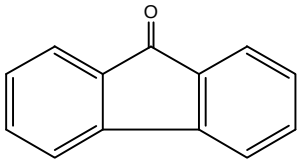
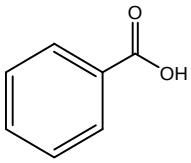
ACID/BASE SEPARATION OF A MIXTURE

Review Chapter 15 in Zubrick

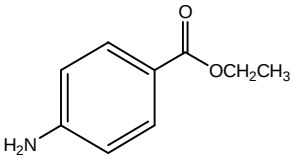
You will separate a mixture of 9-fluorenone, benzoic acid, and ethyl 4-aminobenzoate by successive acid/base reactions and extractions from ether. This process takes advantage of the fact that an organic compound will have increased solubility in an organic solvent when neutral, and an increased solubility in water when it is ionized to a salt. Instead of writing a stepwise procedure for your prelab assignment, you should develop a complete flow chart—including all chemical reactions—for the steps you will perform. Your instructor may provide more information.



9-Fluorenone
mp 82-85°C



Benzoic Acid
mp 122°C



Ethyl 4-Aminobenzoate
mp 91-92°C

PROCEDURE

To a separatory funnel add 50 mL ether, 50 mL 5% HCl, and 3 g of a mixture of equal weights of 9-fluorenone, benzoic acid, and ethyl 4-aminobenzoate. Shake well **[CAUTION: Vent frequently!]** a few times to dissolve and react the solids. Allow the layers to separate and draw off the lower aqueous layer.

Cool the aqueous solution in an ice bath and make it moderately basic by adding 3 *N* NaOH dropwise, checking with pH paper (make sure you use it correctly). Continue chilling the solution, vacuum filter the collected solid, wash it with cold water, and air dry.

To the ether layer in the separatory funnel, add 50 mL 5% NaOH. Shake repeatedly (with venting), remove the aqueous layer, and acidify it with 6 *N* HCl. Collect the solid by vacuum filtration.

To the ether layer still in the separatory funnel, wash with 50 mL saturated aqueous NaCl.

Dry the ether layer over sodium sulfate and let the solution stand. Decant into a pre-weighed 100-mL rotovap flask and rotovap to dryness.

When the crystals are dry, report their mass and melting point, and obtain IR spectra (if required). If possible, use the IR spectrum to comment on purity as well.