

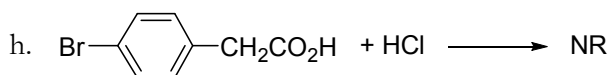
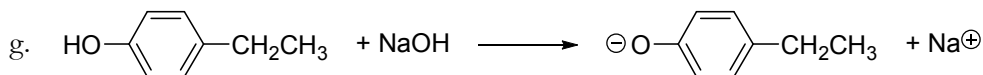
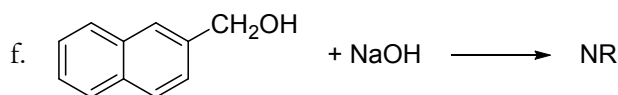
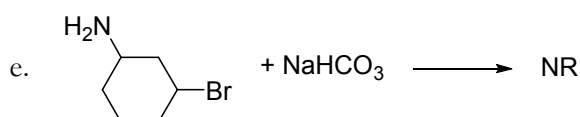
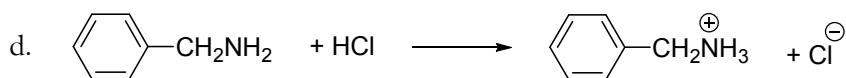
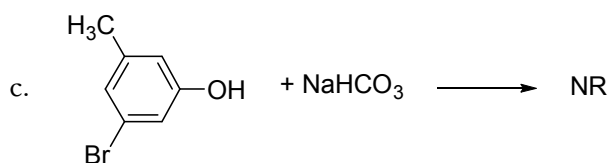
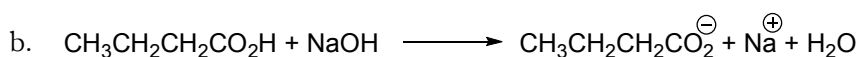
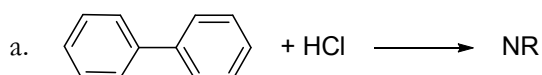
Experiment 4

Extraction: Separation of Benzoic Acid and Phenanthrene

Study Questions

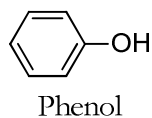
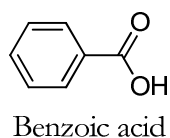
- 1) The organic layer is “dried” by adding anhydrous sodium sulfate. How do you know when you have added enough drying agent? **Answer:** When freshly added drying agent stops clumping or becoming wet looking. Add enough drying agent to cover the bottom of the flask, then filter off old drying agent and add fresh drying agent to the filtered solution if it becomes wet looking or clumped.
- 2) The handbook covers both vacuum and gravity filtration techniques. Explain why vacuum filtration is the method of choice for separating the benzoic acid from the neutralized aqueous solution. **Answer:** Vacuum filtration is quick and efficient, and since it's the solid that is desired, product will not be lost.

- 3) Problem 8 in the Extraction Study Problem section of the *Handbook for Organic Chemistry Lab*.

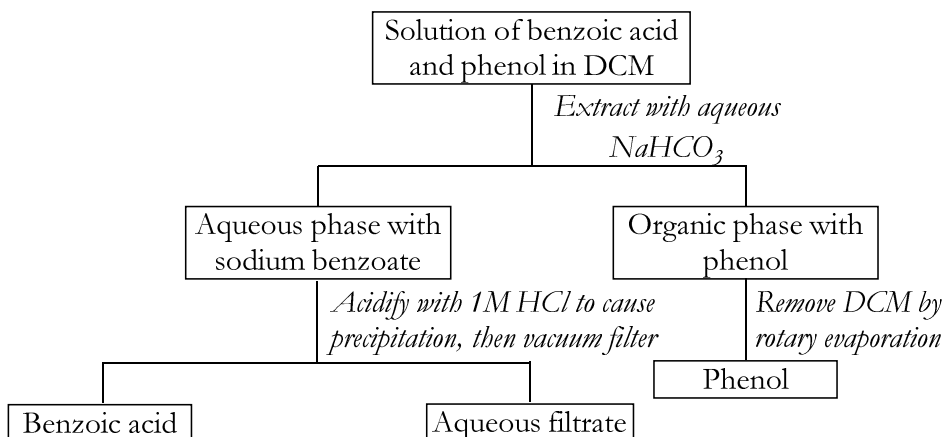


- 4) Carboxylic acids and phenols can be separated by chemically active extraction if the pH of the aqueous layer is properly chosen. Prepare in your notebook a detailed flow chart showing how you would separate a mixture of benzoic acid and phenol:

Experiment 4: Extraction



Answer: It is important to use aqueous NaHCO_3 and *not* NaOH . This is because NaHCO_3 will deprotonate only the benzoic acid, allowing it to go into the aqueous layer while the phenol is left behind in the organic layer. If NaOH (a stronger base than NaHCO_3) were used, it would deprotonate both compounds, and they would both go into the aqueous layer together.



- 5) You have 10 grams of compound A dissolved in 100 mL of solvent X, and want to extract it into solvent Y using 100 mL of solvent Y. You know that the partition coefficient, K , is given by:

$$K = \frac{C_y}{C_x} = 2$$

C_y is the concentration of A in solvent Y (g/mL) and C_x is the concentration of A in solvent X (g/mL).

- a. How much compound A remains in X if you extract with 100 mL of solvent Y?

Answer: We'll use x to represent the amount of compound in solvent X.

$$\frac{(10 - x/100)}{(x/100)} = 2$$

$$(10 - x/100) = 2(x/100)$$

$$10 - x = 2x$$

$$3.333 = x$$

- b. How much compound A remains in X if you extract with 2 x 50 mL portions of Y?

Answer: We'll use x_1 to represent the amount of compound in solvent X after the first extraction, and x_2 to represent the amount of compound in solvent X after the second extraction. The setup is the same as before, only now we have to do it twice in a row.

$$\frac{(10 - x_1/50)}{(x_1/100)} = 2$$

$$(10 - x_1/50) = 2 (x_1/100)$$

$$10 - x_1 = x_1$$

$$5 = x_1$$

$$\frac{(5 - x_2/50)}{(x_2/100)} = 2$$

$$(5 - x_2/50) = 2 (x_2/100)$$

$$5 - x_2 = x_2$$

$$2.5 = x_2$$

- c. How much compound A remains in X if you extract with 4 x 25 mL portions of Y?

Answer: We'll use x_1 to represent the amount of compound in solvent X after the first extraction, x_2 to represent the amount of compound in solvent X after the second extraction, x_3 to represent the amount of compound in solvent X after the third extraction, and x_4 to represent the amount of compound in solvent X after the fourth extraction.

$$\frac{(10 - x_1/25)}{(x_1/100)} = 2$$

$$(10 - x_1/25) = 2 (x_1/100)$$

$$10 - x_1 = 0.5 x_1$$

$$6.6667 = x_1$$

$$\frac{(6.6667 - x_2/25)}{(x_2/100)} = 2$$

$$(6.6667 - x_2/25) = 2 (x_2/100)$$

$$6.6667 - x_2 = 0.5 x_2$$

$$4.4444 = x_2$$

Experiment 4: Extraction

$$\frac{(4.4444 - x_3/25)}{(x_3/100)} = 2$$

$$(4.4444 - x_3/25) = 2 (x_3/100)$$

$$4.4444 - x_3 = 0.5 x_3$$

$$2.963 = x_3$$

$$\frac{(2.963 - x_4/25)}{(x_4/100)} = 2$$

$$(2.963 - x_4/25) = 2 (x_4/100)$$

$$2.963 - x_4 = 0.5 x_4$$

$$1.975 = x_4$$

- d. What rule is suggested by your answers to a, b, and c? **Answer:** It's more efficient to perform multiple small extractions than it is to perform one big extraction, even if both options use the same total amount of solvent.