

“This Caffeine Extraction lab is Tea-riffic!” Merrit DeBartolo, Fall 2002

Tea and coffee have been popular beverages for centuries, primarily because they contain the stimulant caffeine. It stimulates respiration, the heart, and the central nervous system, and is a diuretic (promotes urination). It can cause nervousness and insomnia and, like many drugs, can be addictive, making it difficult to reduce the daily dose. A regular coffee drinker who consumes just four cups per day can experience headache, insomnia, and even nausea upon withdrawal from the drug. On the other hand, it helps people to pay attention and can sharpen moderately complex mental skills as well as prolong the ability to exercise.

Caffeine may be the most widely used and abused drug in the United States. During the course of the day an average person may unwittingly consume up to a gram of this substance. The caffeine content of some common foods and drugs is given in table below.

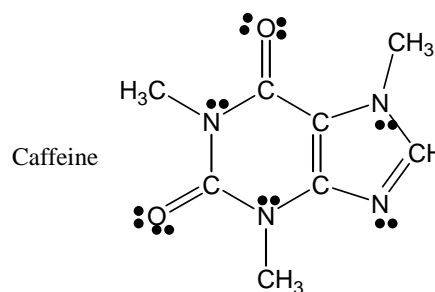
#### Caffeine Content of Common Foods and Drugs

Espresso	120 mg per 2 oz
Coffee, regular, brewed	103 mg per cup
Instant coffee	57 mg per cup
Coffee, decaffeinated	2 to 4 mg per cup
Tea	30 to 75 mg per cup
Cocoa	5 to 40 mg per cup
Milk Chocolate	6 mg per oz
Baking Chocolate	35 mg per oz
Coca-Cola, Classic	46 mg per 12 oz
Jolt Cola	72 mg per 12 oz
Anacin, Bromo-Seltzer, Midol	32mg per pill
Excedrin, Extra Strength	65 mg per pill
Dexatrim, Dietac, Vivarin	200 mg per pill
Dristan	16 mg per pill
No-Doz	100mg per pill

Caffeine belongs to a large class of compounds known as alkaloids. These are of plant origin, contain basic nitrogen, often have a bitter taste and complex structure, and usually have physiological activity. Their names usually end in “ine”; many are quite familiar by name if not chemical structure—nicotine, cocaine, morphine, strychnine.

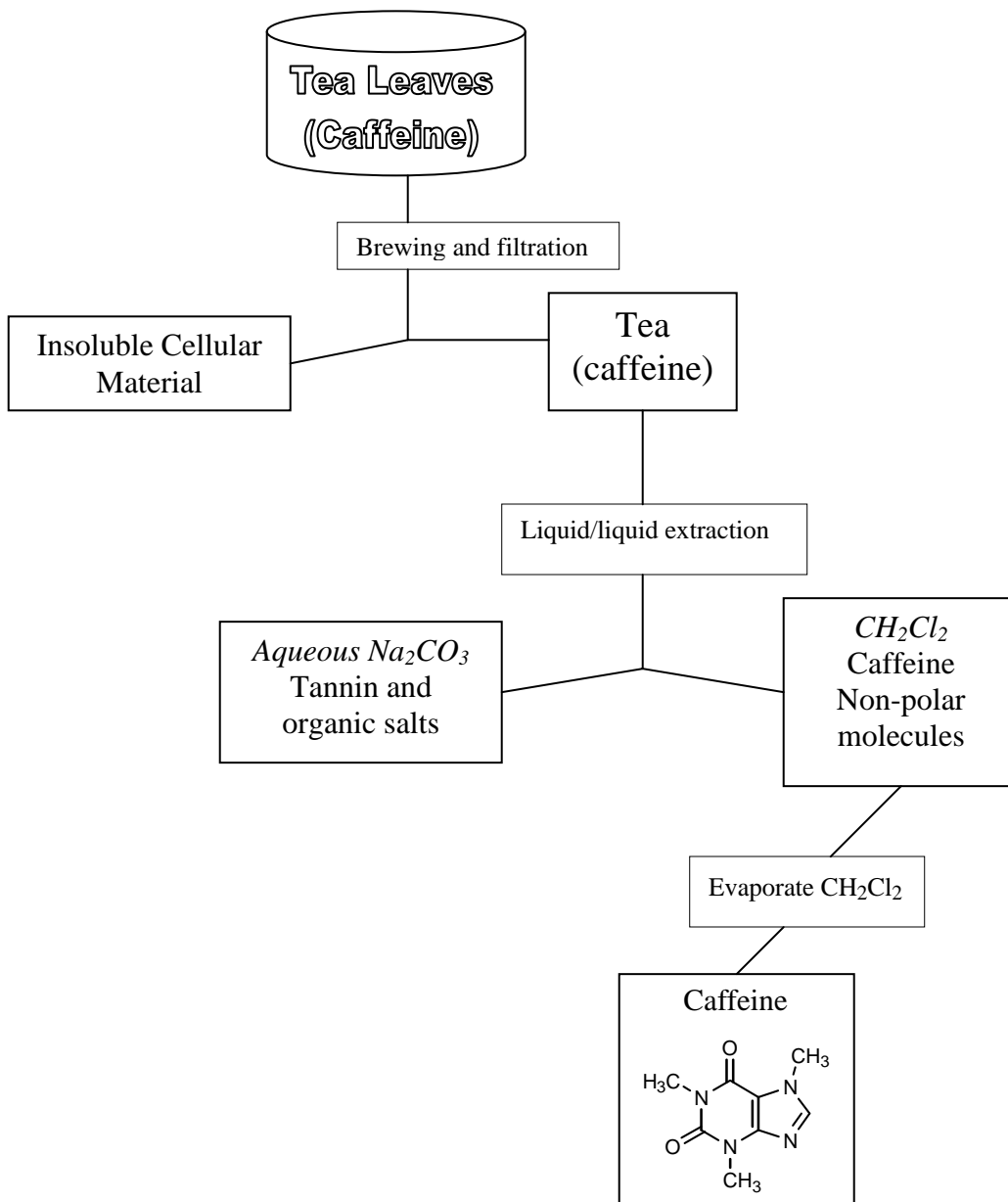
Tea leaves contain tannins, which are acidic, as well as a number of colored compounds and a small amount of undecomposed chlorophyll (soluble in dichloromethane). To ensure that the acidic substances remain water soluble and that the caffeine will be present as the free base, sodium carbonate is added to the extraction medium.

The solubility of caffeine in water is 2.2 mg/mL at 25°C, 180 mg/mL at 80°C, and 670 mg/mL at 100°C. It is quite soluble in dichloromethane, the solvent used in this experiment to extract the caffeine from water.



Caffeine can be extracted easily from tea bags. The procedure one would use to make a cup of tea - simply "steeping" the tea with very hot water for a few minutes - extracts most of the caffeine. Since caffeine is white, slightly bitter, odorless, crystalline solid, it is obvious that water extracts more than just caffeine. When the brown aqueous solution is subsequently extracted with dichloromethane, primarily caffeine dissolves in the organic solvent. Evaporation of the solvent leaves crude caffeine, which on sublimation yields a relatively pure product.

When the concentrated tea solution is extracted with dichloromethane, emulsion can form very easily. There are substances in tea that cause small droplets of the organic layer to remain suspended in the aqueous layer. This emulsion formation results from vigorous shaking.



**Procedure:****A. Crude Caffeine:**

1. Obtain and weigh 5 tea bags.
  2. Add 5 tea bags, 5 g of sodium carbonate and about 200 mL water to a 400 mL beaker.
  3. Heat the water to a vigorous boil with a hot plate. Stir to dissolve  $\text{Na}_2\text{CO}_3$
  4. Allow the mixture to cool for 5 min, and then decant into another beaker.
  5. Gently squeeze the tea bags to liberate the rest of the water.
- Cool the aqueous solution to near room temperature.
  - Continue cooling in an ice bath. Your tea must be cool (about  $20^\circ\text{C}$ ) before coming in contact with dichloromethane (boiling point =  $40^\circ\text{C}$ ).
  - Dichloromethane will irritate your bare skin. Always wear gloves when working with  $\text{CH}_2\text{Cl}_2$

Extract the solution three times with 30-mL portions of dichloromethane ( $\text{CH}_2\text{Cl}_2$ ). Do not get dichloromethane on your hands. Take great care not to shake the separatory funnel so vigorously as to cause emulsion formation, bearing in mind that if it is not shaken vigorously enough the caffeine will not be extracted into the organic layer. Use a gentle rocking motion of the separatory funnel. Take your time. Save and combine the dichloromethane extracts after each extraction.

- the steps of extraction are

- a) pour in 20 mL of dichloromethane.
- b) Stopper the separatory funnel.
- c) Keep your fingers on the stopper and carefully shake the sep funnel.
- d) Vent the sep funnel periodically with the spigot pointing away from you to relieve gas pressure.
- e) When the contents have been sufficiently shaken put the sep funnel back on the ring stand and let the two layers separate out.
- f) Remove the stopper.
- g) Drain the bottom layer into a flask.
- h) Repeat steps a) through g) two more times.

Dry the combined dichloromethane solutions and any emulsion layer with anhydrous Sodium Carbonate ( $\text{Na}_2\text{CO}_3$ ). Add sufficient drying agent until it no longer clumps together on the bottom of the flask – about 1 teaspoon usually works well.

- Carefully gravity filter the dichloromethane solution into a tared (previously weighed) beaker.
- Evaporate the  $\text{CH}_2\text{Cl}_2$  solvent in a hot water bath in the hood. A wooden stick works better than a boiling chip to promote smooth boiling because it is easily removed once the solvent is gone.
- When all the solvent is removed you should observe a residue of greenish-white crystalline caffeine.
- Weigh the beaker plus crude caffeine. You can calculate the mass of your crude caffeine.

- Dispose of used dichloromethane in the liquid waste container provided.
- The tea leaves go in the nonhazardous solid waste container.
- The aqueous layer can be washed down the drain.
- The drying agent can be placed in the nonhazardous solid waste container.

### B. TLC of crude caffeine. (Remember our first experiment?)

1. Prepare a TLC chamber with a dichloromethane/methanol solvent system.
2. Dissolve a small amount of your crude caffeine in a few drops of dichloromethane.
3. Spot a TLC plate with your crude caffeine and the caffeine standard.
4. Develop and visualize under a UV light.
5. Calculate the  $R_f$  value of caffeine and any other compounds that show up on the TLC plate.

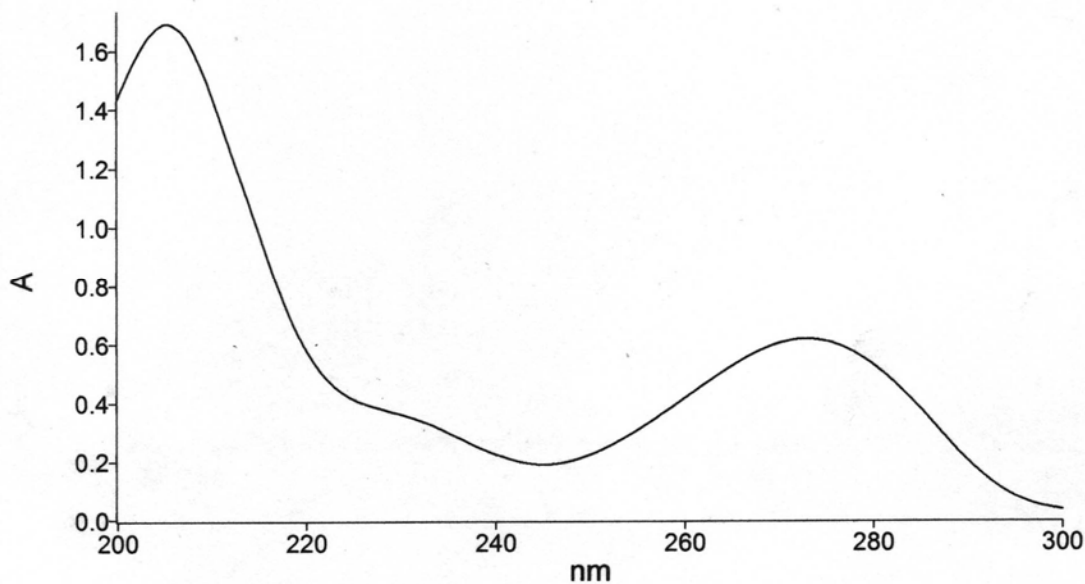
### C. Preparation of UV-vis and HPLC sample

Hand in about half of your recovered caffeine in a properly labeled vial:

- 1) your name, 2) contents of vial [crude caffeine from tea], and 3) date

This will be analyzed by High Pressure Liquid Chromatography (HPLC) and UV-vis spectroscopy

UV-vis spectrum of caffeine in methanol:

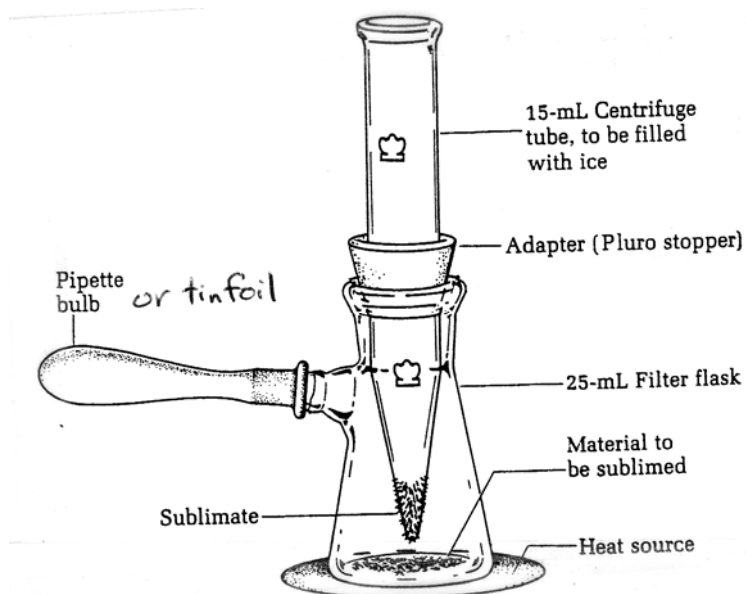


## D. Sublimation of Caffeine

Sublimation is a fast and easy way to purify caffeine. Set up the apparatus depicted below. Use the rest of your crude caffeine left over from part C. If you do not have much substance, combine yours with your hood-mate. Place the dry caffeine powder at the bottom of the flask. Fill the test tube with crushed ice. The bottom of the test tube should rest about 1 cm from the bottom of the filter flask. The test tube and flask should be clean and dry.

Sublime the crude caffeine at atmospheric pressure by placing the flask directly on a pre-heated hot plate. Caffeine melts at  $238^{\circ}\text{C}$  and sublimates at  $178^{\circ}\text{C}$ .

Collect your sublimed caffeine. Put this in a labeled vial and hand it in for analysis.

**Surfing the Web**-----

<http://domin.dom.edu/faculty/jbfriesen/chem253lab/caflab/caflab.htm> Several photos of the Dominican University "extraction of caffeine from tea" by those brave souls who have gone before you.

[http://ull.chemistry.uakron.edu/organic\\_lab/cola/](http://ull.chemistry.uakron.edu/organic_lab/cola/) The extraction of caffeine from 10 mL of cola syrup (Experiment 4) is very clearly demonstrated in 14 color photos.

Checklist for completing the "Prelab" section: (refer to Laboratory Syllabus for complete directions)

\_\_\_ *Title and date*

\_\_\_ *Purpose.* Refer to procedure

*Physical constants.* Create a table of physical constants, solubility, and safety data for:

\_\_\_ caffeine

\_\_\_ dichloromethane (methylene chloride)

\_\_\_ methanol

\_\_\_ sodium carbonate

\_\_\_ **References**

*Structures and equations.* (none)

\_\_\_ *Flowchart.* Refer to "Procedure"

\_\_\_ *Calculations.* If you assume that one teabag makes one cup of tea, about how much caffeine would you expect to extract in this experiment? (Refer to the table on the 1<sup>st</sup> page.) Show your calculations.

\_\_\_ Define the following terms as they apply to HPLC:

1) peak, 2) retention time, 3) adsorbent, 4) eluant

\_\_\_ Safety Question: Dichloromethane is considered to be a volatile respiratory irritant. What two precautions may you take to keep dichloromethane vapors out of your lungs?

Tea, although an Oriental, Is a gentleman at least; Cocoa is a cad and coward, Cocoa is a vulgar beast. <b>G. K. Chesterton</b> , The Song of Right and Wrong
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Experimental Observations and Data:

Hand in a copy of your experimental observations and data before you leave lab.

*Experimental Observations.*

- \_\_\_ What was the appearance of each reagent?
- \_\_\_ Mass of  $\text{Na}_2\text{CO}_3$  used
- \_\_\_ What was the volume of tea at the beginning of your extractions?
- \_\_\_ What did extractions look like?
- \_\_\_ Have you clearly labeled parts A, B, C & D?
- \_\_\_ What were the results of TLC? Calculate  $R_f$  values. Trace slide into notebook. Label clearly
- \_\_\_ How did the crude caffeine differ from the sublimed?
- \_\_\_ Did you record interesting sights and smells?

*Data*

- \_\_\_ Record the mass of your tea bags.
- \_\_\_ Record the mass of your crude caffeine.

Lab Report Checklist:

*Results.*

- \_\_\_ Calculate the percent yield of caffeine from tea. Show your calculations.  
(mass of crude caffeine x 100 / mass of tea leaves)
- \_\_\_ Report your  $R_f$  values from your TLC experiment.
- \_\_\_ Compare the “student sample” UV spectrum with the UV spectrum of pure caffeine. Both can be found on the chem253lab webpage. What are at least two similarities? What are at least two differences?
- \_\_\_ Compare the “student sample” HPLC chromatogram with the HPLC chromatogram of pure caffeine. Both can be found on the chem253lab webpage. What are at least two similarities? What are at least two differences?

*Discussion and Conclusion.*

- \_\_\_ How did your yield compare with your calculations in the prelab? Explain why your observed results (at least 2 reasons) might have been different than the “theoretical yield.”
- \_\_\_ What are two conclusions that you can draw from your TLC, HPLC, and UV data?
- \_\_\_ **Green Question:** Dichloromethane is generally nasty stuff. What could be used to replace dichloromethane in this procedure? Hint: Where does decaffeinated coffee come from? Give sources please.
- \_\_\_ Are you a caffeine user? What effect does caffeine have on you?