**Melting Points**

**Required Reading:** Padias, pages 19, 49-53.

 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. Although called melting points, they are always reported as ranges, with a starting and ending temperature.

**1) Determination of purity:** Although it is called a melting point, because of way a melting point is measured, virtually all melting points are reported as ranges. The more narrow the melting point range, the more pure the compound generally is. A melting range *greater than* 2-3 °C usually indicates an impure compound. There are exceptions to this rule, but not too many. If a compound starts to melt at 60 °C and doesn't finish until 180 °C, it is clearly an impure mixture.

**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 (Padias; Figure 2-4, p 52). 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.

**Experiment**

You will be given an unknown solid and use melting points to determine its identity. Do not forget to record the unknown number and the appearance of your unknown in your lab notebook. First, 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 your unknown sample. Identify the unknown by taking mixed melting points with at least two samples that have similar melting ranges to your compound. Since the Mel-temp apparatus can hold three samples it is recommended that you simultaneously run your two mixed samples along with your pure unknown. Record the melting points of the mixtures and identify your unknown. Record all your data in your record book.

**Possible Unknowns**

|  |  |
| --- | --- |
| Name | Melting point, °C |
| *n*-Butyl-4-hydroxybenzoate | 67-71 |
| Phthalide | 71-74 |
| 4-Chlorobenzophenone | 74-76 |
| Methyl-3-nitrobenzoate | 78-80 |
| 4-Hydroxy-3-methoxybenzaldehyde | 81-83 |
| Ethyl-4-aminobenzoate | 88-90 |
| Tribenzyl amine | 91-94 |
| 5-Chloro-2-methoxy benzoic acid | 98-100 |
| 2-Methyl benzoic acid | 103-105 |
| Cholesteryl acetate | 112-114 |
| Ethyl-4-hydroxy benzoate | 114-116 |
| Mandelic Acid | 118-120 |
| Benzoic Acid | 122-123 |
| Sucralose | 125-128 |
| Malic Acid | 130-132 |

**MELTING POINTS**

**DATA SHEET**

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| **Name:** |  |  | **Section:** |  |

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Answer the following questions:

1. When a second compound is mixed together with a pure compound what is the effect on the pure compound’s lattice structure? Explain your answer at the molecular level.

2. What do you expect to see when you take a melting point of this mixture?

Answer the following using the correct number of significant figures. ***Show your work.***

3. Four students, working together, prepared a solution of KCl in water. The first student added 135.16 mL of water, the second student 65 mL of water and the third 4.6 mL of water. The fourth student added 1.32 g of potassium chloride to the water. You may assume that the KCl does not significantly change the total volume of the solution.

 a. What volume of water was added?

 b. How many moles of KCl were added?

 c. What was the final concentration (M) of the solution?