Purpose

The goal of this experiment is to extract benzoic acid from an aqueous benzoic acid solution using dichloromethane. You will compare the effectiveness of extracting with a single large portion of DCM to the extraction with three smaller extractions. The effectiveness will be evaluated by calculating the distribution coefficient.

Learning Objectives

Perform a liquid-liquid extraction.

Evaluate the effectiveness of a a single extraction vs several small extractions.

Laboratory Skills

Use a separatory funnel.

Perform a liquid-liquid extraction.

Calculate K_d, the distribution coefficient for an extraction process.

Equipment

- Separatory funnel

Chemicals

- Benzoic acid
- Dichloromethane

Introduction

Extraction

This week's experiment begins a three-week study of extraction including acid-base chemistry. Extraction is one of the most common techniques used in organic chemistry. Extraction is a term used for obtaining a substance from a mixture by mixing two immiscible liquids. Liquids are immiscible if they do not mix with each other (such as oil and water).

The density of a liquid tells you where it will be in relation to another liquid. In other words, given a pair of immiscible liquids, the liquid with the greater density will be on the bottom of the immiscible mixture. In an ether-water system, water has a larger density and is on the bottom. In a water-dichloromethane system, the

organic solvent has a larger density and is on the bottom. Look up the densities of water and dichloromethane to confirm that dichloromethane has a larger density.

After an organic reaction, extraction is often performed to isolate the desired product. The extraction of a compound from one liquid into another is an equilibrium process and the extent to which a compound is extracted is measured by its solubility in each of the solvents involved.

Experimental Overview

In our experiment, we start with benzoic acid dissolved in water (aqueous layer). We will use dichloromethane (DCM) for the other immiscible solvent. The extraction process will use a separatory funnel. The purpose of this experiment is to study the theory of extraction. You will begin with an aqueous solution of benzoic acid of known concentration. Benzoic acid is an organic acid that is slightly soluble in water but more soluble in organic solvents such as dichloromethane.

You will "extract" the benzoic acid *from* the aqueous solution *into* the organic solvent. You will then determine the extent to which the benzoic acid was extracted by calculating the distribution coefficient, K_d, for the extraction.

Benzoic acid is much more soluble in DCM than in water, so most of the benzoic acid will migrate to DCM (organic layer). The organic extractions are collected, the DCM evaporated, and the resulting crystals weighed to determine the amount of material that went into the organic layer (and from that, how much was left in the water layer).

Calculation of Distribution Coeffecients

For our experiment, water is the original solvent and dichloromethane is the extracting solvent.The extraction of a compound from one liquid into another is an equilibrium process and the extent to which a compound is extracted is measured by its solubility in each of the solvents involved. In essence, we are looking at a ratio:

$$\frac{\text{material extracted into the organic layer}}{\text{material remaining in the water layer}} \qquad \text{(Equation DC.1)}$$

The ratio of solubilities is called the distribution coefficient, K_d.

$$K_d = \frac{[A]_{S_x}}{[A]_{S_o}} \qquad \text{(Equation DC.2)}$$

where S_x is the extracting phase

S_o is the original phase

$[A]_{S_x}$ is the concentration of the compound in the extracting solvent

$[A]_{S_o}$ is the concentration of the substance in the original solvent

Keep in mind that S_x should be the solvent in which A is more soluble. The concentration of A ([A]) can be measured in any concentration unit, for our experiment, it will be g/mL (grams of benzoic acid in mL of solvent).

To a rough approximation, the ratio of concentrations in this equation is the same as the ratio of solubilities of the compound in the two solvents.

Distribution Coefficient for Benzoic Acid Extraction

In this experiment, you will be extracting benzoic acid (BA) from 70 mL of 0.020 M aqueous benzoic acid with either 1-30 mL portion of DCM or 3-10 mL portions of DCM. Using the data acquired, you will calculated K_d as shown in Example DC.1.

Example DC.1

0.020 M Benzoic Acid solution	70 mL
Dichloromethane	30 mL
Erlenmeyer Flask, empty	104.801 grams
Erlenmeyer Flask + benzoic acid	104.918 grams

Initial Benzoic Acid in Aqueous Layer:

Moles of BA = 0.070 L ×0.020 M (moles/L) = 0.0014 moles of BA

Grams of BA = 0.0014 moles ×122 g/mole = 0.171 grams of BA

Benzoic Acid *After* Extraction:

Grams of BA = Erlenmeyer flask + benzoic acid – Erlenmeyer flask, empty

Grams of BA (organic) = 104.918 – 104.801 = 0.117 grams of BA (organic)

Grams of BA (aqueous) = Initial BA – BA (organic) = 0.171 – 0.117 = 0.054 g

Calculate K_d:

$$K_d = \frac{\text{solubility organic layer}}{\text{solubility aqueous layer}}$$

$$= \frac{\frac{0.117g}{30mL}}{\frac{0.054g}{70mL}}$$

$$= 5.06 = 5.1$$

Procedure

Safety Precautions

Always wear safety glasses.

Benzoic acid is an irritant.

Dichloromethane is a neurotoxin and a suspected carcinogen.

Work in teams of two students: one student in the team will do the "single" extraction and the other student will do the "multiple" extraction. Each person will use their own separatory funnel. Make sure you collect your partner's data before leaving lab so that you use this data for your report.

Procedure for Single Extraction

1. Set up the separatory funnel using a ring and ring stand as shown in the Figure DC.2. Be sure the stopcock is closed before you pour any liquid into the funnel.

2. Label a clean, dry 125 mL Erlenmeyer flask "Organic Layer" and weigh the flask accurately (get a tare weight).

   > Use the same balance to weigh the flask at the end of the experiment.

3. Pour 70.0 mL of 0.020 M aqueous benzoic acid solution into the separatory funnel. Be as accurate as possible in measuring this quantity.

   > When dispensing solutions, please take only the amount required.

4. Add 30 mL of CH_2Cl_2 to the separatory funnel.

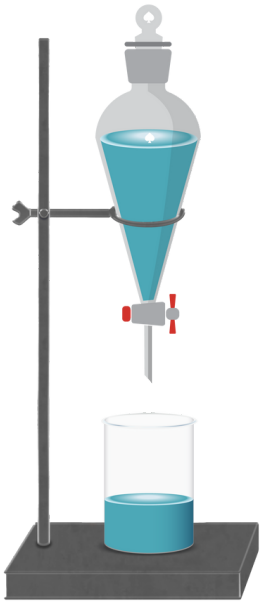

Figure DC.1: Separatory funnel for macroscale separation of organic and aqueous layers

5. Stopper the funnel. Keep one finger on the stopper, invert, and vent by opening the stopcock away from you and others in the laboratory. Close the stopcock. Shake thoroughly, keeping one finger on the stopper and venting the funnel occasionally. Shake well but not too vigorously.

6. Place the funnel upright into the ring and allow the layers to settle and separate.

7. Remove the stopper and drain the lower organic layer into an Erlenmeyer flask labeled ORGANIC LAYER.

8. Pour the aqueous layer out through the top of the funnel into a beaker.

> When performing an extraction, do not throw away any fractions until you are absolutely sure you no longer need that layer.

9. Evaporate the dichloromethane solvent from the organic layer using the recirculating water bath in the hood.

> Your TA will demonstrate and explain how to use this water bath.

10. After the dichloromethane has evaporated, dry the outside of your flask completely and weigh your Erlenmeyer flask.

11. Record masses in the Report Sheet.

12. Clean the glassware.

13. Dispose of solutions and samples in the appropriate waste containers.

Procedure for Multiple Extractions

1. Set up the separatory funnel using a ring and ring stand as shown in the Figure DC.2. Be sure the stopcock is closed before you pour any liquid into the funnel.

2. Label a clean, dry 125 mL Erlenmeyer flask "Organic Layer" and weigh the flask accurately (get a tare weight).

   > Use the same balance to weigh the flask at the end of the experiment.

3. Pour 70.0 mL of 0.020 M aqueous benzoic acid solution into the separatory funnel. Be as accurate as possible in measuring this quantity.

   > When dispensing solutions, please take only the amount required.

4. Add 10 mL of CH_2Cl_2 to the separatory funnel.

5. Stopper the funnel. Keep one finger on the stopper, invert, and vent by opening the stopcock away from you and others in the laboratory. Close the stopcock. Shake thoroughly, keeping one finger on the stopper and venting the funnel occasionally. Shake well but not too vigorously.

6. Place the funnel upright into the ring and allow the layers to settle and separate.

7. Remove the stopper and drain the lower organic layer into an Erlenmeyer flask labeled ORGANIC LAYER. Leave the aqueous layer in the funnel

8. Add another 10 mL of CH_2Cl_2 to the separatory funnel and repeat the shaking steps.

9. Remove the stopper and drain the lower organic layer into the same Erlenmeyer flask labeled ORGANIC LAYER. Leave the aqueous layer in the funnel.

10. Add a third aliquot of 10 mL of CH_2Cl_2 to the separatory funnel and repeat the shaking and extraction steps.

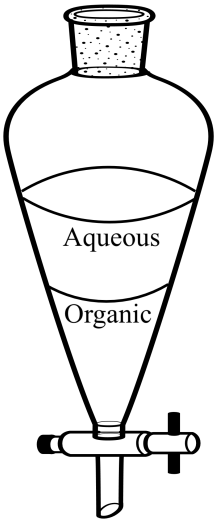

Figure DC.2: Separatory funnel for macroscale separation of organic and aqueous layers

11. Remove the stopper and drain the lower organic layer into the same Erlenmeyer flask labeled ORGANIC LAYER. Leave the aqueous layer in the funnel.

12. Pour the aqueous layer out through the top of the funnel into a beaker.

> When performing an extraction, do not throw away any fractions until you are absolutely sure you no longer need that layer.

13. Evaporate the dichloromethane solvent from the organic layer using the recirculating water bath in the hood.

> Your TA will demonstrate and explain how to use this water bath.

14. After the dichloromethane has evaporated, dry the outside of your flask completely and weigh your Erlenmeyer flask.

15. Record masses in the Report Sheet.

16. Clean the glassware.

17. Dispose of solutions and samples in the appropriate waste containers.

Single Extraction Data

Single Extraction was performed by: _____

1. Benzoic acid in 70.0 mL of 0.020 M BA solution (g): _____

   Space for calculations:

2. Mass of dry Erlenmeyer flask (g): _____

3. Mass of Erlenmeyer flask and benzoic acid after extraction (g): _____

4. Mass of benzoic acid extracted into the organic layer (g): _____

5. Mass of benzoic acid remaining in the aqueous layer (g): _____

6. Calculate K_d from the data collected: _____

   Space for calculations:

$$K_d = \frac{[BA]_{CH_2Cl_2}}{[BA]_{H_2O}} \qquad \text{(Equation DC.1)}$$

Multiple Extraction Data

Multiple Extraction was performed by _____

7. Benzoic acid in 70.0 mL of 0.020 M BA solution (g): _____

Space for calculations:

8. Mass of dry Erlenmeyer flask (g): _____

9. Mass of Erlenmeyer flask and benzoic acid after extraction (g): _____

10. Mass of benzoic acid extracted into the organic layer (g): _____

11. Mass benzoic acid remaining in the aqueous layer (g): _____

12. Calculate K_d from the data collected: _____

Space for calculations:

$$K_d = \frac{[BA]_{CH_2Cl_2}}{[BA]_{H_2O}} \qquad \text{(Equation DC.2)}$$

Comparison of Extractions

13. Compare the single extraction to the multiple extraction. Include the mass of the benzoic acid extracted in each case as well as two K_d values in your argument.

    Amount of benzoic acid removed with a single extraction: _____

    Amount of benzoic acid removed with multiple extractions: _____

    K_d for single extraction: _____

    K_d for multiple extractions: _____

14. Evaluate the effectiveness of the two processes. Is the one single extraction or successive extractions using smaller amounts of solvent more effective? Explain your answer.