

Chemistry 143: Melting Points

1. Check-in

- *Read pages 1-41*

2. Melting points

- *Read pages 150-158*

Possible Unknowns

benzil

benzoic acid

1,3-dinitrobenzene

naphthalene

4-phenylphenol

4-chlorobenzophenone

These chemicals should be recorded in a table of physical constants, together with their melting points and molecular weights. You do not have to draw the structures in the equation portion or your pre-lab.

Obtain a small sample (~0.05 g) of two unknowns. Be sure to record the number or letter code of your unknowns.

Crush each sample into a fine powder on a watch glass. Push the open end of a melting point capillary tube into the pile of powder in order to get a small amount into the mouth of the capillary tube. Tap the tube a few times on the lab bench (closed end down) to get the solid to the bottom of the tube. The depth of the solid in the bottom of the tube should be 1-3 mm.

Place the capillary tubes into the melting point apparatus and begin heating the samples **slowly!** (why?) Observe the solid in each capillary tube while heating. For each capillary tube, record the temperatures at which (a) the first drops of liquid appear, and (b) the entire sample is converted to liquid. The compound may shrink away from the insides of the capillary as the melting point is approached, but this is not the start of the true melting point unless liquid droplets begin to appear. The temperatures (a) and (b) make up the melting point range (for example, 92-94 °C). This range should be reported as the melting point (mp) of your sample.

Repeat the mp determination in order to average the two measurements (why?).

Compare your melting points with the melting points of the possible unknowns. Melting point data of the possible unknowns can be found in the Aldrich catalog in the lab. Identify your unknowns.

3. Mixture Melting points

- *Read pages 154-155*

Check your preliminary identification using mixture melting points. For one of your unknowns, choose the two known compounds that have a mp closest to your observed data. Use these knowns to prepare mixtures of each with your unknown (you should have two mixtures). Crush and mix the two pairs of solids well.

Determine the mp of each mixture as described above.

Compare each mixture mp with the mp of the pure unknown.