CHEM104 Experiment: Physical Characteristics of Organic Compounds 1/26 & 1/27

In this laboratory activity we will explore the properties of density, melting point, boiling point, miscibility, and solubility. Each student will work independently with two compounds: one liquid (limonene or ethyl lactate) and one solid (camphor or vanillin). When you finished with your individual analysis you will meet as a group of four students perform additional analyses. You will then compile and compare your group data. Your data will be entered into a spreadsheet before the end of the period. You will work on the assignment questions as a group until the laboratory time has ended.

Background:

Density is usually given in units of g/cm^3 which is equivalent to g/milliliter or g/ml. Physically, the density is a measure of how closely the individual molecules are packed together in a solid, liquid, or gas. Practically, the density gives us an idea of the compound is more or less dense than water (density of about one g/cm^3 at room temperature).

Physically, the melting temperature is a measure of how much energy is needed to disrupt the solid form of a substance to change it into a liquid. The higher the melting temperature, the more tightly packed together is the solid form. Compounds with a melting temperature above 25 ^oC are typically solids at room temperature although "solids" may take on several different forms. Technically, the melting temperature or "point" of a solid should be represented by a narrow range of temperatures. **It is considered to be scientifically inaccurate to represent a melting "point" as a single temperature.** On the other hand, the melting range of a pure compound should not be more then 5 ^oC, for example 85.5–88.0 ^oC.

The melting points are characteristic of a compound but the melting point itself is not a unique characteristic of the compound. That is, two different compounds could have the same melting point, but two substances of differing melting point are unlikely to be the same compound.

The boiling point is a measure of how strongly the molecules are associated with each other in the liquid form. The more the liquid molecules are attracted to each other the more energy is required to pull them apart to form a gas. Specifically, the boiling point is the temperature at which the vapor pressure of a liquid equals the atmospheric temperature (therefore bubbles are formed). The discussion of boiling "point" versus boiling "temperature range" for pure compounds is similar to that of the previous melting point discussion. However, boiling points are often represented as a single temperature in the literature rather than a range of values.

Solubility characteristics are critical to a compound's chemical reactivity and biological activity. Usually water solubility is the most common value given in databases. The units of solubility are given in mass per unit volume such as g/ml. The temperature at which the solubility is recorded is usually given. As a rule, water solubility increases with temperature. A compound may be considered to be "somewhat soluble" in water between 0.02 and 0.1 g/ml solubility. Above 0.1 g/ml solubility indicates that the compound is "soluble" in water. There are some structure reasons having to do with bond polarity (Zumdahl Sections 12.2 & 12.3) and hydrogen bonding (Zumdahl Sections 14.3 & 15.1) that explain the solubility behavior of a particular compound.

Water solubility is most important because biological systems tend to occupy aqueous environments. However, the compound's solubility in other common laboratory solvents is also very useful.

The solubility of one liquid in another liquid is commonly referred to as miscibility. Immiscible liquids form two layers when mixed together. Miscible liquids form only one layer when they are mixed no matter what their proportions are.

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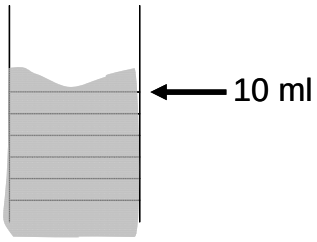
Liquid: _____ Solid: _____

Experimental Procedure:



I. Density:
The density of a liquid can be determined by obtaining the mass of a known volume of liquid.
Obtain a sample of your liquid in an Erlenmeyer flask.

Zero the balance before each measurement.
Weigh a clean dry 10 ml graduated cylinder: _____
Add your liquid to the 10 ml graduated cylinder to the 10.0 ml line.
Use a plastic pipette to transfer the liquid.
Be sure the bottom of the meniscus is on the line →
Obtain the mass the graduated cylinder and 10.0 ml of liquid: _____

Calculate the density of your liquid in g/ml: _____

Observations:

II. Melting point
The melting point of a solid is determined with the compound loaded in a tube.
Obtain a sample of your solid in a 20 ml vial.

The melting point tube is a capillary of about 1mm internal diameter, sealed at one end.
The open end of the tube is pushed onto the solid.
The solid that sticks inside is then shaken to the bottom by tapping the bottom of the tube on a hard surface. (Camphor may require a syringe plunger to poke it to the bottom.)
The height of the sample in the tube is should be 1-2 mm.

Place the sample-loaded capillary tube in the melting point apparatus.
Observe the solid as it heats.
Record the temperature (nearest 0.1 ^{o}C) at which the solid begins to melt. _____

Record the temperature (nearest 0.1 ^{o}C) at which the solid is completely melted. _____

Load another capillary tube and let the melting point apparatus cool to at least 10 ^{o}C below the melting point of the solid. Perform at least two trials
Record the temperature (nearest 0.1 ^{o}C) at which the solid begins to melt. _____

Record the temperature (nearest 0.1 ^{o}C) at which the solid is completely melted. _____
Do a third trial if necessary.
Observations:

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III. Boiling point

The boiling point of the liquid will be measured by placing about 5 ml (measure with a 10 mL graduate) of the liquid in a test tube.
Add one boiling chip to the liquid.
A thermometer will be placed in the liquid and the test tube will be heated in a hot oil bath.
Record the temperature when the liquid starts to boil: _____

Record the temperature after the liquid has been boiling for 60 seconds: _____

Prepare another test tube and let the oil bath cool to at least 10 ^{o}C below the boiling point of the liquid.
Record the temperature when the liquid starts to boil: _____

Record the temperature after the liquid has been boiling for 60 seconds: _____
Do a third trial if necessary.
Observations:

IV. Solubility

Estimate the solubility of your solid in water.
Measure 5 ml of water in a test tube with a graduated cylinder.
Weigh out 0.02-0.03 g of your solid compound on piece of creased weighing paper.
Add the solid to a test tube and shake. Does it dissolve? _____
If "yes" then add 0.09 – 0.11 g of your solid to the same test tube. Does it dissolve? _____
If "yes" then add 0.39 – 0.41 g of your solid to the same test tube. Does it dissolve? _____

Observations:

Estimate the solubility of your solid in hexane.
Measure 5 ml of hexane in a test tube with a graduated cylinder..
Weigh out 0.02-0.03 g of your solid compound on piece of creased weighing paper.
Add the solid to a test tube and shake. Does it dissolve? _____
If "yes" then add 0.09 – 0.11 g of your solid to the same test tube. Does it dissolve? _____
If "yes" then add 0.39 – 0.41 g of your solid to the same test tube. Does it dissolve? _____

Observations:

V. Miscibility:

Obtain a clean dry 20 ml vial for this procedure
Add 5 ml of your liquid to 5 ml of water. How many layers form? _____

Add 5 ml of your liquid to 5 ml of hexane. How many layers form? _____

Observations:

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Group work (to be completed by your assigned group of four):

VI. Mixed melting temperature.

Obtain a mortar and pestle for this experiment.

Mix two solids and grind together before determining the melting temperature (combined mass of 0.5 to 1.0 g).

Mass of vanillin _____ Mass of camphor _____

Record the temperature (nearest 0.1 ^{o}C) at which the solid begins to melt. _____

Record the temperature (nearest 0.1 ^{o}C) at which the solid is completely melted. _____

Observations:

VII. Mixed boiling point.

Mix two liquids in a test tube (total volume between and 5 and 10 ml) and determine their boiling point.

Volume of limonene _____

Volume of ethyl lactate _____

Record the temperature when the liquid starts to boil: _____

Record the temperature after the liquid has been boiling for 60 seconds: _____

Observations:

Before you leave lab: enter all your previous data on a spreadsheet:

Density: Limonene _____ Ethyl Lactate _____

Two melting temperatures: Vanillin 1 _____ Camphor 1 _____
Vanillin 2 _____ Camphor 2 _____

Two boiling temperatures: Limonene 1 _____ Ethyl Lactate 1 _____
Limonene 2 _____ Ethyl Lactate 2 _____

Solubility ("yes" or "no")
Vanillin in Hexane 0.02-0.03g _____ 0.09-0.11g _____ 0.39-0.41g _____
Vanillin in Water 0.02-0.03g _____ 0.09-0.11g _____ 0.39-0.41g _____

Camphor in Hexane 0.02-0.03g _____ 0.09-0.11g _____ 0.39-0.41g _____
Camphor in Water 0.02-0.03g _____ 0.09-0.11g _____ 0.39-0.41g _____

Miscibility: ("1" or "2)
Limonene & Hexane _____ Ethyl Lactate & Hexane _____
Limonene & Water _____ Ethyl Lactate & Water _____

Mixed melting temperature _____ Vanillin g _____ Camphor g _____

Mixed boiling temperature _____ Limonene ml _____ Ethyl Lactate ml g _____

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Postlab Questions:

Data Summary:

Your liquid: _____

Your density _____

Class average of density _____

Literature value (Wikipedia) of density _____

Your solid: _____

Your melting temperature ranges: _____

Class average melting point _____

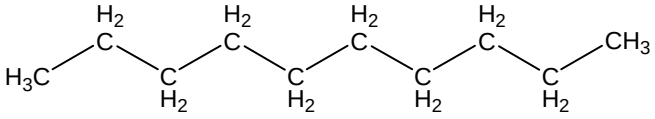
Literature value (Wikipedia) melting point _____

Your boiling temperature ranges: _____

Class average melting point _____

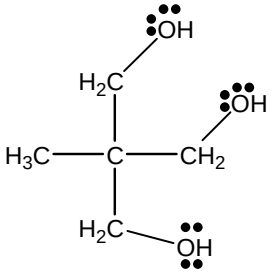
Literature value (Wikipedia) boiling point _____

1. Compare the structure of limonene with decane. How do the boiling temperatures compare? Explain why they are different or similar.



decane - boiling point 174.1 ^{o}C

2. Compare the structure of ethyl lactate with 1,1,1-tris(hydroxymethyl)ethane. How do the boiling temperatures compare? Explain why they are different or similar.



1,1,1-Tris(hydroxymethyl)ethane
boiling point 200 - 203 ^{o}C

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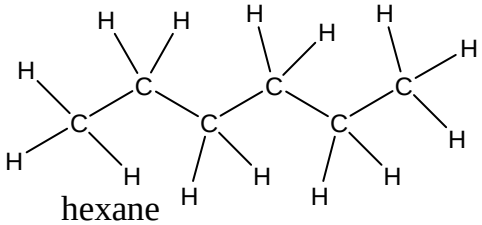
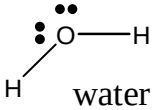
3. Draw the structure of your solid and compare it to the structures of hexane and water. How do structures help explain solubility observations?

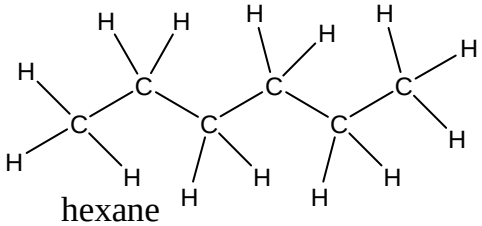


hexane

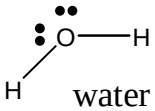


water

4. Draw the structure of your liquid and compare it to the structures of hexane and water. How do structures help explain miscibility observations?



hexane



water

5. In the miscibility experiment how could you determine which liquid is on top if two layers are formed?

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Prelab Questions:

Look up physical data for the following four compounds:
use two different sources: Wikipedia, ACROS Organics, Sigma-Aldrich, etc…

boiling point of limonene _____ _____
other names: (R)-(+)-limonene, (+)-limonene, or D-limonene

boiling point of ethyl lactate _____ _____
other names: ethyl (-)-L-lactate, (-)-ethyl L-lactate

melting point of camphor _____ _____
other names: DL-camphor

melting point of vanillin _____ _____
other names: 4-hydroxy-3-methoxybenzaldehyde

Why do you think the literature values for the same compound differ from each other?
("Human error" is not an acceptable answer.)

Draw the Lewis structures of limonene, ethyl lactate, vanillin, and camphor. Show all atoms, bonds, and non-bonding valence electrons.

Safety: What two things should you do if a melting point capillary accidently breaks in your hand and pricks your finger?