[MUSIC PLAYING]

PROFESSOR: Throughout your laboratory experiences, you will frequently need to assess the purity or identity of a crystalline solid. Both of these goals can be accomplished by determining a compound's melting point. When a compound is in a crystalline solid state, the intermolecular forces that are holding the molecules together greatly outweigh the kinetic energy that is trying to pull them apart. Therefore the molecules appear to vibrate in a fixed location.

If energy is introduced into the system in the form of heat, the kinetic energy of the molecules and thus the temperature of the system is increased. At a certain point, the molecular kinetic energy is high enough to overcome the attractive forces that are holding the molecules together. As a result, the crystal lattice breaks down and the molecules enter a liquid state.

The temperature at which the solid and liquid phases of a compound are in equilibrium at a certain pressure is called the melting point. In reality, a solid melts over a temperature range instead of at a specific temperature. Because this range is a physical characteristic, it is reproducible for a pure compound. For example, N-phenylacetamide melts between 114.2 and 114.9 degrees Celsius. Therefore the term melting point really implies a melting range.

Although a variety of melting point apparatuses are commercially available, this video will demonstrate the determination of melting point by using the Mel-Temp. The Mel-Temp apparatus consists of an on/off switch, a sample holder, a light source that illuminates the sample, an eye piece that magnifies the sample, a digital thermometer, which indicates the temperature of the system, and a voltage control.

The voltage control dictates the rate of heating with a higher setting corresponding to a faster rate. The voltage control does not control the temperature of the system. Use caution when touching the Mel-Temp and always assume that the aperture is hot, especially the part that is labeled "caution. Unit hot."

The first step in determining a melting point is the preparation of the crystalline solid. To prepare the sample, you will need a spatula, a watch glass, a mortar and pestle, capillary tubes that are sealed at one end, and a long piece of glass tubing, such as a long stem funnel. Before proceeding, ensure that the crystalline solid is clean and dry. If the solid is wet, you can try to pat it dry with filter paper. Do not place the sample in the oven where it may melt or decompose.

If your sample consists of large crystals, you must first crush them into smaller crystals with a mortar and pestle. Large crystals cannot pack together well, which creates air pockets. If air pockets are present in your sample, then a time lag for heat transfer will exist, causing the crystals to heat unevenly. If you do not have a mortar and pestle, you can use the flat end of a spatula to grind the sample instead.

After crushing the crystals, place them on a watch glass. Turned the capillary tube upside down and thrust the end over the crystals. A small amount of crystals should be trapped inside the capillary. Invert the tube to its correct orientation and pack the crystals down to the bottom by gently stroking the tube or by tapping it on your benchtop.

If the crystals fail to fall to the bottom of the capillary, then drop the capillary through a length of glass tubing. A long stem funnel works well for this application. You may have to repeat this action several times to ensure that the crystals are effectively packed down.

Loose material contains air pockets which cause the sample to heat unevenly. Ideally, the smallest manageable amount of material should be used, which correlates to a depth of approximately 1 millimeter of packed material. If the depth exceeds 2 millimeters, inaccuracies may result due to uneven heating.

Prepare four capillary tubes of your sample. One filled capillary tube will be used to determine a crude melting point. And the other three will be used to determine your actual melting point in triplicate. If you do not know the expected melting point of your compound, you must first determine a crude melting point.

Finding the approximate melting point of a sample will save a lot of time when performing the actual measurement. To determine the crude melting point, set the voltage on the melt temp to zero and wait until the aperture has cooled to room temperature. Insert a sample. Increase the temperature at a rate of 10 to 20 degrees Celsius per minute and record a rough temperature when the sample melts.

To determine the melting point of a crystalline solid, set the voltage on the Mel-Temp to zero, turn on the power and the digital thermometer, and then insert a filled capillary tube into the sample holder. If the temperature is lower than 20 degrees Celsius below the expected melting point, then dial the voltage to a setting that enables the temperature to increase at a rate of 10 to 20 degrees Celsius per minute.

After the temperature is within 20 degrees Celsius of the expected melting point, dial down the voltage so that the temperature increases at a rate of 1 to 2 degrees Celsius per minute. Watch the sample through the eyepiece and record the temperature when the first crystal starts to melt, which means when the first droplet of liquid appears. Finally, record the temperature when the last crystal disappears. Turn down the voltage, remove the melted sample, and throw it into the proper waste receptacle.

Allow the Mel-Temp apparatus to cool to 20 degrees below the melting point of the compound and repeat the process two more times with the remaining samples to ensure reproducibility. Never remelt a compound because it may have undergone a chemical change while heating such as oxidation, decomposition, or rearrangement.

The point where the first crystal starts to melt can seem ambiguous to someone taking a melting point for the first time. As stated earlier, this is the point where the first droplet of liquid forms. It is not when the material begins to settle, soften, or shrink. Only the appearance of a liquid droplet indicates the beginning of melting.

One common use for melting point is to assess the purity of a known compound. If an impurity is present in a sample, it will interrupt the crystal lattice and weaken the forces of attraction that are holding the molecules together. Therefore the sample will melt at a temperature that is lower than expected. The sample will also melt over a wide temperature range. As a general guideline, if a compound melts over a range greater than 2 to 3 degrees Celsius, it is most likely impure.

Another common application of melting point is the identification of an unknown crystalline solid. To identify a solid, simply compare the melting point of the unknown with the melting points of known compounds that are listed in a reference book. Typically, just the upper limit is reported in these reference books. When combined with other spectroscopic data, you may be able to identify your compound.

To confirm that your compound is indeed what you think, prepare three samples containing mixtures of your unknown compound and the proposed compound. 20 to 80, 50/50, and 80 to 20 are good ratios to start with. Determine the melting point of each mixture. If all the mixtures melt at the same temperature and within the same range, you may have correctly identified your compound. If they do not, then you definitely have not identified your compound.

There are several ways to obtain inaccurate melting point readings. The most common mistake is increasing the temperature too rapidly. In this situation, the capillary will not have enough time to equilibrate with the heated block. As a result, the melting range will appear narrower than it actually is and the melting temperature will appear higher.

A sample that disappears upon heating indicates that it is subliming instead of melting. To remedy this problem, seal the open end of the field capillary with a Bunsen burner before placing it in the sample holder. A sample that changes color upon heating is probably undergoing decomposition. Decomposition must be reported along with the melting point of the compound.

If the melting point is still incorrect after troubleshooting or if you need to confirm the accuracy of your result, you must calibrate the Mel-Temp apparatus. To do this, find pure compounds with known but dissimilar melting points. This video will use naphthalene, N-phenylacetamide, salicylic acid, and succinic acid. Determine the melting point of each pure compound and compare the value you received with what is expected. Plot the actual melting point of each compound against its observed melting point. Use the resulting calibration curve to determine the expected melting point of your compound.

Determining the correct melting point of a crystalline solid involves good technique in both sample preparation and in taking the melting point. To ensure accurate results, use a minimal amount of solid, pack the crystals tightly together, and increase the temperature slowly. Determining the melting point of a crystalline solid is the best way to assess its purity or establish its identity.

[MUSIC PLAYING]