

# **Extraction of Caffeine from Tea Leaves and Coffee – An Inquiry-Based Experiment**

## **Summary**

Reference: <http://spot.pcc.edu/~chandy/241/CaffeineExtractionCH2CCl2.pdf>

Caffeine can be easily isolated from tea leaves and ground coffee beans in the laboratory. The process usually begins by making an aqueous tea or coffee solution to dissolve caffeine from tea leaves and coffee beans into water, and followed by extracting caffeine from the tea solution with an organic solvent. By choosing the suitable solvent and under the appropriate conditions, caffeine can be extracted very efficiently into the organic solution along with very small amounts of other substances as contaminants. The caffeine can be recovered from the organic solution by concentration, and subsequently purified by sublimation or recrystallization.

Caffeine belongs to a class of natural nitrogen-containing compounds known as alkaloids. The nitrogen atoms render caffeine having different solubility in water at different pH. In mild basic solutions caffeine remains in its neutral form and its water solubility is limited. In acidic solutions, however, caffeine is protonated to become cationic and form the conjugated acid salt, and therefore its water solubility is enhanced. Since tea leaves contain tannins which are acidic, an appropriate base should be added to the tea solution to minimize the solubility of caffeine in water and facilitate the extraction process. Furthermore, the base induces the hydrolysis of tannins. Therefore, the base helps removing the tannins from the product.

In this project you will exercise collaborative efforts to investigate the following issues related to the extraction of caffeine. Attached is a typical experimental procedure for isolating caffeine from dried tea leaves. You may vary part of the experimental details to investigate how these changes will affect the efficiency of the extraction process.

1. The caffeine contents in different brands of tea leaves and ground coffee beans.
2. The best choice of base for maximizing the amount of caffeine extracted.
3. The best choice of organic solvent for maximizing the amount of caffeine extracted.

# Extraction of Caffeine from Tea Leaves and Coffee

## Lab Documentation

### Introduction

Tea and coffee have been popular beverages for centuries, primarily because they contain the stimulant caffeine. Caffeine belongs to a large class of compounds known as alkaloids. They are of plant origin, contain basic nitrogen, often have a bitter taste and complex structure, and usually have physiological activities.

The major plant sources of caffeine are tea leaves, coffee beans and cocoa beans. Coffee beans contain about 0.8 – 2.0 weight percentage of caffeine, while the caffeine content in tea leaves is typically between 2 and 3.5%. The exact amount of caffeine in the beverages, however, depends on the style of brewing. For example, a cup of espresso coffee contains less caffeine than standard brewed coffee do. The table below shows the typical amount of caffeine present in some common beverages.

<b>Beverage</b>	<b>Caffeine (mg/cup)</b>
American coffee	70 – 100
Black tea	20 – 35
Coke	30 – 50

The extraction of caffeine usually begins by making an aqueous tea or coffee solution to dissolve caffeine from tea leaves and coffee beans into water. The compounds extracted along with caffeine are tannins, residual pigments, and trace amounts of glucose, amino acids, proteins, and saponins. A suitable base is often added when preparing the tea/coffee solution. The base helps to maintain the aqueous solution at alkaline conditions to prevent the caffeine from being protonated by acid and forming a water-soluble salt. It enhances the solubility of caffeine in organic medium in the liquid-liquid extraction process, and so the efficiency of the extraction process. Furthermore, the base promotes the hydrolysis of tannins and converts them into some very polar, water soluble substances. Therefore, they can be removed from the organic solution during the liquid-liquid extraction process.

Caffeine is a moderately polar compound and is more soluble in organic solvents than in water. The solubility of caffeine in dichloromethane is roughly about ten times better than in water. Therefore, caffeine can be effectively isolated from the

aqueous solution by solvent-solvent extraction.

Shown below is a typical experimental procedure for isolating caffeine from tea leaves. Besides tea leaves, you may also try to apply this procedure (with some necessary modifications) to other types of samples such as ground coffee beans or instant coffee powders. Furthermore, instead of strictly following the given procedure, you may also change some of the experimental conditions such as varying the type of base or organic solvent used to investigate how varying these factors will affect the efficiency of the extraction process.

## **Experimental Procedure**

### **Materials and Apparatus:**

Solvent: *n*-hexane, dichloromethane, ethyl acetate, diethyl ether, acetone  
Tea bags  
Calcium carbonate  
Saturated sodium chloride solution  
Anhydrous magnesium sulfate  
Stirring hotplate  
Rotary evaporator  
General lab glassware kit

### **Part A: Extraction of Soluble Ingredients from Tea Bags**

Boil 5 tea bags and 5 g of calcium carbonate in 250 mL of boiling water in a 600 mL beaker on a hot plate. Do not break the tea bags. Cover the beaker loosely with aluminum foil to minimize evaporation. Boil the mixture gently for 10-15 minutes. Remove the tea bags and squeeze as much solution from the tea bags as possible.

Filter the hot mixture by suction (vacuum) filtration. The mixture should be kept hot during filtration to prevent precipitation which makes the filtration very difficult. Wash the solid residue with 20 mL of hot water and combine the washing with the filtrate. Transfer the filtrate into a large beaker and concentrate the filtrate to about 100 mL by boiling it on a hot plate.

## Part B: Isolation Caffeine from the Tea Solution by Liquid-Liquid Extraction

*\* In this part of the experiment, the presence of saponins in the mixture favors the formation of stubborn emulsions and makes the separation of layers difficult. To avoid this problem, do not shake the separatory funnel too vigorously.*

Cool down the concentrated filtrate to room temperature and transfer it to a separatory funnel. Add 25 mL of dichloromethane. Shake the separatory funnel gently to extract caffeine into the organic layer. Release the pressure inside the funnel occasionally. Allow the funnel to stand still and let the layers to separate. Remove the stopper and drain the organic (lower) layer into a 100 mL conical flask. Leave the aqueous solution (upper layer) and the emulsion layer in the separatory funnel.

**Note:** Do not add sodium chloride to the mixture even if emulsion forms because a large amount of water soluble organic substances are dissolved in the aqueous layer. Adding sodium chloride will cause those substances salting out from the aqueous solution and form precipitates. It makes the situation even much worse.

Repeat the extraction process twice using 25 mL portions of fresh dichloromethane.

Transfer the combined organic solutions back to the separatory funnel, and extract the organic solution with 20 mL of saturated sodium chloride solution to remove the residual water. Collect the organic solution in a dry 100 mL conical flask and dry the solution with anhydrous magnesium sulfate ( $\text{MgSO}_4$ ). Add the drying agent in portions with swirling until it no longer clumps together.

Remove the drying agent by simple filtration. Wash the drying agent with several small portions of dichloromethane and combine the washings to the filtrate. Transfer the solution to a pre-weighed 250 mL round-bottomed flask. Evaporate the solvent using a rotary evaporator to obtain the crude caffeine.

## Part C: Purification of the Crude Caffeine by Recrystallization

Dissolve the crude caffeine in 5 mL of hot acetone in a small conical flask, and warm it gently with a hot water bath. Add hexane to the solution while it is hot until a faint cloudiness appears. Set the flask aside and allow it to cool slowly to room

temperature.

Cool the mixture with an ice-water bath, and then collect the crystals by vacuum filtration. Determine the weight and the percentage yield of the pure caffeine obtained.

## Extraction of Caffeine from Tea Leaves and Coffee

### Data Sheet

Name of Participants: \_\_\_\_\_

\_\_\_\_\_

Type and Brand of Sample: \_\_\_\_\_

Amount of Sample Used: \_\_\_\_\_

Type and Amount of Base Used: \_\_\_\_\_

Solvent Used for the Extraction: \_\_\_\_\_

Amount of Caffeine Obtained:

Before Recrystallization: \_\_\_\_\_ (        %)

After Recrystallization: \_\_\_\_\_ (        %)

Remarks / Other Comments: