

Experiment name / Determination of melting point

PURPOSE

- a) Determine the purity of a substance using melting point as physical property
- b) Identify an unknown compound using its melting point
- c) Identify an unknown compound using mixture melting point
- d) Learn how to obtain an accurate melting point using a Mel-Temp apparatus

Reasons for determining melting points:

1. The melting point indicates the level of purity of a sample. An impure compound melts over a wider range of temperatures, usually greater than 2 degrees.
2. The melting point helps to identify unknown samples, narrowing the number of possibilities, because a pure solid melts reproducibly over a narrow range of temperatures.
3. The melting point helps to characterize new compounds.

Melting point Definition

When a solid substance is heated, typically it will melt; that is to say, at some temperature the solid will begin to liquefy and by some slightly higher temperature all of the solid will have become liquid. The melting point (actually melting point range) of a compound is then defined as the temperature at which an observer can first see liquid forming from the solid to the temperature where the last particle of solid has become liquid. For example, the melting point of pure sucrose (table sugar) is 1850-1860C. This means that as a small sample of sucrose is slowly heated some of the crystals begin to liquefy at 1850 and all of the crystals have become liquid by 1860C.

Experimental Procedures

A. Preparing the Sample

Place a pea-size mound of one of the listed compounds on a piece of paper and grind it to a fine powder using a spatula. Use the spatula to push a small amount of the solid into the open end of a capillary tube. Then drop the capillary down several times to fill and pack the sample well to cause the solid to fall to the bottom. Repeat this

step until you have accumulated a sample 2-4 mm (≈ 0.5 cm) high in the bottom of the tube (see Fig. 1).

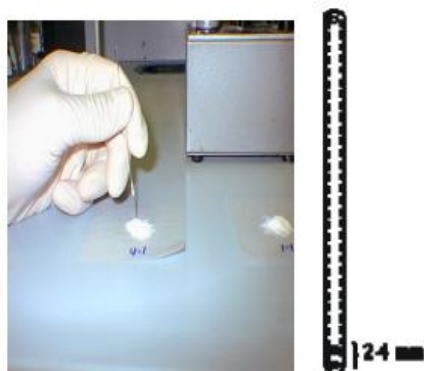


Fig. 1 – Preparing the sample

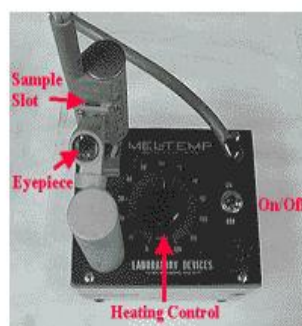
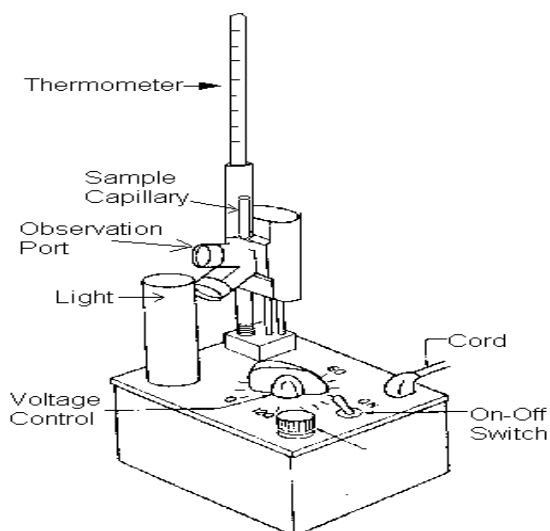


Fig. 2 - Taking a Melting Point

B. Taking a Melting Point

To record the melting point range of a compound fill two capillary tubes each to a depth of 2-4 millimeters with the compound of interest. First, get a rough idea of the approximate melting point range (see Fig. 2). Place one of the tubes in the Mel-Tem apparatus and raise the temperature a relatively fast rate, about $10\text{ }^{\circ}\text{C}/\text{minute}$. Record the range of temperature from the first visible evidence of liquid (the sample appears moist, or a tiny drop of liquid is observed) to the complete liquefaction of the sample. Note the temperature at which the compound first begins to melt. Allow the melting point apparatus to cool to about $20\text{ }^{\circ}\text{C}$ below that temperature, and then insert the second tube. Raise the temperature more slowly this time, at the rate of about $2\text{ }^{\circ}\text{C}/\text{min}$. Note the temperatures at which: I. the first crystals melt, and; II. at which the sample has completely melted. This is the melting point range. For example, a mp range of $164\text{-}168\text{ }^{\circ}\text{C}$ average of $166\text{ }^{\circ}\text{C}$ indicates the sample softened or began to melt at $164\text{ }^{\circ}\text{C}$ and that transition to a liquid was complete at $168\text{ }^{\circ}\text{C}$



Determination of Melting Points

DATA SHEET

Name _____

Name _____

Period _____ Class _____

Date _____

DETERMINATION OF MELTING POINTS

DATA TABLES

Known Compound	Melting Range (°C)

Questions

- 1- If you were given a known substance for the melting point determination how could you prove it to be the specific compound and not another compound with the same melting point ?
- 2- What is the definition of melting point ?
- 3- What is the purpose of determining the melting point ?
- 4- What are the reasons for determining melting point ?