Experiment 1

Determining Melting Temperature

The melting temperature of a compound is the temperature at which it changes from a solid to a liquid. This is a physical property often used to help identify compounds or to check the purity of a compound. The melting temperature is related to the amount of kinetic energy that one adds to a solid substance to overcome the intermolecular attractions that maintain its solid state under given conditions.

It is very difficult, however, to find an exact melting point. Because it is a thermodynamic process, when a substance begins to melt, a dynamic equilibrium is established within which the substance exists in both solid and liquid form. Because the energy transferred to this system is not used entirely to convert the solid to a liquid, a single temperature value is commonly not reported, but rather a temperature range.

Thus, melting temperatures are usually reported as values with a range of 2–3°C. Melting temperature is not a unique physical property of a substance, but it does help you understand more about the substance. It can also help determine the purity of a substance that you have synthesized.

You will use a melt station to determine the melting temperature of a solid substance. Your sample will be one of several possible pure compounds. Your first trial will help you narrow your possibilities. On subsequent trials you will be able to accurately determine the melting temperature of your sample, thus identifying the compound.

OBJECTIVES

- Prepare a solid substance for measuring melting temperature.
- Measure the temperature of a solid substance as it warms to melting.
- Analyze the temperature *vs*. time graphs to determine the rate of heating and the melting temperature of a sample of a solid organic compound.
- Identify the solid from a list of possible pure compounds.

MATERIALS

Chromebook, computer, **or** mobile device
Graphical Analysis app
Melt Station[1]
glass capillary tubes–one closed end
sample of an organic solid
tissues (preferably lint-free)
mortar and pestle (optional)

[1]If using a Melt Station (order code MLT-BTA), an interface such as LabQuest Mini is also required. If using a Go Direct Melt Station (GDX-MLT), an interface is not needed.

PROCEDURE

1. Obtain and wear goggles.

2. Check the control dial on the melt station to confirm that it is in the cooling fan position. Connect the melt station power supply to a powered electrical outlet.

3. Launch Graphical Analysis. Connect the melt station to your Chromebook, computer, or mobile device. Use an interface if necessary.

4. Obtain a small amount of a solid organic compound. The solid should be in powder form. If it is not, use a mortar and pestle to carefully grind the solid to a powder.

5. Prepare a sample for melting.
   a. Pack a capillary tube 3–4 mm (~1/8 inch) deep with your sample by inserting the open end into a small pile of the solid. A small amount of the solid will be pushed up into the tube.
   b. Wipe off any loose solid that is on the outside of the capillary tube.
   c. Tap the closed end of the capillary tube on the desktop to compress the sample into the closed end.
   d. (optional) To further pack down the sample in the tube, drop the capillary tube (closed end down) down a section of glass tubing that has been set up for this purpose.
   e. Carefully insert the capillary tube of solid into one of the three slots in the heating block of the melt station. You may rotate the melt station toward you slightly for a better look at the heating block.
   f. Rotate the melt station up or down slightly to get the best view of the solid sample through the viewing lens.

6. In the first trial, you will want to observe the melting process and make a rough estimate of the melting temperature of your sample. Do not worry if the heating rate is a bit too rapid, and the sample melts too quickly.
   a. Start data collection.
   b. On the melt station, turn the control knob to a setting of 180°C. The red light will turn on indicating active heating.
   c. Carefully observe your sample. When the solid begins to melt, select Graph Tools,  , and then Add Annotation. Enter the temperature. When the entire solid has completely melted, add a second annotation. The two values marked on your graph describe the estimated melting temperature range of your substance.
   d. If the solid does not melt by the time the temperature gets to 150°C, turn the control knob to the 220°C setting. Continue observing your sample, and if the sample begins to melt, mark the temperatures on the graph as previously described.
   e. If the sample has not melted by the time the temperature gets to 190°C, turn the knob to the Rapid Heat setting. When the sample finally begins to melt, mark the graph as previously indicated.
   f. When you have determined the approximate melting temperature range for the sample, stop data collection. Discard the capillary tube and sample as directed by your instructor.
   g. On the melt station, turn the control knob to the cooling fan position to get ready for the next trial. The blue light will turn on indicating that the fan is cooling the melt station.

7. Now that you have a rough idea of the melting temperature, a more accurate determination can be made. Prepare a new sample in a capillary tube, as described in Step 5, to determine the melting temperature.
   a. Start data collection. **Note**: The previous data set is automatically stored.
   b. On the melt station, turn the control knob to the Rapid Heat setting.
   c. Carefully observe the temperature *vs*. time graph. When the temperature is within approximately 10°C of the lowest possible melting temperature of your sample, turn the control knob to a temperature setting corresponding to your expected melting temperature.
   d. Carefully observe your sample. When the solid begins to melt, add an annotation to the graph and record the temperature. When the entire solid has completely melted, add an annotation and record the second temperature. The two values marked on your graph describe the estimated melting temperature range of your substance. When you are finished with this step, stop data collection.
   e. Discard the capillary tube and sample as directed by your instructor.
   f. On the melt station, turn the control knob to the cooling fan position to get ready for the next trial.
8. At the end of the experiment, record the melting temperature range and turn the control knob on the melt station to Off.
9. Complete the Data Analysis section before exiting the data-collection program. Print a copy of your graph and/or save your data, as directed by your instructor.

DATA ANALYSIS

1. What is the code number of your solid sample? What was the melting temperature range of your sample?
2. Use the list of possible compounds, provided by your instructor, to identify your sample.
3. A heating rate of 1–2°C/min is considered ideal for the most accurate determination of the melting temperature of a solid substance. Use the Tangent tool to determine the approximate heating rate during the time that your sample was melting.