Extraction of Caffeine (Experiment)

Caffeine is a minor constituent of tea, coffee, and other natural plant materials. The major constituent of tea is cellulose which is not water soluble. Caffeine is water soluble but so are some tannins and gallic acid which is formed in the process of boiling tea leaves. The latter two components can be converted to their calcium salts which are insoluble in water. The caffeine can then be extracted from the water by methylene chloride in almost pure form. Some chlorophyll is often extracted at the same time.

Procedure

Place 15 g of tea leaves, 5 g of calcium carbonate powder and 200 mL of water into a 600 mL beaker. Boil the solution on a hot plate for 20 minutes with occasional stirring. Cool the solution but, while it is still warm, vacuum filter through a Buchner funnel using a fast filter paper, if available. Normally, hot solutions are not vacuum filtered. Rinse the leaves with 50 mL of water. Carefully press out as much filtrate as possible since the caffeine is in the aqueous layer. Rinse again with 50 mL of water.

Cool the solution to room temperature and pour it into a 500 mL separatory funnel. Extract with 35 mL of methylene chloride. In a departure from normal procedure, it will be necessary to vigorously shake the separatory funnel in order to extract the caffeine. First, relieve the pressure buildup as soon as you mix the two liquids. Then shake vigorously for 10 seconds and relieve pressure, repeat the shaking two more times. An emulsion will probably form.

To break the emulsion formed in the methylene chloride layer, slowly drain the methylene chloride layer through a small amount of anhydrous magnesium sulphate in a powder funnel with a loose cotton plug (a tight plug will prevent drainage).

Extract the aqueous solution once again with a 35 mL of methylene chloride, repeating the steps above to collect the lower layer. Combine the methylene chloride extracts and, if necessary, dry further with additional anhydrous magnesium sulphate.

The methylene chloride solution will be stripped on a roto-evaporator. Tare weigh a 100-mL rb flask and transfer the dried methylene chloride solution to it. Be certain that there is no magnesium sulfate in the solution. Stripping this solution to dryness will take less than 5 minutes. You will be left with a small amount of residue with a greenish tinge. Obtain the weight of crude caffeine by difference.

Add 5-8 mL of hot acetone to dissolve the crude caffeine and transfer the solution to a 50 mL Erlenmeyer flask for recrystallization. Add a few drops of petroleum ether until you reach the cloud point (caffeine is less soluble in this mixed solvent and is just beginning to precipitate) and then cool the solution. If you do not get a precipitate, you may have used too much acetone, carefully boil off the excess on a steam bath using a boiling stick for ebullation.

Suction filter the caffeine using a small Hirsch funnel and petroleum ether as a transfer/rinse solvent. A second crop of caffeine may form in the filtrate as the solvent evaporates. This second crop can also be collected by vacuum filtration but keep it separate from the first crop. After air drying, weigh each crop and record your % caffeine recovered from tea.

The sublimation will be performed as described by your instructor. You will use 50 mg of your caffeine to make a salicylate derivative and sublime the remainder (which should be at least 50 mg).

You will not take a mp of the purified caffeine which would require a sealed capillary to prevent sublimation near the melting point. Save the purified caffeine in a sealed vial. You will use some of this material for TLC analysis next week.

Caffeine is a base which can react with acids to form salts. A well characterized salt of caffeine is caffeine salicylate formed by using salicylic acid. This derivative of caffeine has an accurate melting point. Later this semester, you will be required to make solid derivatives of other compounds.

Preparation of Caffeine Salicylate

Using an analytical balance (there are several top loader balances in the lab across the hall which will quickly weigh to 0.0001 mg), weigh 50 mg of caffeine and 37 mg of salicylic acid (both can be plus or minus 1-2 mg) and dissolve them in 4 mL of toluene in a small 25 mL Erlenmeyer flask by warming on a steam bath. Add 1 mL (dropwise) of petroleum ether and allow the mixture to cool and crystallize. If necessary, cool in an ice-water bath. Collect the crystals by vacuum filtration, air dry, weigh, record the yield, and take a mp (lit mp 137 °C).

طريقة اخرى

Plants and other natural materials are sources of many chemicals. Sometimes you want to isolate a single compound from the thousands that may be present. Here is an example of how to use solvent extraction to isolate and purify caffeine from tea. The same principle may be used to extract other chemicals from natural sources.

Caffeine From Tea: Materials List
2 tea bags
Dichloromethane
0.2 M NaOH (sodium hydroxide)
Celite (diatomaceous earth - silicon dioxide)
Hexane
Diethyl ether
2-propanol (isopropyl alcohol)
Procedure
Extraction of Caffeine:

1. Open the tea bags and weigh the contents. This will help you determine how well your procedure worked.
2. Place the tea leaves in a 125-ml Erlenmeyer flask.
3. Add 20 ml dichloromethane and 10 ml 0.2 M NaOH.
4. Extraction: Seal the flask and gently swirl it for 5-10 minutes to allow the solvent mixture to penetrate the leaves. Caffeine dissolves in the solvent, while most of the other compounds in the leaves do not. Also, caffeine is more soluble in dichloromethane than it is in water.
5. Filtration: Use a Buchner funnel, filter paper, and Celite to use vacuum filtration to separate the tea leaves from the solution. To do this, dampen the filter paper with dichloromethane, add a Celite pad (about 3 grams Celite). Turn on the vacuum and

slowly pour the solution over the Celite. Rinse the Celite with 15 ml dichloromethane. At this point, you may discard the tea leaves. Retain the liquid you have collected -- it contains the caffeine.

6. In a fume hood, gently heat a 100-ml beaker containing the washings to evaporate the solvent.

Purification of Caffeine: The solid that remains after the solvent has evaporated contains caffeine and several other compounds. You need to separate the caffeine from these compounds. One method is to use the different solubility of caffeine versus other compounds to purify it.

1. Allow the beaker to cool. Wash the crude caffeine with 1 ml portions of a 1:1 mixture of hexane and diethyl ether.
2. Carefully use a pipette to remove the liquid. Retain the solid caffeine.
3. Dissolve the impure caffeine in 2 ml dichloromethane. Filter the liquid through a thin layer of cotton into a small test tube. Rinse the beaker twice with 0.5 ml portions of dichloromethane and filter the liquid through the cotton to minimize the loss of caffeine.
4. in a fume hood, heat the test tube in a warm water bath (50-60 °C) to evaporate the solvent.
5. Leave the test tube in the warm water bath. Add 2-propanol a drop at a time until the solid dissolves. Use the minimum amount required. This should be no more than 2 milliliters.
6. Now you can remove the test tube from the water bath and allow it to cool to room temperature.
7. Add 1 ml of hexane to the test tube. This will cause the caffeine to crystallize out of solution.
8. Carefully remove the liquid using a pipette, leaving the purified caffeine.

9. Wash the caffeine with 1 ml of a 1:1 mix of hexane and diethyl ether. Use a pipette to remove the liquid. Allow the solid to dry before weighing it to determine your yield.
10. With any purification, it's a good idea to check the melting point of the sample. This will give you an idea of how pure it is. The melting point of caffeine is 234 °C.

Additional Methods

Another way to extract caffeine from tea is to brew tea in hot water, allow it to cool to room temperature or below, and add dichloromethane to the tea. The caffeine preferentially dissolves in dichloromethane, so if you swirl the solution and allow the solvent layers to separate. you will get caffeine in the heavier dichloromethane layer. The top layer is decaffeinated tea. If you remove the dichloromethane layer and evaporate the solvent, you will get slightly impure greenish-yellow crystalline caffeine.

Safety Information

There are hazards associated with these and any chemicals used in a lab procedure. Be sure to read the MSDS for each chemical and wear safety goggles, a lab coat, gloves, and other appropriate lab attire. In general, be aware the solvents are flammable and should be kept away from open flames. A fume hood is used because the chemicals may be irritating or toxic. Avoid contact with sodium hydroxide solution, as it is caustic and can cause a chemical burn on contact. Although you encounter caffeine in coffee, tea, and other foods, it is toxic in relatively low doses. Don't taste your product!