Melting Point Determinations

Source: AEM Handout

Remainder of Prior Experiment!... Melting Points of Recrystallized Solids

INCLUDE THIS SECTION WITH THE CRYSTALLIZATION LAB THAT YOU DID LAST LAB PERIOD!

If your phthalic acid and benzoic acid crystals have been drying after recrystallization, you are ready to test their purity. (You may want to do these measurements after you have mastered the techniques described in the general melting point guidelines; in other words, calibrate your thermometer and determine your unknown, first, then come back to these samples.) 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 the mass of the very dry crystals prior to removing a small amount to measure the mp. You will also need to calculate % recovery and turn in the crystals for grading when this lab is due. These mp measurements are considered a part of the crystallization experiment and should be included in your notebook with that experiment!

General Melting Point Guidelines

Follow the standard steps for each melting point determination:

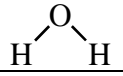
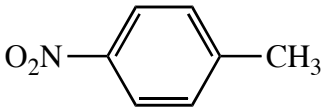
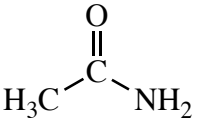
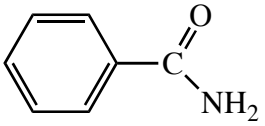
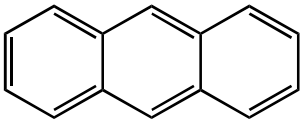
- Break up a tiny amount of the sample.
- Load these small sample pieces into the mp capillary and tap down to the closed end.
- Measure a quick preliminary mp (optional, if you know the expected or literature mp).
- Measure the melting point: place loaded mp capillary in the Mel-Temp and increase the temperature quickly at first and then VERY SLOWLY within 10 to 20º of expected mp;
- Record the mp RANGE!

Calibration of Your Thermometer

The first step for determining melting points is to confirm, with samples known to be relatively pure, how accurately your thermometer reports temperatures. A table of such substances, with their literature melting points is given below. (NOTE: do NOT look up all of these materials for physical and hazardous properties.) Record this list with the melting points in your notebook; include an additional column in the table for your experimental mp data. Measure the temperature of ice/water using your thermometer, and then measure the melting points of the four others listed in the table. (The more samples measured, the more points on your graph, and the better your correction will be.)

For these melting points, be sure to follow the general melting point guidelines given above; be especially careful to raise the temperature only 1º per minute near the melting point and to record the melting point range that you observe. You may want to prepare a mp capillary for each sample, and place the three lowest-melting samples in your Mel-temp first; as the first of these samples melts, you can remove it and replace them with the next higher melting substance. That way, as you move up the temperature range, you can measure the mp's of all of the samples. If the measured mp's deviate significantly from the literature values, measure them once more to confirm your data; the data should agree within 1 degree at either end of the melting point range.

Prepare a thermometer calibration graph using this data by plotting the ending temperature of the measured melting point range on the x-axis relative to the ending temperature of the literature value for the melting point on the y-axis. Use Excel (or some other method) to fit the data with a straight line; be sure to print the line equation on the graph. (Click on the "Truman Fieldguide" link at the Superlab course website, and follow the subsequent links, for more information on making good quality graphs using Excel.) You will need two copies of this graph (one for the permanent page in your notebook, and one for the "copy" page, which will be graded. You will use this equation or this graph to determine the corrected melting points of all other substances throughout the semester. (Note that if you break your thermometer, you will need to calibrate a new one, so treat them with care!)

Compound Name	Compound Structure	Literature Melting Point (degrees C)
Ice/water		0
4-nitrotoluene		53-54
Acetamide		82.3
Benzamide		132.5-133.5
Anthracene		214-217

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. Record the unknown number in your notebook! The sample will be one of the compounds listed below. NOTE: do NOT look up all of these materials for physical and hazardous properties. Record this list with the melting points in your notebook, so that you can choose between them during the experiment.

- 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.
- Third, obtain a small amount of the sample that you think you have, and measure a mixed mp to confirm the identity of your unknown. If your mixed mp indicates that you have not correctly identified your unknown, select another compound with a similar mp from the list and try its mixed mp....

Record all of the above melting point ranges in your notebook. 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. Be sure to explain how and why you determined this identity for your substance in your conclusion.

Options for mp unknown:

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

Cleanup. Any leftover solids should be placed in a hazardous solid waste container. Used mp capillaries can be thrown in the glass waste container.