

Extraction: Isolation of Caffeine from Tea

In this experiment you will isolate a compound from a natural source using two extraction techniques. Such compounds are often referred to as “natural products.” You will use of a series of techniques in isolating caffeine from tea, including both solid-liquid and liquid-liquid extractions. We have learned in another lab about another extraction technique; acidic extractions. The isolated material will be recovered and a percent recovery calculated. The identity and purity of the product will be analyzed by thin layer chromatography.

PRE-EXPERIMENT ASSIGNMENT

Study this chapter of the manual, the procedure relating to thin layer chromatography, and the lecture notes on the Chemistry Department web site for this lab and TLC. Do the first parts of your notebook writeup.

A student who has prepared for the Extraction (Caffeine from Tea) experiment should be able to:

1. Give desirable properties of extraction solvents for liquid-liquid and liquid-solid extractions (dissolves the compound being extracted from a solid, or has a favorable distribution coefficient for liquid-liquid extraction; is not miscible with the other phase in liquid-liquid extraction; has a relatively low boiling point for easy removal by evaporation; does not react with substances present).
2. Explain features of the procedure, including: why the tea is kept basic in the first extraction, what an emulsion is and how to avoid one, and the TLC visualization technique for the colorless caffeine.
3. Calculate the percentage of caffeine in tea given appropriate data, and explain possible sources of systematic error in the process (one hint: the tea bag consists of string and paper as well as tea).
4. Draw the structure given the name, or give the name from the structure, of the compounds used in the day's experiment, and give the role of each (reactant, solvent, catalyst, etc.).
5. Identify and explain safety considerations for this experiment.
6. Perform the day's experiment safely and successfully.

A Word to the Wise

This is a long experiment, so arrive early and work efficiently. As soon as you arrive at lab, begin boiling your water and brewing your tea. The quiz and pre-experiment lecture will not begin until everyone has started their tea brewing. Students who do not arrive on time and who delay the class, your instructor is likely to subtract points.

Brewing the Tea: Solid-Liquid Extraction

Cut the tags from two tea bags and weigh both of the bags together. Record this mass directly in notebook. Place the bags in the bottom of a 100 mL beaker. Add approximately 2 g of solid sodium carbonate and 25 mL of water to the beaker. Add a boiling stick and bring the water to a boil on a hot plate. As soon as the water begins to boil, remove the beaker from the hot plate. Do not allow it to continue boiling. That will cause the liquid to evaporate rather than extracting additional tea.

Turn the hot plate off and unplug. Allow the beaker to stand and cool in a safe place on your lab bench during the quiz and pre-experiment lecture. When the experiment begins again, squeeze the water out of the tea bags and into the beaker by wrapping them around the boiling stick. The bags should be cool by now, but be careful not to burn your fingers or tear the bags. If you think that you have too little liquid or that your tea is not concentrated enough, ask for your instructor's advice before proceeding.

Extracting Caffeine into Dichloromethane: Liquid-Liquid Extraction.

The tea must be close to room temperature (~25°C) before proceeding. If it still feels warmer than that, check its temperature with a thermometer. The next solvent, dichloromethane boils at 40.0°C. If the tea is too hot, when the methylene chloride is added it will immediately boil away. Divide the liquid tea equally between two plastic centrifuge tubes. Use an ice bath, if necessary, to cool the tea. Add 3 mL of dichloromethane to each tube. Cap the tubes tightly and shake moderately. If the tubes are shaken too vigorously an intractable emulsion may form and product recovery may be reduced. Vent the tubes by removing the caps.

Centrifuge the tubes for about 2 minutes to break up the emulsions. Repeat, if necessary. Using a Pasteur pipette, remove the organic layers, which should be the lower layers, from each tube and transfer them to the same small (10 or 50 mL) Erlenmeyer flask. Leave the aqueous layers in the tubes. This will be demonstrated by your professor. A steady hand and patience is beneficial to carrying out this separation effectively. If a small amount of water gets into the flask along with the organic layer, leave it there. It can be removed later.

Add a fresh 3 mL of dichloromethane to each tube and repeat the entire process described in the previous paragraph. Both organic layers should be

added to the same Erlenmeyer flask as in the previous step so that all of the dichloromethane extracts are combined in one Erlenmeyer flask.

Workup and Product Recovery

If more than a few drops of water are floating on top of the dichloromethane solution in your Erlenmeyer flask, use a Pasteur pipette and carefully remove as much of it as you can without removing any of the organic layer. Transferring the solution to a clean test tube may make this easier.

Add a small amount of anhydrous sodium sulfate drying agent to the flask. Swirl it and look at the solid. If it clumps together it has picked up water. If so, add a little fresh drying agent, swirl, and observe the freshly-added drying agent. Keep adding small amounts of sodium sulfate until some of the grains move sort of like sand when the flask is swirled. Note the first clumps will remain. (The term “dry” as used in chemistry labs usually means that water is absent. The dichloromethane should still be present and the solution should still be liquid.) If you add too much drying agent, it may physically absorb all of the liquid and a low to zero percent recovery will be obtained. After some sodium sulfate remains mobile, let the flask sit undisturbed for about 5 minutes. You can use this time to clean up some of your area, weigh a clean dry watch glass, and set up a TLC plate. Instructions for setting up and running the TLC are below.

Decant the dried dichloromethane solution onto a tared (pre-weighed), labeled watch glass in the hood. Do not pour the solution into watch glass on bench and then walk the full watch glass to hood. Obtain a capillary spotter and collect a small portion of the dissolved sample and spot this sample on TLC plate for analysis. More details on the TLC analysis are below. Allow the dichloromethane to evaporate in the hood while you are performing the TLC experiment. What remains on the watch glass after the solvent evaporates should be caffeine. Weigh the watch glass again on the same balance. The difference between the final weight and the tare weight (the weight of the empty watch glass) is the weight of the caffeine.

Thin Layer Chromatography

Obtain a TLC plate. Remember to **not** touch the matte surface of the plate as this will leave greasy fingerprints. Follow the TLC procedure as described in the TLC procedure of this lab manual. Only use pencil, not pen on a TLC plate. Mark a faint straight line approximately one centimeter from the bottom. Label the plate for two spots; authentic caffeine and sample. The mobile phase that will be used is 95% Ethyl Acetate, 5% Acetic Acid. As stated above, the assumption is that the solid recovered is caffeine. You will test that assumption by comparing R_f values, and also assess the purity of your sample.

Spot the TLC plate with your sample; use another spotter to spot authentic caffeine from a solution of caffeine in ethanol that is available in the laboratory.

If your spotter or the product dried out while you were doing something else, dissolve a small amount of your product in ethanol and use that.

Set up a TLC developing chamber. Two or more people may share a single developing chamber. Run only one TLC plate in a chamber at a time. Remember to remove the TLC plate from the chamber after the solvent has moved up the plate more than half way but **before** the solvent reaches the top of the plate.

Pure caffeine is white so it will not be visible to the unaided eye. After the solvent has dried, examine the plate under UV light to observe the spots. (*Safety note: Don't look directly at the UV light.*) Outline the spots with a pencil, place a small dot in the exact center of each spot. Attach the plate to your datasheet. Draw a scale replica in your notebook.

Calculate R_f for each spot. Remember that the starting point for your measurements is the position of the original spots. This is true of the solvent as well as of the spots.

CLEANUP

Discard used Pasteur pipettes in the broken glass disposal box. Place excess dichloromethane in the liquid halogenated waste container in the hood, and place your TLC solvent (95% ethyl acetate and 5% acetic acid) in the container for non-halogenated liquid organic waste. Discard the tea bags and boiling stick in the waste basket. The watch glass can be washed in the sink with soap and water.

Wipe down your work area with a sponge.

POST-EXPERIMENT ASSIGNMENT

Turn notebook pages into lab instructor. Complete datasheet and turn into lab instructor.

Prepare for the isolation of caffeine portion of the next quiz.