Caffeine

Food & Diet, Coffee

1-1 EXPERIMENT 1 ISOLATION OF CAFFEINE FROM TEA In this experiment you will isolate caffeine from tea leaves and prepare the salicylate derivative. Introduction This experiment illustrates the isolation of a naturally occurring product from plant material -- caffeine from tea leaves. The experiment will provide experience in handling relatively small amounts of material and at the same time you will be exposed to several techniques and procedures which are fundamental for survival in an organic chemistry lab. You have been exposed to most of them during your CHEM 250 laboratory session. Please refer to the Laboratory Techniques TECH 1 to 7 and familiarize yourself with the content. For example during this lab you will be doing extraction and TECH 6 describes the theory behind it and how to handle separatory funnel. using a drying agent and in TECH 6-5 you will read about the use of drying agents doing a distillation and the procedure can be found in TECH 5-2 and the apparatus set up in TECH 5-4 carrying out gravity filtration using fluted filter paper and TECH 3-6 shows you how to flute the filter paper. using the rotary Evaporator (Rotavap) to evaporate solvent at a lower temperature (Appendix A) Make sure you are familiar with the above techniques and the apparatus set up. 1-2 . Caffeine is an alkaloid, a class of naturally occurring compounds containing nitrogen and having the properties of an organic amine base [alkaline, hence, alkaloid]. Typical alkaloids are caffeine, nicotine, morphine, codeine, and cocaine. RO O N CH3 C HO Caffeine H3C N O CH3 N N CH3 CO2CH3 N N CH3 O H O O N N CH3 is found caffeine in tea nicotine (from tobacco) (-) cocaine (from coca plant) morphine, R = H codeine, R = CH3 leaves, coffee beans, kola nuts, and cocoa beans. The table belowgives the amount of caffeine in the various

beverages prepared from these natural products. One can develop both a tolerance and a dependence on caffeine. The dependence is real, and a heavy [> 5 cups of coffee per day] user will experience lethargy, headache, and perhaps nausea after about 18 hours abstinence. An excessive intake of caffeine may lead to restlessness, irritability, insomnia, and muscular tremor. Caffeine can be toxic, but it has been estimated that to achieve a lethal dose of caffeine one would have to drink about 100 cups of coffee over a relatively short time. 1-3 Table The amount of caffeine found in beverages* Brewed Coffee 60âˆ’100 mg/100 mL Decaffeinated Coffee 18âˆ’35 mg/100 mL Tea 18-53 mg/100 mL Coca-Cola 12 mg/100 mL Cocoa 3. 5 mg/100 mL * The average cup of coffee or tea contains about 150 mL of liquid. The average bottle of Coke contains about 350 mL of liquid. Because of the central nervous system effects that caffeine causes, many persons prefer to use decaffeinated coffee. The caffeine is removed from coffee by extracting the whole beans with trichloroethylene at 71 °C. Following this, the solvent is drained off, and the beans are steamed to remove any residual solvent. Then, the beans are dried and roasted to bring out the flavor. Decaffeination reduces the caffeine content of coffee from a range of 2% to 5% to the range of 0. 03% to 1. 2% caffeine. The isolation of caffeine from tea leaves presents the chemist with a major problem: caffeine does not occur alone in tea leaves, but is accompanied by other natural substances from which it must be separated. The major components of tea leaves are: 1-4 1. Cellulose âˆ’ the major structural material of all plant cells. Since cellulose is virtually insoluble in water it presents no problems in the isolation procedure. Caffeine âˆ’ one of the major water soluble substances present in tea leaves.

Caffeine comprises as much as 5% by weight of the leaf material in tea plants. Tannins âˆ’ high molecular weight, water soluble compounds that are responsible for the color of tea. The term " tannin" does not refer to a single compound or even to substances having similar chemical structure. Rather, " tannin" refers to a class of compounds that have certain properties in common. They contain phenol groups, are acidic and are used to convert animal hides to leather [tanning]. Flavonoid Pigments âˆ’ water soluble colored compounds that are widely distributed in plant life. Chlorophylls âˆ’ water soluble green plant pigments that enable plants to convert carbon dioxide and water to carbohydrates and oxygen [photosynthesis]. 2. 3. 4. 5. The caffeine isolated from tea leaves can be purified by sublimation. Caffeine melts and sublimes at 2380C. But during this experiment you will identify caffeine not from its melting point but from the melting point of its salicylate. One way to identify an organic compound is to prepare a derivative of it. Caffeine is an organic base and can therefore accept a proton from an acid to form a salt. The salt formed from salicylic acid, even though ioic has a sharp melting point and can thus be used to help characterize caffeine. 1-5 O H3C O N N CH3 CH3 N N O OH OH CH2Cl2 H3C O N O CH3 N N H HO O O N CH3 Caffeine Salicylic Acid Caffeine Salicylate Flow diagram for the separation of caffeine from tea leaves Tea Leaves cellulose caffeine chlorophyll tannic acids flavanoid pigments Extraction with H2O (100oC) H2O insoluable Extracted tea leaves cellulose H2O soluable Aqueous tea soulution caffeine chlorophyll tannic acids flavanoid pigments Exraction with CH2Cl2 CH2Cl2 solution Caffeine Impurities of Tannins and Chlorophylls Reaction with salicylic acid Mother liquor Tannins and chlorophyll impurities Aqueous

Solution Chlorophylls Tannic acids Flavanoid pigments Crystals Caffeine Salicylate 1-6 Prelab Exercise 1. Read the experiment and understand its objectives and the various techniques being introduced. Study the flow diagram, and relate it to the various steps in the experimental procedure. 2. Prepare your prelab write-up. given in Appendix D. Your lab report should follow the example Read through the lab report and complete the relevant sections for your pre-lab exercise. Safety Considerations Dichloromethane is a relatively non-toxic solvent and is often used in place of carbon tetrachloride [CCl4] and benzene, both of which are carcinogenic. It does however, have a high vapor pressure and readily evaporates to give a moderate concentration of CH2Cl2 in the air, which may discomfort some students. Try to contain the CH2Cl2 as much as possible and evaporate it from solution only in a fume hood. 1-7 Experimental Procedure Extraction of caffeine: 1. To a 400 mL beaker containing 5 tea bags add 100 mL of vigorously boiling water. Set the beaker on the hot plate and boil gently for 10 min. Stir the mixture carefully to avoid breaking any of the bags. 2. Decant [pour off the liquid] the hot tea solution into a 250 mL Erlenmeyer flask and press the bags with the bottom of a small Erlenmeyer to recover as much water as possible. Extract the tea bags a second time with 50 mL of boiling water. Heat for a few minutes and then add the second extract to the first in the Erlenmeyer flask. Handle the hot beaker using paper toweling or a J-cloth. 3. Cool the aqueous solution to room temperature using an ice bath and pour it into a 250 mL separatory funnel. Extract [gently shake/swirl the mixture for at least 2 minutes] the tea solution two times, (first time 30 mL and the second time 25 mL) with dichloromethane [CH2Cl2]. Combine both

extracts in a 125 mL Erlenmeyer flask. [Take care not to shake the separatory funnel (TECH 6) so vigorously as to cause an emulsion to form. If you should get an emulsion that does not break, ask your TA to show you how to break it]. 4. Dry the combined CH2Cl2 extracts with anhydrous sodium sulfate (TECH 6-5). Swirl for several minutes to allow enough time for the sodium sulfate to become hydrated with the water. 5. Gravity filter (TECH 3-6) the dry CH2Cl2 solution into a dry pre-weighed 100 mL round-bottom flask. Wash the filter paper and drying agent with about 1mL of CH2Cl2. 1-8 6. Either Remove the CH2Cl2 by simple distillation (TECH 5-4) or your TA will help you to rotavap (Appendix A) the solvent, until about 5 mL of liquid remains in the round-bottom flask. Do not allow the flask to go dry or the caffeine may decompose. 7. Place the round-bottom flask on a steam bath and evaporate the contents to dryness at low-medium heat. Weigh the flask to find the crude mass of caffeine Preparation of the salicylic Acid derivative of caffeine 9. Assuming that the caffeine extracted is pure, add equimolar amount of salicylic acid into the round bottom flask, followed by 2-3 mL of dichloromethane and heat the mixture to boiling 10 Remove the round bottom flak from the heat and add petroleum ether drop wise until a solid white precipitate just begins to form. Now add dichloromethane in drops so as to just dissolve the precipitate. 11 Let the solution cool very slowly to room temperature and then put it on ice to aid the crystallization. 12. Vacuum filter the crystals using a Hirsh funnel and rinse with pertrolium ether. 13 Leave the solid in a tared weigh boat, cover with a filter paper and leave it in your locker to dry until the next lab session. 14. Find the mass and the melting point of caffeine salicylate. Transfer the product into a vial and

submit the labeled vial for marking. 1-9 Chemical Waste Management - tea bags -- waste paper containers at each lab bench - filter paper containing Na2SO4 -- waste containers at the chemical stations - CH2Cl2 from simple distillation -- waste containers at the chemical stations - ether and alcohol wastes -- waste containers at the chemical stations - acetone washings -- acetone waste bottles at the chemical stations - paper waste -- waste paper containers at each lab bench - broken glass -- broken glass container