EXPERIMENT 4

IDENTIFICATION OF A SOLID UNKNOWN BY MELTING POINT DETERMINATION

Materials Needed

Mel-Temp apparatus and capillary tubes, watch glass, spatula. Pure and impure urea samples. Assigned unknown. Samples of all compounds in the Table of Possible unknowns.

Textbook Reading

Smith, Chapter 4.

Background

The melting point (mp) of a solid is the temperature at which it melts to a liquid. The mp is an important physical property of a substance because it is easily measured and helpful in identifying the substance. When properly determined the experimental mp of a solid also reveals whether it is pure or not.

To measure a mp, one generally heats a small sample of the substance while monitoring the applied temperature with a thermometer. This procedure gives a range of temperatures as the mp. The temperature, at which liquid is first seen is the lower end of the melting point range and the temperature, at which no solid remains is the upper end of the melting point range. A pure substance normally has a melting point range no larger than 1-2 ^{o}C.

General Melting Point Procedures

Overview. The procedure we will use is very typical. It uses a very small glass tube called a capillary tube to hold the sample. The capillary tube is placed in a Mel-Temp apparatus and the temperature increased slowly. The experimenter monitors the temperature and the sample and notes both the low and high ends of the mp range.

Loading the capillary tube. Place a 20-30 mg of the solid on a watch glass, and use a spatula to grind it to a powder then use the spatula to gather the powder into a small pile. Stick the open end of a capillary tube into the pile to a then invert the capillary and tap the sealed end on the table top to get the solid to drop to the bottom. (The instructor will also demonstrate some alternative methods for getting the sample to the bottom of the tube.) The height of solid at the bottom of the capillary should about 2-3 mm.

Using the Mel-Temp Apparatus. Place the loaded capillary into one of the 3 slots on the Mel-Temp. Turn the Mel-Temp power switch on and choose a voltage setting. The instructor will supply a heating curve for the Mel-Temp that assists with this. The most important guideline in setting the voltage is that the temperature not be rising faster than 2-3 degrees per minute when the sample is melting. While the sample is heating, watch it while frequently checking the temperature. Note the temperature, at which liquid starts to pool up in the bottom of the capillary. This is the start of your mp range. Continue to heat and observe and note the temperature, at which the sample appears to be a completely transparent liquid; this is the final temperature of the range (Cloudiness indicates solid particles suspended in the liquid, so if it is cloudy it is not completely melted.) Dispose of used capillaries in the broken glass waste container.

Specific Procedures

1. Pure urea. For practice obtain the mp of a pure sample of urea (lit mp = 133-135 °C).

2. Impure urea. To get an idea of how an impure substance behaves in a mp test, obtain the mp of 75% pure urea, to which cinnamic acid (25%) has been added.

3. Unknown. Obtain the mp of the unknown provided to you by the instructor. (Hint: it is a good idea to do a fast determination first to find the approximate melting point range, then do a careful slow determination on a second sample.)

4. Mixture mp experiment. Make a hypothesis as to which compound on the list of possible unknowns below is most likely to be your unknown substance. Then, carry out a mixture mp test in order to conclusively identify the unknown. Mix a small amount of the unknown with the hypothesized substance and determine the mp of the mixture. (Prepare your mixtures with an approximately 1:1 mass ratio and grind together thoroughly before testing the mp.) If your hypothesis is correct what should the result be? If your hypothesis is proved wrong then make a new hypothesis and test it in the same manner. (Alternatively, hypothesize up to three possible identities and test all at the same time making a single run in the Mel-Temp.)

Table of Possible Unknowns

Compound	Literature mp (°C)	Compound	Literature mp (°C)
benzophenone	48-49	*trans*-stilbene	122-124
bibenzyl	50-51	nicotinamide	129-130
biphenyl	70	acetylsalicylic acid	135-136
caprolactam	68-70	salicylamide	140-142
acetamide	79-81	2-methylimidazole	142-143
glutaric acid	95-98	adipic acid	151-153
acetanilide	113-114	benzoin *alpha*-oxime	152-154
benzamide	121-125	salicylic acid	158-161
benzoic acid	122-123	1-naphthoic acid	160

PRE-LABORATORY QUESTIONS

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Name _____ Section _____ Date _____

1. A student obtains the melting point of an unknown white solid and obtains the following result: unknown mp = 145-157 °C. List at least three possible explanations for the mp range being so wide.

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IN-LAB OBSERVATIONS/DATA

Names_____ Date _____

Sample	height in cap tube (mm)	mp range (°C)	Observations
Pure Urea			
Impure Urea			
Unknown # _____			
Unknown Mixed with _____			
Unknown Mixed with _____			
Unknown Mixed with _____			

EXPERIMENT 4. IDENTIFICATION OF A SOLID UNKNOWN BY MELTING POINT

REPORT

Names_____ Date _____

Results

Pure Urea mp and lit value _____

Unknown mp _____

Unknown Identity and mixture mp result_____ _____

Unknown structure:

Questions (type up the answers on a separate sheet).

1. List the four precautions that must be taken in a mp determination so that a pure compound will end up showing a sharp mp range. Explain why each of these causes the mp range to be broad.

2. Use your results to reach conclusions on the mp behavior of pure compounds versus those of impure compounds (mixtures).