**RECRYSTALLIZATION AND IDENTIFICATION OF AN UNKNOWN**

**Required prelab readings**: Padias pages 121-129

**Previous techniques you must understand and be able to perform**: Melting points.

Recrystallization is the first of three new methods of purification you will be learning this term (recrystallization, chromatography and extraction). Recrystallization is probably the most important method for the purification of solids since it can be used in conjunction with other types of purification. Although recrystallization can be performed on virtually any amount of material, one significant advantage of this technique is that it can be performed on very large scale. Kilogram quantities of material can be purified by recrystallization. Purification of that much solid material by other techniques, chromatography for example, would be impractical. It is of utmost importance to learn this technique well since you will use it throughout the year of Organic Chemistry Lab and later in Inorganic Chemistry Lab.

**The General Recrystallization Technique:**

After an appropriate solvent (or solvent combination) is determined by trial and error, the solute is dissolved in a minimum amount of the solvent at or near the solvent’s boiling point. If undissolved impurities are present the mixture is gravity-filtered while still hot to remove the impurities. The filtrate is cooled (usually to 0 °C), sometimes scratched with a glass stirring rod to induce crystallization, and the crystals that form are collected by suction filtration and washed with small amounts of cold recrystallization solvent.

In this experiment youwill use the techniques of recrystallization and acquiring melting points to identify an unknown organic compound. Since you do not know the identity of the compound, you must first determine which solvent is appropriate for the recrystallization process. The solvents that you are to choose from are water, petroleum ether, methanol, toluene, and acetone. Recall that the organic solid should be soluble in the recrystallization solvent when the solvent is hot, and should be insoluble when the solvent is cool.

Once the compound has been recrystallized and dried, a melting point will be taken. You will then identify your compound by comparing the melting point of your compound to the literature melting points of the compounds listed on the third page of this experiment. Once you have decided on the identity of your compound, you must perform a mixed melting point of your sample with the compound that you believe the unknown to be to confirm your choice.

In this report, you will be graded on the purity of your compound after recrystallization, the percent recovery after the recrystallization, and the correct identity of the unknown compound.

**I. Choosing the recrystallization solvent**

• Fill a 250 mL beaker with approximately 100 mL of water and begin heating it on a hot plate to a gentle boil.

• Obtain 1.5 g of an unknown sample to be recrystallized. Write the code from the sample bottle in your lab notebook. Place approximately 0.1 g of the sample into five separate 13 x 100 mm test tubes labeled P, T, A, E, M, W (representing petroleum ether, toluene, acetone, ethanol, methanol and water.)

• Add approximately 2 mL (one Pasteur pipette full) of the appropriate solvent into each test tube. **Make a data table in your notebook** as shown below. You will record whether the compound is soluble or insoluble in each solvent at the various temperatures.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Solvent** | **Petroleum**  **Ether** | **Toluene** | **Acetone** | **Ethanol** | **Methanol** | **Water** |
| **Solubility at room temp** |  |  |  |  |  |  |
| **Solubility near solvent boiling point** |  |  |  |  |  |  |
| **Recrytstallization upon cooling** |  |  |  |  |  |  |

1. Swirl each test tube and record whether the compound is soluble or insoluble in each solvent at room temperature. If the compound is soluble at room temperature in any of the solvents, discard the contents of the test tube in the waste container and rule out that solvent as the appropriate recrystallization solvent for the compound.
2. For the remaining tubes, add a boiling chip and heat each tube in the boiling water bath. Be sure to point the tubes away from anyone in case the contents should spurt out while heating. The boiling points of petroleum ether (30-60 °C) and acetone (56 °C) are fairly low so be careful not to boil them to dryness. If you do boil them to dryness, you will need to add more solvent to finish the test. Record whether the compound is soluble near the boiling point of each solvent. If the compound is insoluble in any of the solvents near the boiling point, discard the contents of the tube and rule out that solvent as an appropriate recrystallization solvent for your compound.
3. Allow any remaining test tubes to cool to room temperature and record which test tube had the maximum recovery of crystals. The best recrystallization solvent should be the one in which the compound is insoluble at room temperature, but is soluble at the boiling point of the solvent, and gives the best return of crystals upon cooling.
4. Given this criteria, YOU need to make the final decision on which solvent to use to recrystallize the remaining 1g of compound.

**II. Recrystallization of the remaining sample**

1. Dispose of the remaining test tubes. Record the mass of the remainder of the original unknown compound and place it in a 50 mL Erlenmeyer flask.
2. Recrystallize the compound in the solvent chosen in the test tube analysis. Given that 0.1 g of the compound was tested in 2 mL of solvent in the test tube, calculate the amount of solvent that should be initially used for the amount of the original compound that remains.
3. Remember that when you obtain crystals by suction filtration, you should always wash the crystals with a small amount (1-2 mL) of ice cold solvent that you used for the recrystallization.
4. Record the melting point of the purified dry compound. Your unknown will be one of the compounds listed in the Table below. In order to unequivocally identify your sample you must also take a mixed melting point.
5. Place your sample in a vial labeled with your name, the sample name, sample mass, and sample melting point and turn it in to your instructor.

**Possible Unknowns**

|  |  |
| --- | --- |
| Name | Melting point, °C |
| *n*-Butyl-4-hydroxybenzoate | 67-71 |
| Phthalide | 71-74 |
| 4-Chlorobenzophenone | 74-76 |
| Methyl-3-nitrobenzoate | 78-80 |
| 4-Hydroxy-3-methoxybenzaldehyde | 81-83 |
| Ethyl-4-aminobenzoate | 88-90 |
| Tribenzyl amine | 91-94 |
| 5-Chloro-2-methoxy benzoic acid | 98-100 |
| 2-Methyl benzoic acid | 103-105 |
| Cholesteryl acetate | 112-114 |
| Ethyl-4-hydroxy benzoate | 114-116 |
| Mandelic Acid | 118-120 |
| Benzoic Acid | 122-123 |
| Sucralose | 125-128 |
| Malic Acid | 130-132 |

**PURIFICATION AND IDENTIFICATION OF AN UNKNOWN**

**DATA SHEET**

|  |  |  |  |
| --- | --- | --- | --- |
| **NAME:** |  | **Section:** |  |

|  |  |
| --- | --- |
| **Unkown #:**  **Unknown Name:**  **Mass of unknown recrystallized:**  **Mass of recovered unknown:**  **Percent recovery**  **Recrystallization solvent** | **Structure of unknown** |

**Melting Point (purified material):**

**Mixed melting point:**

**a) + Unknown:**

(known) (mixed mp)

**b) + Unknown:**

(known) (mixed mp)

**Percent Recovery calculation:**

**Post-Lab Questions**:

1. What is the purpose of recrystallization?

2. At room temperature what types of solid organic compounds are soluble in water, insoluble? Give specific examples of each with structures.

3. Why is gravity filtration unnecessary in this experiment?

4. You have allowed your solution to cool in an ice bath, and no crystals have formed. Assuming the solution is supersaturated, what can you do to get the crystals out of solution at this point?

5. The solubility of a compound is 10.1 g per 100 mL in boiling water, 4.0 g per 100 mL in cold water, whereas its solubility in ethanol is 9.0 g per 100 mL at 78 °C and 3.1 g per 100 mL at 2 °C. Which solvent would be better for the recrystallization of the compound? Explain.

6. Draw the structure of four benzoic acid molecules and as many water molecules as you need to illustrate how the solid state of benzoic acid might look in cold water prior to recrystallization. In a second drawing illustrate what happens if the water is heated to near 100 °C, where benzoic acid is soluble. In each drawing identify, by name, specific examples of the types of intermolecular interactions involved. Based on your drawings explain why benzoic acid is soluble in hot water and not soluble in cold water. Answer the question at the molecular leveldiscussing the specific molecular interactions involved in your drawings.