Extraction
Liquid-Liquid
Finding the Distribution Coefficient
EXTRACTION

Not this painful – Really!
Purpose

• Learn standard extraction techniques
• Calculate distribution coefficients
• Evaluate extraction efficiency
Types of Extraction

• Solid – Liquid
  – Very common in every day life

• Liquid – Liquid
  – Very common in organic chemistry lab
Extraction

The Goal – to separate a mixture into its components

The Method – differential solubility
Theory of Liquid – Liquid Extraction

- Liquids (solvents) must be immiscible.
- Extraction efficiency depends on differential solubility in the two immiscible solvents.

![Diagram showing liquid-liquid extraction process]

- Add clean immiscible aqueous solvent phase
- Shake or stir to allow molecules to partition
- Phases settle and separate with density

- organic product
- impurity
Theory of Liquid – Liquid Extraction

• Distribution Coefficient

\[ K_D = \frac{\text{solubility in organic solvent (g/100mL)}}{\text{solubility in water (g/100mL)}} \]

- \( K_D < 1 \) The substance is mostly in \( H_2O \)
- \( K_D = 1 \) The substance is mostly in Both
- \( K_D > 1 \) The substance is mostly in Organic
Theory of Liquid – Liquid Extraction

- Properties of extraction solvents
  - high solubility of the organic compound
  - immiscible with water
  - relatively low boiling point
  - nontoxic, nonreactive, available, inexpensive

- Density determines top and bottom layer
  - How can you determine which layer you have if you are confused?

- Common extraction solvents
  - methylene chloride, CH2Cl2
  - diethyl ether, CH3CH2OCH2CH3
  - hexane, C6H14
  - ethyl acetate, CH3CO2CH2CH3
Theory of Liquid – Liquid Extraction

• If you have a limited amount of solvent with which to do the extraction and you want to get the maximum amount of product, should you use all the solvent in one portion, or should you go with two or three smaller portions?

Use smaller, multiple extractions!!
Extraction Mechanics

- **Shaking**
  Establish equilibrium concentration of solute between the two immiscible solvents.

- **Venting**
  Release pressure build-up.

- **Separating layers**
  Always remove *bottom* layer through the bottom
  Allow time to separate
  Drain some, repeat to get all of the solvent

- **Drying**
  Remove traces of water from organic solvent.

- **Remove solvent**
  - Isolate extracted compound.
The Particulars

- Determine the distribution coefficient of benzoic acid in methylene chloride.
- Compare the extraction efficiency of smaller and larger volumes given the same total volume.
In this example, we'll use an organic solvent that is more dense than water. So it will be the bottom layer.

1. Compound X
2. Shake
3. Let stand
4. Evaporate solvent (low heat)
5. Use pipet to transfer organic layer
6. Pipet
7. Let it cool, then weigh it.
8. Reheat, cool, and reweigh if time allows.
**K_D Sample Calculation**

<table>
<thead>
<tr>
<th></th>
<th>methylene chloride/H_2O</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial mass of unknown</td>
<td>0.097g</td>
</tr>
<tr>
<td>Solvent volume</td>
<td>1.97mL</td>
</tr>
<tr>
<td>Water volume</td>
<td>2.05mL</td>
</tr>
<tr>
<td>Mass of unknown in solvent</td>
<td>0.059g</td>
</tr>
<tr>
<td>Mass of unknown in water</td>
<td>0.097g-0.059g=0.038g</td>
</tr>
</tbody>
</table>

\[
K_D = \frac{0.059g/1.97mL}{0.038g/2.05mL} = 1.6
\]
Suppose you had 5.0 mmol of benzoic acid to start.

1 extraction: 0.88 mmol of benzoic acid remained in aqueous layer after extracting 50 mL of benzoic acid aqueous solution with 20 mL of CH₂Cl₂.
Sample Calculation of $K_d$ con’t

$$K_d = \frac{4.12 \text{ mmol of benzoic acid in CH}_2\text{Cl}_2}{20 \text{ mL}} \div \frac{0.88 \text{ mmol of benzoic acid in water}}{50 \text{ mL}}$$

$$K_d = 11.7$$
Double Extraction

Suppose you had 5.0 mmol of benzoic acid to start

2 extractions: 0.88 mmol of benzoic acid remained in aqueous layer after extracting 50 mL of benzoic acid aqueous solution with 20 mL of CH$_2$Cl$_2$ in two 10-mL extractions.
Double Extraction con’t

\[
K_d = 11.7 = \frac{X \text{ mmol in } \text{CH}_2\text{Cl}_2}{10 \text{ mL}} \quad \frac{(5.0 - X) \text{ mmol in water}}{50 \text{ mL}}
\]

\(X = 3.50 \text{ mmol in } \text{CH}_2\text{Cl}_2\) (end of 1st extraction)

\[
K_d = 11.7 = \frac{X' \text{ mmol in } \text{CH}_2\text{Cl}_2}{10 \text{ mL}} \quad \frac{(1.50 - X') \text{ mmol in water}}{50 \text{ mL}}
\]

\(X' = 1.05 \text{ mmol in } \text{CH}_2\text{Cl}_2\) (by 2nd extraction)

\(X + X' = 4.55 \text{ mmol in } \text{CH}_2\text{Cl}_2\) by both extractions

\textbf{4.55 mmol in } \text{CH}_2\text{Cl}_2 \text{ compared to } \textbf{4.12 mmol in } \text{CH}_2\text{Cl}_2