Organic Chemistry Laboratory
#1 - Melting Point

The melting range of a compound can provide useful information about its purity and identity. In this experiment you will have an opportunity to use the Mel-Temp apparatus to determine the melting range of several compounds. This experiment is divided up into three sections. First, you will use known samples to confirm their melting ranges and gain some experience with the Mel-Temp. Second, you will determine the identity of an unknown sample using its melting range. Finally two mixed-melting points will be taken to confirm the identity of your compound.

Pre-lab Reading:

Read pages 86 - 92 in Chapter 12 in the Zubrick manual.

Pre-lab assignment:

Answer the questions on the last page of this handout. Remember – this prelab assignment is due *before* your lab section begins. You should also notice that 6 of the compounds listed on the table below do not have their melting ranges recorded, using a CRC Handbook, Aldrich catalog, or some other reliable source fill in the missing data. Be extra careful of internet sources as many are not checked for accuracy.

Procedure:

Choose one of the known compounds available in the lab and place a small pea-sized portion of it on a piece of weighing paper. Fill a melting-point capillary according to the directions given in the Zubrick text. Using the known melting point as a guide, confirm the melting point using one of the Mel-Temp's in the lab. *Be careful, as the apparatus may be hot from a previous use!* Insert the capillary tube into the apparatus and turn the power on. Set the control so that the temperature rises at about 10 degrees per minute. Record the temperatures when the sample first begins to melt and when the last solid disappears. Turn the apparatus off and place your used capillary tube in the **glass-waste container**. Repeat the above procedure with a fresh sample of the same known prepared exactly as you prepared the first sample. This time, when the temperature is 15 degrees below the first temperature you recorded slow the rate of heating to 1 or 2 degrees per minute. Record the melting range you observe under these conditions. How do they differ?

Obtain an unknown solid from your instructor and record its number. Your unknown is one of the compounds listed in the table below. Now carefully determine the melting range of your unknown. Which two of the compounds from the table are closest in mp to your unknown?

Place a small sample of your unknown on a piece of weighing paper and a small sample of one of the compounds you believe your unknown to be next to it. With a spatula, thoroughly mix the two samples then determine the melting range of the mixture. Repeat this procedure with the other candidate. How do the two mixtures differ in mp? Which one of the two known compounds is the same as your unknown?

Possible Unknowns

Compound	*MP (°C)*	*Compound*	*MP (°C)*
benzhydrol	65-67	Acetanilide	
biphenyl	69-72	Adipic acid	151-153
Citric acid		p-Anisic acid	182-185
Cinnamic acid		d-Tartaric acid	
Fluorene	115-116	Thiourea	177-178
Hippuric acid		Urea	

Data and Observations:

You should also include a table of the various solids you will be using during this experiment. In your notebook you should prepare a table similar to the one on the last page of this handout for recording your observations. Several questions are included within the procedure. These are intended to guide you as you write your observations in the notebook.

Report:

Include the data table that appears on the final page. Remember - **do not** fill out this page as you go through the experiment but record all data in your notebook then transfer the necessary data to your report. Also, include in your report the answers to the following questions.

Questions:

1. What error might be introduced if the rate of heating is too great? Be specific.

2. A student suspected that an unknown was undergoing a chemical change at its melting point. Suggest a simple method for testing this hypothesis.

3. Ordinarily you will determine melting points in a glass apparatus. This means that glass constitutes an impurity in the system. Why does glass have no noticeable effect on the melting points?

4. Gram for gram, table salt lowers the freezing point of water much more than table sugar. Explain.

Report: Melting Points

Name:_____

Lab section:_____

Date:_____

Known

Name	M.P.	M.P.	Lit. M.P.
_____	____	____	____

.

Unknown

Unknown number	M.P.	M.P.
_____(quick)	____	____
_____(slow)	____	____
Mixed with	Mixed M.P.	Mixed M.P.
_____	____	____
_____	____	____
_____	____	____

Identity of unknown _____

Prelab: Melting Points

Name:_____

Lab section:_____

Date:_____

1. You have an unknown sample that melts at 101-102 °C and you do a mixed melting point with a compound that also melts at 101-102 °C.

   a. What would be the result if your unknown is identical to the known?

   b. If the unknown is not identical with the known?

2. Fill in the missing melting points. You must include a reference to where you obtained the information like Aldrich or CRC. Be sure to copy this information into your lab notebook.

Compound	***MP (°C)***	***Compound***	***MP (°C)***
benzhydrol	65-67	Acetanilide	
biphenyl	69-72	Adipic acid	151-153
Citric acid		p-Anisic acid	182-185
Cinnamic acid		d-Tartaric acid	
Fluorene	115-116	Thiourea	177-178
Hippuric acid		Urea	