Isolation and Identification of Caffeine Crystals

Objectives
The purpose of this experiment is to isolate crystalline caffeine and determine its approximate amount in some common substances, using an extraction technique. The use of Infrared Spectroscopy as a means of identifying substances will also be illustrated.

Background
Caffeine and closely related compounds like theophylline (used by asthmatics) and theobromine (a compound containing no bromine!) are stimulants which are found in a wide variety of plants growing throughout the world. The most common sources are: teas (roasted ground seeds of the coffee shrub), tea (dried leaves of various shrubs found especially in China, Japan, and India), cocoa (roasted ground seeds of the small evergreen cacao tree found in tropical America), mate (a tealike drink made from the leaves of a species of holly—the national drink in many South American countries), and kola nuts (chestnut sized seeds of a tree indigenous to western tropical Africa, the West Indies and Brazil which are reportedly used in the Sudan both for chewing as well as a form of money). It seems that whenever plants having a high caffeine content grow in particular area, the native population uses extracts of the plant as a beverage.

(Jolt is advertised as having twice the caffeine as the other caffeinated drinks: Mountain Dew, Sunkist Orange, Mr. Pibb, Tabb, and Shasta among others. The popular new European sports drink Red Bull is said to be packed with caffeine. Also containing caffeine in lesser amounts are Dr. Pepper, Pepsi and Royal Crown Cola. Non-caffeine pops or soft drinks include 7-Up, Sprite, Fresca, Fanta Orange, RC 100, and most ginger ales. For the caffeine content of specific brands of coffee, tea, and chocolate drinks, see these issues of Consumer Reports: September 1976; October 1979; May 1985; and September 1987.

Although tea leaves contain considerably more caffeine than coffee grounds on a dry weight basis, a cup of coffee will actually contain more caffeine because more coffee than tea is used in making a cup of "drinking strength" brew. The several million pounds of natural (from decaffeinating coffee) and synthetic caffeine produced annually in the United States are used mainly in headache and "stay-away" tablets. Because of its stimulating effect on the central nervous system, caffeine has also been used as an antidote to counteract the depressant effects of morphine poisoning. The lethal dose for caffeine in humans is estimated to be about 10 g (10,000 mg), but no deaths have been reported. This lethal dose would be equivalent to around 100 cups of coffee drunk all at once!

The decaffeinating of coffee used to be done by extracting out the caffeine with liquid chlorinated hydrocarbons - a method similar to the extracting of caffeine from your beverage with methylene chloride in this experiment. Since such chlorinated compounds have been shown to cause liver damage and are implicated as carcinogens, a process using water extraction has now been developed and is widely used. The procedure in this experiment calls for you to dissolve out (to extract) the caffeine from a water solution using the organic solvent methylene chloride, CH₂Cl₂. Caffeine is slightly soluble in water but very soluble in methylene chloride. Thus, mixing these two insoluble liquids together will cause most of the caffeine to dissolve preferentially in the organic methylene chloride layer. The sodium carbonate that is added reacts to form salts with many of the non-caffeine substances (especially tannic acid) which prevents them from also dissolving in the methylene chloride thus contaminating the caffeine. The two liquid layers (water and methylene chloride) are then separated by a type of filtration. A filter paper cone will be wetted with water, and the methylene chloride layer with a small amount of the water layer, will then be poured into the funnel. Once wetted with water, the pores of a filter paper will only allow water to drain

![Caffeine Molecule](image-url)
through them. When the two liquids are poured into this wet paper the methylene chloride layer will stay trapped inside the filter cone while the water layer drains through. The product you get after evaporation of the liquid methylene chloride should consist of brown to snow white caffeine crystals.

The question then arises "How do you know that these crystals are indeed caffeine?" One simple method for identification is a procedure called a melting point determination. Like boiling point and density the melting point is also a physical constant characteristic for a particular substance. Just as all samples of solid water melt at 0°C, all samples of pure caffeine melt at 238°C. By comparing the observed melting point of an unknown compound with recorded melting points for known compounds, evidence for identity can be obtained. Impurities present in your caffeine sample can cause the sample to begin melting at a temperature lower that 238°C. Some coffees may give caffeine samples melting at 190 to 220°C due to impurities. Be aware that caffeine is a material which can sublime (go directly from a solid to a gas phase), so rapid heating of your sample is necessary, and may cause difficulty in observing the melting point. Your instructor may collect some representative samples from your lab to perform melting point determinations.

In the same manner as for other characteristic properties, the response of a molecule to Infrared light is also characteristic. Light in the infrared wavelength range can cause molecules to vibrate along bonds (see Chapters 2 and 3 in your test for a quick review). Thus, infrared spectroscopy is most often used for the quick identification of functional groups in molecules, since the number and type of bonds in a group of atoms gives a specific response. We will use Infrared Spectroscopy to identify our caffeine samples. In the same manner as for melting points (use the Merck Index or the CRC Handbook to find the melting point of pure caffeine), we need to know how a pure sample of caffeine responds to IR light. This response is reported in graphical format called a spectrum. The IR Spectrum for pure caffeine is provided for you to use as a comparison to the spectrum obtained for your sample. Directions for preparing your isolated caffeine sample and collecting an IR spectrum are included in the section on Laboratory Techniques, page XX.

A. To begin the isolation of the caffeine:
1. Clean a 500 mL Erlenmeyer flask with soap and water. Record the kind of sample (if it is an instant tea or instant coffee, or NoDoz) and record its weight.
2. For solid instant coffee: weigh out at least 1.00 g. For solid instant tea, weigh out at least 0.50 g. For NoDoz, use 3 crushed tablets.
3. (a) Dissolve the solid, instant coffee or tea in 100 mL of cold water (in the clean, 500 mL Erlenmeyer flask).
   OR
(b) For the NoDoz, add 3 crushed tablets to 100mL of water in a (very clean, dry, weighed) 250mL Erlenmeyer flask and boil for 10 minutes. The caffeine will dissolve, and the binder will be left suspended in the water. After the liquid suspension cools, extract the caffeine using the same procedure as for coffee or tea.

B. Extraction
1. Dissolve roughly two grams of sodium carbonate (Na₂CO₃) in your sample in the 500 mL Erlenmeyer flask as prepared in one of the steps from Part A above.

2. Add 25 mL of CH₂Cl₂ (methylene chloride) and agitate by "aggressively swirling" for about 10 minutes; your lab instructor will demonstrate. DO NOT SHAKE or a persistent liquid/liquid emulsion will likely form (a frothy layer of water and methylene chloride mixed that does not separate easily).

3. Let the flask contents stand undisturbed for several minutes while you suspend a long stem funnel in an iron ring and place a collection beaker or flask underneath (size unimportant). Fit the funnel with a cone made from a 12 1/2 cm filter paper and wet the paper thoroughly with water. Your lab instructor can show you how to fold the filter paper.
4. Slowly and carefully pour off and discard as much of the (usually dark) upper water layer as possible. (This is called decantation.) Leave behind in the flask all of the more dense methylene chloride organic layer, together with a little remaining water. (Your textbook has more information on density.)

5. Slowly pour the entire contents of your 500 mL Erlenmeyer flask (the bottom layer of methylene chloride + upper layer of remaining water) into the wetted filter paper cone. Keep the liquid level up near the top of the paper cone until all of the flask contents have been thus transferred. You will see the upper water layer wick into the paper cone and drain through it. Left behind trapped in the filter paper will be the clear methylene chloride solution, which may be yellow to dark brown. This solution contains your caffeine. If you have a frothy emulsion in yourfilter paper cone, you may carefully and gently stir it with a smooth-end stirring rod to hasten the separation.

6. While the last bit of water is draining through the filter cone, take a clean and bone dry 50 mL beaker and put your initials on it. (Use a pencil to write on the frosted glass circle on the side of the beaker.) Weigh this beaker to the nearest milligram (0.001 g) using a top loading electronic balance and record data on the report sheet. Don't forget to zero the balance. Your lab instructor can assist you in the use of the balances. Do not leave trash and chemicals on the balances. It is up to all of us to keep the common balance rooms clean!!

7. Using a medicine dropper, transfer to the weighed beaker ONLY the clear (possibly yellow or dark brown) methylene chloride layer remaining in your filter cone. (If desired, your lab instructor can help you clarify any brown coloration in the liquid resulting from coffee or tea samples.) If any water droplets can be seen contaminating your methylene chloride solution, your lab instructor can show you how to remove them also, using anhydrous sodium sulfate, Na₂SO₄.

8. Go to the hood and place your beaker on the hot plates that are provided (you may need to check them out from the stockroom, room 141). DO NOT LEAVE YOUR BEAKER UNATTENDED. Watch the liquid contents closely. Avoid breathing the methylene chloride vapors. When the liquid level in the beaker gets down to about 1/16 of an inch, IMMEDIATELY REMOVE the beaker from the hot plate and set it aside in the hood to cool, while the small remainder of methylene chloride liquid evaporates (under 5 min). Methylene chloride boils at only 40°C, so you can safely grab the beaker with your fingers - a better option than unnecessarily trying to hold the beaker with tongs. Observe and record your observations on the report sheet; also make a sketch of the solid residue in the beaker with the aid of a magnifying glass. Reweigh the beaker to the nearest 0.001 g. (Be sure to use the same balance that you used before.) Now we are ready to try to find out if what we have isolated is really caffeine.
A. Preparation
Appearance and Source of original sample (coffee, tea, leaves, grounds, etc):

B. Extraction
Appearance of solid residue in beaker after evaporation of methylene chloride

Sketch of caffeine crystals

Weight of beaker + dry residue of caffeine

Weight of beaker (empty and dry)

Total weight of residue (caffeine)

C. Identification
Melting point of residue (your lab instructor may get this information for several representative samples)

Melting point of pure caffeine (consult The Merck Index.)

D. Comments and conclusions on the experiment
Attach your infrared spectrum. What are the wavenumbers (in cm\(^{-1}\)) for the 4 peaks that match (in location and shape) the IR Spectrum of pure Caffeine (provided)?
Caffeine Crystals (continued)

Date ____________________________ Lab Section ________

Name ____________________________ Lecture Section ________

1. In the caffeine isolation steps:
   (a) Which layer in the actual extraction step was the methylene chloride - the top or the bottom?
   (b) What physical property of methylene chloride would cause it to be above or below the water layer?

2. Give the brand name and ingredient list of two drugs that contain caffeine.
   (a)
   (b)

3. Spectroscopy is widely used in the structural elucidation of molecules. What structural features of a molecule are best detected using Infrared Light Energy?

4. We collected data on a background sample (the stretched and mounted teflon tape) before placing our caffeine on the tape. We also allowed the solvent to evaporate before collecting data (scanning) for the sample. What is the purpose of collecting the background data? Why allow the solvent to evaporate?

5. The symptoms of withdrawal from an addictive drug are irritability, desire for the addicting drug, and physical discomfort. All of these symptoms are relieved by a dose of the addicting drug. Using these criteria, is caffeine an addicting drug?

6. Do other extremely widely used drugs like alcohol and the nicotine in cigarettes fit the profile of addictive drugs? State one reason why you would (or would not) favor a law making the sale of one or both of these drugs illegal.