Melting Points

Required Reading
Mohrig, pp. 116 - 127

In the organic lab, whenever you isolate a crystalline intermediate or product you will take a melting point. This is a physical property characteristic of crystalline compounds and also serves as an indicator of purity.

1) **Determination of purity:** If you take a melting point of your compound and it starts to melt at 60 °C and doesn't finish until 180 °C, you should suspect that something is wrong. A melting range *greater than* 2 °C usually indicates an impure compound. (As with all rules, there are exceptions. There aren't many to this one though.)

2) **Identification of Unknowns:**

   a) If you have an unknown solid, take a melting point. Many books *(CRC Handbook of Chemistry and Physics*, for example) contain tables of melting points and lists of compounds that may have a particular melting point. One of them may be your unknown. You may have dozens of compounds to choose from. A little difficult, but give it a try. If nothing else, you know the melting point.

   b) Mixed Melting Points: Take your unknown and mix it thoroughly with a compound that you think might be your unknown. Then:

      (1) If the mixture melts at a *lower* temperature, over a *broad range*, your unknown is not the same compound.

      (2) If the mixture melts at the *same temperature*, *same range*, it is probably the *same compound*! Try another one though, with a different ratio of your unknown and this compound just to be sure.

Our laboratories are equipped with the Mel Temp melting point apparatus, Mohrig, p. 120. The Mel-Temp can accommodate up to three capillary tubes.

Helpful Hints for Taking a Melting Point

1) You need literally ONE CRYSTAL to take a melting point, *i.e.*. the very smallest amount you can see.
2) If the melting point is known, raise the temperature rapidly to within 20 degrees and no faster than 1-2 degrees per minute thereafter. Rapid heating through the melting range will give results that are too high and with too large a range.
3) Never repeat a melting point with the same sample in the same open capillary. Discard the sample and capillary after it cools.
4) Always report the melting range as two temperatures, the first when the sample begins to melt and the second when melting is complete. You must see liquid for it to be complete. Be certain to read the thermometer to the correct accuracy.
5) Do a number of samples simultaneously to save time.
6) If doing a mixed melting point, do all the samples at the same time, if possible.
7) Be sure the sample is dry.

In this lab, practice your technique by taking a melting point of one of the known compounds (of your choosing, from the list below. Then obtain the melting point of an unknown sample. Identify the unknown by taking mixed melting points with one or two samples that have similar melting ranges as your compound. Take melting points of the mixtures and identify your unknown. Record all your data in your record book.

Possible Unknowns

Melting Range °C	Name
59-60	1-Nitronaphthalene
69-72	Biphenyl
78-80	Methyl-3-nitrobenzoate
80-82	Naphthalene
98-100	5-Chloro-2-methoxybenzoic acid
103-105	2-Methylbenzoic acid (*o*-Toluic acid)
112-114	Anthranilamide
113-115	Acetanilide
113-115	1,4-Benzoquinone
122-123	Benzoic acid
122-124	*trans*-Stilbene
133-136	Phenacetin (4-ethoxyacetanilide)
146-148	2-Nitrobenzoic acid
149-151	4-Nitroaniline
152-154	Adipic acid
160-163	Triphenylmethanol
182-185	4-Methoxybenzoic acid (Anisic acid)
188-189	4-Aminobenzoic acid (*p*-Aminobenzoic acid, PABA)

MELTING POINTS
DATA SHEET

NAME: _____

Section Number: _____

UNKNOWN SAMPLE NUMBER:_____

MELTING POINT:_____

UNKNOWN STRUCTURE

MIXED MELTING POINTS

	COMPOUND	LITERATURE MP	MIXED MP WITH UNKNOWN
A)			
B)			
C)			

IDENTITY OF UNKNOWN:_____

In general, the addition of an impurity to a pure substance will cause a melting point to decrease in proportion to the amount of the impurity. Explain this phenomenon.