Cerritos Community College

Organic Chemistry 211 Laboratory

Extraction (Part 2)

Separation of Acidic, Basic, and Neutral Components:

a) Extraction of the basic component:
In a centrifuge tube, dissolve 100 mg fo an unknown in 2 mL of diethyl ether. Add 1 mL of 5% HCl, cap or stopper the tube, and shake gently for about 1 minute. Allow the layers to separate and carefully draw off the bottom layer with Pasteur pipette, depositing it in a second (properly labeled) centrifuge tube. Repeat the procedure with two additional 1 mL portions of 5% HCl, combining these with the first portion. Back-extract the combined HCl layers with 1 mL of fresh ether by shaking gently for 1 minute and then carefully draw off the ether layer with a clean pipette. Add this ether layer to the original ether solution of the unknown mixture.
The combined HCl extracts are then treated with 10% aqueous NaOH solution until basic to Litmus paper test, at which point the appearance of a precipitate indicates the presence of the basic component in the original unknown. If present, the basic component can be gravity filtered through a small Hirsch funnel equipped with a plug, and allowed to air dry.

b) Extraction of the acidic component:
Extract the original ether solution with three 1 mL portions of 5% aqueous NaOH solution, each time transferring the NaOH layer with a pipette to a labeled cantrifuge tube. The combined NaOH extracts are back extracted with 1 mL of fresh ether and then acidified by cautiously adding10% aqueous HCl. A precipitate indicates the presence of an acidic component in the original unknown. Separate the solid as discussed in part "a".

c) Isolation of the neutral component:
To separate the neutral component, wash the original ether layer solution with 1 mL of water, dry the ether layer with a drying agent, and after separtating the drying agent, remove the ether solvent by gently heating the original solution on a steam bath.

*Back Extraction: In case there is trace amount of the other layer in the one, after extraction, we do a back extraction to achieve separation as thoroughly as possible.
*Drying Agents: Anhydrous (without water). The most commonly used one is sodium sulfate. It dries the system by absorbing *trace amounts of* water in a phase. The resulting phase after all the water is absorbed into the sodium sulfate matrix is called dry phase, or dry solution. They need to know that DRY means WATER-FREE, not solid. There is really no fixed amount of drying agent for all to use, since they may have different amounts of residual water to be absorbed. So, it should be added in small portions at a time, and swirled around. The way we visually detect that enough drying agent is added is the following:
Wet drying agent is clumped up, and usually sticks to the walls of the container. Dry (water-free) drying agent is granular (like sugar grains). As soon as you notice free grains of drying agent, you should know that enough has been added, and the free grains don't find any water to absorb.