

Exp 02 - Freezing Point

Exploring the properties that define substances and allow us to distinguish one type of matter from another.

Preparation (complete this pre-lab work before coming to lab)

Read the experiment Freezing Point (http://chem220.com)

Start a new experiment in your lab book. Write the following:

- The experiment objective(s)
- (Objectives are the question(s) you are trying to answer with this experiment-be brief.)
- Your procedures as a short bullet lists of steps
- A simple table to capture the data you expect in your experiment.

Intended Learning Outcomes

- * Record an analog or digital measurement with as much precision as your instrument will accurately allow.
- * Recognize the significant digits that can be trusted in a measurement recorded by other people.
- * Identify which of the three intermolecular forces exist in a substance based on it's molecular structure.
- * Determine the relative mp or bp between two substances based on what intermolecular forces exist in each.
- * Recognize and give examples of the three primary states of matter.
- * Understand colligative properties are properties of solutions that depend only on the concentration of a solute.
- * Know freezing point depression and boiling point elevation are colligative properties.
- * Understand that freezing point depression occurs due to solute interference between the intermolecular forces of a solvent.
- * Know the definition of molality (m) and use it to solve for moles of solute, kilograms of solvent and molality.
- * Identify the freezing point of a solution as a plateau in a temperature vs time graph.
- * Know how the freezing point depression constant (K_f) relates molality (m) to freezing point depression (ΔT)
 - * $\Delta T = -K_f m$
- * Solve for the freezing point depression constant (K_i), molality (m) or freezing point depression (ΔT) of a solution from the other two.

Report (prepare a report on your experiment to be submitted in the following lab meeting)

Prepare a report for this experiment according to this experiments report description for the parts we accomplished. Include the questions with answers for this experiment, except any your instructor tells you to omit.

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Analysis (calculations needed in your report analysis)

- 1. Determine the change in freezing point from the observed freezing point of the solution and the freezing point of the pure solvent. (This will be a negative number.)
- 2. Determine the molal concentration, m, from the change in freezing point and the freezing point depression constant.
- 3. Determine the moles of unknown (the solute) from the molality of the solution and the mass of solvent (in kilograms) used to make the solution.
- 4. Determine the molar mass from the mass of the unknown and the number of moles of unknown. solution.

Post-Lab Questions (rewrite these questions with your response and append them to your experiment report) (when calculations are used to produce your answer, show your calculations)

- 1. Determine the number of significant figures in each of the following measurements:
 - a. 503 g b. 210 mL c. 210.0 mL d. 0.0210 cm
- 2. List the following pure substances in order of decreasing melting point.

a.	$CH_3CH_2NH_2$	C.	CH ₃ CH ₂ CI	e.	CH ₃ CH ₂ CH ₃
b.	CH₃CI	d.	CH ₄		

- 3. Sugar (C₁₂H₂₂O₁₁) is dissolved in 2.0 Kg of water to produce a 6.2 molal (m) solution. How many moles of sugar were dissolved?
- 4. A solution that is 2.4 m in sugar (C₁₂H₂₂O₁₁) is found to have a melting point of -4.46°C. The normal melting point of water is exactly 0°C. What is K_{f H20}, the freezing point depression constant, of water?
- 5. Freezing point depression is a colligative property, a property of solutions that depends only on concentration of solute. Freezing point depression occurs because particles of solute interfere with the intermolecular forces that control properties like melting point and boiling point.
 - 1. What would be the melting point of a 3.6 m solution of sugar (C₁₂H₂₂O₁₁) in water?
 - 2. What would be the melting point of a 2.4 m solution of carbon tetrachloride (CCl₄) in water?
 - What would be the melting point of a 2.4 m solution of sodium chloride (NaCl) in water? (hint: unlike sugar and carbon tetrachloride, sodium chloride is an electrolyte.)

Colligative Properties: Freezing Point Depression

Introduction

The properties of a pure solvent may be altered when a solute is dissolved in it. The magnitude of the change is proportional to the amount of solute added. Some properties of the solvent are changed only by the number of solute particles present, without regard to the particular nature of the solute. These properties are called colligative properties of the solution. Examples of colligative properties include changes in vapor pressure, boiling point, freezing point and osmotic pressure.¹

When a nonionizing, nonvolatile solute is added to a volatile solvent, the amount of solvent that can escape from the surface of the liquid at a given temperature is lowered, relative to the case where only the pure solvent is present. Thus the vapor pressure above the solution containing solute will be lower than the vapor pressure of the pure solvent. The dissolved solute molecules physically block the surface of the solvent, preventing some of the solvent molecules from evaporating at a given temperature.



Shown below are two phase diagrams for water. The first diagram illustrates the normal freezing and boiling points for pure water, i.e., without the addition of any nonvolatile solute. The second diagram illustrates the increased boiling point and reduced freezing point for water after the addition of a solute.

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¹ This is why ethylene glycol works as antifreeze in your car's cooling system: it depresses the freezing point of water so the coolant does not freeze during the winter months.



The decrease in freezing point or the increase in boiling point is proportional to the molal concentration m (molality): We will refer to this equation as the

$$\Delta T = K \cdot m_s$$
 We will refer to this equation
colligative property equation

 ΔT = either decrease in freezing point or increase in boiling point of solvent K = either freezing point or boiling point constant for given solvent

 m_s = molality = moles of solute/kg of solvent

Remember K is a constant for a given solvent and represents by how many degrees the freezing point will change when 1.0 mols of solute is dissolved per kilogram of solvent. For example, let's suppose you are investigating the freezing point depression of water. The colligative property equation would take the following form:

$$\Delta T_{\rm f} = K_{\rm f} \cdot \boldsymbol{m}_{\boldsymbol{s}}$$

where $\Delta T_f = T_{pure solvent} - T_{solution}$

In this case $K = K_f$, the freezing point depression constant for water which is 1.86 °C kg mol⁻¹. Similarly, if you are investigating the boiling point elevation of water, the colligative property equation would take the form:

$$\Delta T_{b} = K_{b} \cdot \boldsymbol{m}_{s}$$

where $\Delta T_b = T_{solution} - T_{pure solvent}$

In this case $K = K_b$, the boiling point elevation constant for water which is 0.51 °C kg mol⁻¹.

<u>Purpose</u>

To become familiar with colligative properties and to use then to determine the freezing point depression constant of water and the molar mass of an unknown substance.

Approach

Water will serve as your solvent for this experiment. First you will determine the freezing point of pure water. You will then prepare a 0.500 m sucrose solution and determine its freezing point. Finally, you will determine the freezing point of a solution containing an unknown solute.

Because the presence of solute reduces the freezing point of an aqueous solution below 0°C, the freezing point of pure water, we need a way to cool the solutions below 0°C. To do this we will use the phenomenon itself to provide the conditions necessary for the test. We will make an ice/salt bath by mixing crushed ice with sodium chloride. The temperature of this ice/salt bath (~ -20°C) will be lower than the temperature of a pure ice bath (0°C).

Students work in pairs.

Apparatus/Materials

Centigram balance		Deionized water
Two large test tubes	(24mm x 150 mm)	2 digital thermometers
1- 600 mL beaker		1 – 400 mL beaker
1 box of salt		Crushed ice
Sucrose		Stopwatch/timer
Stirrer		Spatula (to mix ice + salt)

Part A: Preparation of 0.5 m sucrose solution

- 1. Determine the molar mass of sucrose.
- 2.
- 3. Using clean glassware and a centigram balance, add 50.0 g of prepare 50.0 g of a 0.5 *m* sucrose solution.
- 4. Set solution aside for use in part C.

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Part B: Freezing Point of Pure Water

Unlike the boiling point of water, which varies significantly with ambient local pressure, the freezing point of water is almost unaffected by pressure. Since thermometers may vary in accuracy, you will first calibrate your thermometer by using it to determine the freezing point of pure water.

- 1. Work with a partner to collect data. The analysis of the data is to be done individually.
- 2. Quarter fill a 400 mL beaker with crushed ice and pour a ½ inch layer of sodium chloride on top of the ice. Stir and thoroughly mix the ice and salt. Add another quarter layer of ice and pour another ½ inch layer on top of the ice. Stir and thoroughly mix the ice and salt.
- 3. Immerse a thermometer in the ice/salt bath, allow a minute for it to stabilize, and record the temperature of the ice/salt bath.
- 4. Fill a 600 mL
- 5. Quarter fill a test tube with water (you do not need to determine the mass of water) and insert a stirrer and thermometer into the test tube.
- 6. Carefully press the test tube down into the ice bath until the water is completely below the surface of the ice bath. Record the initial water temperature and start your timer.
- 7. Begin collecting time/temperature data every 15 seconds. Continue stirring the water (move copper stirrer using up and down motion) as you collect data.
- 8. The temperature should "plateau" when the freezing process begins. Eventually the water will solidify to the point where you will be unable to stir it.
- 9. Continue taking the temperature of the water until you have collected 30 data points.
- 10. Prepare a plot of your time/temperature data and determine your temperature correction factor.

Part C: Freezing Point of 0.5m Sucrose Solution

- 1. Place the test tube containing the frozen water (with the stuck thermometer/stirrer) from Part A under hot water begin heating. Remove the thermometer and stirrer and dry the test tube.
- 2. Quarter fill the test tube with the 0.50 *m* sucrose solution prepared in Part A and insert a stirrer and thermometer into the test tube.
- 3. Carefully press the test tube down into the ice bath until the water is completely below the surface of the ice bath. Record the initial aqueous solution temperature and start your timer.
- 4. Begin collecting time/temperature data every 15 seconds. Continue to stir the solution as you collect data.
- 5. Continue recording the temperature every 15 seconds for the remainder of the freezing process. Remember that cooling curves for solutions often tend to show a more noticeable slope as opposed to a "plateau" when the freezing process begins. Eventually the solution will solidify to the point where you will be unable to stir it.
- 6. Continue taking the temperature of the solution until you have collected 30 data points.

Part D: Molar Mass of Unknown Solute.

- 1. Weigh a large, clean, test tube to the nearest 0.01 g.
- 2. Quarter fill the test tube with DI water and weigh again. The difference in mass is the mass of the water.
- 3. Using weighing paper, weigh about 0.75 g of the unknown solute to the nearest 0.01 g.
- 4. Carefully add the unknown solute to the test tube and stir gently until dissolved.
- 5. Insert a stirrer and thermometer into the test tube.
- 6. Carefully press the test tube down into the ice bath until the water is completely below the surface of the ice bath. Record the initial aqueous solution temperature and start your timer.
- 7. Begin collecting time/temperature data every 15 seconds. Continue to stir the solution as you collect data.
- 8. Continue recording the temperature every 15 seconds for the remainder of the freezing process. Remember that cooling curves for solutions often tend to show a more noticeable slope as opposed to a "plateau" when the freezing process begins. Eventually the solution will solidify to the point where you will be unable to stir it.
- 9. Continue taking the temperature of the solution until you have collected 30 data points.

Preparation of Finished Lab Report

Follow the steps below to prepare your finished lab report. Your report should contain the sections listed in the *exact* order they are listed below:

- I) Title (front) page (typed)
 - a) Experiment title
 - b) Your name & name of your lab partner
 - c) Date experiment conducted
 - d) Chemistry 220, Canada College, Fall Session, 2014

II) Data (Excel spreadsheet format) (typed). Include data for your three temperature-time curves. The data should be in tabular form. DO NOT SPLIT UP DATA TABLES.²

- a) Time-temperature data for pure water
- b) Time-temperature data for 0.50 *m* sucrose solution (corrected)
- c) Time-temperature data for unknown solute (corrected)

III) Cooling Curve Plots – Use Excel to generate cooling curve plots for each trial. For each plot, graph time on the x-axis and temperature on the y-axis. Properly label each plot. You do not have to show linear equations for liquid and solid phases on these plots.

² If you ran two trials show data for the trial that will be used in your calculations. If you ran two trials and determined an average value, then show data for both.

IV) Calculations (handwritten or typed)

Calculations: 0.5 m Sucrose Solution

- Use the linear regression feature in Excel to determine a linear equation for the liquid phase and a linear equation for the solid phase for the 0.5 m sucrose solution. Solve these equations simultaneously to obtain the freezing point.³
- 2. Calculate the K_f value for water.
- 3. Compare your calculated K_f value to the published value and determine your % error.

Calculations: Unknown Solute

- 1. Use the linear regression feature in Excel to determine a linear equation for the liquid phase and a linear equation for the solid phase for the 0.5 m sucrose solution. Solve these equations simultaneously to obtain the freezing point.
- 2. Using the published K_f value for water, calculate the molality of the solution containing the unknown solute.
- 3. Calculate the molar mass of the unknown solute.

V) Data Summary (typed) – Prepare a table as shown below and enter results

Freezing pt. pure water (this is your temperature correction factor)		
Results for 0.5 m sucrose solution		
Linear equation (liquid phase) – 0.5 <i>m</i> sucrose		
Linear equation (solid phase) – 0.5 <i>m</i> sucrose		
Calculated freezing pt. for 0.5 <i>m</i> sucrose solution		
ΔT (0.5 <i>m</i> sucrose)		
Calculated K _f for water		
Published K _f value for pure water	1.86 °C/ <i>m</i>	
% error for K _f value (pure water)		
Results for unknown solute		
Mass of water		
Mass of unknown solute		
Linear equation (liquid phase) – unknown solute		
Linear equation (solid phase) – unknown solute		
Calculated freezing pt. for solution with unknown solute		
ΔT (solution with unknown solute)		

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³ You do not need to solve linear equations for the pure water freezing curve.

Published K _f value for pure water	1.86 °C/ <i>m</i>
Calculated molality for unknown solute	
Calculated molar mass of unknown solute	

VI) Post Lab Questions (typed or hand written)

1. For a given number of moles of solute, why do ionic substances have a larger effect on the freezing and boiling points of solvents than do nonionic substances?

2. Which aqueous solution has the lower freezing point, 0.10 *m* NaCl or 0.10 *m* glucose? Explain your answer.

3. What mass of NaCl is dissolved in 200. g of water in a 0.100 m solution?

APPENDIX I – COOLING CURVES

Determining a freezing point by visual observation is difficult, if not often impossible. For this reason a graphic method is employed.

Shown below is a typical cooling curve for a pure solvent, i.e., one that does not contain a nonvolatile solute. The cooling curve is generated by taking temperature readings of the cooling solvent at relatively short time intervals and then plotting these as a function of time.



The flat region represents the freezing point of the pure solvent. The freezing point temperature is determined by extrapolating a line to the y axis (Temperature). The value of T is then read off the y-axis. Sometimes pure liquids exhibit a phenomenon known as "super cooling" which occurs because the solution is cooling faster than the molecules can lock into position in a crystalline structure. Often, supercooling may be prevented by stirring the solvent.



The shape of a cooling curve will change upon the addition of a nonvolatile solute to the solvent as shown in the figure below. The blue curve represents a cooling curve for a pure solvent exhibiting supercooling. The red curve represents a cooling curve for the same solvent to which a nonvolatile solute has been added. Notice that instead of achieving an almost flat segment as in the case of pure solvent, the cooling curves for solutions often tend to show a more noticeable slope. The freezing point for the solution may be determined graphically by extrapolating the cooling curves as shown in the figure below. The point where the curves intersect is the freezing point for the solution.



Another approach that may be used in determining the freezing point of a solution involves overlaying trend lines on certain parts of the data and calculating least-square linear curves. This is shown in the figure below for a solution (solvent + solute) that exhibits supercooling. The first graph shows the plot of the time-temperature data without any trend lines



Two trend lines have been added to the second graph. The first trend line (in black) is obtained using the linear regression feature of Excel to generate the best fitting straight line for the data between Points A and B. The second trend line (in orange) is obtained the same way for the data between points C and D.



Equations for both lines are linear and of the form y = mx + b or T = mt + b where T = Temperature, t = time in seconds and m = slope of line. The intersection of these lines represents the freezing point. Thus by solving the equations simultaneously one may determine mathematically the freezing point of the solution.

Exp #	Name: Student ID: Class Section:			
Chemistry Experiment Report				
Experiment Title:				
Unk#: (write N/A if no unknown for this experiment)	Bench / Locker :/			
This box is for use by your instructor.				
Unknown Composition or Identity:	pre-lab report total			

A report must be submitted for all lab experiments conducted to receive credit for your participation. You may only report on experiments you conducted.

Reminder: All experiment reports should include each of the following sections. Each section should start on a new page and be clearly labeled.

- ➡ <u>COVER PAGE</u> (this page)
- ➡ 1. <u>Pre-Lab</u>
 - ➡ Describe the objectives of this experiment (what you hope to conclude).
 - Any pre-lab questions must be considered and answered before the start of the lab period. Your instructor will initial those pages and they must be included in your lab report to receive full credit for them.
 - If there is a pre-lab quiz it will be graded and returned to you during that lab period. It should be attached to your report to ensure you receive full credit.
- ➡ 2. <u>Data</u>
 - Empirical data, only what you observed and others could if they repeated your experiment. Observations both qualitative and quantitative (measurements).
- ➡ 3. ANALYSIS (CALCULATIONS)
 - Provide demonstrations (justification) of how your conclusions were developed from your observations. Most often accomplished with dimensional analysis or algebra (calculations). For some experiments other methods will be demonstrated and need to be applied.
 - ➡ It is only "known" if it can be justified ... otherwise it's a guess.
- ➡ <u>4. CONCLUSIONS</u>
 - Your final answers. What you came to know by doing this experiment. Be clear and brief, conclusions should not be more than one page and are often only a few sentences.
 - If other factors effected your final answer, briefly describe them on this page and suggest how they may have effected your experiment.
- ➡ <u>5. QUESTIONS</u>
 - ➡ Attach and answer any post lab questions.