

**Molecular Weight of a Water Soluble Solid unknown By
Freezing Point Depression**

Name

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Xxxday, Month Day, Year

PURPOSE/INTRODUCTION

The purpose of this experiment is to determine the molecular weight of an unknown substance by measuring the freezing point depression of an aqueous solution of the unknown. The hypothesis is that as freezing point drops the solvent freeze out, leaving the solution more and more concentrated. The point at which the solvent begins to freeze will be the freezing point of the solution. This will allow for the molecular weight of the solute to be determined. The hypothesis that the freezing point would decrease as more as the solution becomes more concentrated. This is because the addition of solute lowers the vapor pressure of the solution. As the vapor pressure lowers, the freezing point also lowers as the removal of solvent increases the concentration or the number of particles.

The freezing point of a solution of a nonvolatile solute is less than that of the pure solvent. The freezing point depression or change in freezing point is given by

$$\Delta T_f = K_f m \quad \text{where } \Delta T_f = T_f^\circ - T_f \quad (1)$$

T_f is the freezing point of the solution in °C and T_f° is the freezing point of the pure solvent, also in °C. K_f is the molal freezing point depression constant whose value depends only on the solvent. (For water, $K_f = 1.86^\circ\text{C/molal}$). Finally, m is the molal concentration of the solute:

$$m = \frac{\text{moles solute}}{\text{kg solvent}} = \frac{(\text{grams solute})/MW}{\text{kg solvent}} = \frac{\text{grams solute}}{(\text{kg solvent})(MW)} \quad (2)$$

The molecular weight of the solute is MW. To determine the molecular weight of an unknown from the freezing point depression, the freezing points of the pure solvent and of the solution are measured, and ΔT_f calculated. The molality of the solution is then calculated from eqn. (1). The molecular weight of the solute is obtained by rearranging eqn. (2) to solve for MW:

$$MW = \frac{\text{grams solute}}{(m)(\text{kg solvent})} \quad (3)$$

In the preceding discussion, we considered the effect of a *nonionizing* solute. The molal freezing point constant is a property of the solvent, not the solute.

MATERIALS

Materials required to test the hypothesis in a lab include:

- | | |
|---|-----------------|
| ✓ Freezing point depression apparatus (see diagram below) | ✓ Water |
| ✓ Beaker, 600 mL | ✓ Rock Salt |
| ✓ Erlenmeyer flask | ✓ Ice |
| | ✓ Unknown solid |

METHOD

Part One: The freezing point of pure water is determined by cooling a sample of water and measuring its temperature as a function of time.

Clean and dry the smaller test tube of the apparatus and add to it approximately 20 mL of deionized water. Insert the stirrer, stopper and thermometer into the tube. Make sure that the thermometer is immersed in the water, and that the stirrer moves up and down freely.

Place a layer of crushed ice 3-4 cm deep at the bottom of a 600 mL beaker. Sprinkle a good amount of rock salt on the ice. Continue to alternate the layers of ice and salt until the beaker is full. Stir the ice-salt mixture well, immerse the smaller test tube that contains the water into the ice-salt bath, and allow the water to cool to about 4°C. Remove the test tube from the bath, place it inside the larger test tube, and immerse the entire test tube assembly into the ice-salt bath. Stir the water, measure its temperature, and continue taking temperature measurements every 30 seconds. Estimate the temperature to the nearest 0.01°C. Stir the water in the test tube continuously at a uniform rate with the wire stirrer. Continue taking temperature-time readings until 6 successive readings are equal to one another. Repeat this measurement once.

Part Two: To determine the freezing point of a solution of an unknown substance, use the same set up and thermometer used to determine the freezing point of water. Weigh out 3.5 g to 4 g of the unknown to the nearest 0.001g. Record the unknown number and masses.

Using an electronic balance with a precision of ± 0.001 g for all weighings, dry the smaller test tube and weigh it and a small beaker to hold the test tube upright. Add 20 mL of deionized water and weigh the test tube in beaker again. Add the weighed unknown to the test tube. Stir carefully until the solid compound is completely dissolved. Weigh the test tube again.

Prepare a fresh ice-salt bath and reassemble the assembly as in part one making sure the thermometer bulb is immersed in the solution.

Immerse the test tube containing the solution of unknown in the bath and allow it to cool to about 1°C. Remove it from the bath, place it in the larger test tube and place the entire test tube assembly back into the bath. Repeat the procedure in Part one. That is, take temperature time readings to the nearest 0.01°C every 30 seconds. Stir the solution continuously at a uniform rate during the course of the determination. Continue taking readings until the solution solidifies. Do another determination with a second portion of your unknown.

DATA

Data for Unknowns:

Unknown # 3	Mass of	Trial 1, mass/g	Trial 2, mass/g
First weighing	Paper and solid	3.631	2.289.
	Paper	0.018	0.015
	Mass of solid	3.613	2.274
Test tube weighing	Test tube/beaker	60.644	60.678
	Test tube/beaker + solid	64.186	62.949
	Test tube/beaker + solid + water	84.059	82.772
	Water in test tube	20.013	19.823
	Solid in test tube	3.542	2.271
	Volume of water in test tube		20 mL

Time/sec	Pure water		Solution	
	Trial 1 Temp/°C	Trial 2 Temp/°C	Trial 1 Temp/°C	Trial 2 Temp/°C
0.0	4.56	4.03	3.10	1.55
0.5	3.75	3.44	1.82	0.19
1.0	2.91	3.01	0.04	-1.15
1.5	2.41	2.31	-1.77	-2.16
2.0	1.81	1.55	-3.20	-3.12
2.5	1.11	0.96	-4.16	-3.91
3.0	0.59	0.51	-5.34	-4.52
3.5	0.05	-0.06	-6.15	-5.01
4.0	-0.41	-0.31	-6.19	-5.22
4.5	-0.78	-0.65	-5.86	-5.05
5.0	-1.03	-0.85	-5.21	-4.75
5.5	-1.33	-0.70	-4.63	-4.55
6.0	-1.29	-0.55	-4.41	-4.49
6.5	-0.75	-0.23	-4.29	-4.63
7.0	-0.36	-0.05	-4.31	-4.85
7.5	-0.14	0.00	-4.39	-5.13
8.0	-0.05	0.01	-4.47	-5.37
8.5	-0.01	0.00	-4.58	-5.71
9.0	0.00	0.00	-4.74	-6.26
9.5	0.00	0.00	-4.97	-6.88
10.0	-0.01	-0.23	-5.19	-7.58
10.5	-0.04	-0.55	-5.69	-8.46
11.0	-0.28	-1.12	-6.56	
11.5	-0.79	-1.78	-7.34	
12.0	-1.41	-2.36		
12.5	-2.05	-3.11		
13.0	-2.55	-3.55		

RESULTS

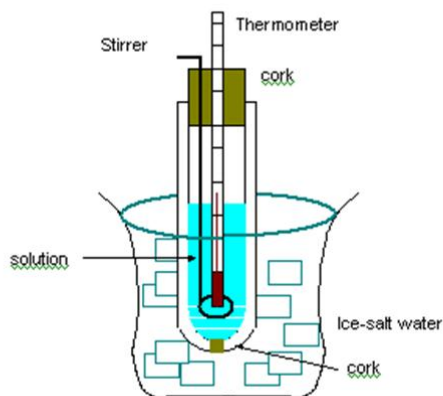


Figure 1 Apparatus for freezing point determination.

The method requires the determination of the freezing points of the pure solvent, water, and of a solution of the unknown dissolved in water. The freezing point of pure water is determined measuring a sample of cooling water's temperature as a function of time. The resulting data are used to construct a cooling curve, shown in Figure 2. The data was plotted using Microsoft Excel. The phase regions listed in the graph are for Trial 1, the blue graph.

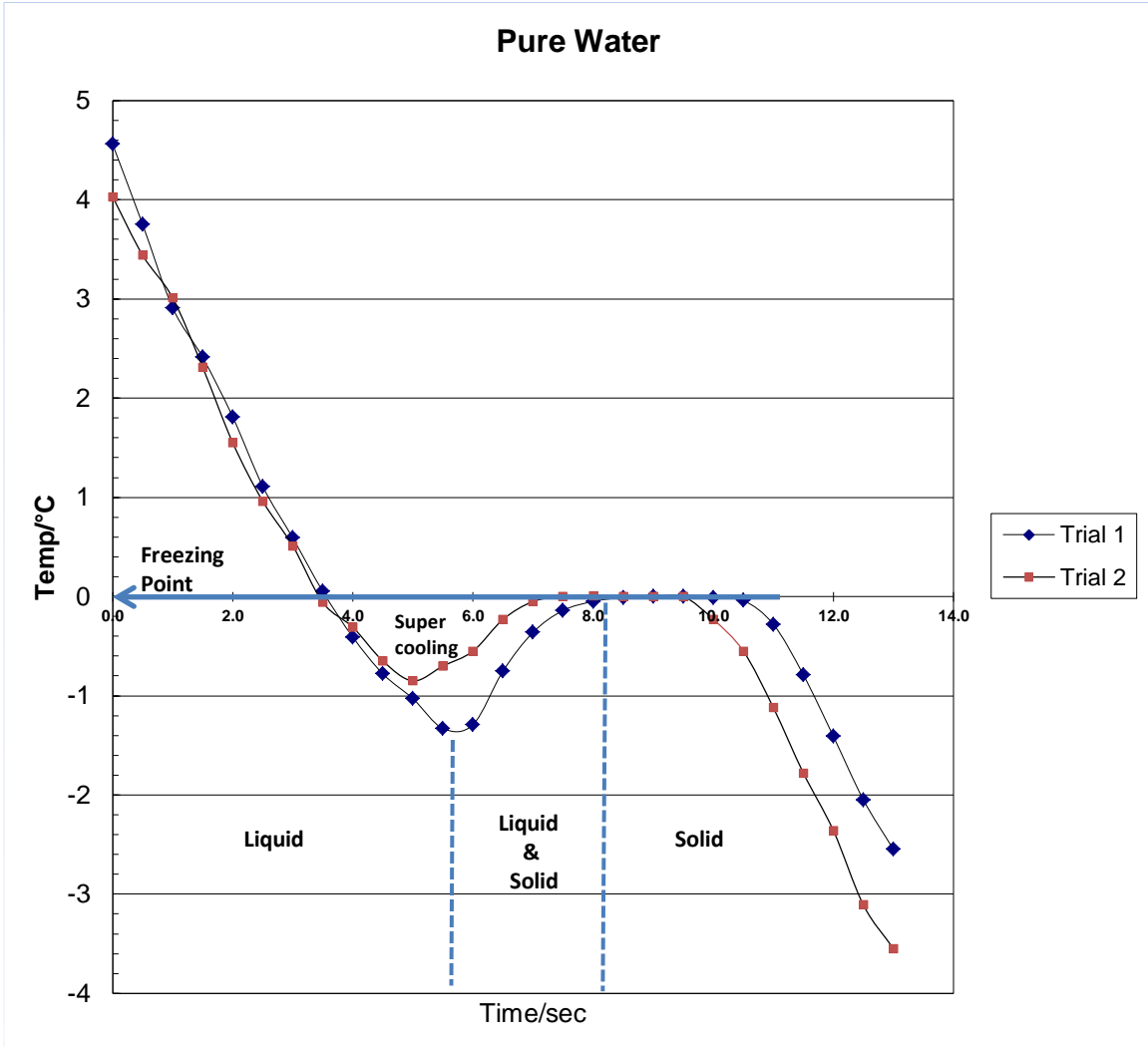


Figure 2. Cooling curve for pure water

The liquid cooled to a temperature below the equilibrium freezing point. This is referred to as "supercooling" (See Figure 2). Supercooling can be minimized by continuously stirring the liquid as it cools. At the freezing point the temperature remains constant because as the liquid solidifies, heat equal to the enthalpy of fusion is released. The true freezing point corresponds to the horizontal portion of the cooling curve. In Figure 2 note the different phases that are present in the solution. The vertical lines are specific to Trial 1, the blue line.

The curve for the solution does not become horizontal at the freezing point. Rather a "kink" or discontinuity occurs at the freezing point. This discontinuity may be obscured by supercooling. If so, it will be necessary to extrapolate by drawing the line **ab** as shown in figure 3.

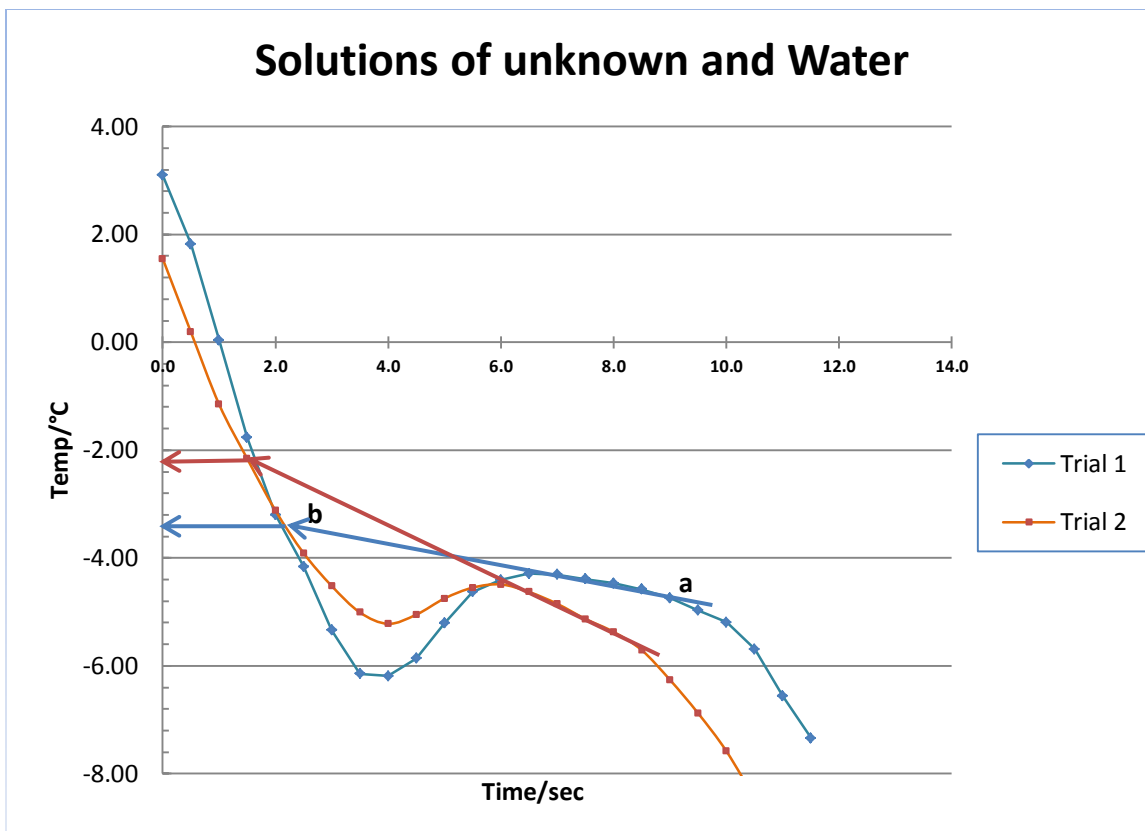


Figure 3. Cooling curves for solutions.

The reason that the cooling curve is not horizontal at the freezing point is that as the solution freezes, the composition of the liquid changes. The solid phase forms at the freezing point is pure solvent. Consequently, as freezing occurs, the concentration of the solution increases, and the freezing point decreases.

ANALYSIS AND DISCUSSION

Pertinent data for the analysis:

	Trial 1	Trial 2
Water in test tube	20.013 g	19.823 g
Solid in test tube	3.542 g	2.271 g
Freezing points	-3.35	-2.16°C
Freezing point depression constant, K_f	1.86°C/m	1.86°C/m
Freezing point of water, from graph	0.00°C	0.00°C

The molality of each solution is calculated using the freezing point depression equation, equation 1, $\Delta T_f = K_f m$, and solve for molality, m :

Equation	Trial 1	Trial 2
$m = \frac{\Delta T_f}{K_f} = \frac{T_f^o - T_f}{K_f}$	$m = \frac{[0.00 - (-3.35)]^{\circ}C}{1.86^{\circ}C/m} = 1.80\text{ m}$	$m = \frac{[0.00 - (-2.16)]^{\circ}C}{1.86^{\circ}C/m} = 1.16\text{ m}$

To determine the molecular weight, equation 3 is used:

$MW = \frac{\text{grams solute}}{(m)(kg\ solvent)}$	$\frac{3.542\text{ g}}{(1.80\text{ m})(0.0200\text{ kg})} = 98.4\text{ g/mol}$	$\frac{2.271\text{ g}}{(1.16\text{ m})(0.0198\text{ kg})} = 98.9\text{ g/mol}$
	<i>m</i> has units of mol/kg	

The average molecular weight is calculated:

$$Ave = \frac{\sum_{i=1}^n (\text{sample})_i}{n} = \frac{98.4 \frac{g}{mol} + 98.9 \frac{g}{mol}}{2} = 98.6\text{ g/mol}$$

The average molecular weight of unknown #3 was found to be 98.6 g/mol. The percent error cannot be determined without knowledge of the substance to compare with the established value. As can easily be seen from the above calculations, the molality of the two trials were considerably different, 1.80 m vs. 1.16 m, the end results were quite similar.

Though the two calculated molecular weights are close, the second trial sample should have been larger to decrease the error. Losing some sample by dropping the unknown sample container prevented a larger second sample. If the glassware was not cleaned correctly or the sample itself contained impurities, the final results would have been off by these impurities. The thermometer was calibrated correctly as seen from the experimental freezing point of pure water being the same as the literature value. A more precise thermometer would have allowed for a more precise final answer.

Freezing point depression is a colligative property that decreases the vapor pressure of the solvent and is only dependent on the number of particles in solution. It was presumed that the solute did not ionize or associate with the solvent. This is a fairly accurate assumption. The freezing point depression with the van't Hoff factor, *i*,

$$\Delta T = i K_f m$$

One way to test out the assumption made in this experiment that the van't Hoff factor is 1.00, is to make up a series of concentrations of the substance using water as the solvent and determine the freezing points experimentally and compare them to the calculated freezing point. If the values are not within the range of the error, then the assumption is incorrect. Also, to prevent association with the solvent or with other solute molecules, the method would not be good for anything but dilute solutions were there are no interactions between molecules.

CONCLUSION

Freezing point depression is one of four colligative properties of solutions. The others are Boiling Point Elevation, Osmotic Pressure, and Vapor Pressure Depression. Colligative properties of a solution depend on the relative numbers of particles of solute and solvent molecules and not on the detailed properties of the molecules themselves.

The results suggest that the freezing point depression method is a viable way to determine molecular weights of substances. Looking at the following equation

$$\Delta T_f = K_f m = \frac{K_f(\text{mass})}{(MW)(kg\ solvent)}$$

it is easy to see that as the molecular weight of a given sample size increases, the temperature change decreases. Thus for large molecular weight substances, this method would not be useful.

The hypothesis was that as the temperature drops below the freezing point of the solvent, the concentration increases as the solution becomes more concentrate with the freezing out of solvent molecules was confirmed. The resulting data can be used to determine the molecular weight of a substance.

During the course of this lab the freezing point of deionized water was discovered through test. This allowed us to later be able to attain other important values. Water was used as the solvent to make solutions 1 and 2 and find the molecular weight of an unknown compound. In this case, the average molecular weight of unknown #3 was found to be 98.6 g/mol.

REFERENCES

Text book Authors. (2008). *Chemistry, What Edition*. New Jersey: Publisher.

Model Science Software Product, Model ChemLab, vs 2.5, Name of Lab

OBSERVATION SHEET goes here