

ISOLATION OF CAFFEINE FROM TEA

EXPERIMENTAL TECHNIQUES REQUIRED

[Extraction](#) (T 6), [drying agents](#) (T 7), [filtration](#) (T 3), [rotary evaporation](#) (T 8), [recrystallisation](#) (T 2) and/or [sublimation](#) (T 9)

EXPERIMENTAL PROCEDURE (Individual work)



Wear gloves when handling dichloromethane. It has harmful vapours.

Avoid skin contact and avoid breathing the vapours.

Keep in the fumehood whenever possible.

Add 100 mL of boiling distilled water to a large beaker and add three preweighed tea bags (note the paper of a single tea bag weighs about 0.15 g). Allow the solution to sit for 10 minutes stirring periodically. Carefully remove the tea bags and press them with a spatula to obtain as much of the tea solution as possible, then rinse the tea bags with some distilled water and repress them to extract as much as possible. Cool the solution by adding about 75ml of crushed ice and standing the beaker in an ice-water bath.

IMPORTANT : *The tea solution must be cooled to room temperature using the ice-water bath before it is used in the extraction step.*

Pour the cooled tea solution into a separatory funnel. Slowly add 20 mL of dichloromethane and shake the separatory funnel to mix the two layers together. Allow the layers to separate (this may take a few minutes, be patient. Note that an emulsion may form, in which case gently stir it with a glass rod to try to encourage it to break up. If it doesn't break up, talk to your TA). Once the layers have separated, carefully drain off the lower organic phase and any emulsion that remains into a 125 mL Erlenmeyer flask. Allow as little as possible of the aqueous layer to escape through the stopcock. Repeat the extraction procedure twice more using fresh 20 mL portions of dichloromethane and combine these two dichloromethane layers with the first. Put the aqueous layer to one side (just in case you need it).

Return the combined dichloromethane organic phases to the separatory funnel and wash with 20 mL of 6 M aqueous NaOH by adding the NaOH solution to the separatory funnel, shaking then allowing the mixture to settle. Remove the lower dichloromethane layer then collect the aqueous layer. Return the dichloromethane layer to the separatory funnel and repeat the wash with a second 20 mL portion of 6 M aqueous NaOH and then wash once with 20 mL of distilled water. At this point be patient and allow the mixture to separate as much as possible. Drain the lower dichloromethane layer into a dry 250 mL Erlenmeyer flask taking care not to allow any of the aqueous layer out of the separatory funnel. If the flask contains any water, you should repeat the separation. Dry the dichloromethane solution by adding about 1 teaspoonful of anhydrous magnesium sulphate. Swirl the flask and allow it to settle. If there is fine

powdery magnesium sulphate still visible then the solution is dry (if not, add more magnesium sulphate), then remove the drying agent by gravity filtration through a filter paper in a stemmed filter funnel into a dry 250 mL Erlenmeyer flask. Transfer the solution to a pre-weighed 100 mL round bottom flask (don't overfill the flask, it can be done in several cycles if needed) and remove the dichloromethane using the rotary evaporator (make sure to ask your TA for assistance). Weigh the flask containing the crude product and determine your crude yield. The crude caffeine may be further purified *either* by recrystallisation *or* by sublimation:

Recrystallisation (recommended)

Dissolve the crude caffeine in the **minimum amount** (< 2.5mL) of hot acetone on a steam bath and quickly transfer it to a large test tube. Cool the solution in an ice bath, crystals should start to form. If crystals don't start to form, reheat the acetone solution and then add drop-wise enough low boiling (30-60C) petroleum ether to turn the solution faintly cloudy (note this may require up to a few mL of pet ether), then cool in an ice bath. Once crystallisation is complete, collect the product by vacuum filtration using a Hirsch funnel, wash with cold petroleum ether and transfer into a pre-weighed sample bag.

Sublimation

Loosely fit a "cold finger" (insert a test-tube containing ice-water into the mouth of the pre-dried Büchner flask, and loosely seal the "cold finger" by means of the small cone filter adapter) into the Büchner flask so that the end of the tube is about 3cm above the base of the flask. Gently heat the flask with a Bunsen burner where the crude caffeine is situated. If a lot of water droplets collect on the cold finger, it may be necessary to remove it and wipe it dry. As the sublimate collects, periodically remove the "cold finger" very carefully and scrape the pure caffeine onto glassine paper or into a preweighed vial. Replace the "cold finger" and allow further sublimate to accumulate, weigh the product. As the ice in the 'cold finger' melts, water may be removed with a pipette.

Determine the weight of the pure product and then calculate the weight percent of caffeine in tea based on the initial amount of tea used. Show your properly labeled sample of caffeine to your laboratory TA. The caffeine sample needs to be kept and used in the chromatography experiment.

CLEAN UP

- The magnesium sulfate should be allowed to dry on a paper towel in the fume-hood. It should then be placed in the solid waste container.
- Waste dichloromethane should be put into to the organic waste container in the fume-hood.
- The aqueous waste should be put into the white aqueous waste drum.

REPORT

Before writing any Chem 351 laboratory report, we strongly recommend that you review section 8 in the introductory section of the [student laboratory manual](#) that discusses how to write reports and/or from “[writing reports](#)” on the course website. Students often don’t get the grades they would like because they make errors that are addressed in that section of the manual. These are avoidable errors.

The report for this experiment is a formal report. Remember that more is not necessarily better. It is important to be accurate and concise rather than verbose and vague. Proper English should be used and it should be written in your own words. A **guide** to the sections/information you should include is shown below.

REPORT SECTIONS FOR THE EXTRACTION OF CAFFEINE

MAXIMUM 4 PAGES including the following content (line spacing 1.5, Arial (font size 12)):

1. Title, Date of the experiment, laboratory section (B xx), TA name.
 2. Introduction.
 3. Experimental procedure.
 4. Results (for example: weight(s) of product(s) obtained, appearance, yields, *etc*).
 5. Discussion of results.
 6. Conclusions.
 7. References (if required)
 7. Questions: (to answer these questions, make sure you’ve read the [extraction technique document](#)).
- a. Consider a compound “X” that has a distribution coefficient, K_D (ether : water) = 2. If 1g of “X” was dissolved in 25mL of water, and this aqueous solution was then extracted once with 25mL of diethyl ether, how many g of “X” would be extracted into the diethyl ether layer ?
 - b. When carrying out an extraction, which of the following maximises the yield of extracted product from 25mL of solution, using 1 x 75mL or doing 3 successive extractions of 25mL each. Explain your reasoning.