Recrystallization Lab (V-B)

Background Reading

Beran, J. Laboratory Manual for Principles of General Chemistry (10th Ed).
Laboratory Techniques: Technique 11A-E (Separating a Liquid or Solution from a Solid).
Zanger, M. and McKee, J.R. Small Scale Syntheses. pp 26 -28.
Zubrick, J. W. The Organic Chem Lab Survival Manual.
Ch 13: Recrystallization (ignore water traps & aspirators).

Key Words

recrystallization, crystallization, filtrate, solubility, immiscible

Experimental Data

- Discuss how recrystallization works as a purification method and the role of the solvent in your Principles section.
- Include acetanilide, benzoic acid, benzoin, and salicylic acid, along with the four solvents, in your Substances table.
- No reaction or mechanism section is needed.
- Illustrate and describe gravity filtration and vacuum filtration in your Apparatus section.
- Note the color, shape, and overall appearance of both sample and product crystals in the Observations section.
- In the Data section, create a **table** of the sample's solubility in the four solvents, as well as in the solvent pair (if used). Note their solubilities in the ice water bath, at room temperature, and in the warm water bath.
- Be sure to include your sample's 3-digit number.
- Record the identity and amounts of solvent used in step 9 through 11.
- Record sample mass used in step 9 and the mass recovered in part 17. Determine the recovery percentage based on these values.
- Use the MP range to determine the unknown's identity. (See table on last page.)
- Confirm the unknown's identity with a mixture melting point test.

Procedure

1. Obtain an unknown and record its ID number. Grind unknown to powder by placing inside a folded piece of filter paper and crushing with a small Erlenmeyer flask.

2. Obtain four small (3") test tubes with a rack. Add approximately 50 mg of unknown to each tube. Add 2 ml of water to one test tube. Add 2 ml of 95% ethanol to the second test tube, petroleum ether to the third, and toluene to the fourth.

3. Agitate the tubes with a glass stirring rod and observe the solubilities at room temperature.

4. Place any test tubes with undissolved sample in a warm water bath using a 250-ml beaker on a hot plate. For ethanol and petroleum ether, keep bath T below 60 ^{o}C. The other two solvents can be heated to 80 ^{o}C. Keep the test tubes off the bottom of the beaker, so that they are not heated excessively. Observe the respective solubilities.

5. Cool all test tubes in an ice water bath. Observe the respective solubilities, that is if solid appears. Make a table showing solubilities with each solvent at each temperature.

6. If sample is soluble in one solvent at room temperature or higher, but insoluble in that same solvent when cold, skip steps 7 and 8. Verify the solvent's properties with a second test, and then use that solvent as the recrystallization solvent if its properties are ideal. Otherwise, use a solvent pair as described.

7. Use data collected in steps 3 through 5 to select a solvent pair. One solvent should dissolve the solid easily when warm, while the other solvent should dissolve the solid poorly. Also, the solvents must be at least partially miscible. For instance, water is completely immiscible with toluene and petroleum ether, so water cannot be combined with either solvent.

8. Proportions should be adjusted so that solid will dissolve completely when warm, but will crystallize when cooled. For instance, if the unknown dissolves well in ethanol at all temperatures, addition of water may improve crystallization when cold.

9. Weigh ~ 2 g of solid, and add to a 50-ml Erlenmeyer flask, along with a boiling chip and 10 ml of your recrystallization solvent. Warm flask in a hot water bath, and stir occasionally, until solid is just completely dissolved. Add more solvent in 1-ml increments as necessary.

   These dissolved crystals will be purified by recrystallization. In that process, insoluble contaminants (if visible) will be filtered out, while very soluble contaminants will remain dissolved with the solution after crystallization of the product. This will effectively remove all contaminants with different solubility properties than the product compound.

10. Add 1-2 ml of recrystallization solvent, so that solution is no longer saturated and will not crystallize during filtration.

11. If any undissolved solid contaminants are visible, remove by decanting or filtering. Hot fluids are always filtered only by gravity filtration using a stemless funnel. Büchner funnels and stemmed glass funnels will become clogged due to crystallization in the stem. If product crystallizes on filter paper, use a small amount of clean, hot solvent to dissolve it. Adjust the proportions of the two solvents in the solvent pair if needed.

    Save the filtrate to obtain the product crystals in the following steps.

12. Obtain a small amount of decolorizing charcoal (30 - 50 mg). Add charcoal slowly and carefully to minimize foaming. Cooling filtrate first, if possible without crystallization, will also minimize foaming. Observe that the charcoal remains solid and does not dissolve.

13. Warm solution in a water bath, then either gravity filter with a stemless funnel or decant into a 50-ml Erlenmeyer flask. Cover flask immediately after filtration to prevent solvent vaporization.

14. Allow crystals to form. Place flask in ice-water bath to improve crystallization, if necessary. Observe the crystals' appearance. They should be white if the colored impurities have been removed.

15. Collect the product crystals by vacuum filtration using a 70-mm filter paper and Büchner funnel. Weigh the filter paper prior to collecting crystals, so that the yield can be obtained. Decant most of the liquid into the funnel prior to transferring the solid crystals into the funnel. This will to prevent the filter from clogging before the solvent has passed through.

    After pouring sample in funnel, add 1-2 ml of ice-cold solvent to wash the product. This wash will remove the mother liquor (residual liquid with impurities) from your sample. Be sure to cool the wash solvent in an ice-water bath first so that it does not dissolve your product also!

16. Dry the crystals and filter paper on a watch glass. A 50 ^{o}C oven may be used to evaporate water or ethanol, but do not use the oven to evaporate toluene or petroleum ether. Air-dry these two solvents in the hood only. Dry overnight at room temperature if necessary.

17. Weigh the dried product crystals. Obtain recovery % as the product crystal mass divided by the sample mass dissolved.

18. Obtain MP range of crystals with at least two trials after weighing them. Match the MP range obtained with the values in the following table (next page). Then, perform a mixture melting point test to confirm the substance's identity by melting a mixture of the crystals with a reference sample of the substance.

Waste Disposal

Place all solutions, charcoal, and crystals in the appropriately-labeled waste container(s). Place filter paper in a trash receptacle after all of the crystals have been scraped off of it. Place used capillary tubes in the broken glass container.

Melting Point Ranges for Unknowns

Acetanilide	113 – 115 ^{o}C
Benzoic Acid	121 – 123 ^{o}C
Benzoin	134 – 138 ^{o}C
Salicylic Acid	158 – 161 ^{o}C

Post-Lab Q's

1. How does the recrystallization process remove both insoluble and soluble impurities from a compound?

2. How would the reduced pressure of a vacuum filtration affect a hot volatile solvent? How would this affect the amount of solute that remains dissolved in the solvent? What would be the potential consequence to such a filtration in step 11?

3. What is a boiling chip and what is its function? What could happen if you boil a liquid without a boiling chip?

4. What is the purpose of the charcoal in step 12? What is the potential consequence of adding too much charcoal or adding charcoal too quickly?

5. What is removed by washing the crystals in step 15?