Isolation of Caffeine from tea

In this experiment, caffeine will be extracted from tea leaves (where it is about 5% present) using hot water. This is essentially the same procedure used to decaffeinate drinks such as coffee and tea.

Procedure

Add about 10.0 g of tea leaves to around 100 mL of water in a 400-mL beaker (cut open the tea bags with some scissors to get at the tea leaves). Add 5 g of calcium carbonate and boil this suspension for about 10 minutes. The caffeine will dissolve in the hot water, but so will some other compounds, known as tannins (a type of carboxylic acid). The calcium carbonate should convert these tannins into insoluble salts, which will then drop out of solution.

Add 3 g of filter acid (celite) and decant the mixture into a Buchner funnel (decanting means to pour the liquid, but try to leave the solid behind). Any solid caught by the filter paper can be discarded. The caffeine is located in the filtrate (the liquid that passed through the filter). You will probably have around 50 mL of filtrate. Less than that is fine, but if you have more, boil it down to about 50 mL.

Cool the filtrate to room temperature and pour it into a 125-mL separatory funnel. Add 10 mL of methylene chloride to the funnel and shake gently (don't shake it too vigorously or it will form a foam that is hard to get rid of). Gases will be produced, so it is important to vent the mixture periodically by taking the cap off (otherwise the pressure will eventually cause it to shoot off). The caffeine is more soluble in the nonpolar methylene chloride than the water, and as a result, will move to the methylene chloride layer (known as the organic layer). Since the organic layer is denser than water, it will be the layer on the bottom.

Open the stopcock on the funnel and pour the organic layer into another beaker. Do not let any of water layer (which is brown) get transferred. If you do so by mistake, it is best to pour everything back in the funnel and try again. Sometimes it is necessary to leave a little bit of the organic layer behind to ensure that you do not collect any water.

Add 10 mL of fresh methylene chloride to the funnel (with the brown water) and once again shake gently with venting. This is just giving another chance for any caffeine left behind in the water layer to migrate to the organic layer. Once again, open the stopcock to allow the organic layer out (combine it directly with the other organic layer) but do not collect any of the brown water layer.

The caffeine should be pure at this stage, but it is still dissolved in the methylene chloride. You will need to evaporate this in a machine called the N-Evap. Your instructor will show you how to use this machine. By applying nitrogen gas in a warm water bath, the methylene chloride will quickly evaporate away, leaving only the caffeine behind. Remember, though, that you cannot evaporate the organic layer unless there is no visible water. (If you see a brown droplet, try to extract it with a Pasteur pipette). The final caffeine should be white. Occasionally, it has a greenish tinge, which is fine. Any yellow or brown color is a sign of impurities.

Waste & Safety

Methylene chloride is a likely carcinogen and should be handled carefully. Although it should be evaporated in this particular experiment, if you have any leftover that needs to be discarded, place it in the Halogenated Organic Waste container.

The brown water layer left behind in the separatory funnel is essentially just decaffeinated tea, and can be poured down the drain.

Pre Lab Questions

1. Define the following:
   (a) Decant

   (b) Tannins

   (c) Filtrate

2. How much methylene chloride should you collect in total from the separtory funnel?

3. How will you know if your final caffeine is pure?

4. What is the purpose of the calcium carbonate in this experiment?

Post Lab Questions:

1. Look up the structure of caffeine in the Merck Index and draw the structure below.
   (a) What is the formula?
   (b) What functional groups are present?

2. People often say that chocolate also contains caffeine. This is not strictly true. chocolate instead contains a compound called theobromine, which is extremely similar but not quite the same as caffeine. Look up its structure in Merck. What is the difference between theobromine and caffeine?

3. In the US, the amount of caffeine that ends up in a drink is carefully regulated. Coca-cola has 4mg per ounce. A typical cup of brewed coffee contains 25mg of caffeine per ounce. Even decaffeinated coffee still contains some caffeine, but usually only half a milligram per ounce. Coffee is usually served in 6 oz cups. Coke, in contrast, is usually served in 12 oz cans or bottles. Caffeine is technically toxic, but only at high doses. You would need about 15,000mg of caffeine in an hour to achieve a lethal dose.
   (a) How many cups of coffee would you have to drink in an hour to reach a lethal dose?
   (b) How many cans of coke would you have to drink in an hour to reach a lethal dose?
   (c) How many cups of decaffeinated coffee would you have to drink in an hour to reach a lethal dose?

4. On the back of this page or on a separate sheet of paper, summarize the major steps of the procedure in the format of a flow chart.