MELTING POINT DETERMINATION

The physical properties of a substance, such as density, color, odor, crystalline form, melting point, and boiling point, are used to characterize a substance. These are particularly useful for identifying organic compounds. For a solid substance, melting point is one of the most important characteristics.

Melting points can be readily determined using electric melting point apparatus such as that shown in Figure MP-1. Melting point apparatus can be equipped with a thermometer for reading temperature, or may contain a digital thermometer or be interfaced with a computer.

A small sample of the compound is placed in a capillary tube (see directions for loading sample into capillary tube) which is then inserted into the heating block of the melting point apparatus, which is located directly in front of the thermometer.

The melting point apparatus is turned on and the temperature control knob is adjusted to give a heating rate of approximately 5 to 10°C per minute.

There is a light bulb in the apparatus that illuminates the sample so the melting point can easily be observed.

Most pure substances will melt over a 1°C range.

If the approximate melting point of the sample is known, the temperature can be rapidly increased to about 20°C below the melting point, then slowly increased to the melting point temperature. Note that there is a time lag on the temperature reading for rapid increases in temperature. After lowering the heating rate, the temperature reading may continue to 5 to 10°C.

After the melting point is determined, the temperature control knob is returned to the zero position, the capillary tube containing the sample is removed and discarded in the proper waste container.

Figure MP-1. An electric melting point apparatus

Loading a sample into a capillary tube

Obtain a clean, new capillary tube.

Place a small amount of powdered sample onto a piece of weighing paper or a watch glass.

Push the open end of the capillary tube into the sample. (See Figure MP-2) Repeat this procedure until the plug of sample in the capillary tube is approximately 2 mm long.

Pack the sample into the bottom of the capillary tube by tapping the closed end on the bench top. Alternatively, the sample can be packed into the bottom of the tube by dropping it, closed end down, through a piece of glass tubing onto the bench top. (See Figure MP-3).

Figure MP-2. Loading the sample into the capillary tube by pressing the open end into the powdered compound.

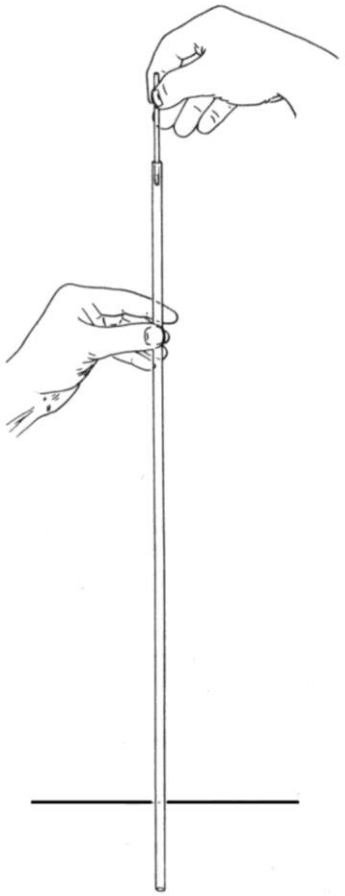

Figure MP-3. Packing the sample into the capillary tube by dropping it through a piece of glass tubing.