Microscale Acid-Base-Neutral Extraction

General Procedure:

Obtain an unknown that contains benzoic acid, ethyl 4-aminobenzoate, and 9-fluorenone.

Weigh 0.150 g of unknown mixture into a 15 mL centrifuge tube and dissolve in 4 mL of diethyl ether. Mix with stirring rod.

Separation of the Basic Component

Using a calibrated Pasteur pipet, add 2 mL 3 M hydrochloric acid are added to the centrifuge tube while cooled in an ice bath, and the resulting two-phase system is then mixed thoroughly for several minutes. After the layers have separated, the bottom aqueous layer is removed using a pasteur pipet, and transferred to a small test tube labeled ethyl 4-aminobenzoate. This step is now repeated using an additional 2 mL of the 3 M acid solution and the aqueous layer again is transferred to the same labeled test tube. This test tube is now capped, and set aside.

Isolation of the Ethyl 4-Aminobenzoate (Basic component)

To the acidic aqueous solution from above, add 6 M NaOH dropwise until the solution is distinctly basic to litmus paper. Cool the test tube in an ice bath for about 10-15 minutes. Collect the solid precipitate using vacuum filtration with a Hirsch funnel. Wash the precipitate with two, 1 mL portions of cold distilled water. Dry the precipitate on weighing paper, weigh, and determine the percentage of ethyl 4-aminobenzoate in your unknown. Obtain a melting point of your sample and compare with the literature value.

Separation of the Acidic Component

To the remaining ether solution now add 2 mL of 3 M NaOH using a calibrated Pasteur pipet. The system is mixed as before and the aqueous layer is separated and transferred to a test tube labeled benzoic acid. This step is repeated using an additional 2 mL of 3 M NaOH and the aqueous layer is separated and transferred to the same labeled test tube, capped and set aside.

Isolation of the Benzoic Acid (Acidic component)

To the aqueous alkaline solution from above, add 6 M HCl dropwise until the solution is distinctly acid to litmus paper. Cool the test tube in an ice bath for about ten minutes. Collect the solid precipitate using vacuum filtration with a Hirsch funnel. Wash the precipitate with two, 1 mL portions of cold distilled water. Dry the precipitate on weighing paper, weigh, and determine the percentage of benzoic acid in your unknown. Obtain a melting point of your sample and compare with the literature value.

Separation of the Neutral Component

Wash the remaining ether solution with two 1 mL of distilled water. Remove and discard the water. To the wet ether solution, add 250 mg of anhydrous sodium sulfate. Set the mixture aside for about 10 minutes. If the drying agent clumps, add additional sodium sulfate.
Isolation of the 9-Fluorenone (Neutral component)

Transfer the dried ether solution with a tared 50 mL beaker that contains 1 or 2 boiling chips. Rinse the drying agent with an additional 1 mL of ether and transfer to the same tared beaker. Concentrate the solution to dryness on a hot plate on the lowest setting. Weigh the dry beaker and calculate the percentage of 9-fluorenone in your unknown. Obtain a melting point of your sample and compare with the literature value.

Flow Chart of Separation

Discussion Items:
- Did the percentages of the three components sum to equal 100 percent.
- Were the colors of each component appropriate.