

## 4/ Extraction: Separating the Components of an Organic Mixture

### procedure

- 1 Obtain a vial containing a mixture of benzoic acid, ethyl 4-aminobenzoate and 9-fluorenone. Transfer the contents of the vial to a centrifuge tube and add about 4 mL of diethyl ether. Cap the tube and gently shake the mixture to dissolve the solid. Be sure to vent the tube by slowly removing the cap to relieve any pressure.
- 2 Cool the centrifuge tube in an ice bath and slowly add 2 mL of 3 M HCl while swirling the tube. Replace the cap and mix the two layers by inverting the tube several times. Once the layers have separated, use a clean Pasteur pipet to remove the aqueous layer, placing it in a **labelled** Erlenmeyer flask. Then, add another 2 mL of HCl to the tube and repeat the extraction. Add this aqueous layer to the first aqueous layer. Set the ether layer in the centrifuge tube aside.
- 3 To the acidic aqueous layer, add 6 M NaOH a drop at a time until the solution is basic to litmus paper. Cool the flask in an ice bath for several minutes. A solid should form in the flask. Collect the solid in a Hirsch funnel, washing with two 1 mL portions of distilled water. Allow the solid to air-dry by pulling air through the funnel. Record the mass and melting point of this dried solid.
- 4 To the ether layer in the centrifuge tube, add 2 mL of 3 M NaOH. (If the two layers are not approximately equal in volume, add 1 - 2 mL of diethyl ether.) Mix the two layers by capping and inverting the tube. When the layers have reformed, transfer the aqueous layer to a labelled Erlenmeyer flask. Perform a second extraction of the ether layer using another 2 mL of 3 M NaOH. Set the centrifuge tube and its contents aside.
- 5 To the basic aqueous layer, add 6 M HCl until the solution is acidic to litmus paper. Cool the flask in an ice bath for several minutes. A solid should form in the flask. If the quantity seems small, add several drops of saturated NaCl solution to promote crystal formation. Collect the solid on a Hirsch funnel, washing the solid with two 1 mL portions of cold distilled water. Dry the solid product and record its mass and melting point.
- 6 Retrieve the centrifuge tube containing the ether layer and wash this layer by adding 1 mL of water, shaking the tube, and removing the water layer. Perform this a second time. [It is a good practice to save the various washings until the end of the day's work, in case you have accidentally removed the wrong fraction.] Filter the ether layer through a Pasteur pipet containing about 1 - 1 1/2 inches of Na<sub>2</sub>SO<sub>4</sub>. Collect the filtrate in a *weighed* round bottomed flask and remove the solvent on the Rotovap. Determine the mass and melting point of the solid.

### health & safety notes

Diethyl ether is very flammable.

HCl is corrosive and NaOH is caustic. Avoid contact with skin.

Benzoic acid and ethyl 4-aminobenzoate are irritants. Avoid skin contact.