

Acid-Base Extraction

In this experiment, you will separate a mixture of three organic compounds—an acid (benzoic acid), a base (m-nitroaniline), and a neutral compound (benzil)—by exploiting their differing solubilities in acidic and basic solutions.

New techniques:

Quantitative transfer
Extraction with a separatory funnel
Washing and drying an organic solution

Reading (in Pavia):

Extraction: chapter 12, pp. 163-164, 164-167 (skim), 167-171, 177-185.
Recrystallization: chapter 11, pp. 139-147, 153-161.
Melting points: chapter 9, pp. 116-120, 121-125.

Prelab: 1) Create a flow chart that outlines the separation of the components of your mixture and that indicates where and in what form each component of the mixture is to be found. 2) Find the structure, melting point and solubility characteristics for the three compounds used in the mixture. 3) Record the densities of water and diethyl ether.

For the lab report: Your report should contain a calculation of the percent recovery of benzil, benzoic acid and m-nitroaniline, and a judgment on their purity. Explain the basic theory behind why this extraction method works, and which compound is extracted at each step in the extraction process.

Procedure: Obtain around 2 g (know the exact quantity) of the mixture of benzoic acid, nitroaniline, and benzil, and dissolve it in 50 mL of ether. Transfer the mixture to your separatory funnel. To ensure complete transfer, wash your beaker with 3-5 mL of ether, and pour the solution into the funnel. Add 10% aqueous sodium hydroxide until you see two phases (about 10-15 mL). In order to ensure to ensure deprotonation of the benzoic acid, shake the funnel as follows: Stopper the funnel (*lightly* grease the stopper), and holding the stopper in, turn the funnel over. Point the bottom of the funnel away from you (and everyone else) and open the stopcock to release the pressure. Close the stopcock, and flip the funnel right-side up and then upside down again, and again open the stopcock. Continue this until no more gas is evolved. After a few careful flips, shake the funnel vigorously to ensure complete reaction. In any extraction, always remember to vent the separatory funnel frequently, even if no gas is evolved—many organic solvents (especially ether) have significant vapor pressure, and will blow out the stopper and contents if you are not careful. After you have shaken the funnel for a total of one minute or so, place it in the iron ring, remove the stopper immediately, and allow the layers to separate. Drain the aqueous layer (top or bottom? Remember to test to make sure) into a

flask, and repeat the process with two more 15 mL portions of 10% sodium hydroxide, collecting all three aqueous layers together. The organic layer can remain in the separatory funnel while you work with the aqueous layer.

To the aqueous layer (what does it contain?), add 6M HCl until the pH is 1 (about 14 mL). Collect the resulting precipitate. When dry, take their mass and melting point. You can recrystallize from water for a better melting point. *What is this compound?*

Extract the organic layer in your separatory funnel with three 15 mL portions of 3M HCl, following the same procedure as with the hydroxide extraction. Again, leave the organic layer in the separatory funnel and add 10% aqueous sodium hydroxide to the combined aqueous layers to a pH of 10 (about 80 mL), and collect the resulting precipitate. When dry, obtain a mass and melting point of your crystals. You can recrystallize them from water for a better melting point. *What is this compound?*

Dry the remaining organic solution in the separatory funnel with three 15 mL portions of saturated sodium chloride solution using the basic extraction technique as above. Combine the aqueous layers and set aside. Pour the organic layer (out the *top* of the funnel) into an Erlenmeyer flask and add anhydrous sodium sulfate until you see no more clumping. Filter the solution by gravity, and rinse the contents of the flask with additional ether. Allow the ether to evaporate at room temperature, and collect the crystals. When dry, note their mass and melting point. This compound can be recrystallized from ethanol or ethanol/water. *What is this compound?*

Turn in samples of all three of your recovered compounds in a clean, labelled vial.

Cleanup: Combine all aqueous layers, dilute with water neutralize with acid or base, and pour down the sink. Any excess ether must go in the organic solvents waste container.