the solvent in the test tube. Do not let it drizzle down the sides of the tube in order to prevent your unknown from evaporating away. Hold both the tube and the dropper vertically while dispensing. Weigh it again and record this mass on your data sheet.
4. Repeat Steps 2-13 of Part I. You may use the same temperature sensor set up and salt-water ice bath as in Part I.
5. Use the freezing point obtained in Part I and in Part II to calculate the molality of the solution of unknown and cyclohexane. Then use this information to find the molar mass of the unknown.

Experiment 20—Data Sheet

Name: _____

Part I: Freezing Point of Cyclohexane

1. Trendline for "cooling" line (from graph): _____

2. Trendline for "frozen" line (from graph): _____

3. Time at freezing point: _____
show calculation:

4. Temperature at freezing point (°C) _____
show calculation:

Part II: Determination of the Molecular Weight of the Unknown

1. Unknown Number: _____

2. Trendline for "cooling" line (from graph): _____

3. Trendline for "frozen" line (from graph): _____

4. Time at freezing point: _____
show calculation:

5. Temperature at freezing point ($^{\circ}\text{C}$)
show calculation:

6. Freezing Point Depression (ΔT_f , $^{\circ}\text{C}$)
show calculation:

7. Molality of solution (m , kg/mol)
show calculation:

8. Mols of unknown (mols)
show calculation:

9. Molar Mass of unknown (g/mol)
show calculation:

A corollary colligative property to freezing point depression is boiling point elevation. In this case, a solution will begin to boil at a higher temperature than its corresponding pure solvent from the following equation:

$$\Delta T_b = K_b * m$$

Here, ΔT_b is the increase between the pure solvent's and the solution's boiling points, K_b is the molal boiling point elevation constant individual to the solvent, and m is the solution's concentration in units of molality.

Strong electrolytes completely dissociate into separate ions when dissolved in aqueous solution. Each ion adds to the number of moles of solute, which in turn increases the change in freezing point or boiling point. The van't Hoff factor (i), is equal to the number of moles that 1 mole of solute becomes when dissolved in solution. A nonelectrolyte, such as $C_6H_{12}O_6$, would have $i = 1$. A strong electrolyte, such as $ZnCl_2$, would have $i = 3$ (Zn^{2+} and 2 Cl^-).

The equations for boiling point elevation and freezing point depression should also include the van't Hoff factor.

$$\Delta T_b = i * K_b * m \quad \text{and} \quad \Delta T_f = i * K_f * m$$

3. Determine the boiling point elevation of a solution prepared by dissolving 11.2 g NaCl into 42.7 g H_2O . The K_b for water is $0.512 \text{ }^\circ\text{C}/m$.

4. Benzene has a K_b of $2.53^\circ\text{C}/m$ and a normal boiling point of 80.2°C . Calculate the molality of a solution of benzene and an unknown nonelectrolyte compound that boils at a temperature of 95.6°C .

5. If the solution in (4) was created by dissolving 15.62 g of unknown into 42.3 g benzene, calculate the molecular weight of the unknown compound in g/mol.

Experiment 20—Pre-Lab Assignment

Name: _____

For all calculations, show all work and draw a box around the final answers.

1. Calculate the molality of a solution prepared from 0.9813 grams glucose ($C_6H_{12}O_6$) dissolved into 8.9547 g water.

2. Find the new freezing point for the solution in (1). (K_f for water is $1.86^\circ C/m$).

3. Why is it important to drop your liquid unknown directly into the solvent rather than allowing it to run down the inside surface of the test tube?

4. A solution is prepared by dissolving 0.8763 g of nonelectrolyte unknown into 10.4832 g of water. The freezing point of the solution was found to be $-3.12^\circ C$. Find the molar mass of the unknown.