

## Organic Chemistry 211 Laboratory **Melting Point Measurement**

### Theory:

- When a contaminant (impurity) is present, the melting point of the impure sample is ALWAYS lower than that of the pure (colligative property: melting point depression), and the range is always broader (for the substances that have an melting point range instead of a sharp point).
- Most of the organic compounds have a melting range, rather than a point. A sharp melting point is generally accepted to have a range of 1 to 2 °C.
- To determine a melting point range, a small sample of the solid in close contact with a thermometer is heated in a metal heating block so that the temperature rises at a slow, controlled rate. The rate of heating would be controlled so that the melting range is as narrow as possible.

For example, if the pure has a melting point range of 180-182 °C. An impure will never be above 182 °C. It is always lower and has broader range such as 176-181 °C.

### Technique:

- Watch the video: How to pack a sample in an mp capillary tube that is sealed at one end.
  - Fill capillary tube with approximately 5 mm of sample and drop tube through long glass tubing to compact the sample.
  - Repeat dropping / packing several times to pack the sample uniformly, and note the number of times. All subsequent packing should be done in exactly the same manner.
- About the Mel-Temp (the melting point measuring apparatus)
  - Insert capillary tube into the Mel-Temp apparatus.
  - The variable voltage control is used to regulate the rate of heating. Do not turn up the voltage selector knob at the beginning. You should increase the voltage gradually; monitor the increase in the temp. If the temp increase slows down (e.g. wait until temperature levels off) and the sample does not melt, then increase the voltage one notch at a time.
  - To pre-measure a rough melting point range, apply heat at a rate that will rise 10-20 degrees per minute watching for any sign of melting or “sweating”. Record temperature when melting first occurs and when sample has completely melted.
  - Repeat the procedure. Heat rapidly to approximately 15 degrees below the first temperature you previously recorded, and then slow to 1-2 °C per minute to allow time for a better equilibrium to be established among sample, heating source, and thermometer.
- About the melting process:

Most of the organic compounds have a RANGE of melting.

  - First you will see “Sweating” of the sample. Record the first instant of observing this “Sweating” as the low end of the range.
  - Then the sweating will increase and at some point you will see a part melted-part solid sample, as the melting process continues.
  - Record the temperature at which the entire sample turns into liquid as the upper boundary for the range.

Duplicate results should agree within 1 to 2 °C. At least run duplicate samples that agree.

(cont')

**Suggested Data Table Format:**

	Run 1	Run 2
Initial Temp. ( $T_i$ )		
Final Temp. ( $T_f$ )		
Average		