Laboratory Investigation: Melting Points of Pure Compounds and Mixtures

Given the huge number and variety of organic compounds, chemists have sought ways to identify specific compounds. Simple physical characteristics have been used since the very beginnings of chemistry to help identify compounds. Melting point and boiling point are examples of such physical characteristics, and provide useful information, especially when the samples measured are pure compounds. Before today’s ability to determine the identity of compounds using spectroscopic information, organic chemists depended on melting point determination to help identify compounds. In the past, organic chemists even discredited previous chemist’s work based on incorrect melting points.

In today’s laboratory investigation you will measure the melting points of both pure compounds as well as the melting behavior of mixtures. The melting point information from your group members will be combined in a melting point curve. The information from the curve about the melting points of mixtures will be applied to prediction and observation of the solid and liquid transitions of a mixture two additional compounds.

Although there are different specific versions of conveniently measuring the melting point, the simplest place a small sample in an apparatus where the temperature is increased and measured (via a thermometer) while the sample is observed. In common versions a sample to be melted is packed into a small thin tube of glass with one closed end (called a melting point capillary tube.) This capillary tube of sample is placed in an indentation in a block of metal that also holds a thermometer. As the block is heated the sample is viewed through a magnifying glass and the temperature recorded when the melting begins and when it is complete. Sometimes an oil bath is used as the hot environment. One common design is sketched below. However, even a glass tube of heated oil or other liquid that contains a capillary tube of sample adjacent to a thermometer bulb will work.
Scenarios for Melting Point Investigation 1

_Scenario 1:_

Consumer products often are prepared from organic chemicals with biological origins, for example, bar soaps prepared from a 20% mixture of coco fatty acid and 80% tallow fatty acids have the optimal amount of lather. Bar soap fatty acid is being sold at auction. Before bidding, you want to know if the soap stock is mostly coco fatty acid, mostly tallow fatty acid, or the 20% coco and 80% tallow mixture of fatty acids. How can you use the melting point of the fatty acid in the tank car to find if the car contains, mostly coco fatty acid, mostly tallow fatty acid, the preferred bar soap fatty acid mixture 20% coco and 80% tallow fatty acid, or something else? (Note that coco fatty acids are mostly lauric acid and tallow fatty acids are mostly stearic acid.)
Melting Point Investigation 1

In this experiment you will work in groups of four to measure the melting point of either pure lauric acid, which also has the name dodecanoic acid \([\text{CH}_3(\text{CH}_2)_{12}\text{CO}_2\text{H}]\), or stearic acid, which also has the name octadecanoic acid \([\text{CH}_3(\text{CH}_2)_{16}\text{CO}_2\text{H}]\), or mixtures of lauric acid and stearic acids. The mixtures of lauric and stearic acid will be prepared for you. The group results from the pure lauric and stearic acids plus the various mixture of lauric acid and stearic will be combined to give a melting point curve. The lowest melting point on the melting point curve defines a eutectic mixture of lauric acid and stearic acid. (In the past, since the melting point of the eutectic is sharp, it was sometimes mistaken for a pure compound.) You can identify the eutectic’s approximate location from your graph of melting point temperature versus mole fraction. You may also be asked to determine the melting point of an unknown that is suspected of being pure lauric, pure stearic or a mixture of lauric and stearic acids.

![Lauric acid and Stearic acid structures](image)

Group assignments

Pair One will measure the following melting points:

Student #1
- pure lauric acid
- mixture of 20% lauric and 80% stearic acid by weight.

Student #2
- mixture of 80% lauric acid and 20% stearic acid by weight
- mixture that is 75% lauric acid and 25% stearic by weight.

Pair Two will measure the following melting points:

Student #3
- pure stearic acid
- mixture that is 25% lauric acid and 75% stearic acid by weight

Student #4
- mixture of 50% lauric acid and 50% stearic acid by weight
- mixture that is 41% lauric acid and 59% stearic acid by weight.
Investigation 1 Procedure (Common procedure for Students 1-4)

1. Crush the sample to be melted into a fine powder such that it will pack tightly into a capillary melting point tube. Invert the tube (open end down) and press the open end into the fine powder packing some into the tube.

2. Tip the tube upright (open end on top) and tap it gently on the counter to pack the material into the bottom of the tube. The packed sample height in the melting point capillary tube should be about 1–2 mm high; larger amounts will result in wider melting point ranges as the greater heat melts a larger sample. The solid can be packed even tighter by dropping the capillary tube down through a piece of glass tubing.

3. Insert both your thermometer (if necessary) and melting point capillary into the melting point apparatus. Heating the sample at too fast a rate will result in inaccurate melting points due to the thermometer and/or sample not being the same temperature as the heated block or bath. If the sample is near the melting point heat at a rate that is about 2 degrees per minute. To save time, if the sample is well below the melting point, you may heat the sample at a faster rate (3 to 6 degree per minute).

   Note: Accurate melting points with alcohol thermometers also require slower heating rates because the alcohol in the thermometer responds more slowly to temperature changes than a metal thermometer.

4. As soon as a sample has completely melted, remove the capillary melting point tube and turn the melting point apparatus heater off. Dispose of the used melting point tube in the (broken) glass disposal.

   Note: Waiting for a melting point apparatus to cool down so you can do another melting point is boring, so always turn off the heater as soon you are done to save time and energy.

Investigation 1 Data Collection and Analysis

1. Work as a team to collect and share the following data:

<table>
<thead>
<tr>
<th>Pair #, Student #</th>
<th>Sample ID</th>
<th>Mixture (Weight %)</th>
<th>Mole Fraction Lauric Acid in Stearic Acid</th>
<th>Melting Point</th>
</tr>
</thead>
<tbody>
<tr>
<td>P#1, S#1</td>
<td>LA</td>
<td>100 % Lauric Acid 0% Stearic Acid</td>
<td></td>
<td>1.0</td>
</tr>
<tr>
<td>P#1, S#1</td>
<td>20LA80SA</td>
<td>20% Lauric Acid 80% Stearic Acid</td>
<td></td>
<td></td>
</tr>
<tr>
<td>P#1, S#2</td>
<td>80LA20SA</td>
<td>80% Lauric Acid 20% Stearic Acid</td>
<td></td>
<td></td>
</tr>
<tr>
<td>P#1, S#2</td>
<td>75LA25SA</td>
<td>75% Lauric Acid 25% Stearic Acid</td>
<td></td>
<td></td>
</tr>
<tr>
<td>P#2, S#3</td>
<td>SA</td>
<td>0% Lauric Acid, 100% Stearic Acid</td>
<td></td>
<td>0.0</td>
</tr>
<tr>
<td>P#2, S#3</td>
<td>25LA75SA</td>
<td>25% Lauric Acid, 75% Stearic Acid</td>
<td></td>
<td></td>
</tr>
<tr>
<td>P#2, S#4</td>
<td>50LA50SA</td>
<td>50% Lauric Acid, 50% Stearic Acid</td>
<td></td>
<td></td>
</tr>
<tr>
<td>P#2, S#4</td>
<td>41LA59SA</td>
<td>41% Lauric Acid, 59% Stearic Acid</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
2. Construct a graph of melting point temperature versus mole fraction of lauric acid for the lauric acid stearic acid system. To do this, calculate the mole fraction of lauric acid in the lauric acid/stearic acid mixtures for each of the above weight percent ratios. (If you need help calculating the mole fraction, consult a textbook, partner, or instructor.)

Investigation 1 Questions

*Note: Answers to the questions can be discussed with your partner and group members but must be written in your own words.*

1. Based upon the graph of melting point versus mole fraction, answer the following questions:
   a. What does the addition of 20% stearic acid do to the melting point of pure lauric acid?
   b. What does the addition of 20% lauric acid do to the melting point of pure stearic acid?
   c. What is the melting point of a 50/50 mixture of lauric and stearic acid stearic acid? Why is this melting point not predictable from the melting points of the pure compounds?
   d. What is the approximate composition of the low melting eutectic for this lauric acid, stearic acid system? What other mole fraction mixtures should you test to establish the composition of the low melting eutectic mixture of this lauric acid and stearic acid system?

2. The two compounds in this mixture, lauric acid and stearic acid have properties of both their function groups—the carboxylic acid, the COOH group, at one end and the hydrocarbon functional group, CH3(CH2)x, at the other end. The long hydrocarbon chains on the ends are hydrophobic, but the carboxylic acid functional groups are capable of hydrogen bonding with each other as “dimers” as illustrated below. (The broken line refers to the hydrogen bond.)

How many unique dimer structures are possible from the mixture of lauric acid and stearic acid if two fatty acids are melted and mixed together in the liquid phase? Draw the dimers below.

*Note: The very broad hydrogen bonded H—O stretching absorption of the carboxylic acid in infrared (IR) spectroscopy is evidence of this dimer formation. Be sure to recall that characteristic of carboxylic acids if you have already studied Infrared (IR) Spectroscopy or recall this dimer structure when you study IR spectroscopy of carboxylic acids and other functional groups.*
Melting Point Investigation 2

Melting point is the key property behind the behavior of a fascinating group of compounds called liquid crystals. In 1888, Frederick Reinitzer, an Austrian botanist was studying the compounds formed from two hydrophobic natural materials, (fatty acids of oils and cholesterol.) His careful study of the melting behavior of pure “cholesterol benzoate” showed it consistently changed at 145 °C from solid crystals to a milky solution which upon further heating consistently changed to a clear solution at 179 °C. Reinitzer called the milky phase the “liquid crystal phase” after also discovering other compounds with similar properties. Today, liquid crystals are common and discovering new liquid crystal compounds with unique properties as well as new liquid crystal applications is a current area of research. Two compounds similar to the first liquid crystal compound discover by Reinitzer are cholesteryl pelargonate and cholesteryl oleyl carbonate. The structural formulas of these compounds and their respective reported phase transitions are below.

Pre-Activity Questions

Note: Answers to the questions can be discussed with your partner but must be written in your own words.

For Pair #1 (Students #1 and #2): Based upon the melting point behavior of mixtures, at what temperature would you expect a mixture of 0.125 g oleyl cholesteryl carbonate and 0.050 g cholestryl pelargonate to melt? Give a reason for your guess. Is your guess significantly above room temperature? Why or why not? (You may predict either the melting point to the liquid crystal phase or the clear (isotropic) liquid phase.)

For Pair #2 (Students #3 and #4): At what temperature would you expect a mixture of 0.050 g oleyl cholesteryl carbonate and 0.125 g cholestryl pelargonate to melt? Give a reason for your guess. Is your
guess significantly above room temperature, why or why not? (You may predict either the melting point to the liquid crystal phase or the clear (isotropic) liquid phase.)

**Procedure**

For Pair #1 (Students #1 and #2): Combine 0.125 grams oleyl cholesteryl carbonate and 0.050 g cholesteryl pelargonate in a small beaker or vial, (50mL or less) warm and melt on a hot plate and swirl to mix together or stir with a stirring rod. Once the mixture has melted together, remove the melted mixture from the hot plate and observe as it cools on black background.

For Pair #2 (Students #3 and #4): Combine 0.050 grams oleyl cholesteryl carbonate and 0.125 g cholesteryl pelargonate in a small beaker or vial, (50mL or less) warm and melt on a hot plate and swirl to mix together or stir with a stirring rod. Once the mixture has melted together, remove the melted mixture from the hot plate and observe as you allow it to cool on a black background.

**Post-Activity Questions**

*Note: Answers to the questions can be discussed with your partner and team but must be written in your own words.*

For Pair #1 and Pair #2 as a team:

1. What is observed in terms of solid to liquid and/or liquid to solid transitions?
2. What special effect is observed?
3. Does this special effect take place in the clear liquid (isotropic) phase, the liquid crystal phase or the solid phase?
4. Liquid crystal compounds that are chiral exhibit twisted conformations like a screw axis, where each layer of the aligned rod like (liquid crystal) molecules is turned relative to the next layer. Temperature and the energy of the molecules determine the amount of rotating (turning) between successive layers. The distance (pitch) between matching identical orientations (of the rod shaped molecules of the screw) corresponds to the wavelength of reflected light.
   a. What are the observed color changes (upon cooling) and why do they occur in the order observed?
   b. If you have begun the study of chiral compounds, place a star on at least two of the centers of chirality in cholesteryl pelargonate shown below:
5. Which of the mixtures (Pair #1 or Pair #2) should pack together tighter based upon the structure of the different groups (pelargonate, or oleyl carbonate) on the common cholesteryl group? Describe how tighter packing affects the observed transitions.

For Your Information: For full credit you must submit the Data Table, your Graph of Melting Point vs Mole Fraction Stearic Acid in Lauric Acid, and answers to all questions from Investigation 1 and Investigation 2. (Answers to the questions can be discussed with your partner and group members but must be written in your own words.)