Melting Point Experiment

Source: AEM Handout, revised 7/10/17

Standard Steps for Melting Point Determination

- pulverize a tiny amount of the sample
- load the mp capillary (open at one end, closed at the other!)
- measure a preliminary mp (optional, if you know the expected or literature mp)
- measure the experimental melting point range: place loaded mp capillary in the Mel-Temp and increase the temperature quickly at first and then VERY SLOWLY within 20° of expected mp
- Record the mp RANGE!

Cleanup. Dispose of the melting point capillary in the glass waste bin.

Melting Points of Recrystallized Solids from Previous Experiment

If your phthalic acid and benzoic acid crystals have been drying after recrystallization, you are ready to test their purity. If you did not isolate them on the previous lab day, then you'll need to finish the recrystallization and let them dry for a day or more prior to mp determination.... Remember to measure and record the mass of the very dry crystals (to calculate % recovery) prior to removing a small amount to measure the mp. Also, since your crystals will be graded, don't pulverize them all, just a small amount for the mp! These mp measurements are also considered a part of the crystallization experiment and should be included in your notebook with that experiment.

Determining the Identity of an Unknown by Melting Point

You will be given an unknown solid sample whose melting point can be used for identification purposes. The sample will be one of the compounds listed below.

- Record this Options for mp Unknown list with the melting points in your notebook, so that you can choose between them during the experiment. (Note: do NOT look up all of their physical properties!)

Compound	**mp (°C)**	**Compound**	**mp(°C)**
Benzophenone	49-51	Benzoin	137
Steric acid	69-70	Anthranilic acid	145-147
Vanillin	80-81	Cholesterol	149-150
Naphthalene	80-82	Adipic acid	152-153
1-Naphthol	95-96	Citric acid	153-155
Acetanilide	113.5-114	Salicylic acid	158.5-159
2-Naphthol	121-122	Itaconic acid	162-164
Benzoic acid	121.5-122	Sulfanilamide	165-166
Cinnamic acid	132.5-133	Succinic acid	184.5-185
Urea	132.5-133	3,5-Dinitrobenzoic acid	205-207
Benzamide	132.5-133.5	*p*-Terphenyl	210-211

- Record your unknown number in your notebook.
- First take a preliminary, fast melting point of the sample to get an idea of its mp.
- Second, very carefully and slowly measure the sample's melting point range.
- Finally, measure at least TWO mixed mps; choose the known sample that you think is your unknown and also another one that has a similar literature mp. For each mixed mp, obtain a small amount of the known material and mix it in about equal proportions with your unknown using your spatula. The known substance that matches your unknown should have a mp very similar to the carefully measured "slow" mp (usually within 1° at either end of the range). If your mixed mp measurements indicate that you have not correctly identified your unknown, select another compound with a similar mp from the list and try its mixed mp....
- Before you turn in this experiment, record in your notebook the structure, physical, and hazardous properties of only the material that you identify as your unknown.

Cleanup. Any unknown that has been removed from its container and is no longer needed should be put in the solid hazardous waste. The rest of the unknown sample should be returned. Please do not remove the unknown code.