THE EXTRACTION OF CAFFEINE FROM COFFEE

PURPOSE

- To carry out an extraction of an aqueous solution of coffee with an organic solution.
- To isolate caffeine from coffee.
- To determine the melting point of caffeine.

DISCUSSION

Every day, throughout the world, people start out their morning by extracting a family of natural products called alkaloids from naturally occurring materials and then drinking this extract. The most common of these alkaloids caffeine, 1,3,7 - trimethylxanthine, occurs naturally in coffee



beans and tea leaves, but is added to soft drinks and stimulants such as Vivarin and No-Doz, dieting aids such as Dexatrim and Diatac and painkillers such as Anacin and Excedrin.

Alkaloids are a broad category of nitrogen containing organic metabolites produced by plants. Since they contain nitrogen, they behave like bases (alkalis) and hence, they are termed alkaloids. Other common alkaloids are morphine, quinine, cocaine and codeine. These substances are extremely bitter and/or toxic. By producing them, plants make their leaves unattractive to eating by insects and higher animals.

In this experiment, you will extract caffeine from coffee using methylene chloride (CH_2Cl_2) . Methylene chloride is an organic solvent that is somewhat nonpolar. In this experiment, we take advantage of the same principles we used in thin layer chromatography, especially "like dissolves like". Methylene chloride is a liquid but it isn't like water so they form two

layers, just like vinegar and oil. The caffeine molecule is closer in structure to methylene chloride, so it will dissolve in that layer. The two layers can then be separated, and the methylene chloride, which has a very low boiling point, can be evaporated in the hood, leaving a residue of caffeine.

In order to verify that you have obtained caffeine, you will take a melting point of this residue. Melting points are a unique property of pure substances. For example, pure water melts at 0 degrees C. Pure caffeine melts at 238 °C. If the material you collect melts at a temperature close to 238 degrees, you can be more confident that it is caffeine. The material that you have extracted will probably have a slightly lower melting point.

PROCEDURE

A. Extraction of the Coffee Solution

- 1. To a clean 500 mL Erlenmeyer flask, add 100 mL of coffee extract. This extract will have been prepared for you in advance.
- 2. Add approximately two grams of sodium carbonate $(NaCO_3)$ to the coffee solution. This will react with some of the substances in the coffee extract and make them extremely water soluble. Swirl the mixture until all the sodium carbonate dissolves.
- 3. Add 25 mL of methylene chloride (CH_2Cl_2) , and vigorously swirl the mixture for 10 minutes. Do not shake the mixture or an emulsion will form.
- 4. Allow the mixture to stand and separate into two layers; a dark aqueous top layer and a clear methylene chloride bottom layer.
- 5. Carefully pour off into a beaker, as much of the top layer as you can, without removing the bottom layer. This process is called decanting.
- 6. Place a 12.5 cm fluted filter paper in a long stem glass funnel. Put the funnel in a small iron ring and suspend it over a 250 mL Erlenmeyer flask.

- 7. Using a squeeze bottle of water, thoroughly wet the filter paper.
- 8. Slowly and carefully pour the methylene chloride/water mixture into the fluted filter paper. The excess water will drain through and the methylene chloride solution of caffeine will remain on the filter paper.
- 9. Using a pipet, transfer the methylene chloride solution to a 50 mL Erlenmeyer flask. To this solution, add a scoop of anhydrous sodium sulfate (Na_2SO_4) , in order to remove the last traces of water.
- 10. While the solution is drying, weigh (tare) a 50 mL beaker to the nearest 0.001 g on a balance. Record this tare weight in Table 1 on the DATA SHEET.
- 11. Using a pipet, transfer the dried solution to the tared 50 mL beaker.
- 12. Evaporate most of the methylene chloride in the hood on a warm hot plate. When only a fraction of a milliliter of liquid is left, remove the beaker from the hot plate. Allow the beaker to stand in the hood for a minute or two. The heat remaining in the glass will cause the last amount of methylene chloride to evaporate and produce a solid residue of crude caffeine.
- 13. In order to determine your recovery of caffeine, reweigh the cool beaker and record this mass in the Table.
- 14. By difference, determine the mass of the caffeine in the beaker and record this value in the Table.
- 15. Pure caffeine is a white solid. Describe the appearance of your product in the Table.

B. Melting Point Determination

1. To a melting point tube, add a small amount of your caffeine. This can most easily be done by pressing the open end of the tube down on the caffeine, turning the tube right-side-up and tapping the tube on the bench until the solid falls to the bottom of the tube. Tap the tube on the bench a few more times so that the caffeine is compacted.

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- 2. Place the tube in the slot of the melting point apparatus. Turn on the switch of the instrument. This will turn on a light that illuminates the sample. By looking through the magnifying glass, you should have a clear view of your sample.
- 3. Turn the heating dial to a setting of about 40. This will cause a rapid heating of your sample initially, but should not cause it to melt. As the temperature increases, there will be a decrease in the rate of heating. Since your sample will melt above 200 °C, you may need to periodically increase the setting of the heating dial. Ideally, the temperature increase at the melting point should be only 1-2 °C per minute.
- 4. Record the range of the melting range of your sample as follows. In the Table record the temperature at which you first see liquid beginning to form. The sample will continue to melt. When the last of the sample has melted, record the second temperature. These two values are separated by a dash, i.e., 63-65 °C. For a relatively pure compound, this temperature range will generally be only a few degrees.

DATA SHEET - THE EXTRACTION OF CAFFEINE FROM COFFEE

Tare weight of the empty beaker (g)	
Mass of the beaker and the caffeine (g)	
Mass of the caffeine (g)	
Appearance of the caffeine	

Melting range of the caffeine

QUESTIONS

- 1. a. Is methylene chloride more or less dense than water?
 - b. What evidence do you have to support your answer?
- 2. Was your melting point the same as the reported melting point? If not, why not?

3. Why was sodium carbonate added to the coffee solution?

4. Explain why the caffeine that you obtained in this experiment may not have been white in color.

- 5. List some substances that contain caffeine other than coffee.
 - a.
 - b.
 - c.
 - d.
 - e.