Lab Report: Melting Point

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Purpose:

There are several reasons to determine a compound's melting point: it is useful in supporting the identification of a compound, as well as serving as a rough guide to the relative purity of the sample. The purpose of the Melting Point lab is to learn to determine melting points accurately. This is an important technique that will be used in many of the experiments in the organic lab.

Experiment 1 -- Melting Point

Solids vs. Liquids

Melting refers to a transition between a solid (frozen) and a molten (liquid) state. When a compound melts, its constituent molecules become free from their neighbors and are able to adopt many more flexible conformations and positions. Molecules in the liquid state can also move relative to one another and thus gives fluids the ability to "flow".

The more a molecule is able to bond to its neighbors, the more energy it takes to free it from its intermolecular interactions to reach the liquid state. A classic example comes from comparing ordinary salt and paraffin wax, two compounds that are both solids at room temperature, but require vastly different amounts of thermal energy to reach the liquid phase. In the case of salt, each atom in the salt crystal interacts with its neighbors through strong electrostatic interactions in the form of strongly attractive opposite ionic charges. In contract, paraffin wax is a mixture of long chain hydrocarbon chains that are uncharged, non-polar, and interact only through much weaker Van-der-Waals transient dipole interactions. As a result, converting solid wax to a liquid involves overcoming weak intermolecular interactions and can be accomplished at relatively low temperatures (~50 ℃). Melting salt, on the other hand, requires much more energy to overcome the strong electrostatic interactions that form a salt crystal. Ordinary salt becomes a molten liquid at about 800 ℃.

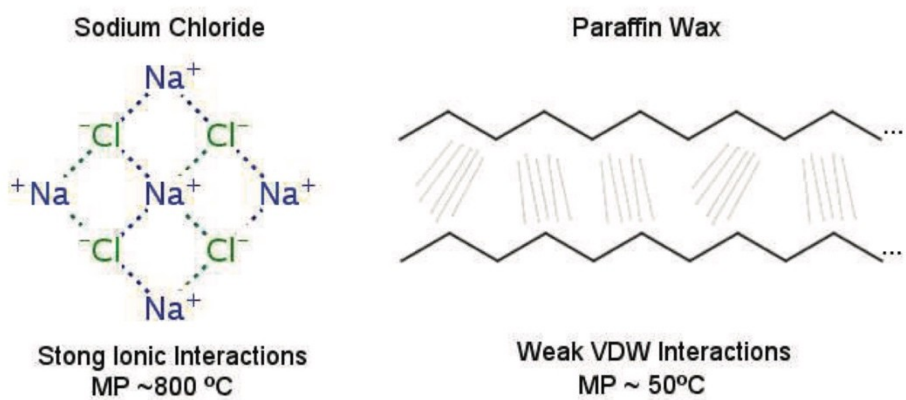


Figure 1.1

Figure 1.1 Melting Points for Sodium Chloride and Paraffin Wax. The dashed lines on the left represent strong ionic interactions within the salt lattice while the faint stripes on the right represent the far weaker Van-der-Waals (VDW) interactions between the alkane chains of paraffin wax (entire chains are not shown).

Melting Point as a Physical Property

A pure non-ionic crystalline organic compound usually has a sharp and characteristic melting point. In the laboratory, the melting point of a substance is defined as the temperature at which the solid and liquid phase of the substance are in equilibrium at atmospheric pressure.

In a pure substance, each molecule resides in an identical environment has the same set of interactions with its neighbors. Thus, as the substance is heated, each molecule should break free of its neighbors at the same temperature. Provided a sample is absolutely pure and can be heated evenly, it should convert from solid to liquid all at once. Moreover, the temperature at which this uniform transition occurs is a physical property of compound and should never vary. Though perfectly even heating is impossible, keeping sample size small will provide more uniform heating and better measurement of melting temperature.

However, if a substance is not pure, each molecule of the mixture is not in an identical environment. Adding a small amount of an impurity to a mixture disrupts the regular pattern of interactions within an ordered lattice.

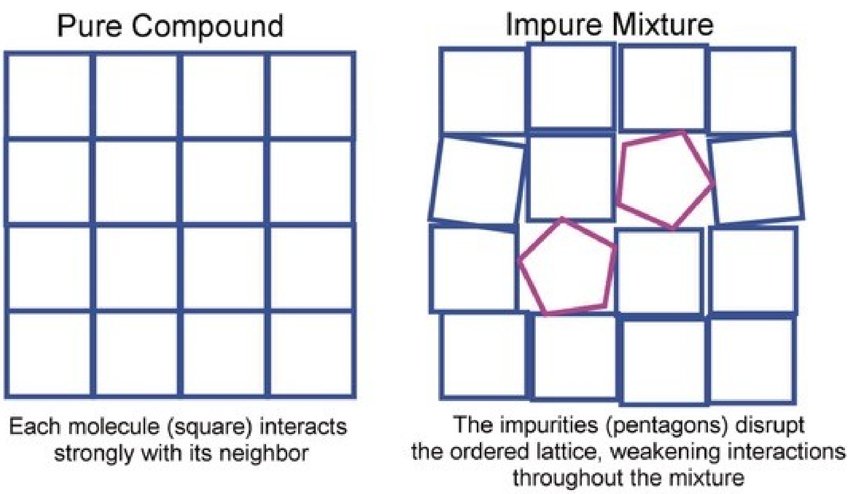


Figure 1.2

Figure 1.2 Impurities typically lower and blur a melting point. The lattice on the left represents a crystalline pure compound while the disrupted lattice on the right reveals the disruptions caused by the addition of impurities.

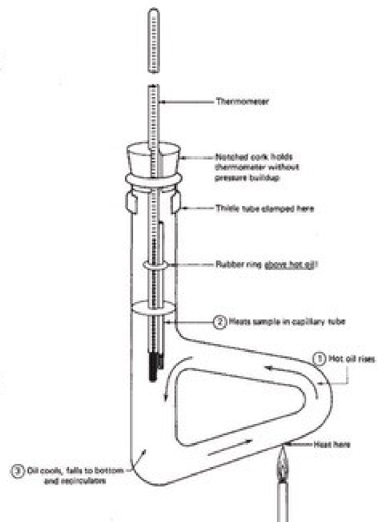
To a first approximation, the disrupted lattice on the right in Fig 1.2 is easier to convert to a liquid as the connections between adjacent molecules are weakened. Thus, the presence of impurities usually results in a lowered melting point. Because the intermolecular interactions in the disrupted lattice are not identical from molecule to molecule, parts of the mixture can melt easier than others and, on the whole, the melting an impure lattice occurs over a broader temperature range than is the case with a

We will employ two common techniques for measuring melting point, the Thiele-Dennis Tube and a Melting Point Apparatus.

Thiele-Dennis Tube:

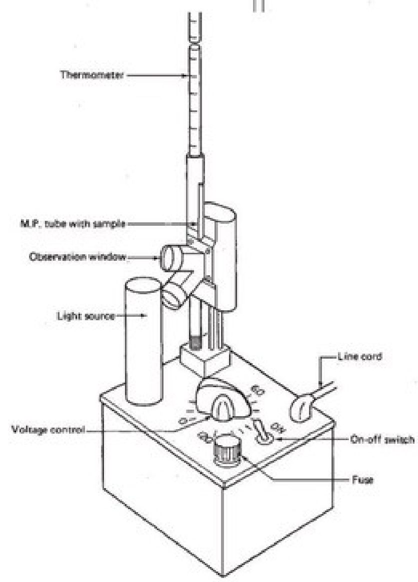
The Thiele-Dennis tube is a glass tube designed to contain heating oil (usually di-n-butyl-phthalate) and a thermometer to which a capillary containing the sample tube is attached. The shape of the Thiele-Dennis Tube allows convection currents to form in the oil then the apparatus is slowly heated. These currents maintain a uniform temperature distribution throughout the tube (ensuring the sample and thermometer are in thermal equilibrium).

The tube is slowly heated using a Bunsen Burner and the rate of temperature increase must be regulated carefully. The rate near the melting point should be low (about 1 °C per minute) to ensure that the temperature increase is not faster than the heat is transferred to the sample (again, equilibrium).



Melting Point Apparatus (Mel-Temp®):

A typical Melting Point Apparatus is an electronically heated device which allows several samples to be melted simultaneously. Such instruments contain a heated block, thermometer, viewing port, and heating controls. These controls typically allow various rates of sample heating. Use slow heating as your sample approaches its melting point and, as always, be sure the instrument is below your sample's melting point before you insert your sample.



Capillaries:

The use of either instrument shown requires that a small sample be loaded into a thin-walled glass capillary prior to heating. Loading a sample can be accomplished by first tamping sample into the mouth of the capillary then tapping the closed end on the lab bench until the sample is completely packed at the closed end of the tube. Alternatively, the sample can be packed by dropping the capillary from a height of one meter or so (usually while contained in a pipe or channel of some sort).

(Figures here taken from: http://what-when-how.com/)

EXPERIMENT:

1) Melting Point of Pure Sample

Crush and grind to a fine powder a small amount of sample using a mortar and pestle. Push a melting point capillary into the sample and force the powder down into the capillary (see above). Using either the Thiele-Dennis tube or Melting Point Apparatus, determine the melting points for both Cinnamic Acid and Urea. Record your values below.

2) Melting Points of Mixtures

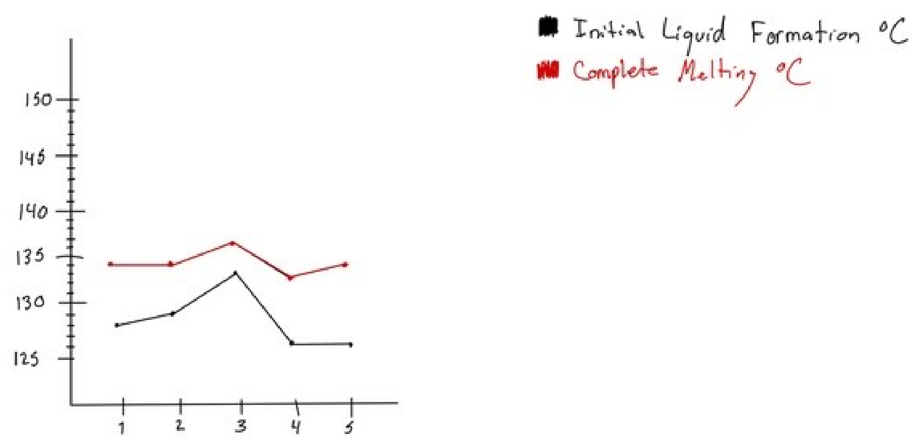
Prepare mixtures of Urea and Cinnamic Acids in the following ratios: 1 to 4, 1 to 1, and 4 to 1 (use a balance to be accurate -- for example for a 1 to 4 mixture of Urea to Cinnamic Acid, you could mix 0.1g of Urea with 0.4g of Cinnamic Acid and grind/mix as before). Determine the melting points of each of these mixtures. Record all data below

3) Unknown

Prepare a sample of the unknown given to you by your instructor. Measure and record its melting point in the table below.

Sample	Initial Liquid Formation °C	Complete Melting °C
Urea 1	128.3	134.2
Cinnamic Acid 2	128.5	134.2
1:4 Urea/Cinnamic Acid 3	133.5	136.1
1:1 Urea/Cinnamic Acid 4	126.2	131
4:1 Urea/Cinnamic Acid 5	126.2	134.2
Unknown		

Should your sample melt upon insertion into the melting point instrument, you <u>must</u> repeat your experiment and get accurate data. Compare your results to your labmates, if you have any doubts, repeat your experiment.



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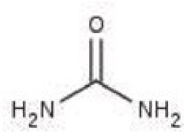
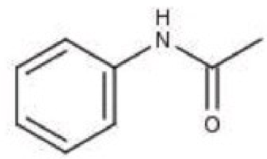
Reference Data:

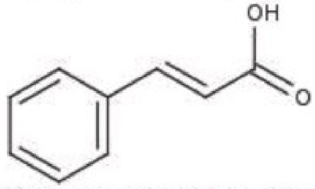
Compound structures and melting points.



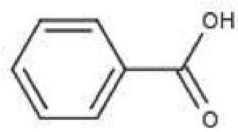
Urea (133-135 °C)



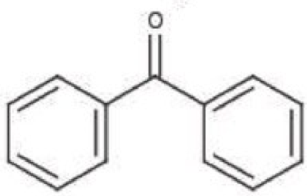
Acetanilide (113-114 °C)



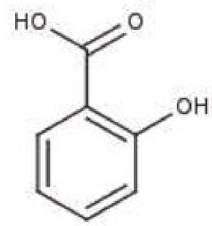
Cinnamic Acid (133-135 °C)



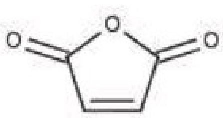
Benzoic Acid (121-122 °C)



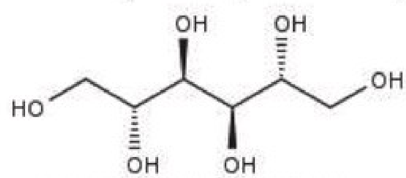
Benzophenone (47-48 °C)



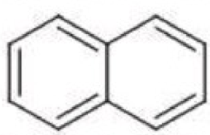
Salicylic Acid (158-159 °C)



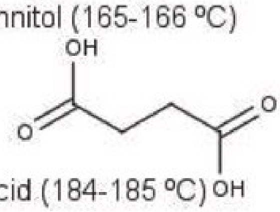
Maleic Anhydride (52-56 °C)



Mannitol (165-166 °C)



Naphthalene (80-82 °C)



Succinic Acid (184-185 °C)

Lab Report:

1) Make a graph of your melting point data. Plot melting point (y-axis) versus composition (x-axis).

2) From the accompanying reference data above, identify your unknown.

3) Answer the following questions

- What two pieces of information can melting point reveal about a substance?
- What effect would the incomplete drying of a sample (e.g. if a recrystallized sample was not completely purged of solvent) have on melting point and why?
- Both Urea and Cinnamic Acid have similar melting points, yet mixtures of these two compounds have very different melting points -- how can this be?
- Rank the compounds in the above reference data in terms of polarity (10 total) based on their given melting points. In term of the relationship between crystalline lattices and melting points given above, how does polarity contribute to melting point?
- ADVANCED QUESTIONS FOR ORGANIC CHEMISTRY:

  Can you conceive of a situation wherein two compounds with equivalent melting points form a mixture (non-covalent) with a higher melting point than the individual constituents?

  Give correct IUPAC names for cinnamic acid, urea, salicylic acid, and succinic acid.