

February 20, 2018

## **Melting Point Apparatus Procedures**

The Capillary Melting Point Apparatus—located in W5007 SERC—consists of a capillary melting point apparatus Mel-Temp Thermo Scientific and a digital thermometer Fluke 51-II. It provides an efficient, fast and accurate way to measure melting points of compounds of up to 500 °C. Mel-Temp has castings that conduct and radiate heat uniformly to capillaries and thermometer. The samples are illuminated and clearly visible through the precision 6-power lens.

### **Using the Capillary Melting Point Apparatus**

#### *Sample Preparation*

1. Grind solid samples into fine powder before loading them into the capillary tube.
2. Press the open end of the capillary tube into the powder.
3. Pack the sample by inverting the capillary tube and tapping it until the sample is at the closed end of the capillary tube.
4. Repeat the cycle of loading until 1–2 mm of the sample is in the capillary tube. Do not pack the capillary with too much sample because it will cause a broad melting point range.

NOTE: For samples that sublime before melting, sealing the open end after packing is necessary.

#### *Measuring the Melting Point*

1. Turn the power switch ON of both the melting point apparatus and the digital thermometer.
2. Insert the capillary tube with the sample into the capillary holder in the melting point apparatus. The melting point apparatus can hold up to three capillary tubes; however, the leftmost slot is already occupied by the digital thermometer probe.
3. Set the power level by turning the control knob clockwise to obtain the desired heating rate at the anticipated melting range. A good practice is to heat the sample relatively quickly (10 °C/minute), until you reach the temperature ~20 °C lower than the anticipated melting point. Once at that temperature, the heating rate should be lowered to 2 °C/minute and the melting process carefully observed.
4. Observe the sample with the eye about 6" (15 cm) from the lens.
5. Take note of the temperature that the substance has started to melt (first drop of liquid) and the temperature at which it has completely melted (last crystal melts, the entire sample is a liquid). By pressing the °C/°F/K button, you can view the temperature in different temperature scales.

#### *Cleanup*

1. Turn the thermometer and the melting point apparatus OFF. Throw away your capillary tube(s)—you should never reuse these samples, not even to obtain a second reading of the melting point.