Separation Of A Mixture By Extraction


You will use a common separation technique that takes advantage of the differences in physical properties and chemical reactivity of organic compounds. The difference in physical properties often relies on solubility differences and the difference in reactivity is usually based on the acidic/basic behavior of the functional groups.

Procedure

Caution! You are working with ether; it is volatile and flammable. Stopper all solutions when not in use. NO FLAMES!

In a 125 mL Erlenmeyer flask, dissolve approximately 3.0 g of a mixture of benzoic acid and biphenyl in 25 mL of ether (diethyl ether). Pour the solution into a separatory funnel. Rinse the flask with 5 mL of ether and add the ether to the separatory funnel. Extract the mixture with 25 mL of 5% NaOH. Be sure to vent the separatory funnel to relieve any pressure buildup. Allow the layers to separate, and then draw off the lower aqueous layer into a clean Erlenmeyer flask labeled NaOH extract. Extract again with 25 mL of 5% NaOH, draw off the aqueous layer, and combine with the previous NaOH extract. Extract the remaining ether layer with 10 mL of water and combine the aqueous layer with the NaOH extract.

Pour the ether layer through the top of the separatory funnel into a 50 mL Erlenmeyer flask and add a tiny scoop of anhydrous MgSO₄. Stopper the flask and allow the ether solution to stand for 15 minutes. (Some MgSO₄ will clump together, but there should be a small amount of free-floating powder when the solution is completely dry. If necessary, add additional MgSO₄.) Place a small plug of cotton into the bottom of a conical funnel or Pasteur pipet. Add 1-2 boiling chips to a small beaker and weigh the beaker. Gravity filter the solution through the small plug of cotton in the funnel and into the beaker. Decant as much of the solution as possible through the funnel. Wash the remaining MgSO₄ in the flask with 1-2 mL of ether and filter this solution through the funnel. Place the beaker in the fume hood on a hot plate and allow the ether to evaporate under low heating. Since biphenyl has a low melting point, the residue will be liquid even though all the ether has evaporated. When the residue no longer bubbles, remove it from the hot plate and allow to cool in your locker for several days. The residue will be the biphenyl. Determine the weight and melting point of the compound. Calculate the percent recovery.
Cool the aqueous NaOH extract in an ice bath and then acidify by carefully adding 6M HCl. Use pH or Universal paper to make sure that the solution is acidic. The benzoic acid will precipitate out. Collect the product by suction filtration and wash the crystals with ice-cold water. Allow the product to dry over several days, weigh it, and determine the melting point. Calculate the percent recovery.

**Follow-up questions**

1. Draw the reaction that occurs when you add NaOH.
2. What is the purpose of adding MgSO₄?
3. Why must you be sure the solution is acidic after adding HCl?
4. a) Draw the reaction that would occur if you added aqueous HCl to a diethyl ether solution containing benzoic acid and methamphetamine (shown below). *Hint: which is the most basic functional group/atom among benzoic acid and methamphetamine?*
   b) How would this reaction be helpful in separating benzoic acid and methamphetamine using extraction? *Hint: how can you get benzoic acid and methamphetamine into different layers?*

![Structure of benzoic acid and methamphetamine](image)

**Conclusion:** Summarize your results, including your percent recovery of each compound from the total original mixture. Cite any important considerations that should be observed when performing an extraction.