

Organic Chemistry Laboratory

#3 - Extraction

Extraction is a very important technique in organic chemistry. During the semester, and more so next semester, you will need to rely on extraction to separate a compound from a mixture of compounds. Primarily, you will do this to isolate and purify reaction products. Extraction can also be used to isolate compounds from plant or animal sources. Your success in this experiment is directly related to your understanding the pre-lab reading. Make sure you read this prior to the exercise and if you have questions try to get them answered.

Pre-lab Reading:

Read Chapters 15 in the Zubrick manual, which deal with the extraction process and the theory of extraction respectively. You should also read Chapter 10 in the Zubrick text that discusses drying agents.

Pre-lab assignment:

Sometimes a flowchart is helpful in understanding what is happening during a given process such as an extraction. The last page of this handout contains an incomplete flowchart for the separation of aniline, benzoic acid, and naphthalene. Complete this handout prior to the lab period. Also, for each of the following pairs of immiscible solvents indicate which would be on top and which would be on bottom, in a separatory funnel: **a)** water, ether; **b)** water, dichloromethane; **c)** 10% NaOH, hexane.

Procedure:

Obtain an unknown sample and weigh the container with the sample, record this weight. Dissolve your unknown in 50 mL of ether and transfer it in a 125 mL separatory funnel. (*Be sure that the stopcock is closed!*) You should rinse the original container with a small amount of ether and add this to the separatory funnel. Then weigh the empty, dry container and determine the mass of the unknown mixture.

To separate the basic component of your unknown extract the solution with three 15 mL portions of 5% aqueous HCl. Set up a ring stand to hold the separatory funnel while the layers separate. Combine all the aqueous layers into a 125 mL Erlenmeyer flask. Label this flask and set it aside for later.

Next, separate the acidic component of the mixture by extracting the remaining unknown solution with three 15 mL portions of 5% aqueous NaOH solution. Combine these aqueous extracts into a fresh 125 mL Erlenmeyer flask. Label this flask and set it aside.

Now wash the ether solution in the separatory funnel by adding 10 mL of water. Drain the water into a beaker and pour the ether solution into a 125 mL Erlenmeyer flask. Rinse the separatory funnel with a few mL's of fresh ether and add to the Erlenmeyer flask. Dry the ether solution with sodium sulfate as directed on page 69 of the Zubrick text. Filter the ether through a cotton plug into a clean dry flask and evaporate the ether on a steam bath. If a neutral compound is present in the ether solution, it should remain as an oily residue or a crystalline solid.

To recover the basic component of the unknown, add 10% NaOH solution to the acidic extracts until the solution is basic to pH paper. **NOTE: Do not dip the test paper into the solution instead, dip a glass rod into the solution and transfer one drop of solution to the test paper! (page 113)** When the aqueous solution becomes basic enough any basic organic will separate from the solution as either an oil or solid. If a solid appears collect it vacuum filtration and rinse the solid with some cold water while still in the filter funnel, then set the filter paper and product aside to dry. If an oil appears, then place the solution in the separatory funnel and extract with fresh ether (3 x 10 mL). Combine the ether extracts and dry with sodium sulfate, filter, and evaporate the ether on the steam bath.

To recover the acidic component of the unknown, slowly add 10% HCl solution to the basic extracts, swirling as the acid is added. Add acid until the solution is acidic by pH paper. **NOTE: See note above!** Most likely any organic acid in the solution will precipitate quickly and can be collected by vacuum filtration, washed with some cold water, and set aside to dry. If the organic acid separates as an oil then you will need to extract it from the water with ether (3 x 10 mL), dry with Na₂SO₄, and evaporate the solvent.

For each solid obtained, spread the compound out on weighing paper and allow to dry. Determine the weight and melting point for each compound then identify the compound using the table below. If any identification is questionable, then you may need to recrystallize the compound or determine a mixed melting point with an authentic sample.

Possible Unknowns

<i>Acids</i>	<i>MP</i> (°C)	<i>Bases</i>	<i>MP</i> (°C)	<i>Neutral</i>	<i>MP</i> (°C)
o-Toluic acid	104	p-Chloroaniline	72	Naphthalene	80
Benzoic acid	122	Ethyl p-aminobenzoate	89	Benzil	95
trans-Cinnamic acid	133	2-Aminobenzophenone	106	Phenanthrene	101
m-Nitrobenzoic acid	140	p-Phenylenediamine	140	Fluorene	114
m-Bromobenzoic acid	155	4-Aminoacetanilide	162	2,6-Dichloronaphthalene	136
p-Toluic acid	180				

Data and Observations:

Record the physical constants for the compounds used in this experiment in your notebook. You may use the CRC Handbook, Aldrich Catalog or any other reference book

available to obtain any information in addition to what is contained in this handout. You may wish to view MSDS reports for any of the compounds used. The MSDS are available on the web site of several manufacturers and obtainable through the course web site under *Chemistry Links*.

You should include a table of data that resembles the table on the next page.

Report:

Include the data table that appears on the final page. Remember - **do not** fill out this page as you go through the experiment but record all data in your notebook then transfer the necessary data to your report. Also, include in your report the answers to the following questions.

Questions:

1. What practical advantage does an extraction solvent that is heavier than water have over one that is lighter than water?
2. The distribution coefficient, $K_D = (C_{\text{ether}})/(C_{\text{water}})$ between ether and water for aspirin (acetylsalicylic acid) is 3.5 at room temperature. What volume of ether would be required to extract 0.95 g of aspirin from a solution of 1.0 g of aspirin in 100 mL of water in a single extraction?
3. Chloroform is an excellent solvent for extracting caffeine from water. The distribution coefficient, $K_D = (C_{\text{chloroform}})/(C_{\text{water}})$ for caffeine in chloroform-water is 10 at 25 °C. What weight of caffeine could be removed from a solution of 4.0 g of caffeine in 100 mL of water by a single extraction of 100 mL of chloroform?
4. In the above question; what weight of caffeine could be removed by two extractions using 50 mL of chloroform each time?
5. Suppose that during an extraction, you lost track of which layer was the aqueous layer. What could you do to determine which layer is which?
6. Using the sample flowchart as a guide, draw a flowchart to describe your particular extraction.

Name: _____

Lab section: _____

Date: _____

DATA

Unknown No. _____

Total mass _____ g

Acidic comp. Present Y / N

Weight _____ g

MP range _____ °C

Identity _____

Basic comp. Present Y / N

Weight _____ g

MP range _____ °C

Identity _____

Neutral comp. Present Y / N

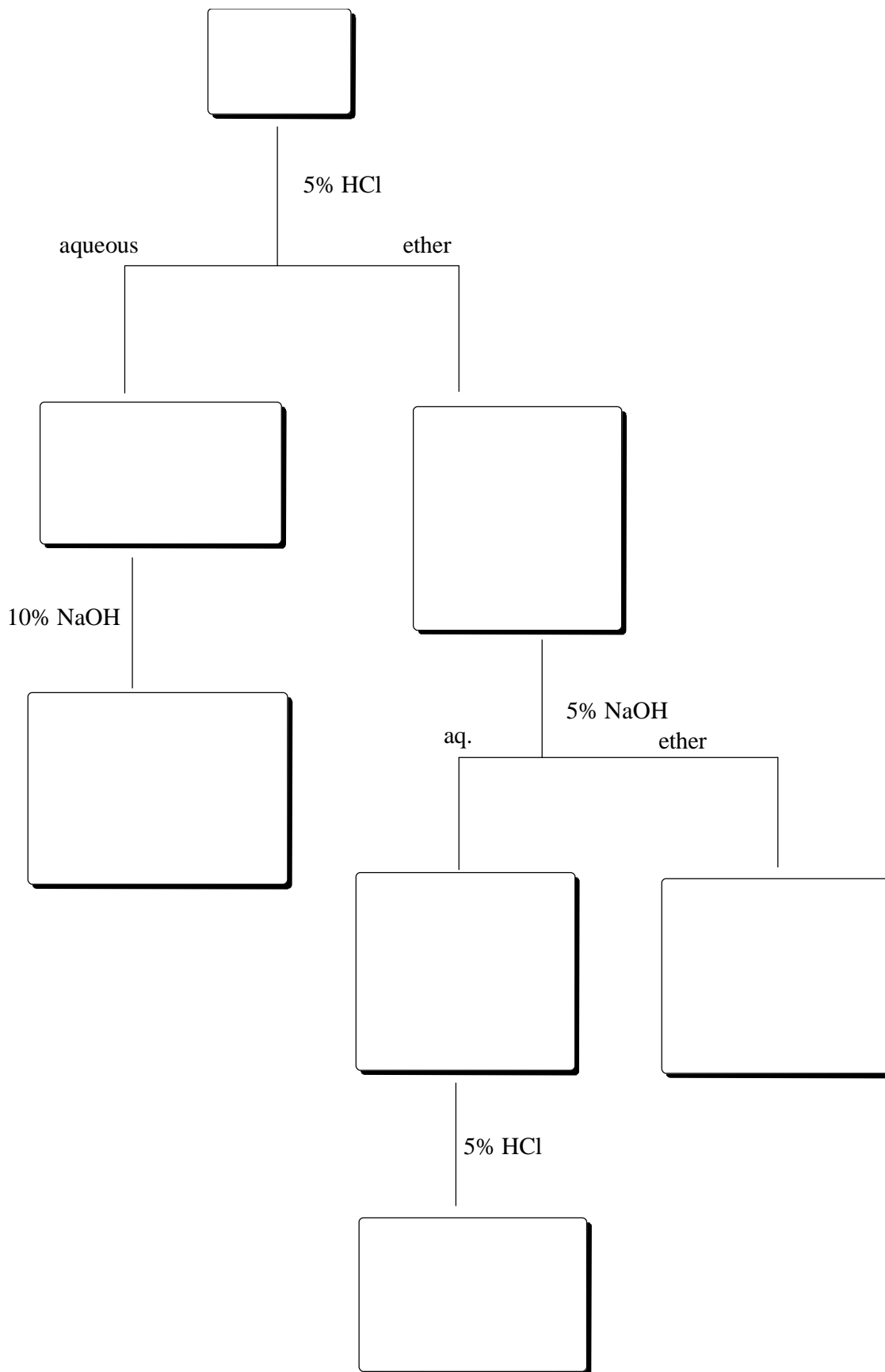
Weight _____ g

MP range _____ °C

Identity _____

Total mass
recovered _____ g

Percent recovered _____ %



Extraction Flowchart

