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Experiment 3 – Graphing Freezing Points

Discussion

The freezing and melting of a substance occur at the same temperature, which is measured when the solid and liquid phases are at equilibrium (therefore, the terms "melting point" and "freezing point" can be used interchangeably). Accurate measurement of this temperature is useful for identification purposes, since few chemicals share exactly the same melting point. The unofficial definition of freezing point is the temperature where crystals first start to form. Officially, freezing point is determined by graphing a "cooling curve", which is a plot of temperature versus time as a substance is being cooled. The temperature will decrease at first as the liquid is cooled; then the temperature will remain constant during the phase change from liquid to solid. This "plateau" is the freezing point. When all the liquid has frozen, the temperature will again decrease.

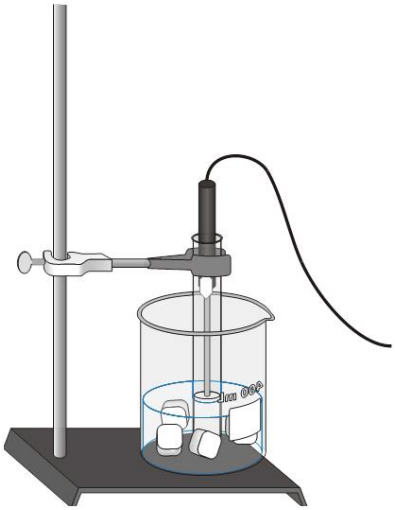
An impurity will disrupt the crystal structure of a substance; therefore, it must be cooled below its normal freezing point to achieve crystallization. This depression of the freezing point has some interesting applications: measuring the amount of impurity, identification by mixing a known with an unknown substance, lowering the freezing point of water using antifreeze, etc.

Occasionally, a substance will become colder than its normal freezing point and still remain a liquid. This unstable condition is called "supercooling". "Shocking" the system by stirring or jarring usually will cause crystallization and the substance will rapidly rise to its normal freezing point.

The goal of this experiment is to determine freezing points by graphing two cooling curves: (1) pure (or glacial) acetic acid with stirring – this usually eliminates supercooling; and (2) pure acetic acid without stirring – supercooling will probably occur.

Procedure

Trial 1: With Stirring



1. Fasten a test tube clamp to a stand. Take a small-sized test tube and a stopper to the fume hood. Add 5 mL of acetic acid to the test tube and close the test tube with the stopper before removing from the fume hood.

2. Place the test tube in the test tube clamp to minimize spillage. Adjust the temperature of the acetic acid to about 25 ^{o}C by warming or cooling the test tube in a beaker of water.

3. Prepare your cooling mixture by placing about 150 mL of ice in a 250 mL beaker; then add water to barely cover the ice.

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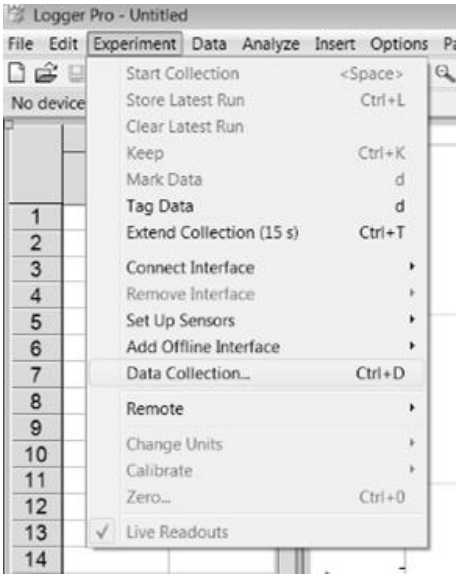
4. Obtain the following equipment: Chemistry Department laptop (with Logger Pro software), Vernier LabQuest Mini, USB cable, thermometer probe. (***Note:*** *an alternate procedure is available that uses LabQuest 2 devices instead of laptop computers.)*

5. Carefully insert the thermometer probe into the stopper & be sure the metal probe is touching the acetic acid.

6. Connect the thermometer probe to the LabQuest Mini (any CH) and then use the USB cable to connect the LabQuest Mini to the laptop.

7. Open the Logger Pro software.

8. Click on the Experiment menu and select Data Collection… Set the Duration: 10 minutes and the Sample Rate: 5 samples / minute and click Done.



9. When you are ready to start the Trial, lower the test tube clamp until the acetic acid in the test tube is below the surface of the ice water and click the green Collect button.

10. Use the thermometer as a gentle stirrer during this trial. Continue recording temperatures for 10 minutes where Logger Pro will stop automatically. If the temperature has stabilized for several minutes (flat part of the graph) and the acetic acid is completely frozen, you may click the red Stop button to end the run.

11. To prepare the acetic acid for your next run, raise the test tube clamp and remove from the ice bath. Replace with a beaker of warm water to melt back to about 25 °C.

12. On the displayed graph, analyze the flat part. Press the mouse button and hold it down as you drag across the flat part to select it. Click Statistics. The mean temperature value for the selected data is listed in the statistics box on the graph. Record this value as the freezing temperature (in the Post Lab Questions). To remove the statistics box, click the upper-left corner of the box.

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Trial 2: Without Stirring

1. Prepare Logger Pro for your next trial: From the Experiment menu, choose Store Latest Run (to save for later comparison). You may either leave Trial 1 on the screen (Trial 2 data will overlay in another color of line) or you may hide the curve by: click the Temperature label of the graph, click More, and uncheck the Run 1 Temperature box. Click OK.

2. Replenish the ice bath if necessary.

3. Repeat the same timing procedure as Trial 1 but do not stir.

4. If the temperature falls to about 4 ^{o}C without forming crystals, you are observing supercooling. If supercooling persists for more than 2 minutes, "shock" the solution by moving the thermometer until crystals form.

5. Continue recording the temperature for the full 10 minutes or until completely frozen. When stopped, be sure to Store Latest Run.

6. On the displayed graph, analyze the flat part using the Statistics function again and record this value as the freezing temperature in the Post Lab.

Completing the Graphs for your Experiment:

1. If not already both displayed, click the Temperature axis label, click More, and check the Run 1 Temperature and Latest Temperature boxes. Click ok.

2. Label both curves by choosing Text Annotation from the Insert menu, and "Stirring" (or "Without Stirring") in the edit box. Then drag each box to a position near its respective curve. Adjust the orientation of the arrowhead by clicking and dragging to the desired position.

3. You will need to show both Trial 1 & Trial 2 graphs to your instructor by bringing the CHEM Dept. laptop to them for credit on this experiment.

<u>Hazardous Waste</u>

Thaw the acetic acid with a beaker of warm water and then remove the temperature probe. Using your wash bottle filled with water, rinse the temperature probe off into a beaker from your drawer. Take the stoppered test tube, your wash bottle, and the beaker to the fume hood. Pour the acetic acid out of the test tube into the provided "Recycled Acetic Acid" bottle. Before you leave the fume hood, use your wash bottle to rinse the acetic acid out of the test tube into your beaker. ***Please do not contaminate the recycled acetic acid with water!*** You can then pour the water that is in your beaker down the sink drain and wash your test tube and beaker before you put them away.

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Post-Lab Questions

Use your graphs to answer the following questions. Note: The accepted freezing point of acetic acid, according to the *CRC Handbook of Chemistry and Physics*, is: 16.6 °C

1. a. What is the experimental freezing point of acetic acid in Trial 1? _____

   b. What is the experimental freezing point of acetic acid in Trial 2? _____

   c. Calculate the percent error in the Trial 1 measurement of the freezing point:

      SHOW CALCULATION:

      _____

2. What is supercooling? Did you observe supercooling in your experiment? Explain.

3. What is the difference between melting and freezing a substance?