# Extraction of Caffeine from Tea Leaves

**Introduction**

 Extraction is a common technique used in organic chemistry to separate or isolate a desired compound from a mixture. The two major types of extractions used in the organic laboratory are solid-liquid extraction and liquid-liquid extraction. Solid-liquid extraction is often used to isolate a natural product from its biological source, such as the leaves or bark of a tree. For example, the anti-cancer drug paclitaxel (Taxol®) can be isolated from the bark of the Pacific yew tree by solid-liquid extraction with methanol. Liquid-liquid extraction is the most common extraction technique, and involves partitioning a solute between two immiscible liquid phases, usually an aqueous (water) phase and an organic solvent phase. The relative concentration of solute in each phase depends on the solubility of the solute in each phase and is given by the partition coefficient (K):

 [g/100 mL] in organic phase

 Partition coefficient (K) =

 [g/100 mL] in aqueous phase

Caffeine is an *alkaloid* (a class of naturally occurring compounds containing nitrogen and exhibiting the properties of an alkali, hence the term *alkaloid*) which is found in many sources, such as tea and coffee. In fact, the amount of caffeine in tea leaves can be as much as 5%.



Caffeine

In this experiment, you will first use an aqueous medium (sodium carbonate in water) to extract caffeine (along with tannins, gallic acid, flavinoid pigments, and some other compounds) from tea leaves by a solid-liquid extraction process, in other words, you will make tea (albeit tea that is basic). Caffeine, tannins, gallic acid, and the flavinoid pigments are soluble in this alkaline medium, but the main component of tea leaves – cellulose – is not. You will then perform a liquid-liquid extraction of the tea solution with an organic solvent, methylene chloride, to separate the caffeine from the basic tea solution and away from the tannins, gallic acid and the remainder of the tea components. Since caffeine is more soluble in the organic solvent methylene chloride (CH2Cl2) than in water, it partitions primarily into the methylene chloride. The tannins and gallic acid, being acidic compounds, are converted to water soluble salts by the sodium carbonate, and remain in the aqueous layer during the extraction. The other components of tea are not very soluble in methylene chloride. Therefore, the extraction of tea solution with methylene chloride removes virtually no other compound but caffeine. The solid caffeine can be then be isolated by evaporation of the low-boiling methylene chloride.

**Required Reading:**

*Lehman “The Student’s Lab Companion”:*

“Planning an Experiment” (pp. 337 – 340)

“Extraction” (pp. 95 – 106)

“Drying Liquids” (pp. 148 – 153)

“Evaporation (pp. 107 – 112)

**Special Safety Notes:**

Methylene chloride is a somewhat toxic solvent, so handle it as much as possible in a hood, and avoid spilling it on yourself.

**Procedure:**

Preparation of Tea

 Place about 20 mL of water into a 100 mL beaker (use the beaker markings to estimate the volume). When the solution has reached a gentle boil, remove the watch glass and place a tea bag (the instructor will give you the weight of the tea in the bag) into the gently boiling water so that it is covered as completely as possible by the water. Note the approximate level of the liquid in the beaker at this point. Replace the watch glass and continue gently boiling the mixture for about 15 minutes. Water may evaporate during this step, so do not be afraid to add water to maintain the liquid level. Occasionally push down (GENTLY!) on the tea bag with the closed end of a test tube to maintain contact of the tea in the bag with the solution. After the 15 minute boiling period, remove the beaker from the hot plate and squeeze any excess liquid out of the tea bag into the beaker (use a stir rod or test tube to roll the tea bag against the side of the beaker – the tea bag will be HOT!), then add 1.1 g of sodium carbonate (Na2CO3) and stir the hot solution with a stir rod until the sodium carbonate is dissolved. Allow the tea solution in the beaker to cool to room temperature. While this initial tea solution is cooling, place the tea bag into a 50 mL beaker, add 5 mL of water, cover with a watch glass, and heat this to a gentle boil for about 2 minutes. Remove this beaker from the hotplate, squeeze the excess liquid out of the tea bag into beaker, then add the contents of this beaker to the tea solution in the original beaker. ***Be sure the tea solution has cooled to room temperature before you proceed to the next section!*** The tea bag from this step can be discarded.

Extraction of Caffeine from the Tea Solution

 Pour the tea solution into a 60 mL separatory funnel (***be sure that the stopcock is closed before you add the tea solution to the separatory funnel!***). Extract the tea solution with 6 mL of methylene chloride (CH2Cl2, also called dichloromethane or DCM). Mix the two layers by inverting the funnel gently several times (do not shake the funnel too hard or an emulsion will form which is very difficult to separate). Vent the funnel frequently to avoid any pressure buildup. Place the funnel in a ring and allow the layers to separate. Drain the methylene chloride extract (the lower layer) into a **dry** 25 mL Erlenmeyer flask. Avoid draining any of the aqueous layer or any emulsion (a green-brown layer between the top aqueous layer and the lower methylene chloride layer) into the flask. Perform a second extraction in the same manner with another 6 mL of methylene chloride. Combine this methylene chloride extract with the first one by draining it into the same 25 mL Erlenmeyer flask. If there are any visible water drops in the methylene chloride solution, pour the extract back into the separatory funnel, allow the layers to separate, then drain the methylene chloride layer into a **dry** 25 mL Erlenmeyer flask. Dry the methylene chloride solution by adding a small amount of granular anhydrous sodium sulfate. Allow the methylene chloride solution to remain over the drying agent for about 10 – 15 minutes.

Isolation of the Caffeine

 Decant the dried methylene chloride solution into a **dry**, **tared** 30 mL beaker. Avoid transferring any of the sodium sulfate drying agent to this beaker (a Pasteur pipet works well for the transfer). Place the beaker on a hot plate (***IN THE HOOD!***) and evaporate the methylene chloride over a low heat setting. The evaporation can be accelerated by directing a slow stream of air over the mouth of the beaker. As soon as you see solid forming in the beaker, remove it from the heat (leave it in the hood). The residual heat should be enough to complete the evaporation of the methylene chloride. Do not heat the beaker after the solvent has evaporated, otherwise you may sublime the caffeine. Determine both the weight of the caffeine and its melting point.

**References**

Pavia, D. L.; Lampman, G. M.; Kriz, G. S.; Engel, R. G. *Introduction to Organic Laboratory Techniques, A Microscale Approach;* 3rd ed.; Brooks/Cole: Pacific Grove, CA, 1999; pp. 125 – 127.

Mayo, D. W.; Pike, R. M.; Butcher, S. S. *Microscale Organic Laboratory;* John Wiley & Sons: New York, 1986; pp. 124 – 125.

Hill, R.; Barbaro, J. *Experiments in Organic Chemistry;* 3rd ed.; Contemporary Publishing Company: Raleigh, NC, 2005; pp. E4-1 – E4-4.

**Extraction of Caffeine from Tea Leaves: Pre-Lab Worksheet**

1. What is the purpose of this experiment?

2. Look up the structures and MSDS’s for the compounds used in this experiment (at the Fisher scientific website, for example) and complete the following table:

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Compound | *Structure/Formula* | *LD50* | *Flash Point* | *Corrosive?* |
| Caffeine |  |  |  |  |
| Methylene Chloride(Dichloromethane) |  |  |  |  |
| Sodium Carbonate |  |  |  |  |
| Sodium Sulfate |  |  |  |  |

3. Look up the physical properties for the compounds used in this experiment (at the Fisher scientific website, for example) and complete the following table:

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Compound | *Molecular Weight* | *Melting Point* | *Boiling Point* | *Density* |
| Caffeine |  |  |  |  |
| Methylene Chloride(Dichloromethane) |  |  |  |  |

4. Prepare a “Flow Diagram” for this experiment similar to the example found at the top of p. 338 of your lab text.

5. Describe the principle of liquid-liquid extraction.

6. What is the purpose of the sodium sulfate used in this experiment?

**Extraction of Caffeine from Tea Leaves: Post-Lab Worksheet**

Recovery Data

1. Mass of Tea Leaves: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

2. Mass of Caffeine Recovered: \_\_\_\_\_\_\_\_\_\_\_\_\_\_

3. Percentage Recovery of Caffeine from Tea: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
(show calculation)

4. How does your answer in Question 3 compare to the amount of caffeine normally found in tea, and why might it be different?

Melting Point Data

5. Melting point of your caffeine: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

6. Literature melting point of caffeine: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

7. If the melting point of your caffeine is different from the literature value, why might it be different?

Questions

8. Several organic solvents can be used for extraction of organic materials from aqueous solutions. Some of the more common solvents (other than methylene chloride) are (a) chloroform, (b) toluene, (c) ethyl acetate, and (d) diethyl ether. For each of these solvents, draw the structure and indicate, for an extraction from aqueous medium, whether the solvent will be the top layer or the bottom layer. Hint: what is the density of the solvent in question?

9. Why was sodium carbonate added to the water used to make the tea solution?

10. Caffeine is fairly soluble in ethanol (grain alcohol). Why would ethanol **not** be an appropriate solvent for extracting caffeine from an aqueous solution of tea?