Experiment

FREEZING POINT OF GLACIAL ACETIC ACID

The CCLI Initiative

Computers in Chemistry Laboratory Instruction

LEARNING OBJECTIVES

The objectives of this experiment are to . . .

- illustrate how an experiment program is created and executed using the ***MicroLAB*** Interface.
- demonstrate the general features of a cooling curve.
- measure the freezing point of a compound.

BACKGROUND

In this experiment you will measure the freezing point of an organic compound, glacial acetic acid. A sample of the compound, which is a liquid at room temperature, will be cooled and stirred until the temperature is well below it's freezing point. The temperature will be monitored with a temperature probe. A graph of temperature *versus* time (called a cooling curve) will be made which should look like the graph in the Figure 1 below.

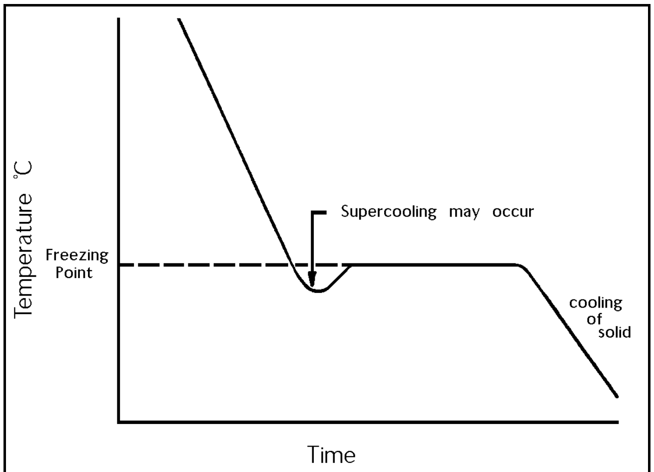

Figure 1. An idealized cooling curve. **NOTE**: The dip in the curve before it plateaus is due to **supercooling**, in which the molecules must be cooled below the normal freezing temperature before they are able to line up and crystalize.

Note that the graph shows the temperature first dropping as the liquid is cooled, then remaining constant when both liquid and solid phases are present. It finally drops again when only solid phase is present. The freezing point is the constant temperature of the solid-liquid mixture. It is not uncommon for liquids to exhibit supercooling as shown in the figure.

Temperature measurement in the experiment is made with a temperature probe. Temperature probes are temperature sensitive circuits which respond with linearly increasing voltage output wit increasing temperature. By calibrating temperature probe at known temperatures, the interface will read temperature directly in °C.

BEFORE PERFORMING THIS EXPERIMENT

...You will need a ***MicroLAB*** program that will measure time and temperature, and, display them in the **Graphing, Spreadsheet** and **Digital Display** views. Choose one of the **temp.vs.time.exp** experiments located in **Time and Temperature** tab for this experiment.

This experiment program is easily created by selecting commands from a menu. The experiment program is stored in a file and later loaded for execution.

SAFETY PRECAUTIONS

Glacial acetic acid is a pure organic compound sold commercially as a 5% solution known as vinegar. It is a weak organic acid. Any skin area contacted with the acid should be washed thoroughly with soap and water. Safety goggles must be worn at all times in the lab.

EXPERIMENTAL PROCEDURE

Loading and running the experiment program

If you do not know how to start the ***microLAB*** program, review the procedures in **MEASUREMENT, The Basic Science**, in the section on **Starting your *MicroLAB* program.**

Calibrating your temperature probe.

Obtain a temperature probe from your instructor. They are fairly sturdy, but should be handled with reasonable care. You will need an ice-water slurry about 50 % ice in one 250 mL beaker and heat about 200 mL of water to approximately 65°C in a second 250 mL beaker. You will need to obtain at least three calibration points for the temperature probe to insure a good calibration. Be sure to connect the temperature probe to the appropriate CAT-5 input jack as indicated under **Input** in the **Data Sources/Variables** view, then click on the temperature probe in the **Data Sources/Variables** view, click on **Edit**, and follow the instructions for **Performing a New Calibration**.

Do not take the temperature probe and thermometer out of the liquid to read them or your calibration will be in error. They must be read while the thermometer bulb is immersed in the liquid. When completed, you will be returned to the Main Screen. As soon as the calibration is completed, *thoroughly dry the temperature probe and stirrer* using a paper towel. Save the ice and hot water baths for later in the experiment.

Testing the program and temperature probe

As soon as you complete the calibration, you should see "live" readings in the **Digital Display** view. Blowing on the temperature probe tip or holding it in a warm hand should result in an observable temperature change on the screen. You may change the sampling frequency to the desired rate by double clicking on the **Experiment Steps** statement **Repeat Every – Seconds**. A new window will open where you can change the numerical value. If all is well, continue to set up the experiment. Otherwise, check with your instructor.

Setting up the apparatus

The apparatus for this experiment is shown in the figure above, originally designed for phenyl salicylate. However, you will be using glacial acetic acid in this experiment. Obtain the test tube assembly from your instructor. Assemble the apparatus as indicated but do not insert the test tube with the glacial acetic acid, temperature probe, and metal stirrer into the nesting test tube. The inner beaker contains an ice-water slurry. The beaker sizes are 400 mL and 600 ml. The double test tubes are to reduce the heat flow so that equilibrium conditions are obtained more readily through-out the glacial acetic acid. The double beakers accomplish the same thing for heat flow to the outside. When the apparatus is assembled and you are ready to begin the experiment, place the test tube containing the glacial acetic acid into the larger test tube.

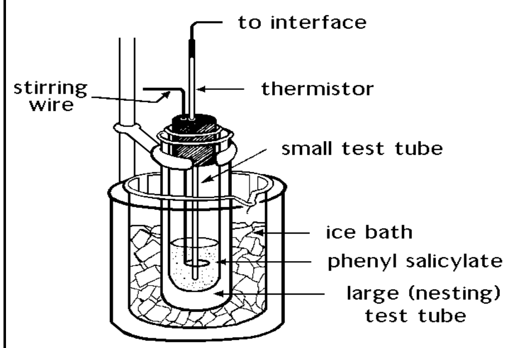

Figure 2. Set up for measuring the freezing point of Acetic acid. **NOTE**: You may be using a temperature probe other than a thermistor, and you will be using glacial Acetic acid rather than phenyl salicylate.

Running the experiment

As soon as you click on the "Start" button, the ***MicroLAB*** interface begins measuring temperature. Note the temperature of the glacial acetic acid displayed on the monitor.

1. For the first experiment, immerse the test tube containing the glacial acetic acid directly into the ice bath, and **DO NOT** stir the liquid. Clamp the test tube in the utility clamp and allow the system to cool undisturbed. When the glacial acetic acid has solidified and a downward cooling trend is observed, **Stop** the experiment. **Save this data file with a unique name you will recognize later, e.g. gl.HOAC.Coolng1.** Remove the sample tube carefully from the ice bath and place it in the hot water bath to melt **all** of the solid.

2. For the second experiment, set up the apparatus exactly as shown in the diagram above. As soon as the tube containing the acetic acid is immersed in the ice mixture, begin stirring constantly and vigorously as the cooling curve data is collected. Continue stirring until solid forms and stirring is no longer possible. At that point, allow the program to continue taking data until the sample completely solidifies, the temperature of the solid drops below the freezing point and you have collected enough of the cooling curve beyond the freezing point to get a good linear regression line.

3. Once the glacial acetic acid begins to freeze, the curve will flatten out, and begin to descend again after all of the liquid has solidified. Observe your curve as it forms to insure that you have a good flat portion of the curve. If there is no or only a very short portion of the flat section, this means that the cooling has taken place too rapidly, your data treatment will not be as satisfactory and you will have to use the cooling or downward portion of the curve. After you have collected a reasonable portion of downward cooling curve, stop the experiment by clicking on the "Stop" button and save your data.

4. Remove the sample tube carefully from the ice bath and place it in the hot water bath to melt the solid. Leave the test tube of glacial acetic acid in the warm water bath until only liquid is present. **Repeat the above procedure at least two (2) more times to be able to do a statistical analysis on the data.**

5. After the final data run (resulting in at least three (3) good data sets), remelt the glacial acetic acid, remove the temperature probe, rinse it with water in the sink, return it to its storage container and to its storage place. Discard the glacial acetic acid into the appropriate waste container. ***Do not attempt to remove the temperature probe and stirrer from the sample tube while it is frozen solid***.

DATA ANALYSIS

1. Reload your data files, one by one, and perform the following analyses:

2. Right click in the graph view and reset the curve so there is no line connecting the data points.

3. You will now proceed in one of two ways:
   a. For each of the data tables with a flat freezing section,
      i. Using the **Domain** button, define the region from where the temperature just flattens out after rising from the supercooling and "click drag" to the end of the flat region. If necessary, repeat this zoom process to remove any points that don't fit into the trend of the desired region.
      ii. Using the **Analysis** button, perform a **First Order (Linear)** curve fit of that data.
      iii. Go to the **Data Sources/Variables** view, scroll down to **Analysis**, and place the cursor on that line.
      iv. You will not be able to read the equation of the first order curve fit.
   b. For each of the data tables without a flat freezing section:
      i. Scroll in the data table to where the temperature begins to drop after peaking, and using the **Domain** button, define the region from this point to the end of the downward sloping region. If necessary, repeat this zoom process to remove any points that don't fit into the trend of the desired region
      ii. Using the **Analysis** button, perform a **First Order (Linear)** curve fit of that data.
      iii. Go to the **Data Sources/Variables** view, scroll down to **Analysis**, and place the cursor on that line.
      iv. You will now be able to read the equation of the first order curve fit.

4. In each case, review your selections to insure that the plotted line actually is the best straight line through the desired portion of the curve. If not, repeat the above sequence of steps to improve it.

5. Use this **First Order (Linear)** equations and the **Interpolation** function under **Analysis**, calculate the temperature for the Y intercept. This is the best estimate of your freezing point for that experimental run. Record this value in your lab book.

6. Repeat steps one through four for each freezing point determination.

Determining the freezing point

If you have taken the data carefully, you should have at least three sets of data that give freezing points very close to each other.

Open a new ***MicroLAB*** program in **Hand Enter** mode, enter the freezing point data for each of your determinations, **Right click** on the freezing point column and select **Column Statistics**, then print out the statistical information for your experiment.

For your report

Construct a data table like this in your lab notes. Write a brief computer processed report which includes a brief discussion of the experiment, the average value of your freezing point, along with the standard deviation for your data, this table, and the answers to the following questions.

TABLE 2: DATA FOR GLACIAL ACETIC ACID

Run #	T1	Row #	T2	Row #	mp T

1. On one of your freezing point graphs, label each section of the graph and in your report discuss what is happening in terms of the kinetic and potential energy of the system at each section of the curve.

2. What is the purpose of the double test tubes and the double beakers?

3. Briefly explain what is happening to cause supercooling.

4. Why is it important to have a flat portion of the curve following the supercooling?

5. In the Handbook of Chemistry and Physics, look up the accepted value for the freezing point of glacial acetic acid, then calculate the percent error for your data. Discuss this in your report.

6. Explain any significant differences in 5 above.