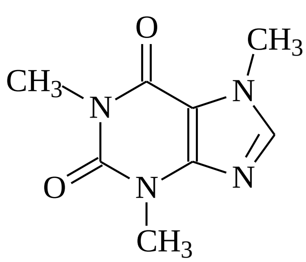
Experiment 9: Extraction of Caffeine

Introduction

Caffeine

Caffeine occurs naturally in tea leaves and coffee beans. Cocoa beans, used to produce chocolate, contain a compound that is nearly identical in structure to caffeine. Caffeine is also added to many types of soda and energy drinks. Caffeine is a white solid material at room temperature. It is classified as an alkaloid—a nitrogen-containing basic (as opposed to acidic) compound that is obtained from plants and has physiological effects in the body. Caffeine is a stimulant and mildly addictive. Withdrawal symptoms may include headache and irritability. There is no conclusive evidence that caffeine causes cancer or heart disease. However, animal studies suggest it may be a weak teratogen (an agent that causes birth defects in an embryo or fetus), so pregnant women are advised to limit their intake of caffeinated beverages.

Structure of caffeine:



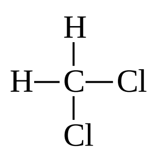
Extraction

Extraction is a technique in which a solvent is used to remove/isolate a compound of interest from a liquid substance. For example, coffee is a liquid which contains dissolved caffeine. In this experiment you will extract (remove) caffeine from coffee. The extraction will be carried out by simply adding a portion of solvent to the coffee. The caffeine is more soluble in the solvent than in the coffee, so it "moves out of" the coffee and into the solvent. The solvent, now containing dissolved caffeine, is then separated from the coffee. At this point the caffeine has been extracted from the coffee because it is now dissolved in the solvent, rather than the coffee. If the solvent is allowed to evaporate or is removed in some manner, the caffeine will be left behind as a white powder.

The solvent that is used for an extraction must be immiscible with the other liquid. Immiscible liquids are two liquids which are not soluble. In other words, when they are mixed together they separate into two layers. The denser liquid will form the bottom layer. (Conversely, miscible liquids are soluble in each other.) In the extraction of caffeine, a solvent that is immiscible with coffee will be added to the coffee. The mixture will be shaken in order to make the solvent and coffee mix together as much as possible, enabling the caffeine to move out of the coffee and into the solvent. As soon as shaking is stopped, the two immiscible liquids will separate into two layers. The mixture is placed into a separatory funnel, enabling the easy separation of one layer from the other—the lower, denser layer can be drained out of the bottom of the separatory funnel, leaving behind the upper layer.

Coffee is mostly water, so the solvent chosen for the extraction of caffeine must be immiscible with water. Dichloromethane is a water-immiscible solvent that works well, because caffeine is highly soluble in it (which means that most of the caffeine will easily move out of the coffee and into the dichloromethane). Dichloromethane is also often called methylene chloride.

Structure of dichloromethane:



CH_2Cl_2
Also called methylene chloride

Once the caffeine has been extracted into the dichloromethane solvent, the dichloromethane will need to be removed in order to obtain dry caffeine powder. This could be done by simply allowing the dichloromethane to evaporate, but that would take a long time. Instead, distillation will be used to remove the dichloromethane quickly. The distillation technique will be explained in the pre-lab lecture.

Name: ____________________________

Experiment 9: Extraction of Caffeine
Pre-Lab Exercise

1. What is an alkaloid?

2. What is a teratogen?

3. What does it mean for two solvents to be immiscible?

4. Complete each of the following conversions ('g' is grams; 'mg' is milligrams). (1000 mg = 1 g).

2.143 g = ________ mg	284 mg = _________ g
0.024 g = ________ mg	52 mg = _________ g
1.024 g = ________ mg	65.3 mg = _________ g
0.3 g = ________ mg	5000 mg = _________ g

5. The structure of caffeine is shown in the introduction. However, some of the carbon atoms are eliminated to simplify the structure, making it similar to a line formula. This is a common practice. Redraw the structure of caffeine, showing <u>all</u> carbon and hydrogen atoms.

What is the molecular formula for caffeine? (For example, $C_{12}H_{22}O_{11}$ is a molecular formula.)

Use the molecular formula of caffeine to calculate its molecular weight.
(C = 12 g/mol; H = 1 g/mol; N = 14 g/mol; O = 16 g/mol)

Experiment 9: Extraction of Caffeine
Procedure

1. **CAUTION:** It is best not to get dichloromethane on your skin. You may choose to wear gloves during this experiment. Otherwise, wash thoroughly if you do get some on your hands.

   CAUTION: Gas will build up in the separatory funnel while you are shaking it, so you must vent the funnel occasionally to prevent the stopper from popping out, causing a mess and possibly getting dichloromethane on your skin.

   Obtain a separatory funnel and close the stopcock. Set up a ring stand on your lab bench to support the "sep funnel." Place 75 mL of coffee into the sep funnel. Add 20 mL of dichloromethane and place the stopper in the top of the sep funnel. Shake the funnel for 1-2 minutes following the instructions given during the prelab lecture. Place the funnel back in the ring stand and allow the two layers of liquid to separate—this may take a few minutes. The lower layer will be dichloromethane. There may be an emulsion at the boundary between the two layers—an emulsion is a region where "bubbles" of one liquid are suspended in the other liquid, and the two refuse to separate. If an emulsion occurs, use a glass stirring rod to poke around in the emulsion to break it up. Ask the instructor for help if the emulsion seems particularly stubborn.

   Be sure the stopper is removed from the top of the sep funnel for the following drainage step—otherwise the liquid will drain out of the funnel extremely slowly. Once the two layers have separated, or are nearly separated, drain the lower dichloromethane layer into a clean, DRY 250 mL flask. **Be careful not to drain any of the COFFEE into the flask!** It is best to leave a very small amount of dichloromethane at the bottom of the sep funnel so as not to accidently allow some coffee to escape.

   The dichloromethane you just drained contains dissolved caffeine that has been extracted from the coffee. However, it isn't possible to extract all the caffeine with a single portion of dichloromethane. Therefore, you will do two more extractions using fresh portions of dichloromethane in order to extract the majority of the caffeine that is in the coffee.

2. Set the flask containing the first portion of dichloromethane aside. Add a fresh 20 mL portion of dichloromethane to the coffee in the sep funnel. Shake the funnel for 1-2 minutes. Allow the two layers to separate again. Then drain the lower

dichloromethane layer into the 250 mL flask that already contains the first portion of dichloromethane—**be sure to leave a very small amount of dichloromethane in the funnel and don't allow any coffee to drain out!** At this point the two portions of dichloromethane are together in the flask.

3. Repeat step 2. At the end of this step, the flask will contain all three portions of dichloromethane. If everything went well, most of the caffeine from the coffee should be dissolved in this solvent.

4. The dichloromethane in the flask is certain to contain small amounts of water from the coffee, even if you can't actually see visible water droplets. Therefore, it must be "dried" to remove any water. If there are some visible droplets of water/coffee present, decant the dichloromethane into a new dry flask, attempting to leave the water droplets behind when decanting. If no visible droplets of water are present, skip the decanting step.

   When there are no visible water droplets remaining, weigh out approximately 2 grams of anhydrous sodium sulfate and add it to the dichloromethane (it is not necessary to obtain exactly 2 grams). Swirl the container; then let it sit undisturbed for about 10 minutes. The sodium sulfate will absorb any water (which you can't see) that is present. While waiting, proceed to step 5.

5. Weigh a clean, dry 125 mL flask and record the exact mass on the report sheet. Obtain a condenser, two rubber hoses, and a "still head" (a bent glass tube with a rubber stopper at each end) for carrying out distillation. Attach the rubber tubes to the water outlets on the condenser. Fill a 600 mL beaker about 1/3 full with distilled/purified water and place it on a hot plate. Do not turn the hot plate on yet.

   After the dichloromethane has dried for ~10 minutes, decant the dichloromethane into this flask, being careful to leave all the sodium sulfate behind. You no longer need the sodium sulfate, so it can be washed down the drain.

6. Assemble a distillation apparatus as follows. Place the flask containing the dichloromethane into the beaker of water on the hotplate, so that the dichloromethane is entirely submerged in water. Use a clamp from the cabinet under your lab bench to clamp the neck of the flask so that it stays in place at the proper level. Place one stopper of the still head into the mouth of the flask. Attach the condenser to the rubber stopper on the other end of the still head. Place a waste beaker under the open end of the condenser to catch liquid that will drip out during the distillation. If necessary, use another clamp to hold the condenser in place.

One rubber hose should already be connected to each of the water outlets of the condenser. Attach the other end of the lower rubber hose (the hose furthest from the still head) to the white plastic tube coming out of the water faucet on your lab station. You may have to first remove the black rubber attachment from the end of the white plastic tube.

Place the end of the upper rubber hose (the hose nearest to the still head) into the sink so that water coming from the hose can drain into the sink. **Ask the instructor to check your distillation apparatus before proceeding.**

When you are sure the rubber hoses are attached correctly and tightly, turn on the water faucet slowly and allow water to flow into the hoses and condenser. Adjust the water flow so that it is not too fast (otherwise one of the hoses might blow off). Turn the hotplate on “high.” The water in the beaker will begin to boil and its heat will cause the dichloromethane inside the flask to boil. At that point, you will soon see dichloromethane begin to drip out of the condenser into the waste beaker.

7. As the dichloromethane becomes hot and vaporizes (boils), the dichloromethane vapor will travel into the cool condenser where it will condense back to liquid and drip out of the bottom of the condenser into the waste beaker. The caffeine should not vaporize and travel with the dichloromethane. Rather, it should be left behind in the flask.

   Once all the dichloromethane has boiled out of the flask and the flask is dry, turn off the heat and disassemble the apparatus. There should be caffeine residue inside the flask.

   Record the appearance of the caffeine on the report sheet. Wipe the outside of the flask completely dry and weigh the flask containing the caffeine. Record the total mass on the report sheet, and then calculate the mass of caffeine extracted by subtracting the mass of the empty flask. Complete the lab report by calculating your percent recovery of caffeine.

8. Pour the distilled dichloromethane into the bottle labeled “recycled dichloromethane.” The coffee in the sep funnel can be discarded down the drain. Wash the sep funnel with water, rinse it with acetone from the bottles with red lids near the sink, and return the funnel. The caffeine can be washed out of the flask using water (and acetone if necessary). Return the 125 mL flask (do not put it in your drawer)

Name: ______________________________

Lab Partner: ________________________

Desk: _________ **Date:** _________

Experiment 9: Extraction of Caffeine

Report Sheet

Data

1. Mass of empty flask ________ g

2. Description of extracted caffeine: Include physical state (solid, liquid, gas) and color.

3. Mass of flask containing caffeine ________ g

Calculations and Conclusions

4. Mass of caffeine extracted ________ g = ________ mg

5. Actual amount of caffeine in coffee sample ________ mg

The coffee was prepared by the stockroom manager. According to the recipe used, the coffee had a caffeine concentration of 1 mg/mL. Calculate the actual amount of caffeine in the coffee sample you used for the extraction. Show work here:

6. Percent recovery of caffeine in coffee sample ________ %

This is the percent of caffeine you were able to extract from the actual amount of caffeine in the coffee sample. <u>Pay careful attention to units</u>!! Show work here:

Experiment 9: Extraction of Caffeine

Post-Lab Exercise

1. On the report sheet you calculated your percent recovery of caffeine.
   - If your percent was less than 100%, give at least one reason why it was <u>less than</u> 100%.
   - If your percent was more than 100%, give at least one reason why it was <u>more than</u> 100%.

   Do NOT simply say "human error." Be specific about the types of errors (human or otherwise) that may have occurred, leading to a recovery that is not exactly equal to 100%.

2. Based on what you observed during this experiment, is dichloromethane denser OR less dense than water? Explain your reasoning.

   Based on what happened in this experiment, is caffeine more soluble in water, OR more soluble in dichloromethane? Explain your reasoning.

3. Ethanol is <u>miscible</u> with water. (After all, alcoholic beverages don't separate into an "alcohol layer" and a "water layer.") Assuming that caffeine is soluble in ethanol, could you use ethanol as a solvent to extract caffeine from coffee (rather than using dichloromethane as the extraction solvent)? Explain why or why not.

4. A 12 oz can of Coke contains 46 mg of caffeine. One fluid ounce is equivalent to 29.57 mL (1 oz = 29.57 mL). What is the concentration of caffeine in Coke, in units of mg/mL? Show your work.