Chem 51Lb Exp 6  
Compound Separation and Identification (Unknowns) Project

Reactions: None    
Techniques: Melting Point, Extraction, TLC, Recrystallization, Spectroscopy

In this two-lab-period project you will be assigned a mixture of two unknown solids. Your job will be to identify the components of and separate the mixture.

Reading Assignment:
➢ Review previous techniques.

Caution

The compounds you are assigned may be hazardous if inhaled or absorbed through the skin. Avoid contact and inhalation.

Dichloromethane is toxic, an irritant, absorbed through the skin, and harmful if swallowed. Wear gloves and wash your hands thoroughly after handling it. Avoid contact with skin and eyes. Dichloromethane is a suspected carcinogen when inhaled in large quantities.

Diethyl ether, hexanes, and ethyl acetate are volatile and flammable. Use in the fume hood and avoid inhalation.

Do not heat diethyl ether on a hot plate! Use an air stream for evaporation.

Hydrochloric acid solutions are corrosive. Avoid contact with skin and eyes.

Sodium hydroxide solutions can cause burns to skin and eyes. Avoid contact.

Experimental Notes:
This project is designed to be completed in two lab periods. A good general plan is to identify your unknown mixture components in the first lab period and separate them in the second lab period. You will need to arrive at each lab period with a plan of what you will accomplish that day and how you will accomplish it. If you do not plan ahead, you will probably not be able to complete the entire project. You may attempt to separate your mixture during the first lab period if you have time, but most likely this will need to wait until the second lab period. Be sure to complete the experiment progress report at the end of each lab period and give it to your TA.

Below you will find a list of possible components of your unknown mixture. Before your first lab period you will need to look up structures and properties of these compounds.

• p-acetophenetidide  
• benzhydrol  
• biphenyl  
• 2'-bromoacetanilide
Identifying the Components of Your Mixture
Each unknown mixture is comprised of ~300 mg of one compound and ~100 mg of a contaminant compound. You may use spectroscopy, melting point and TLC data to identify the components of your mixture. You will be given NMR spectra for your compounds to help with confirmation of your unknown identities. In lab you will be provided with melting point and TLC standard samples of each of the possible compounds for comparison. Recommended eluting solvents for TLC are 100% hexanes, 9:1 hexanes/ethyl acetate, 8:2 hexanes/ethyl acetate, 7:3 hexanes/ethyl acetate, 6:4 hexanes/ethyl acetate, and 5:5 hexanes/ethyl acetate. You do not need to run every sample in every solvent mixture. Think about what is the best strategy to identify the components of your unknown mixture in a minimal amount of time and using a minimal amount of materials. If you have a spot that is streaking on your plate but does not appear to be streaking because of excessive sample concentration, you can try adding a single drop of glacial acetic acid to your eluting solvent.

Separating the Components of Your Mixture
All unknown mixtures can be separated by either acid-base extraction or recrystallization. Based on the components of your mixture, you will need to decide which method to use. You must have a general procedure written for your chosen method before you will be allowed to proceed! You are responsible for isolating and confirming the identity of the major component of your mixture. In most cases you will not be able to isolate the minor component. Be sure to confirm the identity of the isolated major component using melting point and TLC.

General Recrystallization Information
All mixtures requiring recrystallization can be recrystallized from ethanol and water. Benzhydrol tends to precipitate initially upon cooling rather than crystallizing. If this happens try adding a seed crystal of pure benzhydrol to initiate formation of crystals.

General Extraction Information
Solutions of NaOH (1-2 M) and 6M HCl will be provided for acid-base extraction. Use either diethyl ether or dichloromethane (~15-20 mL) as your initial organic solvent. Remember to pay attention to which layer is which and to where your desired compound is! Magnesium sulfate and sodium sulfate will be provided as drying agents.

**DO NOT HEAT DIETHYL ETHER ON A HOT PLATE TO EVAPORATE! USE A STREAM OF AIR INSTEAD!**

POST-LAB
Write a full and complete lab report detailing the identification and separation of the components of your unknown mixture.