Recrystallization

Objectives:

1. To purify a compound by recrystallization
2. To evaluate the effectiveness of the recrystallization by comparing melting points.

Discussion: Recrystallization

Most reactions in organic chemistry result in a mixture of the desired product and smaller amounts of contaminants (consisting of unreacted starting material or products of other competing reactions). The job of the organic chemist is to separate the pure desired product from the contaminants. The resulting compound must be pure enough for analysis, characterization, or for use in manufacturing, depending on the reaction involved. There are many techniques used for separation and purification, and selecting one for a particular situation requires a thorough knowledge of the physical and chemical properties of the substances, as well familiarity with various instrumental and traditional lab techniques.

Recrystallization is the most common technique used to purify solid products contaminated with other solids. When an impure product is contaminated by a solid with different solubility characteristics, a solvent is chosen which will dissolve the desired compound but will not dissolve the impurity. (Example: salt contaminated by sand could be dissolved with water, leaving the sand behind.) When this situation exists, the solvent is added, the mixture decanted or filtered to remove the solid impurity, and the product recovered by evaporation of the solvent. Unfortunately, many times in organic chemistry, the impurity has about the same solubility characteristics as the product itself, and the situation becomes a little more difficult. In this case, the mixture is dissolved in a minimum amount of hot solvent. As the solvent cools down, the solution will become saturated in product, and fairly pure crystals of the product will begin to form. (The solution is usually not near the saturation point in contaminant, because the contaminant is present in much smaller amounts). The product will continue to crystallize and the contaminant remain in solution as the solution cools. Then the mixture is filtered and the crystals of product are washed to remove any traces of contaminant clinging to them. A second “crop” of crystals may be obtained by boiling away some of the liquid, but this increases the chance that the contaminant will precipitate too.

Recrystallization to separate two similar solids is a widely-used technique, but it has two disadvantages. First, some contaminant does adhere to the crystals of product as they precipitate. This gives a product which is not completely pure since the contaminant becomes encapsulated in the forming crystals and cannot be rinsed off. The product can be recrystallized again, though, and each subsequent recrystallization yields a purer product. Second, the yield is not near 100% because a portion of the product remains dissolved in the liquid that contains the contaminant (and this is usually discarded). Despite these limitations, the method is routinely used to purify products.

However, if an attempt is made to recrystallize *very small amounts* of impure product, the product may be "lost", and not recrystallize from even a few drops of solvent. Newer methods (chromatography, for example) are used with extremely small samples.

In this experiment, you will be given a mixture of "product" and "contaminant" to separate, and told what solvent to use. You will take a melting point of your impure sample as received, and compare it to the value for the pure "product." Then you will recrystallize your "product" and check the melting point to evaluate your technique.

Procedure: Recrystallization:

1. Obtain a 0.5 gram sample of contaminated salicylic acid. Use a little of it to fill three melting point capillary tubes. (This is for comparison of the melting points before and after recrystallization.) Weigh the remaining sample plus container, empty the sample into a 50-ml Erlenmeyer flask, and reweigh the empty container. Subtract to obtain the exact sample mass. Pour about 12-14 ml of deionized water into the flask, and place the flask and contents on a wire gauze held by a ring and stand. Heat the mixture to boiling with a Bunsen burner, stirring with a glass rod until all of the solid is dissolved. Rinse the sides of the flask down with small amounts of deionized water from your wash bottle.

2. When all the solid has dissolved, shut off the Bunsen burner and allow the flask to cool. As it cools you will observe the crystallization of the pure salicylic acid. After the flask has cooled to room temperature, cooling in an ice bath will complete the process. Also chill a little deionized water in your water bottle for use in washing the crystals.

3. While the flask is cooling, rinse the container in which you received the crystals (save uncapped until the next lab period). Also take a melting point of the impure mixture, using the capillaries already prepared.

4. Obtain a Hirsh funnel and filtering apparatus and set it up as demonstrated, making sure to secure the filter flask and trap with clamps. Place the small filter paper disk carefully in the funnel, and when you are ready to filter, wet the disk with a little deionized water, turn on the suction (all the way), and pour the contents of the flask into the funnel. Use a spatula or the rubber "policeman" on the stirring rod to help transfer the crystals. (Be careful not to poke a hole in the paper.) Rinse the remaining crystals from your flask with a small amount of chilled water, and pour this onto the crystals in the filter to help rinse them. Continue rinsing the crystals with small amounts of chilled water to remove all of the mother liquor, which contains the contaminant (urea).

5. Draw air through the crystals in order to dry them as thoroughly as possible. Spread the crystals out on a watch glass and leave in your locker until the next lab period.

6. (Next lab period.) Weigh the empty dry container. Transfer your crystals to the container and reweigh. Subtract to get your "recovered amount" of salicylic acid. Calculate your percent recovery by using your original sample weight and the weight of your recovered (purified) sample. Take a melting point of your purified sample. Hand in your purified sample. Label the vial with your name, section number, and the compound name.

Name_____

Report: Recrystallization Section #_____

A. Impure salicylic acid

Sample Appearance:

Mass sample + container _____

Mass container _____

Mass sample _____

Melting point range _____

B. Purified salicylic acid

Sample Appearance:

Mass sample + container _____

Mass container _____

Mass sample _____

Melting point range _____

Literature melting point range for salicylic acid _____

Questions:

1. Calculate the percent recovery of salicylic acid. _____ percent

$$\% \text{ Recovery} = \frac{\text{g recovered (yield)}}{\text{g sample}} \times 100 =$$

Give 2 reasons why your percent recovery will be less than 100%. Be specific.

2. Compare the melting points of your impure and purified product. Did your attempt at recrystallization improve the quality of the product? _____ Explain.

3. Compare the melting point of your purified product with the literature value. Would a second recrystallization improve the quality? _____ Explain.