

## Experiment

# FREEZING POINT OF PHENYL SALICYLATE

### The CCLI Initiative

Computers in Chemistry Laboratory Instruction

### LEARNING OBJECTIVES

The objectives of this experiment are to . . .

1. demonstrate the general features of a cooling curve.
2. measure the freezing point of a compound.

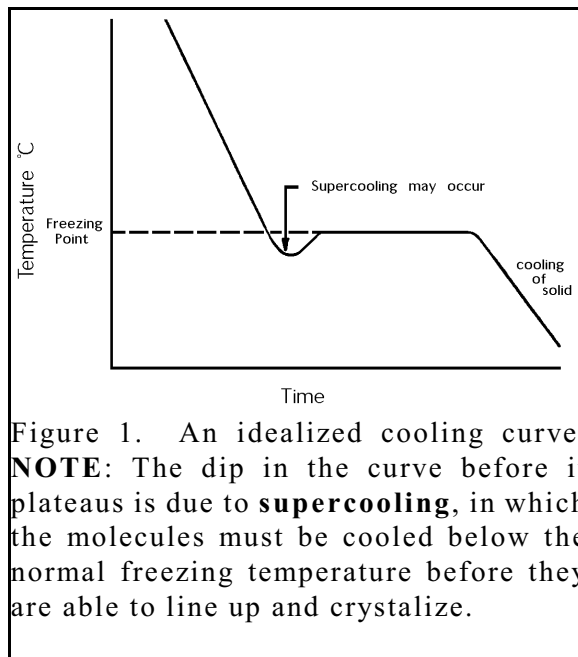
### BACKGROUND

In this experiment you will measure the freezing point of an organic compound, phenyl salicylate. A sample of the compound, which is a solid at room temperature, will be warmed until it melts and the temperature of the liquid is well above its freezing point. The sample then will be cooled and stirred, and its temperature 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 below.

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 integrated circuit chips designed to produce 18 mV per degree change in temperature, and has a linear response over the entire range from -18 °C to +150 °C. By calibrating at known temperatures, the interface will read temperature directly in °C.

The program to run the MicroLAB interface is a simple temperature/time relationship in which the **Temperature** probe is selected and calibrated, and **Time** is selected to start when the **Start** button is clicked. The variables have been assigned to their respective places in the **Graphing, Spreadsheet** and **Digital Display** views. Choose the **freezing point** experiment located in **Time and Temperature** tab for this experiment.



## SAFETY PRECAUTIONS

Phenyl salicylate is used topically as an analgesic and antipyretic but is also listed as an irritant. Any contacted skin should be washed with soap and water. Safety goggles must be worn at all times in the lab.

## EXPERIMENTAL PROCEDURE

### Preparing the experiment program

Turn on the computer and monitor. Turn on the power to the interface box by depressing the switch on its right back corner.

Open *MicroLAB Experiment* by double clicking in its icon on the desk top. If you are unfamiliar with how to do this, look it up in the **Measurement Manual**.

### Temperature probe calibration

Obtain a temperature probe from your instructor. You will need a cold water and a hot water temperature source for calibrating the temperature probe. Prepare an ice-water slurry in one 250 mL beaker and heat about 200 mL of water to approximately 75°C in a second 250 mL beaker. Connect the temperature probe to the **CAT-5A** input on the interface. Be sure that the interface power is on (lower right back button).

Select the temperature probe by clicking on it, then click on **Edit**. The **Calibration** window will open. Calibrate the probe at a minimum of three points spanning the temperature range of your experiment by mixing in various proportions the ice water and hot water. You will be asked to read and enter the temperature from the thermometer. Be sure to estimate all temperatures to the nearest 0.1°C. When completed, you should be returned to the main menu. As soon as the calibration is completed, *thoroughly dry the temperature probe* using a paper towel. Save the heated water for later in the experiment. As soon as your probe is calibrated, you should see a live reading in the **Digital Display** view.

### Setting up the apparatus

The apparatus for this experiment is shown in Figure 2. Assemble the apparatus as indicated but do not insert the test tube with the phenyl salicylate, temperature probe, and metal stirrer into the nesting tube. The inner beaker contains an ice-water slurry. The beaker sizes are 400 mL and 600 mL. When the apparatus is assembled, place the test tube containing the phenyl salicylate into the 250 mL beaker of 65°C water. When the solid begins to melt, use the stirrer to agitate the mixture gently. When all of the solid is melted, continue warming and stirring the liquid for about five minutes. Toward the end of this warming period, begin the preparations described in the following section.

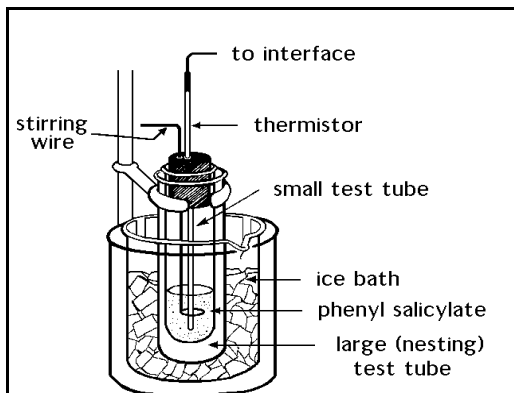


Figure 2. Set up for measuring the freezing point of phenyl salicylate.  
NOTE: you may be using a temperature probe other than a temperature probe.

## Running the experiment

As soon as you click on the “Start” button, the *MicroLAB* interface begins measuring temperature. Note the temperature of the phenyl salicylate displayed on the monitor.

1. For the first experiment, immerse the test tube containing the phenyl salicylate 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 phenyl salicylate 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.PhSa.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 phenyl salicylate 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 phenyl salicylate 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 phenyl salicylate 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 phenyl salicylate, remove the temperature probe, rinse it with water in the sink, return it to its storage container and to its storage place. Allow the phenyl salicylate to cool, replace the stopper and return to the reagent desk. *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 now 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 phenyl salicylate

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 phenyl salicylate, then calculate the percent error for your data. Discuss this in your report.
6. Explain any significant differences in 5 above.