Separation of Solids by Extraction

I. PURPOSE: To learn how a mixture can be separated into its constituent compounds by employing differences in solubility in various liquids.

II. READING ASSIGNMENT: Landgrebe 123-141(*108-123 5th ed*), 387-389 (and handouts. View the movie on how to use the separatory funnel.

III. EQUIPMENT AND SUPPLIES:

A. From Stockroom: An additional 125 mL separatory funnel.
B. From Lab Assistant: Unknown mixture to be separated.

IV. GENERAL INSTRUCTIONS:

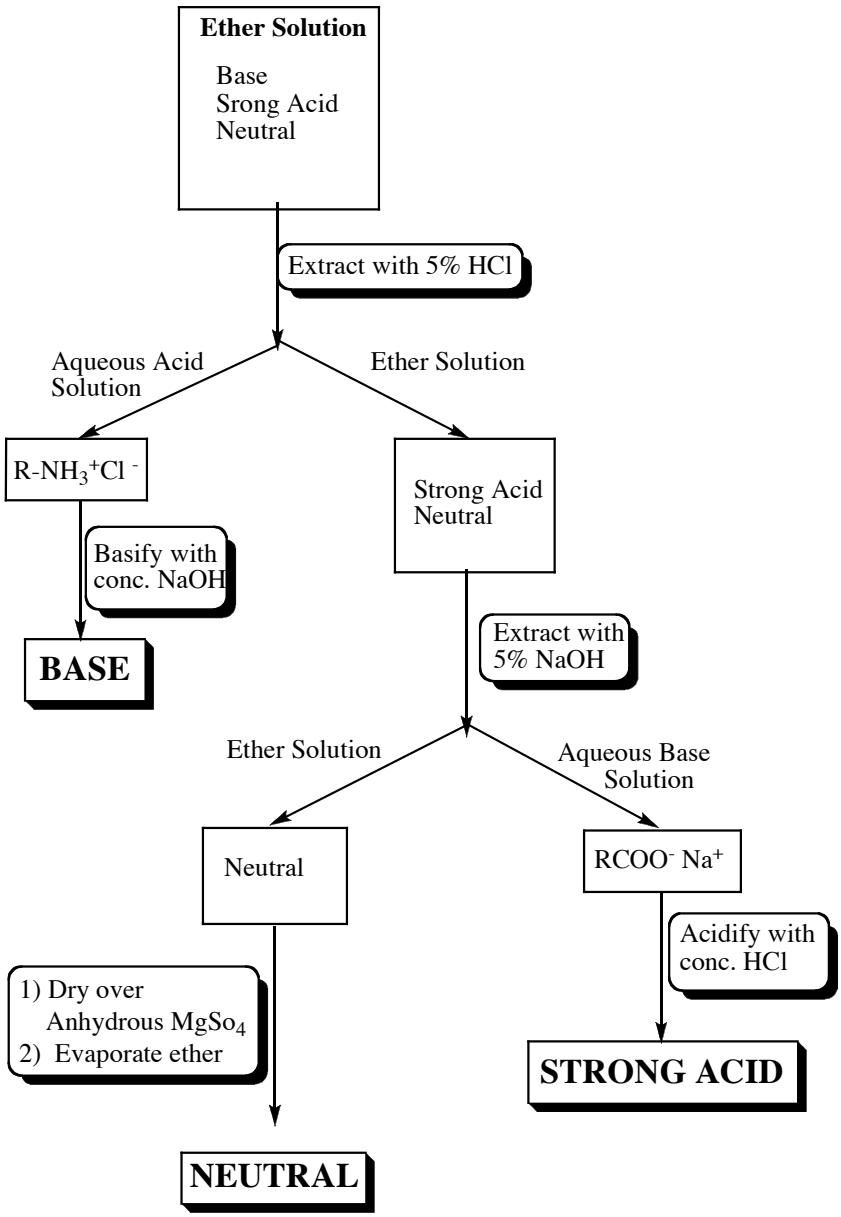
1. You will work individually on this experiment
2. We will be using diethyl ether in the lab today, a compound which is much more flammable than gasoline. **NO FLAMES IN THE LAB TODAY.**
3. Pressures can build up in the separatory funnel. Safety Goggles must be worn at all times.
4. Our first procedure will be to separate and purify benzoic acid from a mixture with triphenylmethanol. You will use the procedure on pp 387-389. Record the weight of dry, recovered benzoic acid and it's melting point. Calculate the % recovery of benzoic acid from the mixture.
5. We will also separate an unknown mixture into its individual components. This unknown mixture may contain three of the following: (a) Neutral Compound, (b) Strong Acid, (c) Base. The possible compounds are listed in the Table at the end of the handout.

V. OVERVIEW OF PROCEDURE FOR BENZOIC ACID AND TRIPHENYLMETHANOL

1. Follow procedures A and B on page 387-389. Get 3.5 g of the mixture and dissolve in 15-20 mL of methylene chloride. Extract with 3M NaOH solution. Obtain a % recovery and melting point of the dried extract.

VI. OVERVIEW OF PROCEDURE FOR UNKNOWN:

1. Get an unknown from the Lab Assistant and weigh the mixture. It will be composed of equal lparts of acid, base and neutral compounds. Follow the flow chart below to separate the mixture.



2. Dissolve 3.0g of the unknown in 50mL of diethyl ether and transfer the solution to a 125 mL separatory funnel. Add to the funnel about 15mL of the acid (5% HCl) or base (5% NaOH) solution you wish to extract with. Remember, you ***extract out*** the organic acid with the base solution and the organic base with the acid solution. Shake well. Draw off the lower aqueous layers. Extract a total of 3x15mL. Combine the three extracts together (~45mL total). .

3. Acidify the basic extracts with conc.HCl (BE CAREFUL, bicarbonate gives off CO_2). Check with litmus or pH paper. Basify the acidic extract with conc. NaOH. Check with litmus or pH paper. Your acid or base should precipitate out at this point. Collect this solid by vacuum filtration and leave to dry over the intervening period in you bench. Weigh the dry acid and base compound the following lab period.

4. Dry the remaining diethyl ether solution over anhydrous magnesium sulfate (~1g for 5 min), decant off the dry ether solution (discard the "used" drying agent into a pre weighed beaker and set aside for the ether to evaporate in the hood (write your name on the beaker). Re-weigh the flask to get a weight of neutral product.

5. Take melting points of all three compounds the following lab period.

A. HOW TO USE SEPARATORY FUNNEL

1. **Make sure the stopcock of your separatory funnel is CLOSED before adding liquids!!!!!!!!!!!!!!!!**
2. Make sure that the separatory is firmly supported on the ring stand
3. Hold stopper with index finger, sides of funnel with other fingers
4. Invert once and **immediately** open the stopcock to vent the pressure
5. Repeat until no pressure release is evident
6. Invert repeatedly for a total of 20 times with only occasional venting
7. Set funnel on ring stand **with stopper removed** to let the layers separate
8. Either drain out the bottom layer or decant off the top layer. Leave the layer you are to extract next in the funnel. (**Note:** You cannot drain liquid from a stoppered separatory funnel. A vacuum will be created inside which will prevent liquid from leaving the funnel.)
9. Always check a solution by adding water to it before discarding. If two layers form, the solution is **not** aqueous.
10. Label **all** containers of solutions. They get mixed up after several are placed on your bench

B. Types of solutions with which we extract.

1. Saturated, aqueous $NaHCO_3$
    1. Converts a carboxylic acid (which is soluble in polar, organic solvents) into their sodium salt (which is more soluble in water).

2. NaOH
    1. Converts carboxylic acids **and** phenols into their sodium salts (which are more soluble in water).
3. HCl
    1. Converts amines into their quaternary ammonium salts (which are soluble in water)

2. Converts salts of either carboxylic acids or phenols into their acid form (which is more soluble in organic solvents)

C. Types of organic solvents used in extractions

1. Methylene chloride (dichloromethane)
    1. heavier than water
    2. polar
    3. nonflammable
    4. not biodegradable
2. Ethyl ether
    1. lighter than water
    2. polar
    3. highly flammable
    4. biodegradable

D. Types of Drying Agents

1. $CaCl_2$
    1. Inexpensive
    2. complexes with amines and OH containing groups
2. Anhydrous $MgSO_4$
    1. Expensive but high grade
    2. Good drying power
    3. can be used with amines and OH containing groups
3. Anhydrous Na_2SO_4
    1. Inexpensive
    2. Moderate drying power
    3. can be used with amines and OH containing groups

E. Technique of removing water from organic solvents prior to removing the solvent.

1. Cover the bottom of the container with the drying agent.
2. Swirl occasionally over a 15 min period.
3. If lumps form, decant the solvent and repeat steps (1) and (2)
4. Decant or filter off the drying agent and evaporate the solvent.

VII. LAB REPORT

1. Title

2. Procedure and Observations - Describe the procedure you followed, both for the benzoic extraction and the unknown. Illustrate with a flow

chart such as that above. Report any observations made during the process(such as weights, mp's etc.).

3. Calculate the percent recovery of benzoic acid based on the total weight of mixture used.(show calculations)

4. Report the melting point range of your benzoic acid and discuss the implications toward the purity of the sample..

5. Identify the three compounds present in your unknown by mp (from the list of compounds shown on the end of this handout) and give their % recovery from the unknown mixture (suppose an original mixture ratio of 1:1:1 for each component).

6. Answer questions 1,2,3,5 and 7 on pages 141 (*123 5th Ed*) of Landgrebe.