Matter and Motion

Fall 2015

Chemistry Lab 7: Freezing Point Depression of t-butanol

Overview: The goal of this lab is determine the freezing point depression constant K_f for t-butanol and determine the molar mass of a solute by measuring the freezing point depression of t-Butanol when that solute is added to it.

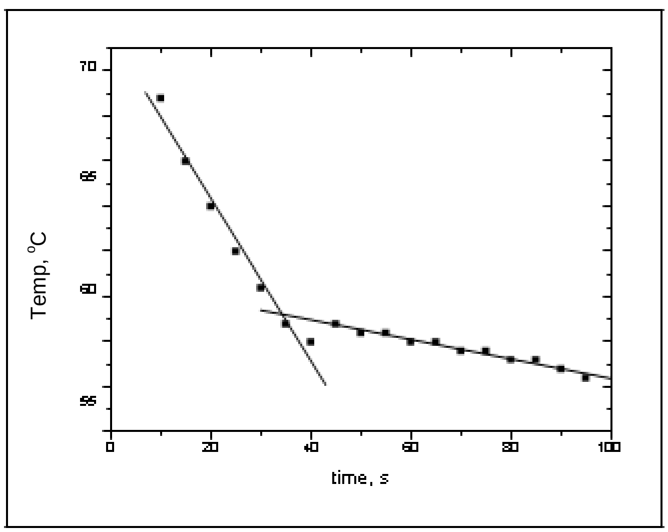
Background

Colligative properties are properties of a solvent, such as freezing point depression and boiling point elevation, which depend on the concentration of solute particles dissolved in the solvent. The decrease in freezing point, ΔT_f (freezing point depression) for a near ideal solution can be described by the equation:

$$\Delta T_f = K_f \cdot m \qquad \text{Eq 1}$$

where K_f is the molal freezing point depression constant of the solvent with units °C · kg solvent/mole solute. *m* is the molal concentration of the solute dissolved in the solvent expressed as moles of solute/kg solvent.

The temperature at which this equilibrium exists is the freezing point of the substance. Sometimes this temperature is difficult to determine, so the use of cooling curves is required. To construct a cooling curve one would warm their sample, pure solvent or solution, to well above its melting point, then allow it to cool. As the sample cools the temperature of the sample is monitored as a function of time. A graph is made by plotting the temperature vs. time. An example of a cooling curve is shown below. As shown in the figure, you will need to draw best fit lines for the liquid cooling phase and the solid phase. The intersection of the two lines indicates the freezing temperature.



In this experiment you will determine the freezing point of pure tertiary butyl alcohol (*t*-butanol) and then determine the freezing point of *t*-butanol with an unknown solute dissolved in it. From these freezing point measurements you will be able to calculate the molar mass of the unknown solute. The *t*-butanol is a good solvent choice for this experiment. Its transition state from solid to liquid occurs near room temperature, so it has a relatively low melting point, thus a low freezing point. It also has a relatively large K_f, 9.10 ºC·kg solvent/mol solute, which is good for estimating the molar mass of a solute because it will allow us to see a greater ΔT_f relative to a solvent with a lower K_f.

Prelab Questions

Read the entire lab carefully (including the Post-lab) then complete the Pre-lab before coming to lab. It should be completed in your chemistry lab notebook and presented for a check as you walk in the door.

1. Some of the words in this lab may be new to you. Make a list of any new vocabulary and their definitions.
2. Read the lab carefully. Describe all of the data you will record during this lab. Be as specific as possible, i.e, include the units used and the number of times each measurement made.
3. Do a quick internet search for the MSDS of t-butyl alcohol. How flammable is the solvent we are working with?

Experimental Procedure

Part I: Determine freezing temperature of pure t-butanol

1. Set up an ice water bath in a 400 mL beaker.
2. Fill another 400 mL beaker with water and place it on a hot plate set to 40°C.
3. Gather a clean and dry test tube and 150 mL beaker to support it as demonstrated. Weigh this assembly on a Top-Loading Balance to a precision of 0.01g. Add about 12 g (~15 mL) of t-Butyl Alcohol, and reweigh the assembly. Aim for between 11.5 and 12.5 g of t-Butyl Alcohol. Use the provided Pasteur pipet to add or remove a small amount if needed. DO NOT PUT ANY t-Butyl Alcohol BACK INTO THE ORIGINAL CONTAINER. When obtaining your sample be sure the outside of the container is thoroughly dry as even minute amounts of Water in the t-Butanol will contaminate the alcohol such that it is unusable.)

!Good Practice – Always use the same balance for repeat weighings!

4. Assemble the test tube, looped stirrer and temperature probe as demonstrated and place this apparatus in your warm water bath. Be careful to not expose your t-Butyl Alcohol to any water.
Heat the system until all the solvent has been melted and heated to ~40°C.
5. Remove the test tube from the hot water bath and place it in the ice water bath. The ice bath should be stirred occasionally during the course of your measurements.
6. Measure the temperature of your t-Butyl Alcohol every 15 sec until solidification is complete. This should take approx. 4 or 5 minutes. During this time the solvent should be continuously (carefully!) stirred using the looped stirrer. Record your observations during this time. Record at the temperature at which slush begins to appear. Do not dispose of the solvent when you are done obtaining the Cooling Curve. You will use this same solvent for the next part of the experiment. **Do not try to pull the thermometer and stirrer from the frozen t-butanol!** Doing so may break the thermometer.

Part II: Determine k_f for t-Butyl Alcohol

7. Again, heat the solvent in the test tube until it is melted and again at ~40°C.
8. Use a micropipette to add your assigned amount of known solute (ethanol CH_3CH_2OH) to your t-Butyl Alcohol. We will calculate the mass used from volume and density (0.789 g/mL).
9. Stir the solvent-solute mixture in the large test tube and repeat the determination of the Cooling Curve.

Part III: Use freezing point depression to calculate molar mass of an unknown solute.

10. Repeat Step 3 to obtain a fresh sample of t-butanol.

11. Use a 10 mL graduated cylinder with cork to measure between 0.5 – 1.0 mL of unknown solute. Use the top loading balance to measure the mass of the graduated cylinder + solute. Record this mass. Carefully transfer the solute to the test tube containing the warmed solvent. Be certain none of the solute adheres to the walls of the test tube. If some does, roll the test tube so that the solvent contacts and dissolves the solute. Immediately re-cork the

"empty" cylinder. Re-weigh the graduated cylinder. The difference in mass of the "full" and "empty" cylinders gives the mass of the solute used.

12. Stir the solvent-solute mixture in the large test tube and repeat the determination of the Cooling Curve.

Calculations and Analysis

1. Use the volume of ethanol used and the density provided to calculate the mass of ethanol used and then use the molar mass of ethanol to calculate the moles of ethanol present in your t-butanol solution.

2. Use your moles of ethanol and mass of t-butanol to calculate the molality of ethanol in your t-butanol solution.

3. Use the freezing point determinations from Part I and Part II to calculate the ΔT (the freezing point depression) of t-butanol with an ethanol solute.

4. Navigate to the Program Workspace folder for this lab and put your molality and ΔT data into the provided table.

5. Use your results from calculation 2 and 3 to calculate K_f, the freezing point depression constant, for t-butanol. Compare this to the provided literature value. What can you say about the reliability of our method for determining K_f?

6. Calculate the ΔT of a t-butanol solution with the unknown solute.

7. Use your ΔT from calculation 6, the mass of t-butanol used in Part III, the mass of unknown solute used, and your K_f value to determine the molar mass of your unknown.

References:

ULM, Chem 1010 http://www.ulm.edu/chemistry/courses/manuals/chem1010/experiment_01.pdf

NMT, http://infohost.nmt.edu/~jaltig/FreezingPtDep.pdf