

**Exercise 7**

Fig. X7-1

**Choosing Laboratory Procedures**

Decisions: Should you take the high road or the low road?

**A. Separation and purification.** The techniques of evaporation, filtration (gravity and vacuum), recrystallization, extraction, chromatography (thin layer, column and gas) and distillation (simple, fractional, vacuum and steam) are commonly used for separation and purification. For each problem below, state which method you would try first. Explain your choice.

- a. 10 mL of a 1:1 mixture of methanol and 1-butanol
- b. 10 mL of a 1:1 mixture of methanol and ethanol
- c. 2 grams of aspirin contaminated with sand and naphthalene
- d. The amino acids that result from the breaking of the amide linkages in milk protein (0.01 g).
- e. A mixture containing 1 g of benzoic acid, 1 g of 2-aminonaphthalene and 1 g of naphthalene
- f. A 10 mL contaminated sample of styrene (Note that styrene polymerizes when heated to its atmospheric pressure boiling point)
- g. Isolation of carvone from spearmint leaves
- h. Separation of 1 g of a 1:1 mixture of anthracene and 9-acetylanthracene

**B. Product workup.** After a reaction is run to synthesize a product, work up procedures usually starting with extraction followed by recrystallization or distillation are performed. For each of the synthesis given below, describe the work-up procedure you would use to separate and purify the desired product.

- a. Bromobenzene is reacted with magnesium to make a Grignard reagent. The phenyl magnesium bromide in ether is subsequently reacted with dry ice to make benzoic acid. What steps should follow?
- b. 20 mL of 3,3-dimethyl-2-butanol is dehydrated with 8 M sulfuric acid. How would you isolate the alkene?
- c. Cyclohexanone is reacted with sodium borohydride in slightly basic aqueous solution. After reaction is complete, 6 M HCl is added until the solution is slightly acidic. How would you isolate and purify the cyclohexanol?

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- d. Vanillin is reacted with sodium borohydride in 1 M NaOH solution. After reaction is complete, 6 M HCl is added until the solution is slightly acidic. How would you isolate and purify the vanillyl alcohol.
- e. Using sulfuric acid as a catalyst, salicylic acid is esterified in methanol under reflux. How would you isolate and purify the methyl salicylate (oil of wintergreen)?
- f. Aluminum chloride, acetyl chloride and p-xylene are refluxed in methylene chloride. How would you isolate and purify the product?
- g. Furan and maleic anhydride are allowed to sit in an ether solution for several days until a product crystallizes out of the solution. How would you isolate and purify the product?

**C. Analysis or identification.** For each of the problems in B above, describe what you would do to determine the identity and purity of the product.