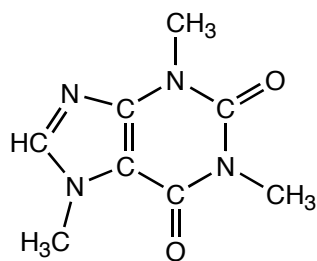


# Isolating Natural Products: Liquid-liquid extraction<sup>1</sup>

## Purpose

To extract a molecule from an aqueous solution into an organic solvent and to consider the role of pH in the process.

## Background



Caffeine

Caffeine is the principle stimulant found in tea and coffee, and it can account for up to approximately 5% of the mass of the tea leaves. Caffeine is soluble in water and a variety of organic solvents, and both can be used to extract caffeine from tea leaves or coffee beans. In fact, a wide variety of solvents including water, water in combination with organic solvents, methylene chloride, ethyl acetate, and supercritical carbon dioxide are used commercially to extract caffeine from coffee. Since we are not interested in maintaining the flavor of our tea when we extract the caffeine, we are going to use a two-step, water followed by organic solvent, extraction technique.

Taking advantage of caffeine's water solubility, we will first brew a *very* strong cup of tea. Once we have extracted the caffeine from the tea leaves we will extract the caffeine from the water using an organic solvent. Finally, we will evaporate the organic solvent to recover our caffeine.

## Procedure:

1. Place two teabags in a small beaker and add 20 mL of water.
2. Boil the tea for 20 minutes. Add water as needed to maintain the volume of the solution.
3. After testing your centrifuge tubes for leaks, transfer the tea in equal portions to two 15-mL screw-cap centrifuge tubes.
4. Add 2 mL of water to the tea bags. After gently but firmly pressing the liquid from the tea bags, transfer the 2 mL of water in equal portions to the two 15-mL centrifuge tubes used previously.
5. Add 0.5 g of sodium carbonate to each centrifuge tube.
6. Cap the centrifuge tubes and carefully shake them. Remember to vent your centrifuge tubes because the sodium carbonate will release carbon dioxide.
7. Once the tea-sodium carbonate suspension has cooled to room temperature, add 3 mL of methylene chloride (also called dichloromethane) to each centrifuge tube.
8. Cap the tubes and shake them. Remember to vent your centrifuge tubes.

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<sup>1</sup> Adapted from *Introduction to Organic Laboratory Techniques: A Microscale Approach*, 3rd edition. Pavia, Donald L., Gary M. Lampman, George S. Kriz, and Randall G. Engel (1999).

9. Spin your tea–methylene chloride suspensions in a centrifuge for five minutes to break the emulsion. Remember to balance your centrifuge tubes in the centrifuge.
10. Identify your organic layer and carefully transfer it from both centrifuge tubes to a third 15-mL centrifuge tube.
11. Add 3 mL of methylene chloride to each of the centrifuge tubes that contain the tea.
12. Cap the tubes and shake them. Remember to vent your centrifuge tubes.
13. Spin your tea–methylene chloride suspensions in a centrifuge for five minutes to break the emulsion. Remember to balance your centrifuge tubes in the centrifuge.
14. Identify your organic layer and carefully transfer it from both centrifuge tubes to the centrifuge tube that contains the methylene chloride from steps 7–10.
15. With a clean dry Pasteur pipet, transfer your methylene chloride solution, which may be contaminated with tea, from centrifuge tube three to a fourth 15-mL centrifuge tube. It is very important that no water (tea) is transferred during this step. (If no tea is present in the third centrifuge tube, this transfer step is not necessary.)
16. Dry the organic solvent with anhydrous sodium sulfate.
17. To remove the solvent, transfer the dry methylene chloride–caffeine solution to a tared 25-mL round bottomed flask.
18. Remove the methylene chloride by simple distillation. Warm the flask and the solution therein in a hot water bath (50 °C max). Continue gently heating the solution until all of the solvent has evaporated.
19. Determine the mass of your crude caffeine, and purify the caffeine by subliming the caffeine. Determine the mass of the sublimed tea.

## Report

Create a table that lists the appearance and amount of crude caffeine, and the appearance and amount of sublimed caffeine.

For example...

		Caffeine isolated from Na <sub>2</sub> CO <sub>3</sub> treated tea
crude	color	
	mass	
sublimed	color	
	mass	

Convert the Kekulé structure of caffeine on the first page to a Lewis structure. Consider the Lewis structure of caffeine and explain why caffeine is a base.

If caffeine picks up a proton, what happens to the charge of the molecule? Would this change cause the molecule to be more soluble in water or in an organic solvent?

Consider your responses to the questions above and decide how the addition of Na<sub>2</sub>CO<sub>3</sub> (a base) could affect the solubility of caffeine.

The tannins that give tea its color are neutral when protonated. What would the addition of a base like Na<sub>2</sub>CO<sub>3</sub> do to the charge of the tannins? In the presence of a base, would the tannins be more or less soluble in water? How does adding a base like Na<sub>2</sub>CO<sub>3</sub> help keep the color of your caffeine white.