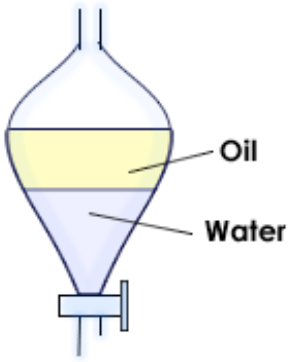
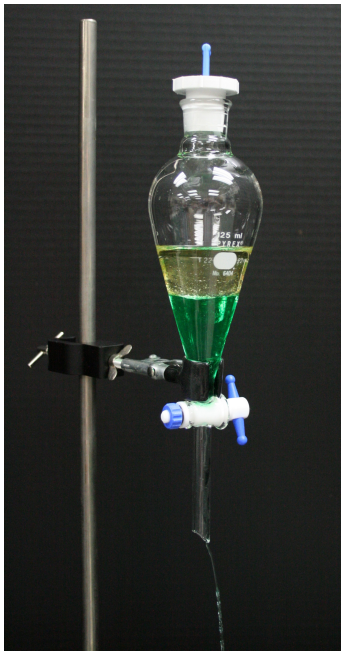
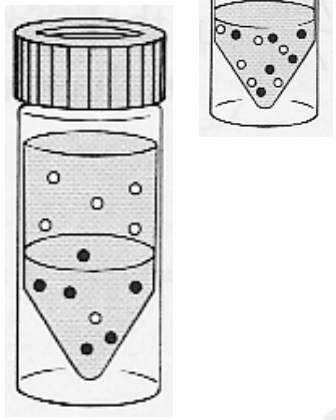
Ex04

Extraction

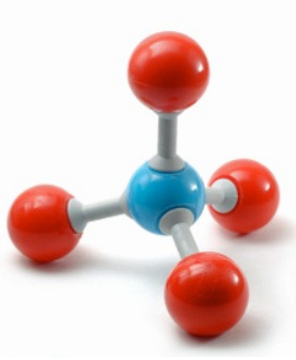
- Extraction
  - Relative Solubility
  - Distribution
    - Solubility into Two Phases
  - Applications
- The Experiment
  - Part A — Extraction of Caffeine
  - Part B — Solute Distribution
- For Next Week





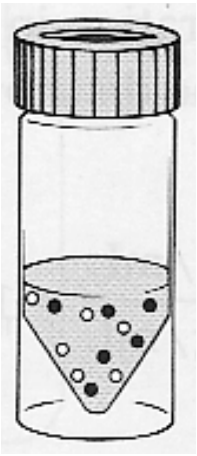


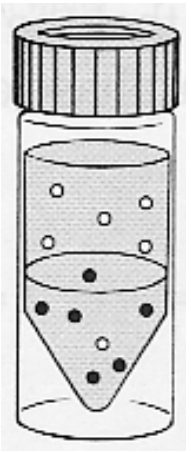
$$K = \frac{50.\ mg/mL}{10.\ mg/mL} = 5.0$$

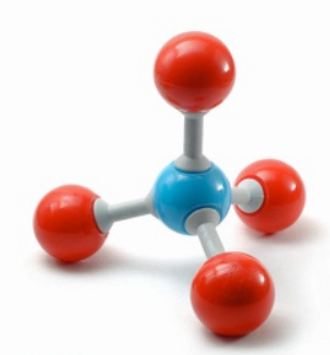


Relative Solubility

- We've seen why solubility exists and how it differs between solvent-solute pairs.
- One way of separating mixtures is to take advantage of this difference causing a chosen substance to loose solubility and separate as (relatively) pure crystalize phase.
- Not all substances can be crystalized.
- Another technique is introduce a second immiscible solvent, in which the desired substance has greater solubility.
  - And undesired solutes have lesser solubility.
- With agitation, the desired solute will preferentially move to the new solvent.
    - Producing a mixture more pure in the desired solute.
- Extraction is a technique for separating a desired substance from a mixture using difference in relative solubility of two solvents.







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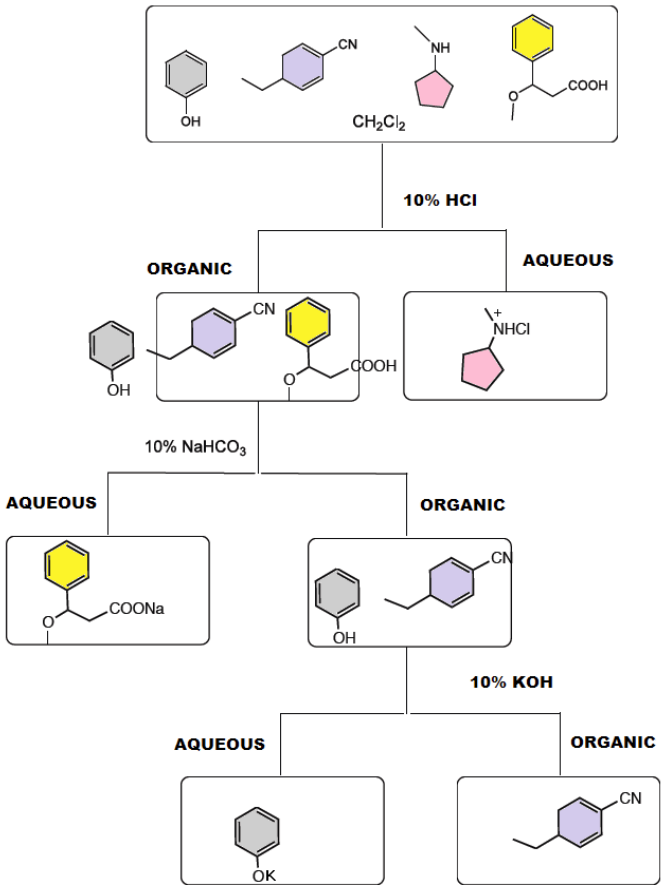

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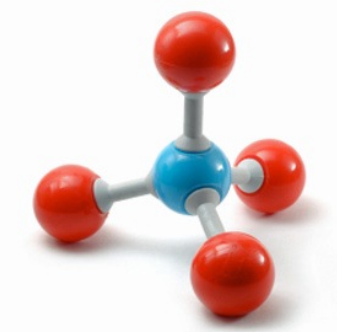
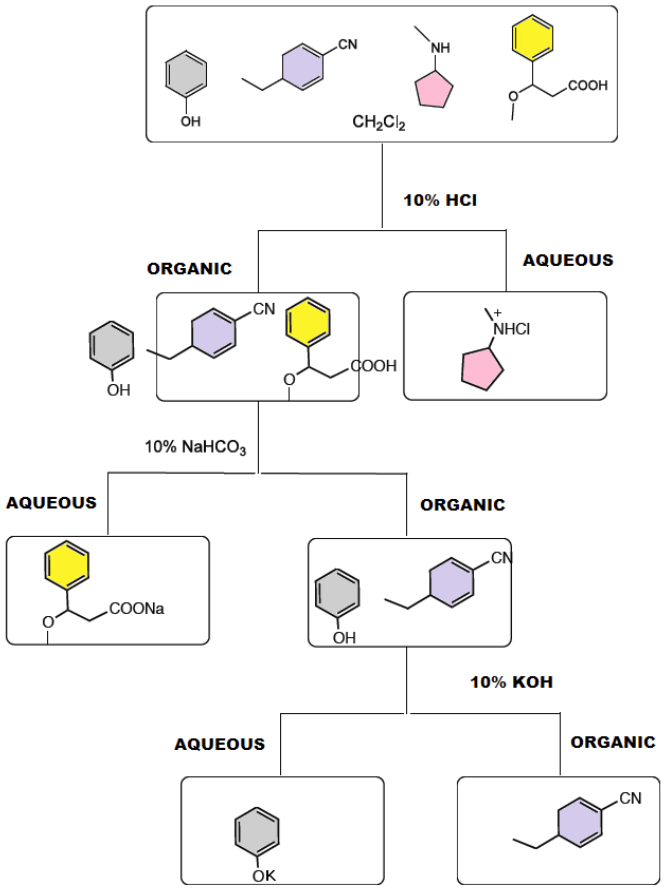
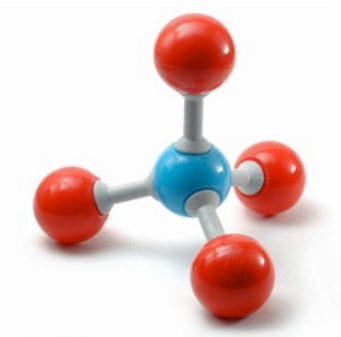


Relative Solubility

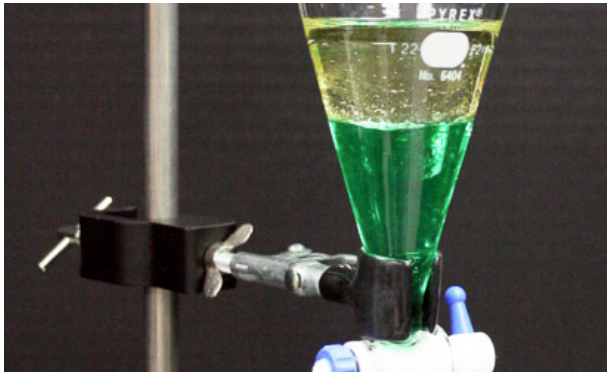
- Extraction requires experimenting with many solvents for a particular
- Identifying factors in the molecular structure will let you predict effective solvent combinations for extraction.
  - Look for:
    - Alkane/Aryl Groups
    - Polar Groups
    - Hydrogen Bonding Groups
    - Acid-Base Groups
- Quantifying the relative solubility of solvent-solute pairs is also valuable.





Distribution

- Distribution coefficient:
  - The ratio of concentrations of a compound in a mixture of two immiscible phases at equilibrium.
  - A measure of the difference in solubility of the compound in these two phases.
    - How a solvent relates to another solvent — for a particular solute.

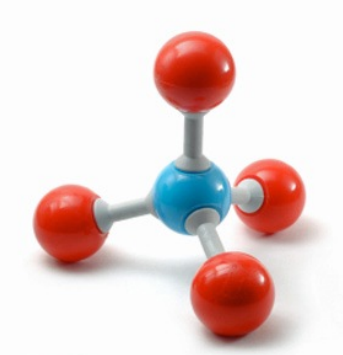


$$K = \frac{C_A}{C_B}$$

$$C_A = \frac{\text{solute dissolved}}{\text{solvent volume}} = \frac{50.mg}{1.0mL} = 50.\ ^{mg}/_{mL}$$

$$C_B = \frac{\text{solute dissolved}}{\text{solvent volume}} = \frac{10.mg}{1.0mL} = 10.\ ^{mg}/_{mL}$$

$$K = \frac{50.\ ^{mg}/_{mL}}{10.\ ^{mg}/_{mL}} = 5.0$$



Start

50.0 mg compound in
1.00 mL water

Extraction

$$K = 10 = \frac{\left(\frac{50.0 - x}{1.50}\ \frac{\text{mg}}{\text{mL ether}}\right)}{\left(\frac{x}{1.00}\ \frac{\text{mg}}{\text{mL water}}\right)}$$

$$10 = \frac{(50 - x)\,(1.00)}{1.50x}$$

$$15.0x = 50.0 - x$$
$$16.0x = 50.0$$

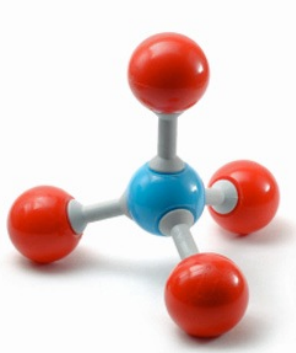
$x = 3.1$ mg in water
$50.0 - x = 46.9$ mg in ether

Finish

(50.0 – 3.1) =

46.9 mg compound in
1.50 mL ether

3.1 mg compound left in
1.00 mL water



Ex04

Extraction

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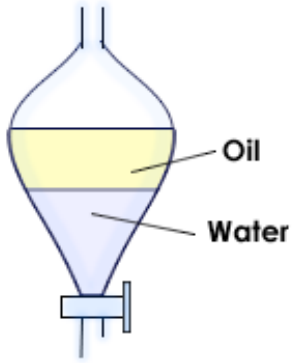
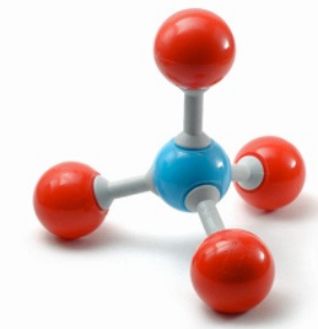
  - Relative Solubility
  - Distribution
    - Solubility into Two Phases
  - Extraction Applied



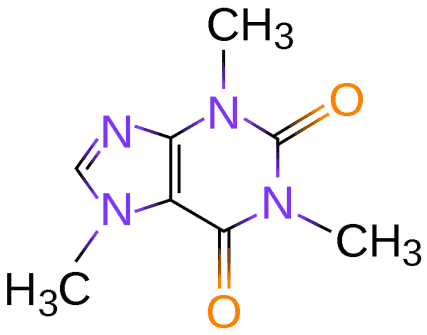
- The Experiment
  - Part A — Extraction of Caffeine
  - Part B — Solute Distribution

- For Next Week

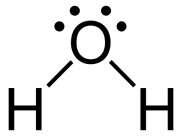


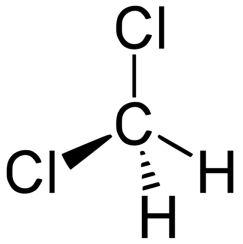
Extraction of Caffeine

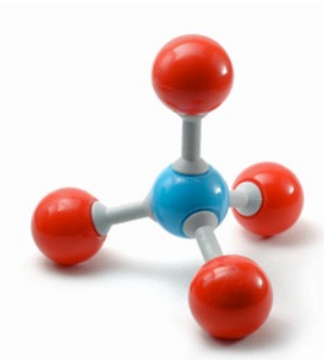
- Goal:
  - Learn how differences in solubility can be used to isolate organic compounds from complex mixtures.
- Objective:
  - Extract caffeine from a water solution.
  - Determine the % of caffeine that you can extract from water into methylene chloride.



Caffeine

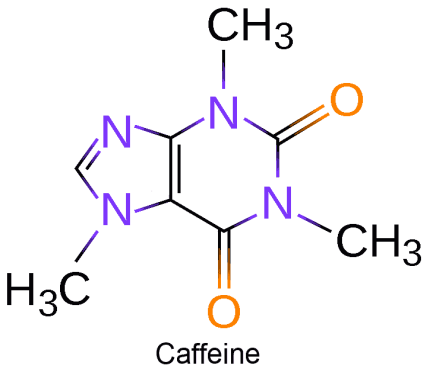




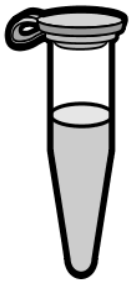


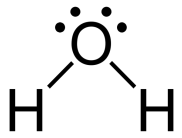
Extraction of Caffeine

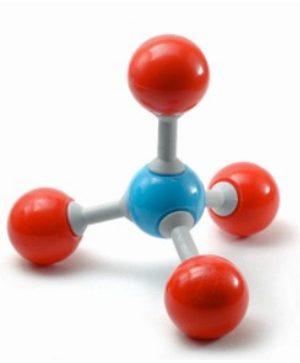
- Preparation:
  - Test your screw cap centrifuge for leaks.
    - Fill it half way with demonized water and cap it.
    - Shake it cautiously and then vigorously to see if any water escapes.
  - Combine 70. mg of Caffeine and 4.0 mL of deionized water in a screw cap centrifuge tube.
  - Shake it vigorously to dissolve the caffeine.
  - Gentle heating with a hot water bath may be required to completely dissolve the caffeine.



Caffeine

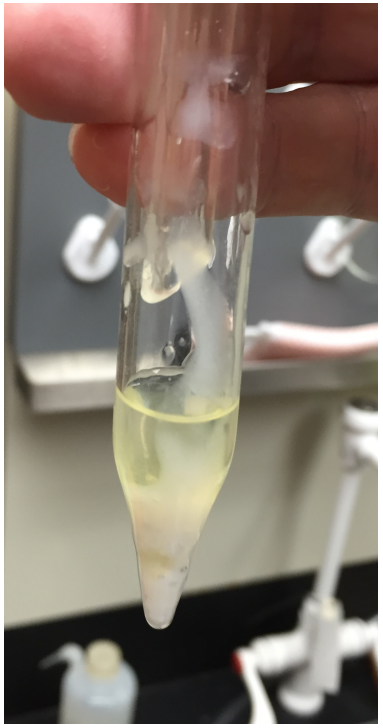


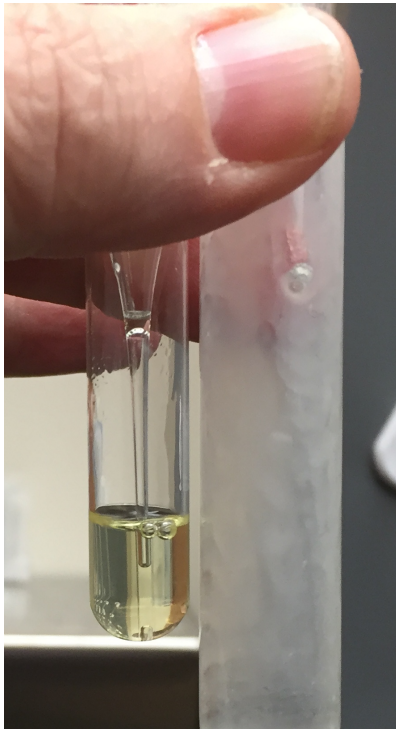


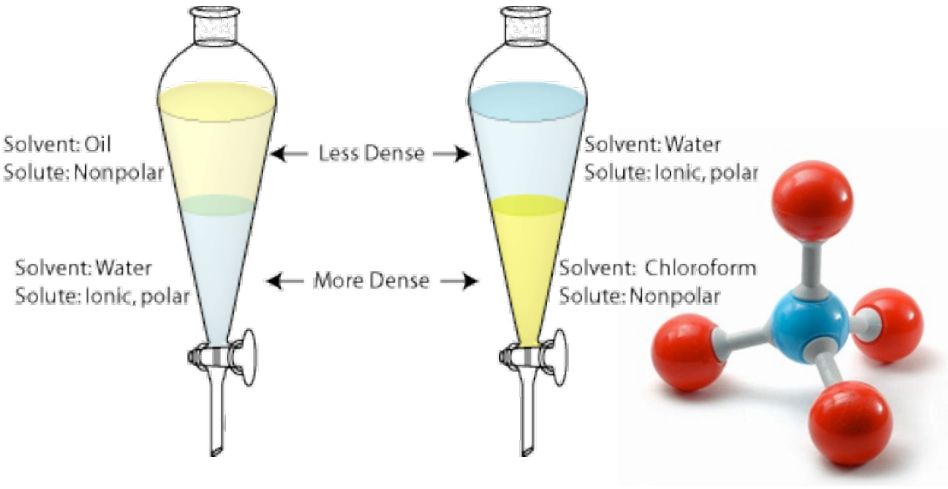


Extraction of Caffeine

- Extraction:
  - Add 2.0 mL of methylene chloride (CH_2Cl_2) to your tube.
  - Seal the tube.
  - Rock the tube:
    - Invert and right it, about twice a second.
    - For about a minute.
  - Let the tube stand in a rack, until the layers separate.
  - Identify the layers
    - (halogenated solvents tend to be more dense than water, alkanes tend to be less)
  - Using a pipet transfer the methylene chloride layer into a large test tube.
  - Repeat with two more 2.0 mL CH2Cl2, combining the methylene chloride layers.

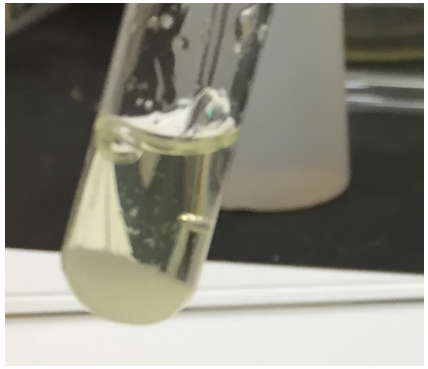


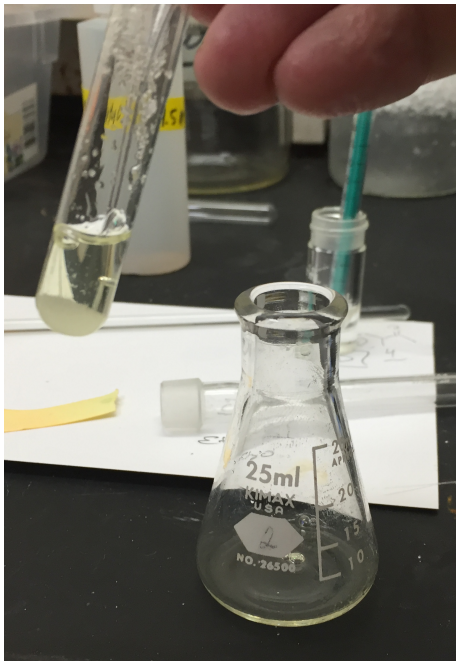


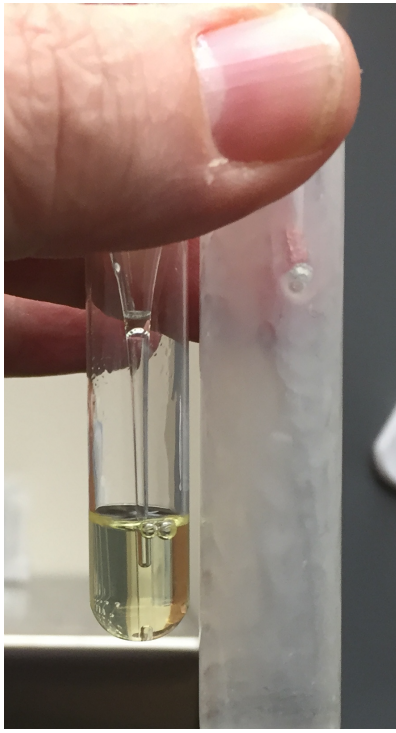


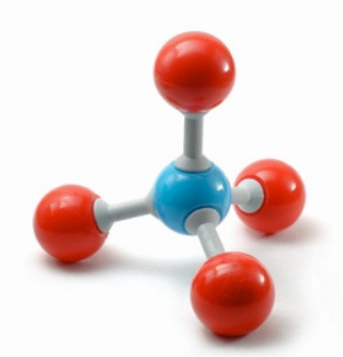
Extraction of Caffeine

- Drying:
  - Using the small end of the spatula, add one measure of sodium sulfate (Na_2SO_4) to your extracts.
    - Look for clumping of sodium sulfate (swirl tube or stir with clean dry micro spatula).
  - Continue adding small quantities of Na_2SO_4 until clumping occurs.
  - You will probably need 2-5 small measures of the drying agent.
    - If you use too much, you may have difficult removing the dried liquid and will get a poor yield.
  - Let solution dry at least 15 minutes.
    - Stir slightly every 2-3 minutes to increase the exposure to the drying agent.
  - Remove the dried solution by pipet to a try pre weighted 10 mL Erlenmeyer flask.



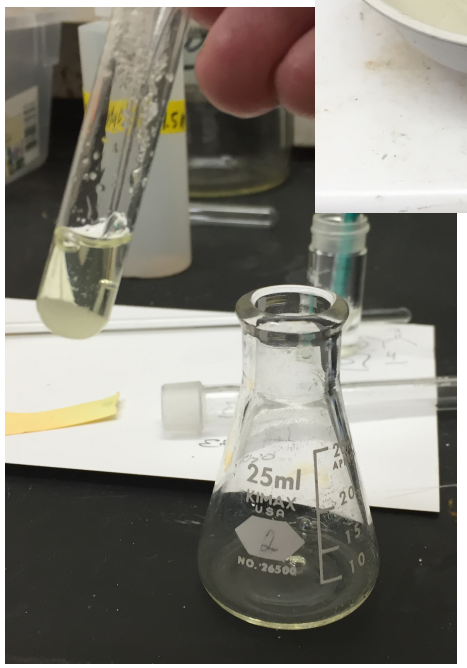


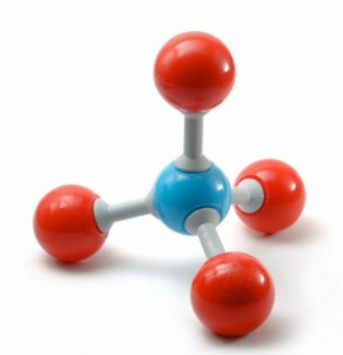




Extraction of Caffeine

- Concentration:
  - Evaporate the solvent using a 45˚C water bath.
    - DO THIS IN THE HOOD
  - Determine the weight of the substance recovered and report the percent caffeine you were able to extract using this technic.





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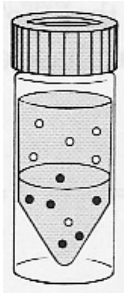

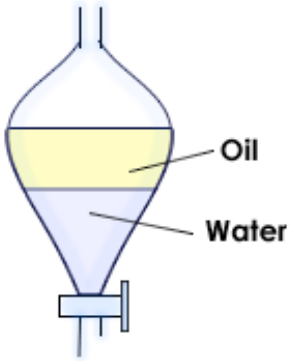

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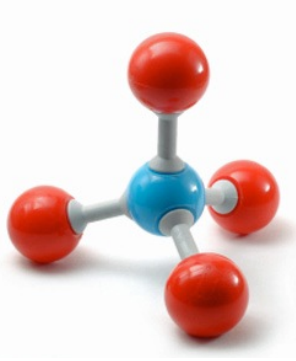
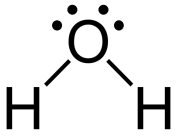
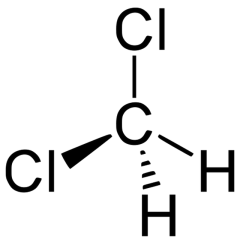
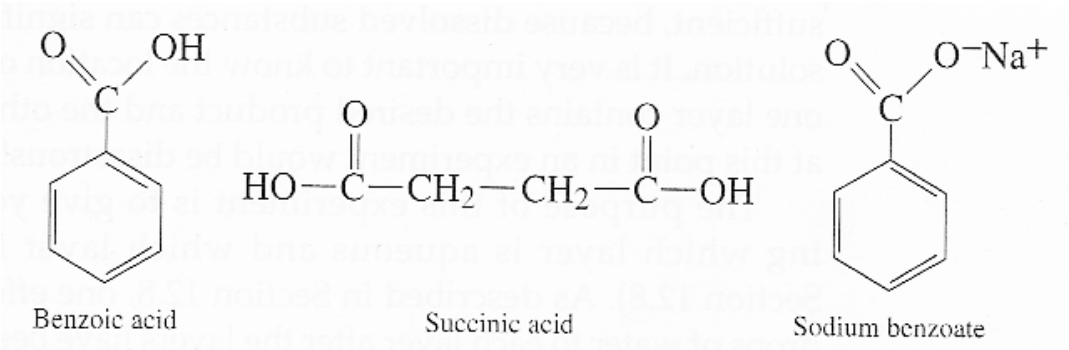


Distribution between Solvents

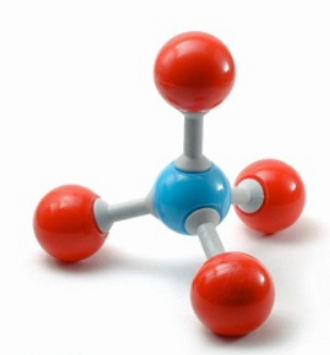
- Goal:
  - Learn how different substances distribute themselves between solvents with varying solubility.
- Objective:
  - Determine the distribution coefficient of substance 1, 2 or 3 between methylene chloride and water.







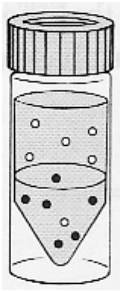
1 2 3

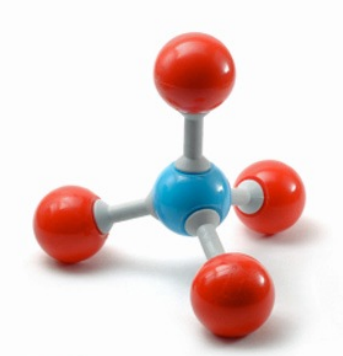


Distribution between Solvents

- Preparation:
  - Determine which unknown you will be working (assigned by instructor)
  - Record your unknown number.

- Procedure:
  - Add 50. mg of your unknown into a 5 mL conical screw cap vial.
  - Add both:
    - 2.0 mL of methylene chloride
    - 2.0 mL of water
  - Cap and shake the vial until all solid has dissolved.
  - By glass pipet transfer the methylene chloride to a test tube and dry with Na2SO4
  - Transfer the dried methylene chloride to a pre-weighted test tube and concentrate to a solid.
  - Determine the weight of the material found in the methylene chloride layer.





Distribution between Solvents

- Analysis:
  - Record the weight of your unknown found the they methylene chloride.
  - By difference determine the weight of your unknown remaining in water.
  - Determine the concentration in mg/mL of unknown in each solvent.
    - Discuss the experiment with two other students and determine the concentration values they determined.
  - Calculate and report the K value for each of the three unknowns.

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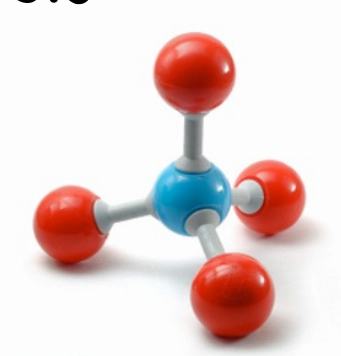

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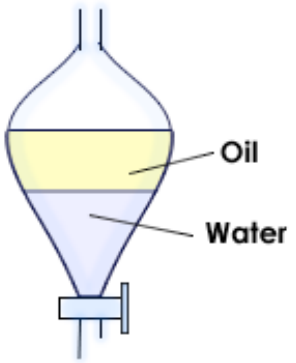
→ For Next Week



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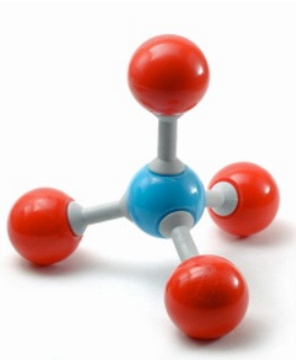
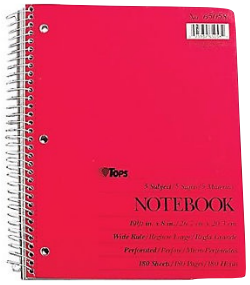


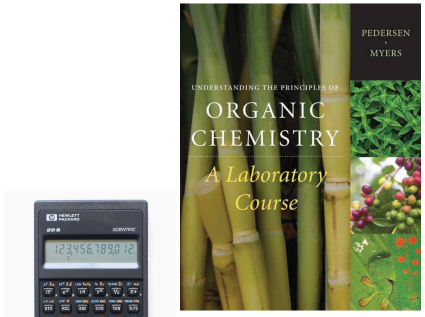
Next Meeting

- For next Meeting:

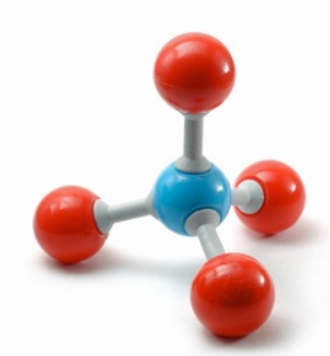
Read: Technique 19 - Column Chromatography
Technique 20 - Thin Layer Chromatography

Do: Identify Objectives
List Materials w/ Properties
Organize Procedures





We will start with a quiz about the experiment and reading.



Questions?