

4 Determination of Melting Points

Purpose: The object of this experiment is to identify a solid from its melting point.

Introduction

The melting point of a compound is the temperature at which it changes from a solid to a liquid. This is a physical property and is often used to identify compounds or to check the purity of the compound. Organic compounds have melting points that are low enough to measure them conveniently with the MelTemp apparatus which has a 0 to 260 °C thermometer. (Thermometers from 0 to 400 °C are still low for ionic compounds.)

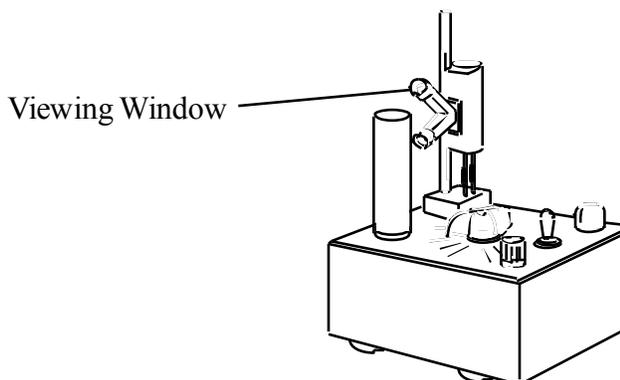
Sometimes compounds melt in a 2 to 3 degree range, so you may observe a melting “range” rather than a melting point. This is sufficient to identify the compounds in this experiment.

Note: The substance must be dry, otherwise the solvent present will depress the melting point. All samples used in this experiment will be dried.

Apparatus

Your instructor will demonstrate how to use the MelTemp device. This type holds three sample capillary tubes, so you can make more than 1 measurement at a time.

(CAUTION: Use extreme care when removing or replacing the thermometer.)



Procedure

Measuring melting points

1. Practice by measuring the melting point of a known compound. (Samples are already placed on weighing paper.) The capillary tubes have one open end and the other is closed.
2. To fill the capillary tube, place its open end on the solid sample to force some crystals in, then invert the tube and tap to allow the solid to settle to the bottom. You will need about 3-4 mm of densely packed solid in the tube. Place the filled capillary tube into the MelTemp. (If you are testing more than one compound at a time, repeat this step)
3. Do a careful (slow heat-up) until you observe the compound melt. Record the temperature on the data sheet and compare it with the value listed in the Table on the next page.

Note: Allow the melting point apparatus to cool before making another measurement. It takes about 7 minutes to cool from 200 °C to 100 °C.

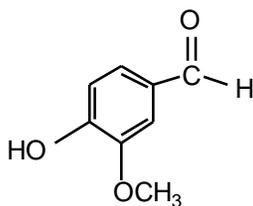
4. Repeat steps 2 and 3 with unknown compounds. Identify the compound from the data in the Table below. If the measured melting point is not close to just one of the listed compounds, repeat the measurement.

Drexel Science in Motion

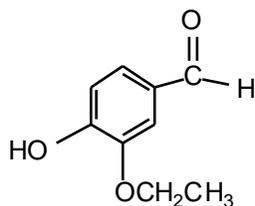
Melting Points of Solids. Structures are shown below.

Compound	m.p. °C	Compound	m.p. °C
ethyl vanillin	77.5	acetaminophen	169
naphthalene	80	para-aminobenzoic acid	188
vanillin	81.5		
acetyl salicylic acid	135		
salicylic acid	158		

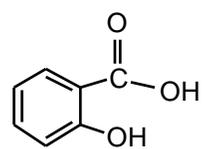
Structures



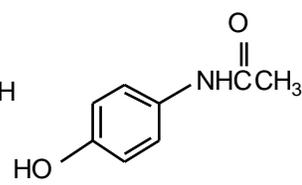
Vanillin



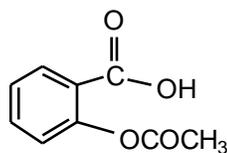
Ethyl vanillin



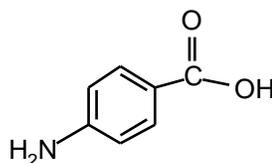
Salicylic acid



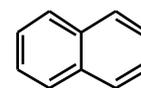
Acetaminophen



Acetyl salicylic acid



p-Amino benzoic acid



Naphthalene

Data and Results (Melting Points)

Name(s) _____

Sample	m.p. measured °C	m.p. from table °C *	Compound
Known			

* Choose the one that is closest to your measured value.

Question: Why can't the MelTemp be used to identify ionic compounds such as NaCl?

Instructor's Guide
#4 Determination of Melting Points

(Data and Results)

Instructors: For each sample, place a small amount onto a piece of weighing paper. Identify each with a code number written on the weighing paper.

Sample	m.p. measured °C	m.p. from table °C *	Compound
Known	75-77	77.5	<i>ethyl vanillin</i>
1	169-174	169	<i>acetaminophen</i>
2	158-161	158	<i>salicylic acid</i>
3	186-188	188	<i>p-aminobenzoic acid</i>
4	135-138	135	<i>acetyl salicylic acid</i>
5	81-83	81.5	<i>vanillin</i>

* Choose the one that is closest to your measured value.

Question:

Why can't the MelTemp be used to identify ionic compounds such as NaCl?

Most ionic compounds have melting points that are too high for the MelTemp apparatus (up to 400 °C) such as NaCl, which melts at 801 °C.

Instructor's Guide
Determination of Melting Points (cont'd)

Time: 30 – 40 min

Equipment and Materials per group

Items	Number	Comment
MelTemp & power cord	1	
thermometers	1	
capillary tubes	10	
known sample	1	
unknown samples	5	
spoon spatulas	6 per class	
Safety glasses	1 per student	
weighing papers	package	1 piece for each sample
marker	1 per class	To write code letters on weighing paper

Ideas/ Information

1. Some compounds, such as sucrose and other sugars, decompose, turning brown as they melt.

2. Compounds must be molten when electrolyzed to break them down into elements. One ionic compound that has a relatively low m.p. is potassium hydroxide, KOH, 360°C, the compound used in Davy's discovery of potassium by electrolysis of KOH.

The high melting point of Al_2O_3 , 2045°C, caused problems in producing cheap Al from electrolysis. The Hall process solved the problem by adding cryolite to aluminum oxide to lower its melting point.