Determination of Physical Properties of Organic Compounds

Introduction

The purpose of this experiment is to acquaint you with the experimental techniques used to measure certain classic physical properties of organic substances. These properties include the boiling point, density, and refractive index for liquids, and melting point for solids. These procedures are explained in more detail in your book.

Organic compounds have a number of physical properties that allow their precise characterization. These include the classical physical constants: boiling point, density, and refractive index for liquids and melting point solids. The rapid development of chemical instrumentation has made determining the identity of a compound easily achievable and fast. Not only are physical properties used to identify a particular compound, they are often used to compare different compounds and are useful in developing separation techniques. Example: two compounds with vastly different melting points can easily be separated by crystallization/recrystallization.

The various classical physical constants of a large number of organic substances appear in the CRC handbook of Chemistry & Physics, Lange’s Handbook and the Aldrich Catalog Handbook of Fine Chemicals. The Aldrich Catalog also contains references to published infrared (IR) and nuclear magnetic resonance (NMR) spectral data for many of the compounds.

Here we will learn how to determine the identity of a substance using its melting point. Many times during an organic synthesis reaction, a melting point of a solid is taken to identify the identity of a compound.

Method 1: Determination of Melting Points

In a microscale lab, two different types of melting point determinations are carried out: (1) simple capillary melting points and (2) evacuated melting points. In this next procedure, you will have to do determine the melting point of two substances and a 50:50 mixture of the two substances using the simple capillary method.

How to use the Thomas-Hoover Uni-Melt apparatus: The Thomas-Hoover Uni-Melt utilizes a electrically heated stirred silicone oil bath covered by a metal and contains a small opening for the thermometer fitting. Melting points are determined in capillary tubes. The capillary is loaded by introducing aprox. 1 mg of material into the open end of the capillary then placing the tube into one of the three slot of the apparatus. A glass window is present for viewing the filled capillary as the temperature is increased by the instrument. Before you start, you must first cool the temperature so that it is at least 20
degrees below the range you believe the melting point will be. If you don’t know, start at room temperature. Turn the dial to number 4. At 4, the temperature should rise at 2-3 degrees per minute. This is optimum, if it rises faster or slower, then readjust the settings. To fill the capillary with the compound, weigh 1 g of substance on a petri dish or weighing paper. Place the solid in a mortar and grind the solid with a pestal several times until the solid is a fine powder. Place the solid back onto wax paper. Tap the open end of a capillary tube against the paper a few times until about 2-3 mm of the solid is at the top of the capillary tube. Then invert the capillary and tap the closed end gently against the table until the solid falls to the closed end. Be careful not to tap too hard as to break the capillary. Place the capillary tube into the Melt-Temp apparatus closed end down and watch the compound as the temperature increases. **Do not leave the Melt-Temp, BE PATIENT.** Melting point transitions occur fast so you need to keep looking for a transition. Measure the point at which you see the first signs of melting then the point at which the last drop has melted. Remember, melting point is a range and you need to record the full range. A 99.99% pure substance will have a range of 1-2 degrees. Less purity means larger range.

**Preparation of Evacuated Melting points:** Many organic compounds decompose before or at their melting point. The decomposing is usually due to oxidation and is mainly at the surface. The decomposed compound will act as an impurity and cause spreading of the melting point range. A convenient way to measure the melting point of a compound that decomposes is to use a closed end-evacuated capillary tube. To do this, take the small end of a Pasteur pipet and treat it with a flame to close the end. Then load 1-2 mg of compound into the Pasteur pipet and use a copper wire to flush the compound down to the sealed end. Next, set up a vacuum (can be an aspirator; and a flame next to each other. **Be careful not to have the flame near and flammables!!!** Flame the middle of the pastuer pipet closer to the closed end while the large open end is adjacent to the vacuum. This will close the other end under a vacuum. Be careful not to allow the solid to move into the vacuum. Your instructor will demonstrate this delicate technique. Be sure to turn off the flame when you are done.
Experiment: Determining the Melting Point of a Compound

In part A this experiment, you will determine the melting point of cinnamic acid, urea and a 50:50 mixture using the methods outline above. The melting point range is sensitive to impurities so it should be expected that the 50:50 mixture will melt over a broader range the each of the pure substances. When you are finished, describe in your report what effect was observed for the 50:50 mixture and why. In part B of this experiment, you will determine the identity of an unknown by measuring its melting point. First determine the melting point of the unknown and find which of the given samples is the unknown (on page 4). Then, to confirm that you chosen the correct compound, prepare a 50:50 mixture of the unknown with each of the given reagents and measure their melting points. Record all data on the table and hand it in before you leave. In your lab report, describe what is your unknown and why based on melting point data.
Chemicals:

1. Acetanilide
   a. Molecular Formula: CH$_3$CONHC$_6$H$_5$
   b. Formula Weight: 135.17 g/mole
   c. bp: 304 °C
   d. mp: 113-115 °C
   e. density: 1.219 g/mL
   f. Dangers: toxic, irritant

2. Caffeine
   a. Molecular Formula: C$_8$H$_10$N$_4$O$_2$
   b. Formula Weight: 198.21 g/mole
   c. mp: 234-236.5 °C
   d. Dangers: flammable, irritant

3. Cinnamic acid
   a. Molecular Formula: C$_9$H$_8$O$_2$
   b. Formula Weight: 148.1 g/mole
   c. mp: 132-133 °C
   d. Dangers: Flammable, irritant

4. Urea
   a. Molecular Formula: CON$_2$H$_4$
   b. Formula Weight: 60.0 g/mole
   c. mp: 132-133 °C
   d. Dangers: highly toxic, flammable
CHEM 315 - Melting Points Experiment
Determination of Melting Points

Experimental:
A. Determination of the melting points of pure cinnamic acid, pure urea, and a 50:50 mix.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Start</th>
<th>Finish</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cinnamic Acid</td>
<td>__________</td>
<td>__________</td>
</tr>
<tr>
<td>Urea</td>
<td>__________</td>
<td>__________</td>
</tr>
<tr>
<td>50:50 Mixture</td>
<td>__________</td>
<td>__________</td>
</tr>
</tbody>
</table>

B. Determination of Identity of an Unknown

Melting Range of Unknown _______________ °C

Mixed melting points (50:50 mix of unknown with compounds of similar melting ranges from table on page 1-2):

<table>
<thead>
<tr>
<th>Mixture</th>
<th>Melting Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unknown &amp; ______</td>
<td>__________ – __________ °C</td>
</tr>
<tr>
<td>Unknown &amp; ______</td>
<td>__________ – __________ °C</td>
</tr>
<tr>
<td>Unknown &amp; ______</td>
<td>__________ – __________ °C</td>
</tr>
<tr>
<td>Unknown &amp; ______</td>
<td>__________ – __________ °C</td>
</tr>
</tbody>
</table>

Identification of Unknown ___________________________________________________

for Unknown #___________