Lab #1- The Separation and Extraction of Caffeine

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CHM 2123 E00

Introduction

The purification of chemical compounds is an important technique used in many organic chemistry experiments and in everyday life. There are three technique that can be used to purify a compound for the purpose of this lab. The first is the extraction in which the extraction is performed using two immiscible compounds to isolate the organic layer containing our target compound. The organic layer will contain compounds with nonpolar molecules and have a low solubility level while the aqueous phase will contain polar compounds and have a high water solubility level, this is why they don't mix. The second technique is sublimation with the use of a cold finger which is the process in which a solid state compound converts to a gas state compound without the liquid intermediate. The cold finger containing dry ice and the heat of a hot plate allows the compound to vapourize and for small crystals to condense onto the outside of the cold finger. This gives a purified version of the target compound. The last technique is thin layer chromatography(TLC) where the use of polar and nonpolar interactions is the focal point to identify the purity of the compound. The TLC plate is covered with a very polar substance(silica gel) where the starting compound, purified compound and reference compound are compared to each other with the use of a solvent acting as a mobile phase moving the compounds up with it. If the compound is more polar it will have stronger interactions with the silica gel therefore, not moving very far up the plate. If the compound has nonpolar properties then it will move up the plate a lot easier and quicker with the solvent.

The goal of this experiment is to extract and purify the purest version of caffeine from tea leaves. When the purification is done using a separatory funnel and sublimation, the use of a TLC plate is used to compare the version of caffeine purified in the lab and a very pure version of caffeine as the reference. This way it can be determined how well the techniques were performed.

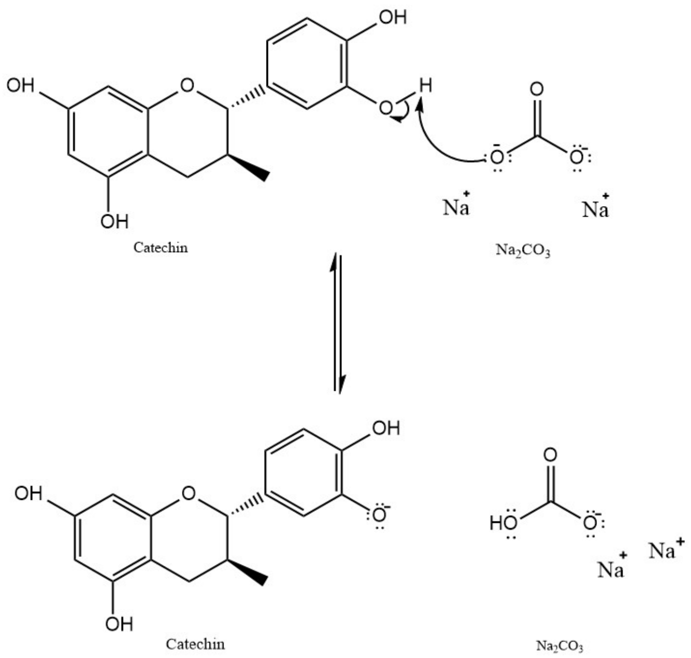


Figure 1. Deprotonation of catechin using sodium carbonate

Experimental Procedure

Refer to "Experiment #1" pages 6-8

Table of Reagents

Reagent	Molar Mass	Amount(g or mL)	Density(g/mL)	Moles	Equivalent
$CH_2Cl_{2(l)}$	84.93	45.0	1.33	0.705	1
$Na_2CO_{3(s)}$	105.99	2.0	N/A	0.0189	N/A
$H_2O_{(l)}$	18.02	60.0	1	3.33	N/A

Table 1. Table of reagents

Observations and Results

Before extraction	- Steeped tea was a dark opaque brown - When Na_2CO_3 was added it turned the tea an opaque black
During extraction	- Dichloromethane(clear liquid) on the

	bottom as the organic phase which included the caffeine - Aqueous phase on the top containing all other compounds from the tea that isn't caffeine - When separation occurs brown bubbles were present in the organic layer(emulsion)
After extraction	- Organic layer is clear with a few brown bubbles - Aqueous layer looks black and also contains a very turbid brown/black on the bottom half with some bubbles - Organic layer is dried and now contains no brown liquid/bubbles - Dried caffeine has mass of 0.01g
During sublimation	- 0.01g of caffeine is a powdery yellowish/white substance - Dry ice causes steam and small water droplets of condensation - White powder forming on cold finger

Table 2. Observations made during experiment

Compound	Molar Mass (g/mol)	Quantity (g)	mmoles	Yield (%)	Melting Point (°C)
Caffeine	194.19	0.01	0.0515	4.7	233-237

Table 3. Table of resulting product

Calculations

Rf value

$R_f = \frac{distance\ travelled\ by\ the\ compound}{distance\ travelled\ by\ the\ solvent\ front}$

$R_f = \frac{4cm}{5.1cm}$

$R_f = 0.78$

Percent Yield

$\%yield = \frac{actual\ yield}{theoretical\ yield}\ x100\%$

$\%yield = \frac{0.01g}{0.2132g}\ x100\%$

$\%yield = 4.7\%$

TLC Results

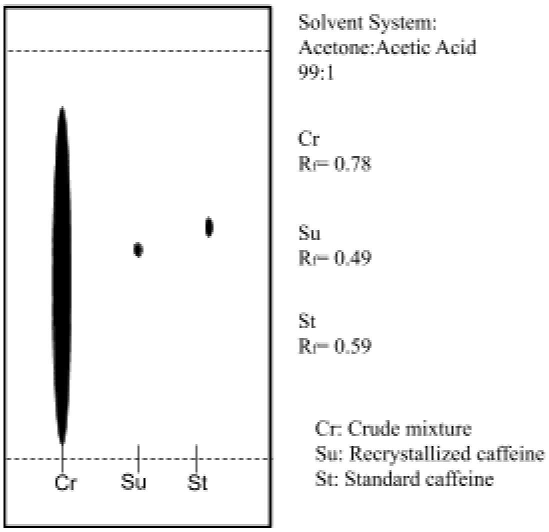


Figure 2. TLC performed in 99:1 Acetone:Acetic acid

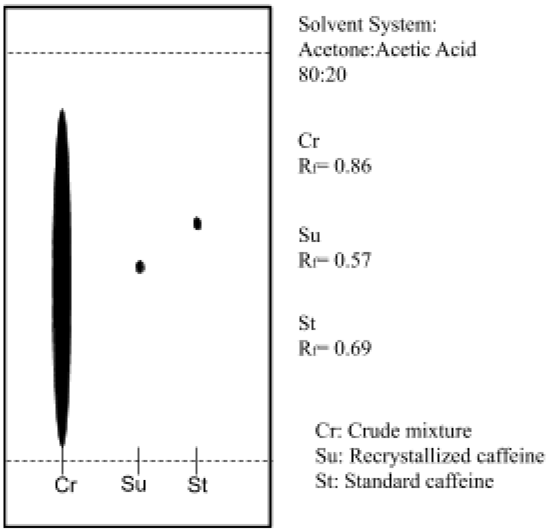


Figure 3. TLC performed in 80:20 Acetone: Acetic acid

Flowchart

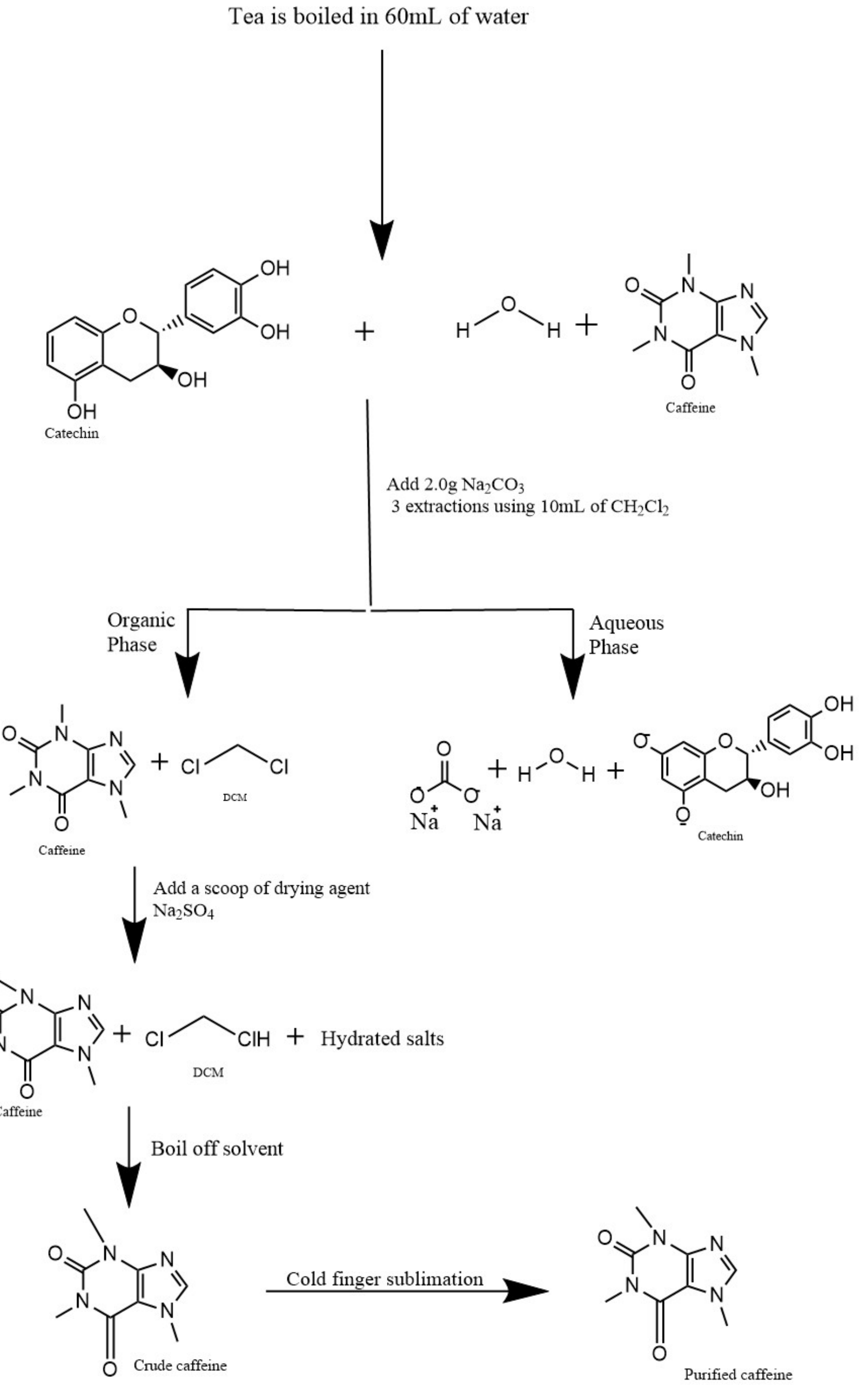


Figure 4. Flowchart of the process of extraction and purification of caffeine

Discussion

The goal of this experiment was to extract and purify caffeine from tea leaves while using two important techniques, extraction using a separatory funnel and sublimation. Before extraction sodium carbonate is added to the steeped tea before transferring it to the separatory funnel because this keeps the acidic compounds soluble in water and allowing the caffeine to move into a more basic environment like dichloromethane. Sodium carbonate acts as a base that deprotonates the tannins in the tea leaves making them more ionic and more soluble in water. This way in dichloromethane, the ionic strength in the aqueous phase is much stronger and will push the caffeine we need into the organic layer. The solution and dichloromethane are added to the separatory funnel, where it's apparent that the organic layer is on the bottom and the aqueous layer is on the top. Since dichloromethane has a higher density than water it will rest on the bottom along with drawing out the caffeine from the aqueous layer. Although caffeine is a more polar substance in this basic solution with the dichloromethane it becomes less polar and therefore is drawn out of the aqueous layer.

During separation, there was some emulsion between the organic and aqueous phases which was the result of not shaking the funnel gently enough but this was resolved by gently shaking the funnel after the two layers had time to separate. This was apparent during the three times dichloromethane was used to extract the caffeine. Since caffeine is semi-soluble in water and the caffeine gets lost in the aqueous phase, more washes of dichloromethane would help increase the amount of caffeine recovered and this is why three washes are performed.

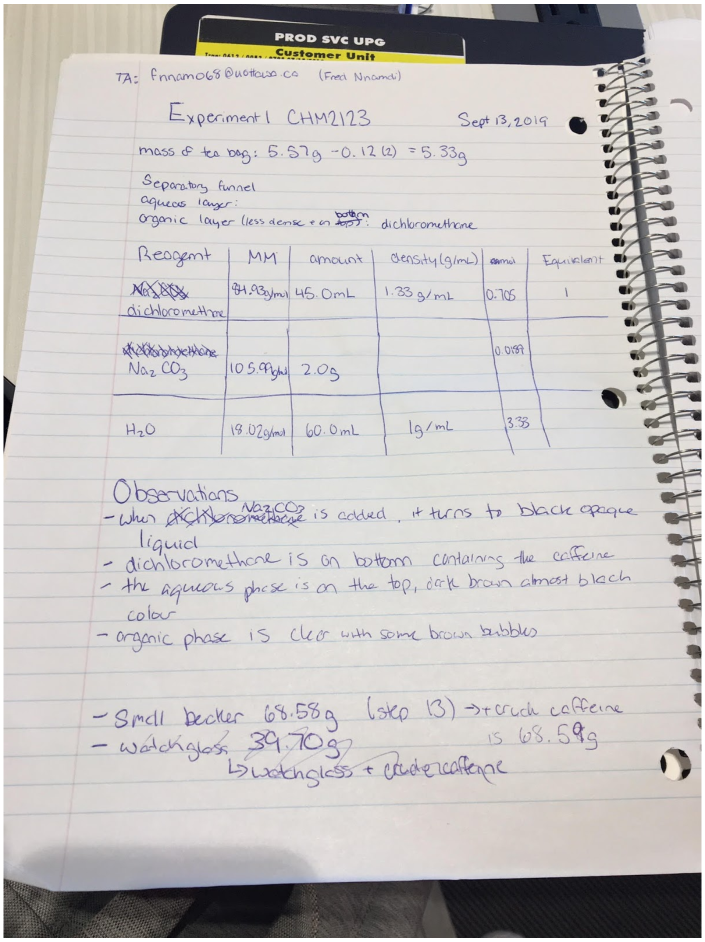
Next, the sodium sulphide is added to the organic layer as a drying agent to ensure that any water present is gotten rid of so the solution containing the caffeine can be boiled away leaving behind the crude caffeine. The drying agent clumps the water together, creating hydrated salts that can be extracted using a funnel and filter paper. After boiling the organic layer in a beaker of water, the crude caffeine is scraped and measured demonstrating a solid has formed. Sublimation is the process in which a solid goes directly through to the gas phase and will deposit crystals of purified product onto the cold finger structure. Sublimation is used to separate the caffeine from the less volatile impurities. The use of dry ice in the cold finger, the heating plate and the constant pressure of a vacuum creates perfect conditions for the solid pure caffeine to deposit onto the cold finger.

A couple sources of error must have occured in regards the the small percent yield of 4.7%. The first source of error is the emulsion in the separatory funnel that prevented a large amount of caffeine from being drawn into the organic layer, it is possible that it got stuck in the aqueous layer and the turbid layer. Even though three washes of dichloromethane were performed, it may not have been enough due to have emulsion present in all three washes. A second source of error is in the sublimation apparatus. We washed the glassware to prevent any contaminants from coming into contact with the purified caffeine but since the water did not dry fast enough, we had to chemical force it to dry using acetone. If there is water on the cold finger it will not allow the caffeine to solidify properly. Also since such a small amount of caffeine is obtained any droplet of condensation could cause the caffeine to dissolve right off the cold finger. We did see a couple droplets of condensation that may have caused this.. It is also possible that the pressure and temperature of the sublimator did not stay constant which would affect the sublimation conditions. These errors can be fixed by being more patient and taking more precaution when transferring solutions and washing the organic phase more carefully to avoid emulsion. Also it would be helpful to do more rinses of dichloromethane to maximize the amount of caffeine being extracted.

The TLC results were very similar between both solvents. Since acetic acid is more polar than the acetone, the 80:20 acetone: acetic acid mixture provided higher Rf values than 99:1 acetone: acetic acid. Since the 80:20 solvent is more polar it brought the more non polar compounds up with it creating the higher Rf values which is seen in *Figure 3.* An error made during the TLC is that there is a streak for the crude mixture which means the mixture was too concentrated. Also the recrystallized caffeine dot was much more faint meaning that the mixture of dichloromethane to caffeine wasn't concentrated enough. But only 0.01g of caffeine was obtained after sublimation so this is the reason why it wasn't concentrated. Being more tedious while extracting the caffeine from previous steps would provide for a better concentration for the reference.

The melting point of the purified caffeine obtained in the experiment was a range of 233-237 °C. The melting point of pure caffeine used as the reference is actually 235-238 °C which is very similar to what was obtained in the lab. Although since it is not the exact same we can conclude that there are some impurities present.

Raw Data



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mass of tea bag: 5.57g − 0.12 (2) = 5.33g

Separatory funnel

aqueous layer:

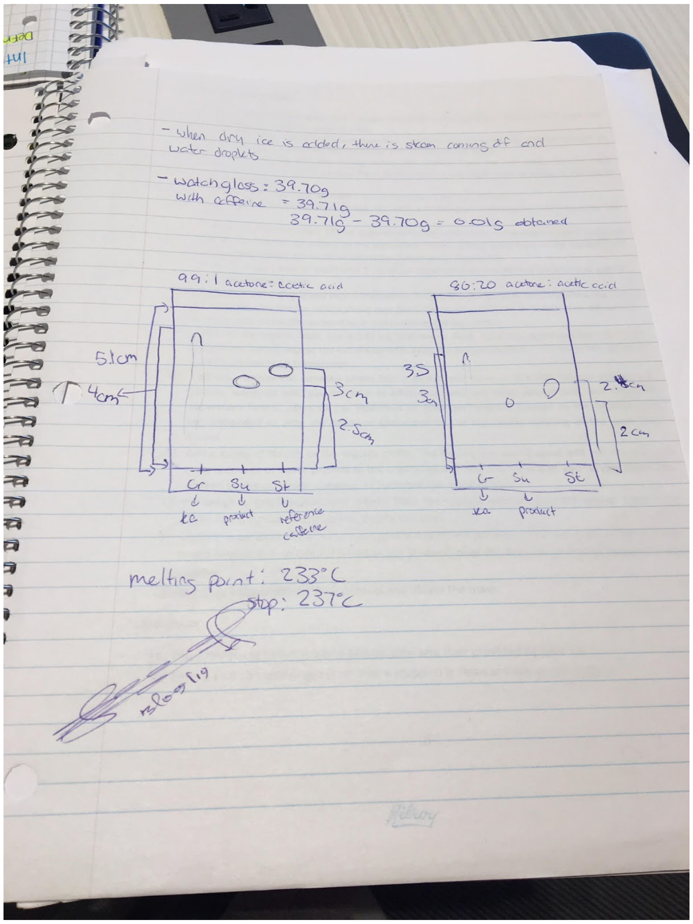
organic layer (less dense + on ~~top~~ bottom): dichloromethane

Reagent	MM	amount	density (g/mL)	mmol	Equivalent
~~Na_2CO_3~~ dichloromethane	84.93g/mol	45.0mL	1.33 g/mL	0.705	1
~~dichloromethane~~ Na_2CO_3	105.99g/mol	2.0g		0.0189	
H_2O	18.02g/mol	60.0mL	1g/mL	3.33	

Observations

- when ~~dichloromethane~~ Na_2CO_3 is added, it turns to black opaque liquid
- dichloromethane is on bottom containing the caffeine
- the aqueous phase is on the top, dark brown almost black colour
- organic phase is clear with some brown bubbles

- Small beaker 68.58g (step 13) → + crude caffeine is 68.59g
- watchglass 39.70g
  ↳ watchglass + crude caffeine



- when dry ice is added, there is steam coming off and water droplets

- watchglass: 39.70g
  with caffeine = 39.71g
  39.71g − 39.70g = 0.01g obtained

melting point: 233°C
stop: 237°C