

## 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 not possible, however, to find an exact melting *point*. Being 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 cannot be given for this process, 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 Vernier 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

In this experiment, you will

- 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

LabQuest or computer interface	glass capillary tubes – one closed end
LabQuest App or Logger <i>Pro</i>	tissues (preferably lint-free)
Vernier Melt Station	sample of an organic solid
	mortar and pestle (optional)

### PROCEDURE

1. Obtain and wear goggles.
2. Check the control dial on the Melt Station to confirm that it is in the Off position. Connect the Melt Station power supply to a powered electrical outlet.
3. Connect the Melt Station sensor cable to a LabQuest or to a computer interface.
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 desk top to compress the sample into the closed end.

- d. To further pack down the sample in the tube, tap the closed end of the tube on the lab bench until all of the material is at the bottom.
- e. Carefully insert the capillary tube of solid into one of the three slots in the heating block of the Melt Station (make sure that there are no broken tubes in the slot you choose). 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. Start the data-collection program, and then choose New from the File menu. You are now set up to take melting temperature data for up to 20 minutes.

7. In the first trial, you will want to observe the melting process and make a *rough estimate* of the melting temperature of your sample. Don't worry if the heating rate is a bit too rapid, and the sample melts too quickly. To do this:

- 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. If the solid begins to melt, click Mark to mark the temperature on your graph (or press the **D** key on the computer or the OK button on LabQuest). When the entire solid has completely melted, click Mark again. 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. If your sample does not melt by 250°C, make a note of this and stop heating.
- f. When you have determined the approximate melting temperature range for the sample, stop data collection. Store the run by tapping the File Cabinet icon in LabQuest, or choosing Store Latest Run from the Experiment menu in Logger *Pro*. Discard the capillary tube and sample as directed by your instructor.
- g. On the Melt Station, turn the control knob to the Fan/Cooling setting to get ready for the next trial. The blue light will turn on indicating that the fan is cooling the Melt Station.

8. 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.
- 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, click Mark to mark the temperature on your graph. When the entire solid has completely melted, click Mark again. 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. Store the run.
- f. Discard the capillary tube and sample as directed by your instructor.

g. On the Melt Station, turn the control knob to the Fan/Cooling setting to get ready for the next trial.

9. At the end of the experiment, record the melting temperature range and turn the control knob on the Melt Station to Off.

10. Complete the Data Analysis section before exiting Logger *Pro* or the LabQuest App. Print a copy of your graph and/or save your data, as directed by your instructor.

### DATA ANALYSIS

- Base on the list of possible compounds, provided by your instructor, what identities do you propose for the 3 samples you tested?
- For each sample use your melting point and solubility data to support your predictions for your 3 compounds and describe the type of bonding and intermolecular forces present (if any) in each compound.

Structure	Compound	Melting Temperature (°C)
<p align="center"><b>Lauric Acid</b></p>	Lauric acid	42 - 44
<p>Palmitic acid C<sub>16</sub>H<sub>32</sub>O<sub>2</sub></p>	Palmitic acid	61–63
<p align="center">benzoic acid</p>	Benzoic acid	122–124
<p align="center"><b>Maleic Acid</b></p>	Maleic acid	138–141
<p align="center">Citric acid, a hydroxy polycarboxylic acid</p>	Citric acid	152 - 154
	Sodium oxalate	259 - 261
	Sodium sulfate	883 - 885

**CLASS SET - DO NOT WRITE ON THIS COPY!**