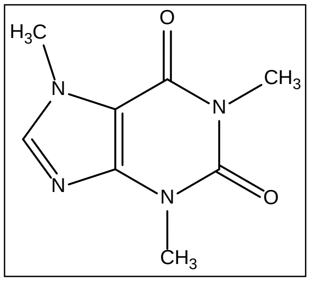
CHEM 30B—Trego

Extraction of Caffeine from Black Tea

Introduction

Caffeine is a naturally occurring alkaloid produced by tea and coffee shrubs. It is a CNS stimulant that is believed to act by serving as an antagonist of adenosine receptors on neurons.



Caffeine is soluble in hot water and is extracted from coffee grounds or tea leaves when these products are brewed. While caffeine is water soluble, it is much more soluble in the organic solvent methylene chloride (CH_2Cl_2). Methylene chloride is immiscible with water and when mixed separates from water to form a two layer mixture. Because methylene chloride is denser than water it usually comprises the lower layer in the two part mixture. By mixing brewed tea with methylene chloride, the caffeine can be extracted into the organic layer. Since the organic layer is immiscible with water, it can be removed after it separates from the water, and the solvent evaporated to give nearly pure caffeine.

Procedure

Preparation of the Brewed Tea

Obtain a tea bag. Weigh the tea bag and record the mass. The instructor will provide the average mass of the empty paper bag so that you can determine the mass of the contents. Place 20 mL of deionized water into a 50 mL beaker. Cover the beaker with your watch glass and heat it on a hotplate until the water is almost boiling. Place the tea bag into the hot water making sure that it lies flat on the bottom of the beaker and is as completely covered with water as possible. Place the watch glass on the beaker and continue heating for 15 minutes. Occasionally push down on the tea bag with a test tube to keep it as fully submerged in water as possible. Be gentle with this step—don't break the bag! If excessive evaporation of water occurs, add more DI water as necessary.

Transfer the brewed tea to two plastic centrifuge tubes using a plastic pipette. Try to keep the liquid in each centrifuge tube equal. Gently press against the tea bag (while it is against the wall of the beaker) with a test tube to squeeze out as much tea solution as possible. Pour 2 mL of hot water over the tea bag while it is sitting on the bottom of the beaker, again pressing gently against the bag with a test tube to release as much tea as possible. Divide this additional liquid equally across the two centrifuge tubes. Add 0.5 g of sodium carbonate (Na_2CO_3) to each tube. Cap the tubes and shake until the solid dissolves. Carefully vent the tubes.

Extraction of the Caffeine from the Tea

When the tea has cooled to room temperature, add 3 mL of methylene chloride (CH_2Cl_2) to each centrifuge tube. Cap the tubes and gently shake for several seconds, carefully vent the tubes to release any pressure buildup by slowly opening the caps. Recap the tubes and shake for about 30 seconds (vent the tube occasionally). An emulsion will likely form during the extraction process. To "break" the emulsion, spin the tubes in the centrifuge for 2-3 minutes. Make sure the centrifuge is balanced with tubes of nearly equal mass opposite of each other in the rotor. The mixture should have two layers—a nearly colorless bottom layer and a dark upper layer. If a third frothy green-brown layer is in between the upper and lower layers, the emulsion is still present and the tubes should be centrifuged again.

Transferring and Drying the Methylene Chloride Solution

Using the plastic pipette, remove the lower organic layer and transfer it to a 25 mL Erlenmeyer flask, try not to transfer any of the dark aqueous layer. Add a fresh 3 mL portion of methylene chloride to each tube, cap and shake for about 30 seconds to extract the tea again. Be sure to occasionally vent the centrifuge tube. Centrifuge the tubes again as described above. Remove the bottom organic layer and combine with the first extracts in the 25 mL Erlenmeyer flask. Again, try not to transfer any of the dark aqueous layer.

Add anhydrous sodium sulfate (Na_2SO_4) to the combined methylene chloride extracts to remove any traces of water. Add small spatula tips full of the sodium sulfate until the crystals no longer clump. Allow this mixture to stand for 10 minutes.

Evaporating the Solvent and Recording Mass of Caffeine

Weigh a second, dry 25 mL Erlenmeyer flask and record the mass on your data sheet. Transfer the dried methylene chloride extracts to the flask using a dry pipette. Be careful not to transfer any of the solids. Place the flask on the steam bath in the fume hood to evaporate the methylene chloride. Remove the flask from the steam bath as soon as all the solvent has evaporated, otherwise you may lose some of the caffeine by sublimation. Dry the outside of the flask with a paper towel, weigh the flask with contents, and record the mass on your data paper.

Measuring the Melting Point of the Recovered Caffeine

Scrape as much of the caffeine from the flask as possible using a spatula. Load a capillary tube with about 2 mm of the caffeine and determine the melting point using the MelTemp apparatus.

Deposit the remaining caffeine in the container in the hood. If time allows, the instructor may demonstrate a method for further purifying the caffeine.

Calculations

1. Determine the mass of the tea leaves (before brewing) by taking the difference between the tea bag (with tea) and the average mass of the empty paper bag itself (provided by the instructor).

2. Calculate the mass percent caffeine in the tea leaves.

3. Suppose your tea bag was used to brew a standard American "cup" of tea (which is actually 6 fluid ounces). Calculate the concentration of the caffeine in the beverage in units of mg of caffeine per fl. oz.

Questions

1. Black tea often contains up to 5% caffeine by mass. How do your results compare with this value? What might account for measuring a percentage lower than this value?

2. The literature melting point of caffeine is 238 ^{o}C. How does your measure melting point compare? What does the melting point measurement tell you about the purity of the recovered caffeine? (Remember, a depressed and broad-ranged melting point is indicative of an impure sample.)