CHEM 267. Week 1. Melting Points. (revised 7/10).

For background information on determining MPs, do the assigned reading. You will use a Mel-Temp device. It is not necessary to read about the other MP devices. View the MP movie on the course website.

The purpose of the Melting Point experiment is to learn to determine melting points (MPs) accurately. This is an important technique that will be used in many of the experiments in the Organic Lab. Always record a MP range - the temperature at which the first drop of liquid appears, to the temperature at which all sample has melted. Note that upon heating, crystals may expand or contract, resulting in movement of the crystals. This should not be mistaken for the beginning of the MP range. Wait for the first tiny drop of liquid to appear on the crystals. A wide MP range indicates that the sample is impure. A MP determination provides two important pieces of information: a physical constant, which can help to identify a compound, and a measure of the purity of a compound. Note too that an impure sample melts at a temperature lower than that of the pure compound. An impure sample therefore melts over a wide range and at a temperature lower than that of the pure material. For your purposes, a compound with a MP range of 2° C or less will be considered to be sufficiently pure.

Apparatus: The height of sample in the capillary tube should be about 2-3 mm (the thickness of two 25 cent coins is about 3 mm). Three capillaries can be heated at the same time, although when learning to determine MPs it is advisable to do one at a time. Heating is accomplished by adjusting the voltage control - note that the settings are not temperature settings. They are given either in volts or in units of 0 to 10, where 0 means zero voltage and 10 means full voltage. A higher setting results in a faster rate of heating. Start at a low setting, observe the temperature for several minutes to get a feel for use of the apparatus, then adjust the setting so that the temperature rises at a reasonably rapid rate to about 10° below the expected MP range. (reasonable rate = rapidly enough so that time is not wasted but slowly enough so that the temperature does not increase past the expected MP range) The setting is then adjusted to achieve the desired temperature rise of 1°/minute near the MP of the sample. Note the settings and temperatures for future reference. A lag time exists between changing the voltage and observing a temperature change on the thermometer. When a voltage change is made therefore, allow time for the temperature to equilibrate. Note that the digital thermometers used here are accurate only to about ± 2° C. **Caution: the heating block in which the sample and thermometer reside gets very hot. WHEN FINISHED USING A MEL-TEMP, ALWAYS TURN THE VOLTAGE TO ZERO, TURN THE SWITCH TO OFF, AND TURN THE DIGITAL THERMOMETER OFF.**

(1.) Determine the melting points (MPs) of naphthalene, urea, and sulfanilamide. Note that the MPs of samples used in the lab may differ slightly from those shown in Williamson. Therefore, use the MP values given in the table below. Samples will be dispensed in labeled crystal dishes on the side bench. Do not move the dishes. Fill the capillaries right there. Determine the MPs of these known compounds in order of increasing MP. This will obviate the need to allow the apparatus to cool between determinations. The values that you find should agree well with those listed and the range should be narrow. If you find a wide range (> about 2°) or a value different from that expected (within ± 2° - remember that the thermometers used here are good only to about ± 2°C), do a second determination. It is important to use a small powdered sample and to raise the temperature very slowly (about 1°C/minute) when you are near the MP of the sample. If a MP must be redetermined, use a fresh sample - once a sample has melted, you must assume that it may have decomposed, and therefore become contaminated with impurities. (What effect would this have on the observed MP range?) Never dispose of used capillaries or any glass in the regular trash. The person who empties the trash

may become injured. Place the used capillaries in the dishes provided. ALWAYS turn the device and digital thermometer off when finished.

(2.) Determine the MP of an unknown compound (List of possible compounds below). To save time, when you determine the MP of an unknown sample, first find the approximate MP by raising the temperature much more rapidly than you normally would, and then redo it with a fresh sample, raising the temperature at about 1°C/minute when you get near the MP. Using the MP, identify the compound and draw its structure.

(3.) To observe the effect of impurities on the MP of a compound, on a piece of weighing paper, using a spatula, mix and grind together a **very small** amount of naphthalene with a **small** amount of your unknown and determine the MP of this "impure" sample. To minimize waste, when making the mixture, keep in mind that only very small samples are needed for MPs. To most effectively demonstrate the effect of small amounts of impurities on the MP of a sample, add only a small percentage of the impurity (naphthalene).

BEFORE LEAVING THE LAB: turn the voltage on the Mel-Temp to zero, turn the switch to off, and turn the digital thermometer off. Clean up your work area, lock up and ask your TA to sign your lab notebook. If the fume hood was used be sure it is left clean and close the sash completely.

WASTE DISPOSAL: Place used MP capillary tubes in the evaporating dishes on the side benches. Place any unused impure sample that you made into the Solid Waste container in the hood.

SAFETY: Burned tissue caused by hot surfaces or flames should be immediately placed under cold tap water and as soon as possible into ice/water. Keep the burned tissue cold until the pain subsides. This will minimize the pain and tissue damage. Keep all lab chemicals off of your skin.

Table. Melting points of known and unknown compounds.

COMPOUND	MP (°C)
4-Methylphenol	35
Benzophenone	48-50
Maleic anhydride	54-56
4-Bromophenol	64-66
4-Aminobenzaldehyde	71
Naphthalene	79-80
3,4-Diaminotoluene	89-90
Acenaphthene	94-96
Isobutyranilide	106-107
Acetanilide	113-115
Benzoic acid	122-123
Urea	132-134
d,l-Glyceraldehyde	145
Adipic acid	152-154
Sulfanilamide	165-167
2-Aminophenol	174
4-Toluic acid	180-182
Succinic acid	187-189

The melting points listed here vary slightly from those given in the Williamson lab text. MPs that you find will be closer to those listed in this handout.

Postlab Questions (These are done as part of the complete report which is due the day after the next lab period.)

1.) What is the effect of an insoluble impurity on the observed MP of a compound?

2.) Two test tubes contain compounds having the same MP. Using MPs, how could you determine whether the two test tubes contain the same or different compounds?

3.) Strictly speaking, why is the term melting *point* incorrect?

4.) In a recrystallization (a technique that you will encounter later in the semester), a solid is dissolved in a solvent and later the solvent is removed. If a MP of the sample is taken while the sample is still moist with solvent, what effect might that have on the MP of the sample?

5.) What two pieces of information can a MP determination provide?