A new consumer group, Consumers for the Education of Caffeine Abusers (CECA), has been formed to educate the general public on the ubiquitous presence of caffeine in the American diet. Currently, consumer foods are not required to publish on the label the amount of caffeine their product contains. Consumers must rely on internet sites such as http://wilstar.com/caffeine.htm that publish the caffeine content of various beverages to determine the amount of caffeine they are ingesting. Typically, the simply publish caffeine content data that has been supplied by manufactures. This data may be deliberately falsified to protect their brand or unintentionally misinterpreted due to poor experimental design.

CECA has heard about our recently acquired expertise on the extraction of caffeine from tea and they are soliciting the help of Dominican University's organic chemistry class to help them develop methods to measure the quantity of caffeine in various consumer products. Your task in this lab period will be to design and perform an experimental procedure to isolate caffeine from the beverage of your choice.

Prelab:

____Choose a caffeine source.

____Research the amount of caffeine you would expect to isolate from this source. Websites such as http://wilstar.com/caffeine.htm and http://www.energyfiend.com/the-caffeine-database/ are a good place to start. Record the amount of caffeine and your reference.

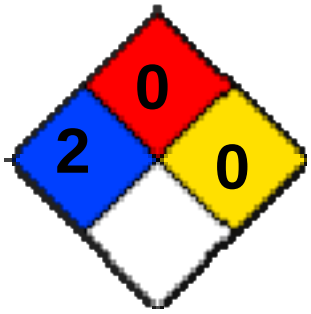
____Design an experimental protocol, based on your experience with the "caffeine from tea" laboratory experiment. Include the glassware and equipment you will use (such as 200 mL beaker and UV-vis spectrophotometer).

____ Consult the OChemOnline 2007 student comments on the "Caffeine" page http://ochemonline.pbwiki.com/Caffeine. Which comment did you find most helpful?

____ Read the Journal of Food Chemistry and Toxicology article, "Caffeine Content of Prepackaged National-Brand and Private-Label Carbonated Beverages" and respond to the following questions.

1) What is the significance (importance) of such as study?
2) From Table 3, how many measurements of RC cola were done?
3) What do the terms "isocratic" and "reverse-phase" mean when applied to HPLC?

____ Safety Question: Here is the NFPA **(**National Fire Protection Association) "fire diamond" for dichloromethane. What does this mean?



In lab:

____Write down a detailed experimental method in your lab notebook of what you did in lab.
____ Include the glassware you used (such as 200 mL beaker and 250 separatory funnel), grams of solid chemicals, and volumes of liquid chemicals.
____ Include the make and model of instruments that are used.
Do not sublime your extracted caffeine.

____Obtain the mass of your extracted caffeine before you subject it to chromatography and spectroscopy.

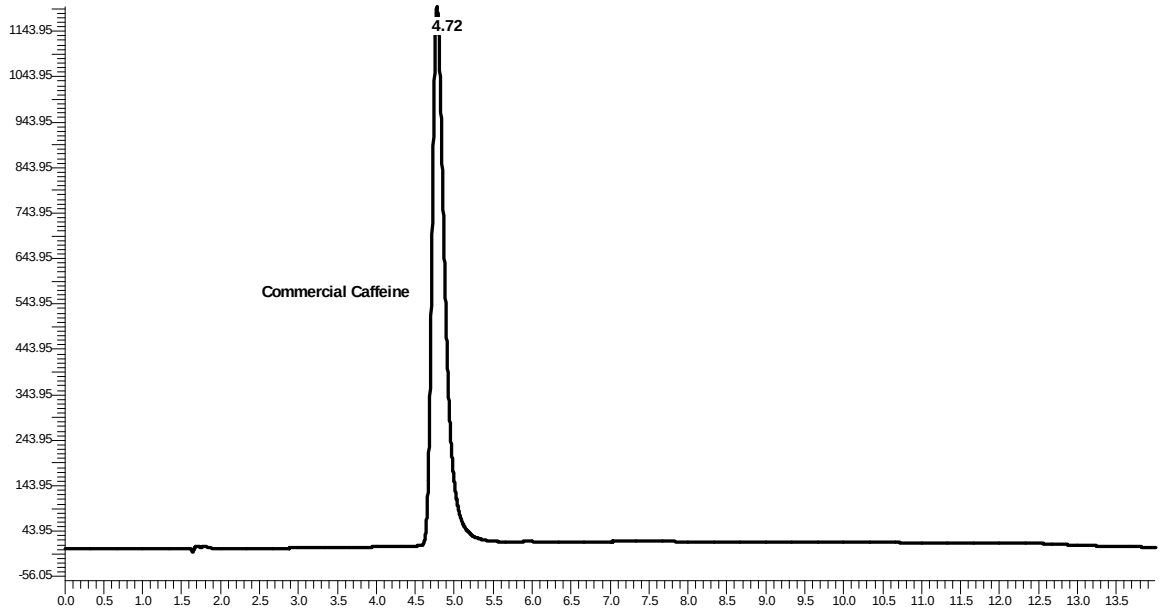
You will perform three experiments to determine the identity and purity of your extracted caffeine.

TLC:

Test your caffeine from this lab side-by-side with your "caffeine from tea" sample and a sample of pure caffeine on a TLC plate (three spots total). Record the results (including R_f values) in your lab notebook. Pay attention to the presence of impurities (non-caffeine spots.

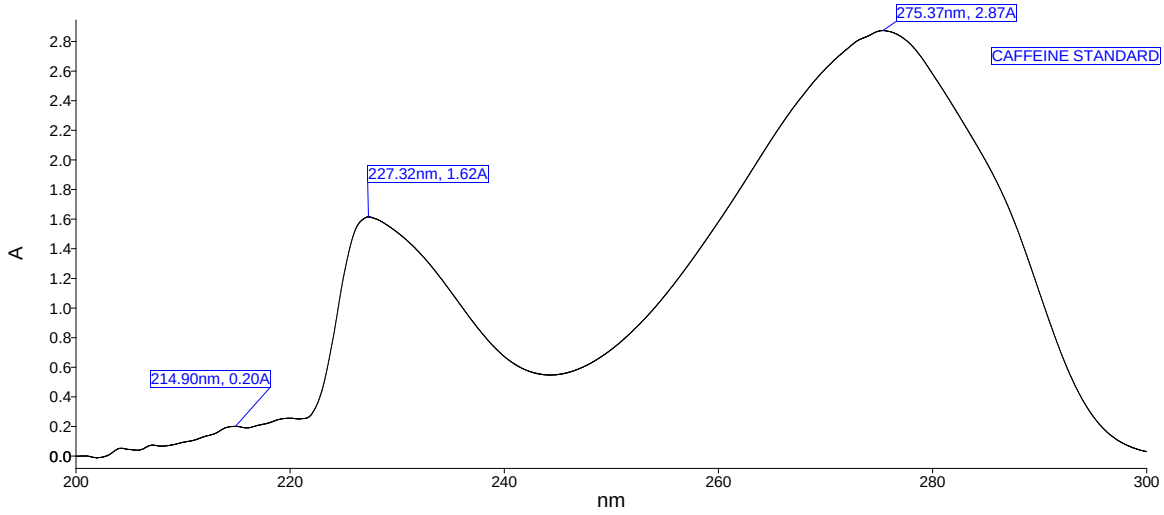
HPLC (High Pressure Liquid Chromatography):

This method gives much the same information as TLC. The caffeine sample is dissolved in a solvent and introduced into a column that separates the compounds in a mixture in much the same fashion as TLC. The eluant of the column is monitored with UV absorption. Compounds show up as "peaks" on the chromatogram.



UV spectrum.

Obtain an ultraviolet (UV) spectrum of your caffeine sample.
You will need to dissolve your sample in dichloromethane to do this.
Determine the wavelength of maximum absorption in nanometers (nm).
Compare your UV spectrum to the spectrum of pure caffeine.



Lab Report:

____ Record the mass of your caffeine.
____ How did your extracted yield compare with your "theoretical yeild?"
____ Interpret the results of the TLC. Where any impurities detected?
____ Interpret the results of your HPLC chromatogram
____ Interpret the results of your UV spectra.

____ Did you isolate caffeine?
____ How pure is your isolated caffeine?
____Back to the Journal of Food Chemistry and Toxicology article, "Caffeine Content of Prepackaged National-Brand and Private-Label Carbonated Beverages."

a) What wavelength of UV light did they use to detect caffeine? According to your results was that an optimal wavelength?
b) How did they determine that their method was accurate in recording the exact amount of caffeine in 12 ounces of the beverage being tested.
c) The HPLC column was run with a pH 3 aqueous buffer. What is the molecular structure of caffeine at that pH?

____ Environmental question. What effect does all this caffeine drinking have on the environment?