

Experiment #4: Acid/Base Extraction

CHEM 213 –Fall 2009

Acid/base extraction can be an extremely useful separation technique in organic chemistry. Using simple acid/base reactions, several different classes of organic molecules can be separated from one another. This procedure is most easily visualized using the flow chart for acid/base extraction on the following page. In this experiment you will be randomly assigned a mixture containing two components; an acid, a base, or a neutral component. The goal will be to separate the mixture by acid/base extraction, recrystallize the solid compounds and identify them based on solubility and melting point determination. You will be given approximately 3 g of the mixture and use the **miniscale** (section 8.2) procedure described in the text.

Objectives:

1. Separate two compounds from an unknown mixture.
2. Purify the unknowns by recrystallization.
3. Analyze and identify the compounds by melting point determination.

Helpful Hints:

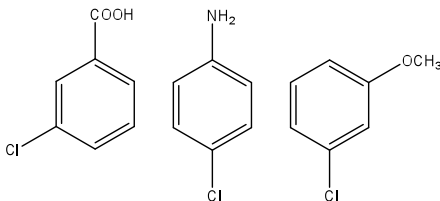
1. Do not dispose of any reagent or solution until you have collected both compounds. Keep solutions in separate flasks, do not create one giant waste beaker.
2. Always label your beakers, flasks, test tubes, etc.
3. Be clear which layer is which; water vs. organic.
4. If you create an emulsion, be patient. If the emulsion does not clear, see your instructor.
5. Drying agents do not need to be weighed out exactly. Add enough drying agent to cover the bottom and swirl the flask. When the drying agent flows freely, the solvent is dry.
6. Do not add drying agent to the water layer!
7. Do not forget to vent your separatory funnel.

Reading:

In addition to reviewing Technique 8 in Mohrig, it is helpful to read sections 19.12 (extraction of carboxylic acids) and 25.9 (amines as bases) from your CHEM 210 lecture text (*Organic Chemistry*, Smith.)

Pre-lab* Questions:

1. What is an easy, convenient method to tell which layer is which if the densities are not readily available during an ether/water extraction?
2. Provide an extraction separation scheme for separating these three organic molecules. (See next page.)



* Acid/base extraction is a purification technique; a balanced equation is not required. The Chemical Data Table should include water, dichloromethane, magnesium sulfate, calcium chloride, sodium sulfate, hydrochloric acid, sodium hydroxide, sodium bicarbonate, and sodium carbonate. It is **not** necessary to include all the unknown choices.

