

Experiment 5

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?

<i>Solubility (in 100 mL of water)</i>	<i>Temperature</i>
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

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that it is probably the compound you intended to isolate.