Separation of a base neutral mixture essay sample

A commonly used method of separating a mixture of organic compounds is known as liquid-liquid extraction. Most reactions of organic compounds require extraction at some stage of product purification. In this experiment you will use extraction techniques to separate a mixture of an organic acid, a base, and a neutral compound. Organic acids and bases can be separated from each other and from neutral compounds by extraction using aqueous solutions of different pH values. Most organic carboxylic acids are insoluble or slightly soluble in water, but these compounds are highly soluble in dilute aqueous sodium hydroxide because the acid is deprotonated by the base producing the sodium carboxylate salt. RCO2H(solv) + OH–(aq) → RCO2–(aq) + H2O(aq) The carboxylic acid can be selectively isolated by dissolving the mixture in an organic solvent that is immiscible with water, and then extracting the solution with sodium hydroxide.

The basic aqueous solution containing the carboxylate salt is acidified, causing the sodium carboxylate salt to convert back to the carboxylic acid, which is not water soluble. The acid will precipitate from the solution, as shown here. RCO2–(aq) + H+(aq) → RCO2H(s) Organic bases (e. g., amines) that are insoluble in water can be separated by extraction with hydrochloric acid. Addition of HCl to the amine produces the corresponding ammonium salt, which is soluble in water but not in organic solvents. The amine can be recovered from the aqueous solution by treatment with a base, converting the ammonium salt back to the amine. The amine is not water-soluble and will precipitate, as shown here. Using your understanding of these properties, separation of a mixture containing a carboxylic acid, an amine, and a neutral compound can be carried out via sequential acid and base

extractions. The precipitates will be collected and characterized by melting temperature analysis.

In this experiment, you will Separate a mixture containing benzoic acid, 3-nitroaniline, and naphthalene. Calculate the percent recovery of each component in the mixture. Measure the melting temperature of each isolated compound.

60 mL separatory funnel four 50 mL Erlenmeyer flasks two 100 mL beakers gravity filtration apparatus vacuum filtration apparatus support ring spatula disposable Pasteur pipets and bulb 10 mL graduated cylinder two watch glasses pH paper balance Part II Melting Temperature weighing paper sample mixture diethyl ether 6. 0 M hydrochloric acid solution 1. 0 M sodium hydroxide solution 6. 0 M sodium hydroxide solution sodium sulfate, Na2SO4, anhydrous cold distilled water in a wash bottle saturated sodium chloride solution ice compressed air

LabQuest or computer interface LabQuest App or Logger Pro Vernier Melt Station glass capillary tubes, one end closed tissues (preferably lint-free) isolated samples from Part I mortar and pestle benzoic acid (optional) 3-nitroaniline (optional) naphthalene (optional)

PROCEDURE

Part I Extraction

1. Obtain and wear goggles. Protect your arms and hands by wearing a long-sleeve lab coat and gloves. Conduct this reaction in a fume hood. 2. Weigh out approximately 1. 0 g of the sample mixture. Record the mass to the

nearest 0. 001 g. Transfer the mixture to a 100 mL beaker and dissolve it in 15 mL of diethyl ether. CAUTION: Diethyl ether is flammable. Be sure that there are no open flames in the room during the experiment. 3. Clamp the support ring onto a ring stand and place the separatory funnel into the ring. Pour the solution into the separatory funnel and add 5 mL of 6. 0 M hydrochloric acid. CAUTION: Handle the hydrochloric acid with care. Can cause painful burns if it comes in contact with the skin. 4. Cap the funnel and gently shake several times, venting frequently to avoid pressure buildup. When venting the funnel, point the tip away from your face and open the stopcock to release the pressure. Place the funnel on a support ring with a clamp and allow the solvent and aqueous layer to separate. Leave the funnel uncapped. 5. Drain the lower aqueous layer into a 50 mL Erlenmeyer flask. Repeat the extraction with another 5 mL of 6. 0 M hydrochloric acid, draining the second aqueous layer into the same Erlenmeyer flask. Save the solvent layer in the separatory funnel for later use. 6. Cool the flask containing the acidic aqueous extracts into an ice water bath. Slowly add 6. 0 M sodium hydroxide with a pipet until the aqueous layer is basic. Use pH paper to test. CAUTION: Sodium hydroxide solution is caustic. Avoid spilling it on your skin or clothing.

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Separation of Organic Compounds by Acid-Base Extraction Techniques 7. Collect the solid using vacuum filtration and save the solid for melting temperature analysis in Part II. Note: Be sure to record the mass of the filter paper before placing it in the vacuum funnel. 8. Extract the saved ether layer in the separatory funnel with three 5 mL portions of 1. 0 M sodium

hydroxide. Drain the aqueous layer into a 50 mL Erlenmeyer flask. Save the ether layer in the separatory funnel. 9. Cool the flask containing the basic aqueous extract in an ice water bath. Using a pipet, slowly add 6. 0 M hydrochloric acid until the aqueous layer is acidic. Use pH paper to test. CAUTION: Handle the hydrochloric acid with care. Can cause painful burns if it comes in contact with the skin. 10. Collect the solid using vacuum filtration and save the solid for melting temperature analysis in Part II. Note: Be sure to record the mass of the filter paper before placing it in the vacuum funnel. 11. Add 10 mL of saturated aqueous sodium chloride solution to the ether solution remaining in the separatory funnel and shake gently. Be sure to vent frequently.

12. Allow the layers to separate and discard the lower aqueous sodium chloride layer. Pour the solvent layer into an Erlenmeyer flask containing approximately 1 g of anhydrous sodium sulfate and allow it to stand for about 10 minutes, swirling occasionally. 13. Gravity filter the contents in the flask, including the sodium sulfate, into a clean 100 mL beaker. Evaporate the ether in the fume hood. CAUTION: Diethyl ether is flammable. Be sure that there are no open flames in the room during the experiment. Do not leave unattended 14. Weigh the recovered solid and record the mass to the nearest 0. 010 g. Save the solid for the melting temperature analysis in Part II. Part II Melting Temperature

15. Obtain a small amount of the isolated solid from the acid extraction. The solid should be in a powdered form. If it is not, use a mortar and pestle to carefully grind the solid to a powder. Pack a capillary tube ~4 mm (~1/8 inch) deep with your sample. 16. Check the control dial on the Melt Station to

confirm that it is in the Off position. Connect the Melt Station power supply to a powered electrical outlet. 17. Connect the Melt Station to a LabQuest or to a computer interface. Choose New from the File menu of the data collection program. 18. Carefully insert the capillary tube of solid into one of the sample holders of the Melt Station. 19. Begin collecting melting temperature data using the Melt Station. 20. Adjust the control dial in order to determine the approximate melting temperature range for the sample. 21. When finished, stop data collection and turn the dial to the Fan/Cooling setting. Record the melting temperature range in your data table.

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Experiment 5 22. Store the run and collect a second run, if desired. 23. Repeat the necessary steps to collect melting temperature data for the isolated solids from the base and neutral extractions. 24. At the end of the experiment turn the control dial on the Melt Station to Off. Dispose of the capillary tubes as directed by your instructor.

DATA TABLE

Part I Extraction Mass of mixture (g) Mass of filter paper (g) Mass of filter paper and benzoic acid (g) Mass of benzoic acid (g) Mass of filter paper (g) Mass of filter paper and 3-nitroaniline (g) Mass of 3-nitroaniline (g) Mass of naphthalene (g) Part II Melting Temperature Measured melting temperature range (C) Benzoic acid 3-nitroaniline Naphthalene

DATA ANALYSIS

1. Draw the structure of each of the compounds. 2. Outline a flow chart describing the separation of the mixture and the isolation of each compound.

3. What was your percent recovery for each of the three compounds? Assume equal amounts of the carboxylic acid, amine, and neutral compound were present in the unknown mixture.

Safety information Essential instructor background information Directions for preparing solutions Important tips for successfully doing these labs

The complete Organic Chemistry with Vernier lab manual includes 26 labs and essential teacher information. The full lab book is available for purchase at: http://www. vernier. com/products/books/chem-o/

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