Separation and Purification of Unknown Mixture

Prelab reading from Mohrig, “Techniques in Organic Chemistry”
- Technique 10.4 (Vacuum filtration)
- Technique 11 (Skip 11.5)—Extraction
- Technique 12 (Drying organic liquids)
- Technique 15 (Recrystallization)

Procedure

Place your unknown mixture (about 0.6 grams) in an Erlenmeyer flask and add 30 mL of diethyl ether, swirling until the solid is dissolved. Use a funnel to transfer the ether solution into a separatory funnel. Add 20 mL of a 1.5 M sodium hydroxide solution to the sep funnel. Shake the funnel, vent, and allow the phases to separate. Drain the lower aqueous layer into a beaker labeled, “basic extraction.” Wash the organic layer in the funnel with 5 mL of water, allow the phases to separate, and drain the aqueous layer into the beaker marked “basic extraction.” Pour the upper ether layer into an Erlenmeyer flask marked “organic extraction.” Add sodium sulfate to dry the solution, allow to stand.

Get 5 mL of 6M sulfuric acid. Add it dropwise to the basic extraction beaker with swirling until a precipitate forms and the aqueous solution is acidic to pH paper. Cool the “basic extraction” beaker with precipitate in an icewater bath. Collect the solid by vacuum filtration until the crystals are fairly dry. Place the crystals on a labeled 50 mL beaker.

Separate the “organic extraction” ether layer from the sodium sulfate by decanting the liquid into a beaker or roundbottom flask. (Ask the AI if you will evaporate the ether using heat or a rotovap. They will direct you in this procedure.) After the ether is removed, you will have a solid residue remaining.

Recrystallize the “basic extraction” solid from water and the “organic extraction” solid from ethanol. (Follow Technique 15, “Recrystallization” from Mohrig.)

Notebook Guidelines: General guidelines for the notebook are found in the syllabus. Follow these guidelines to include all the proper information for this lab in the correct sections. Your notebook will be graded for accurate completion of all these questions/observations as well as the proper format as outlined in the syllabus.

Observations: Include physical characteristics of the original unknown, the crude ether extract, the crude hydroxide extract, the purified ether extract, and the purified hydroxide extract.

Results: Include the mass of the original unknown, the purified ether extract, and the purified hydroxide extract.
Discussion:
Draw a flow chart of the extraction process.

Explain why you performed these steps in the flowchart above:
   A. wash the organic phase with 10 ml of water
   B. add 6M HCl to the aqueous solution
   C. add sodium sulfate to the organic layer

What was the main purpose of the extraction?

Do you expect the product isolated from the “Basic Extraction” to be an acid, base, or neutral compound? Explain.

Do you expect the product isolated from the “Organic Extraction” to be an acid, base, or neutral compound? Explain.

What was the main purpose of the recrystallization?

Why didn’t you just use water as the recrystallization solvent for both of the crude products you isolated?