Experiment 7

Recrystallization: Purification of Crude Benzoic Acid and Phenanthrene

Study Questions

1) What effect would each of the following operations have on the success of the recrystallization of benzoic acid from hexanes? Explain your answers.

   a. The hot solution containing the dissolved benzoic acid is immediately placed in an ice bath. **Answer:** The crystallization will be less successful because the product will be less pure. There will be a rapid formation of solid material with inclusion of impurities rather than a true crystallization. The compound will form irregular, small crystals, not in the proper lattice structure, including materials not normally part of the lattice. Slow cooling encourages pure crystal formation.

   b. After recrystallization has taken place, the cold solution is vacuum filtered and product crystals are collected on a Buchner funnel, then the crystals are washed with hot hexanes. **Answer:** Product will be lost. The crystals would redissolve in the hot hexanes instead of remaining on the filter paper on the Büchner funnel, causing loss of product.

   c. After isolation of the benzoic acid crystals on a Büchner funnel, they are washed with diethyl ether. **Answer:** The crystals may redissolve in the new solvent, or even react with it. It might be okay to rinse with a different solvent, such as diethyl ether, if you know that the compound is totally insoluble in the new solvent.

2) A student crystallizes 5 g of a solid and isolates 3.5 g as the first crop. She then isolates a second crop of 1.2 g solid from the filtrate.

   a. What is the percent recovery in the first crop? **Answer:** 3.5/5.0 = 0.70 or 70%.

   b. What is the total percent recovery? **Answer:** 4.7/5.0 = 0.94 or 94%.

3) The solubility of acetanilide in hot and in cold water is given in the table below. What is the maximum percent recovery if 5.0 g of acetanilide is recrystallized from 100 mL of water?

Solubility (in 100 mL of water)	Temperature
5.5 g	100°C
0.53 g	0°C

   Answer: If 5 g of acetanilide is dissolved in 100 mL of water, it will all go into solution. When the solution cools, 0.53 g of this 5 g will not come out of solution; it will remain dissolved in the cold solvent. The amount of acetanilide that will come out of solution is (5 - 0.53) or 4.47 g. The maximum percent recovery is then 4.47/5 = 0.89 or 89%.

4) The CRC lists the melting point for a compound as 182-183°C. You observe a melting point for

this same compound isolated in your experiment as 177-181°C.What can you conclude about the compound isolated in your experiment? **Answer:** It is a little bit impure. The melting point range is both lower and wider than the literature value. It is close enough to the literature value to indicate that it is probably the compound you intended to isolate.