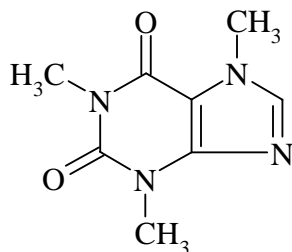


Isolation of Caffeine from Teabags



Caffeine

This experiment will introduce us to 3 new techniques, extraction, recrystallization and melting point determination.

Extraction and Washes

Extractions work by using two non-miscible solvents. Non-polar substances will be more soluble in the non-polar solvent and polar substances will be more soluble in polar solvent.

An extraction is when the majority of the solute (example: caffeine) changes solvents. In this experiment, the caffeine is extracted into methylene chloride from water.

Common polar solvents are water often with acid or base (like NaHCO₃) added to control the pH. Salt (NaCl) is often added to increase the polarity and help exclude organic solutes. Common non-polar solvents include methylene chloride, diethyl ether and petroleum ether (which is a mixture of hydrocarbons, mostly hexane)

A wash occurs when the solute stays in the same phase. We wash our methylene chloride with water(5% NaHCO₃ actually) in order to remove any remaining water-soluble impurities

Melting Point

First, melting point is a misnomer. Solids tend to melt over a range of temperatures. When recording a melting point, you begin with the temperature that the solid begins to melt and you also record the temperature when the solid has completely melted. Please read the web page on melting points. Caffeine actually sublimes.

The Experiment

In this experiment we will isolate caffeine from tea.

In the first step we have to brew a very strong cup of tea. Bring in two tea bags from home. Bring about 100 ml of water to boil in a 150 ml beaker. Add about 4 g of sodium carbonate. Place the tea bags in the boiling water and let them steep for 7 - 10 minutes with a watch glass on the top and the heat on low. Excessive heat can decompose caffeine so do not let the tea dry out.

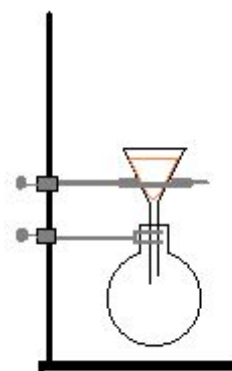
Squeeze the tea bag against the side of the beaker with a stir rod to remove as much of the liquid as possible. Let the solution cool to room temperature.

Pour the tea into the separatory funnel. (Make sure that the stopcock is closed). Add about 10 ml of methylene chloride and shake gently. Vigorous shaking will lead to an emulsion. Allow the layers to separate. Methylene Chloride is denser than water so it will be the lower layer. Draw off the lower layer and add another 10 ml of methylene chloride. Shake and again draw off the methylene chloride layer. Wash the combined organic layers with a 5% NaHCO_3 solution. Dry the methylene chloride layer with anhydrous magnesium sulfate. Water is only slightly soluble in methylene chloride but a quick trick is to reduce the polarity of the mixture by adding a half a ml of hexanes (or petroleum ether). This will increase the rate of water association with the magnesium sulfate. It also provides a quick test for water. If when you add the alkane the solution gets cloudy, there is a good chance that the solution is wet.

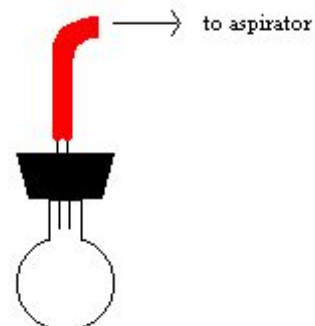


Remove the magnesium sulfate by gravity filtration through filter paper.

Filter into a pre-weighed 50 ml round bottom flask and remove the solvent through low-pressure evaporation. As the methylene chloride evaporates, the flask will get cold. It may help speed the process to have a large dish or bowl filled with room temperature warm water to keep the flask from getting too cold.



Weigh the flask and the product and record the mass and the properties of your crude caffeine.



Find the melting/ sublimation point using the Mel-Temp apparatus.

Post Lab questions:

1. Please read about partition coefficients at

<http://orgchem.colorado.edu/hndbksupport/ext/extsqans.html>

Assume the distribution coefficient, $k = (\text{conc. in } \text{CH}_2\text{Cl}_2 / \text{conc. in water})$ for caffeine is 6.8. Show all work.

- What weight of caffeine would be removed of 10 g of caffeine in 100 ml of water from a single extraction with 100 ml of CH_2Cl_2 ?
- What weight of caffeine would be removed of 10 g of caffeine in 100 ml of water from 4 successive extractions with 25 ml portions of CH_2Cl_2 ?
- Is it better to do one big extraction or multiple smaller extractions or doesn't it matter?

2. Phenol (PhOH) is a weak acid. Its conjugate base is obviously more soluble in water because it is an ionic compound.

a. Will phenol dissolve in a NaHCO_3 solution? (Assume that ionic compounds are soluble and neutral organic compounds are not soluble.) Use pK_a 's and equations and sentences to support your answer.

b. Will phenol dissolve in a NaOH solution? Use pK_a 's and equations and sentences to support your answer.

4. Caffeine (pK_a of conjugate acid is 3.6) is an alkaloid. Alkaloids are organic natural products with an amine functional group.

a. Will Caffeine be more soluble in aqueous solutions of acid (HCl solution) or base (NaHCO_3 solution)? Use pK_a 's and equations and sentences to support your answer.

b. Why was it important to have some base present during the extractions?