CHEM 333
Organic Chemistry Laboratory
Truman State University

Crystallization and Melting Points

Crystallization is a fundamental technique for purifying compounds. In this experiment, you will perform two crystallizations and use the property of melting point to determine the purity of your sample.

New techniques:

Crystallization
Melting points
Unknown identification

Reading (in Pavia)

Recrystallization: chapter 11, pp. 139-147, 153-161.
Melting points: chapter 9, pp.116-120, 121-125.

Prelab: Ascertain the structures, molecular weights and densities of the materials used in this lab. In your lab notebook, outline a basic procedure you will follow.

Part A: Recrystallization of phthalic acid

Compute the approximate amount of water needed for dissolving 1 gram of phthalic acid at 100° C using the following solubility data: in water, 0.54^{14}, and 18^{99}. ("Solubility" is usually reported in grams of solute per 100 mL of solvent at a particular temperature. The particular temperature is represented as a superscript. Thus, 0.54 g of phthalic acid dissolves in water at 14° C.) Knowing this, choose an Erlenmeyer flask that holds at least twice that volume and dissolve about 1 g (record the exact mass used to 3 decimal places) in a minimum amount of boiling water in that flask. Be sure to add a boiling stick when you start adding the water, and also to keep the mixture near the boiling point during the dissolving process. (You do *not* need to keep track of the amount of water added) Decolorize and/or filter any solid impurities from the solution, if necessary. After filtration, you may need to reduce the volume of the solution to make it saturated again. Allow the saturated solution to cool very slowly, without disturbing the flask, to room temperature to grow the best crystals. Cool further in an ice bath for at least 10 minutes. Collect the crystals using suction filtration. After the crystals are very dry (next lab period) measure the mass of the isolated crystals, calculate the % recovery, and measure the melting point. Turn your crystals in for grading.

Part B: Recrystallization of benzoic acid

Recrystallize ~0.25 g (again, record the exact mass to 3 decimal places) of benzoic acid from water using the same procedure that you used for the phthalic acid. Since you do not

have the same data for the solubility of this substance, be sure to add the solvent slowly, allowing time between each addition for the solid to dissolve.... After the crystals are very dry (next lab period) measure the mass of the isolated crystals, calculate the % recovery, and measure the melting point. These crystals will be turned in for grading.

Cleanup. The aqueous mother liquor should be poured down the drain with lots of water.

Part C: Melting Points

1) Determine the melting point of your recrystallized samples from parts A and B above using the method given in recitation.
2) Determine the melting point of the prepared mixtures of urea and cinammic acid of known proportions to see the effect of impurities on the melting point.
3) Use the melting point of an unknown compound by repeated melting point determination or by mixed melting point.

For the lab report:

1) Give your percent recovery of phthalic and benzoic acid (show your calculations), your observed melting points for these two compounds, and the observed melting points for the urea/cinammic acid mixture and your unknown (as well as your unknown's identity).
2) Offer one or two suggestion on how your percent recovery could be improved.
3) Comment on the purity of your recrystallized compounds.
4) Briefly explain how you determined the identity of your unknown.