

## Caffeine Experiment

Caffeine is a minor constituent of tea, coffee, and other natural plant materials. The major constituent of tea is cellulose which is not water soluble. Caffeine is water soluble but so are some tannins and gallic acid which is formed in the process of boiling tea leaves. The latter two components can be converted to their calcium salts which are insoluble in water. The caffeine can then be extracted from the water by methylene chloride in almost pure form. Some chlorophyll is often extracted at the same time.

**Procedure:** Place 15 g of tea leaves, 5 g of calcium carbonate powder and 200 mL of water into a 600 mL beaker. Boil the solution on a hot plate for 20 minutes with occasional stirring. Cool the solution but, while it is still warm, vacuum filter through a Buchner funnel using a fast filter paper, if available. Normally, hot solutions are not vacuum filtered. Rinse the leaves with 50 mL of water. Carefully press out as much filtrate as possible since the caffeine is in the aqueous layer. Rinse again with 50 mL of water.

Cool the solution to room temperature and pour it into a 500 mL separatory funnel. Extract with 35 mL of methylene chloride. In a departure from normal procedure, it will be necessary to *vigorously* shake the separatory funnel in order to extract the caffeine. First, relieve the pressure buildup as soon as you mix the two liquids. Then shake vigorously for 10 seconds and relieve pressure, repeat the shaking two more times. An emulsion will probably form.

To break the emulsion formed in the methylene chloride layer, slowly drain the methylene chloride layer through a small amount of anhydrous magnesium sulphate in a powder funnel with a loose cotton plug (a tight plug will prevent drainage).

Extract the aqueous solution once again with a 35 mL of methylene chloride, repeating the steps above to collect the lower layer. Combine the methylene chloride extracts and, if necessary, dry further with additional anhydrous magnesium sulphate.

The methylene chloride solution will be stripped on a roto-evaporator. Tare weigh a 100-mL rb flask and transfer the dried methylene chloride solution to it. Be certain that there is no magnesium sulphate in the solution. Stripping this solution to dryness will take less than 5 minutes. You will be left with a small amount of residue with a greenish tinge. Obtain the weight of crude caffeine by difference.

Add 5-8 mL of hot acetone to dissolve the crude caffeine and transfer the solution to a 50 mL Erlenmeyer flask for recrystallization. Add a few drops of petroleum ether until you reach the cloud point (caffeine is less soluble in this mixed solvent and is just beginning to precipitate) and then cool the solution. If you do not get a precipitate, you may have used too much acetone, carefully boil off the excess on a steam bath using a boiling stick for ebullation.

Suction filter the caffeine using a small Hirsch funnel and petroleum ether as a transfer/rinse solvent. A second crop of caffeine may form in the filtrate as the solvent evaporates. This second crop can also be collected by vacuum filtration but keep it separate from the first crop. After air drying, weigh each crop and record your % caffeine recovered from tea.

The sublimation will be performed as described by your instructor. You will use 50 mg of your caffeine to make a salicylate derivative and sublime the remainder (which should be at least 50 mg).

You will not take a mp of the purified caffeine which would require a sealed capillary to prevent sublimation near the melting point. Save the purified caffeine in a sealed vial. You will use some of this material for TLC analysis next week.

Caffeine is a base which can react with acids to form salts. A well characterized salt of caffeine is caffeine salicylate formed by using salicylic acid. This derivative of caffeine has an accurate melting point. Later this semester, you will be required to make solid derivatives of other compounds.

Preparation of Caffeine Salicylate. Using an analytical balance (there are several top loader balances in the lab across the hall which will quickly weigh to 0.0001 mg), weigh 50 mg of caffeine and 37 mg of salicylic acid (both can be plus or minus 1-2 mg) and dissolve them in 4 mL of toluene in a small 25 mL Erlenmeyer flask by warming on a steam bath. Add 1 mL (dropwise) of petroleum ether and allow the mixture to cool and crystallize. If necessary, cool in an ice-water bath. Collect the crystals by vacuum filtration, air dry, weigh, record the yield, and take a mp (lit mp 137°C).