Experiment 1: (Density, melting point and refractive index) Mohrig, Chapter 10, 13

Theory: These topics have been covered in general chemistry. However, in **Chem 6A** we use these measurements to identify organic compounds, and in some cases, approximate purity.

A. Density (sample identification)

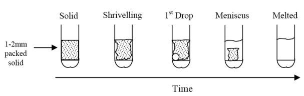
Density can be measured in g/mL or mg/mL. It is a very useful property for identification purposes. Be sure to discuss how to measure the volumes of gases, liquids and solids and how to measure the masses of solids, liquids and gases. Density is determined by finding the weight of a specific volume of a liquid. To do this, we will use 1-mL plastic disposable syringes [no needle] to precisely measure 1 mL. The technique involves 1) weighing the empty syringe, 2) filling the syringe to 1 mL with any of the organic liquids (toluene, cyclopentanone and ethyleneglycol) supplied in the laboratory, and then 3) weighing the liquid plus the syringe. The difference is the weight of 1 mL of the liquid or the density is given by the weight of 1 mL. This part of the experiment also involves determining the density (mg/mL) of a 10 wt% salt water solution? What is the density (mg/mL) of a 1M salt water solution?

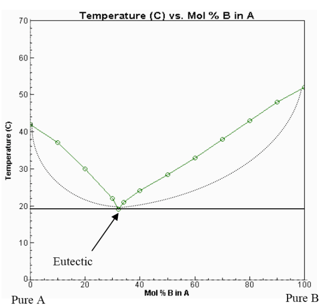
B. Melting point (sample purity and composition)

This experiment is designed to 1) introduce students to the use of a typical "melting-point apparatus," which you will use repeatedly throughout **Chem 6A-B**, 2) to demonstrate that pure compounds have "sharp" melting points and melt over a small temperature range, 3) to demonstrate how an impurity lowers the melting point of a substance and broadens its melting range, and 4) to explain how a "mixed melting-point" procedure can be used to find the eutectic point. The diagram that is constructed can be used to the ratio of components in binary mixtures.

Mp apparatus

This part of the experiment involves determining the melting point of ninemixtures of **cinnamic acid** (mp. 133-134 , MW = 148.16 g/mol) and **urea** (mp. 132-135, MW = 60.06 g/mol). Students will construct a melting point versus composition diagram. Fill a closed capillary with one of the samples to be measured (about 1/8 to 1/4 inch of crystals), and determine the melting point range (show your students how to fill the capillary). Determine the melting point range of a pure compound and the melting point range of a mixture of that compound with another compound. Construct a melting point vs percent composition diagram (either "percent by weight" or "mole percent"). A mixture has a wider melting range and the starting temperature is considerably lower than for pure material. Inevitably, it is more difficult to determine when the melting starts as opposed to when it concludes. The remWatch for shriveling and then record the start at the first sign of a liquid at the edges of a crystal. The end point is recorded when the entire sample is a liquid. The following chart should help you translates the mol % and the wt % of the cinnamic acid/urea mixtures. The weight percent should be on the bottles. The eutectic point is 111 °C with a 63 wt % which equals 41 mol % of cinnamic acid. The distance between the dashed and solid lines indicates the melting range. MW of urea is 60.06 and *E*-cinnamic acid is 148.16.





wt. % of cinnamic acid **A** /urea **B**	mol %
0 (1000 mg B)	0
10 (100 mg A + 900 mg B)	4.3
25 (250 mg A + 750 mg B)	12
50 (500 mg A + 500 mg B)	29
63 (630 mg A + 370 mg B)	41
71 (710 mg A + 290 mg B)	50
85 (850 mg A + 150 mg B)	70
90 (900 mg A + 100 mg B)	78.4
100 (1000 mg A)	100

C. Refractive Index (sample purity and composition)

Not used in many labs, but plays an important role in HPLC detection for compounds.

Lecture Ideas

Explain melting point

Melting occurs when a compound is at the temperature where the solid and liquid phases are in equilibrium. Most pure organic compounds melt over a 'sharp and narrow' range spanning 1-2 °C. This melting range gives us an idea of purity, because the melting point decreases "almost" linearly as the amount of impurity increases.

Explain the eutectic point

The low point in the melting point phase diagram occurs at a very specific ratio of mixtures of compound A and B. This point is called the eutectic point or eutectic temperature. The eutectic mixture is the composition of the mixture of A and B at the eutectic point. At the eutectic point, both compounds are melting simultaneously, resulting in a sharp melting point rather than the broad melting point typically seen for impure compounds. A eutectic mixture is misleading in that it suggests a pure compound!

Explain concept of a mixed m.p.

Give access to the known pure substance, a mixed melting-point can be used to identity an unknown pure compound. Suppose we suspect the unknown to be benzoic acid (m.p. 120–121 °C). We find the mp to be 118–119 °C. This is close, but hundreds of compounds may have this melting-point. We mix a small amount of pure benzoic acid with our unknown. If the melting point is unchanged then we know that the pure unknown compound is benzoic acid. If the "unknown" was not benzoic acid, then benzoic acid would have acted as an impurity, and the m.p. would have been lowered and melted over a broader range.

Explain Refractive Index (N).

$N = \frac{c}{V}$ Defined as the relative speed at which light moves through a material with respect to its speed in a vacuum. The index of refraction, N, of transparent materials is defined through the equation shown above. c = $3X10^8$, which is the speed of light in a vacuum and V is the speed of light in some other medium. Since the speed of light is reduced when it propagates through transparent gasses, liquids and solids, the refractive index of these substances is always greater than 1.0. If the refractive index of the product is 0.0010 below or above the literature value, it indicates that impurities are present.

Quiz ideas (1)

Calculate all molarity from weight % for 40 wt % KOH / H_2O.