Separation and Analysis of an Unknown Mixture

Question: Given a two component mixture, what are the identities of the two organic compounds?

Objectives: In this two-week project you will use extraction to separate a mixture containing an acidic and a neutral compound. The isolated solids will be purified by recrystallization, assayed by TLC, and their identities will be determined by melting point and infrared spectroscopy.

Procedure:

Start with 0.60 g of the unknown mixture. Perform an acid-base extraction to separate the acidic and neutral components of the mixture. The solvents/solutions/reagents at your disposal are the following: t-butyl methyl ether, 1.5 M sodium hydroxide, concentrated hydrochloric acid, saturated sodium chloride solution, calcium chloride. **[Prior to the start of the lab period, you must have written a step-by-step procedure for the extraction in your notebook; otherwise, you will not be allowed to perform the experiment.]**

Once you have isolated the crude acid unknown and the crude neutral unknown, recrystallize the unknown acid from water and the neutral compound from ethanol. Allow both recrystallized compounds to dry until the next laboratory period.

Determine the melting ranges of the two unknown compounds that you isolated and recrystallized. Compare your data to a list of known compounds available in the laboratory. Samples of known compounds are also available for mixed melting points.

Analyze the recrystallized unknowns by TLC, using 0.5% acetic acid : 95% ethyl acetate as the developing solvent. Calculate the R_f values for all spots.

Evaluate the success of the separation and purification using the mp and TLC results.

Obtain an IR spectrum for each of your recrystallized compounds. Analyze each spectrum, and assign the principal vibrational bands in each.