INTRODUCTION

The goal of this experiment is to isolate and purify caffeine from tea. This is first done through an extraction. Extractions make use of two immiscible solvents, often times using water as an aqueous phase and an organic solvent as an organic phase. As they are immiscible, extraction takes advantage of the fact that they have different solubility. In general, the inorganic or polar compounds will dissolve in the aqueous phase, while the organic or nonpolar compounds will dissolve in the organic phase. Through this method, extraction allows for the movement of compounds from one phase to the other while leaving behind any unwanted impurities. With the addition of dichloromethane to the caffeine containing tea, it was possible to wash the caffeine from the aqueous phase into the organic phase, separating it from the various other components in the tea which remain in the aqueous phase. The organic phase can then be extracted.

After extraction of the organic phase, the solvent is boiled off to leave a solid, crude caffeine product. The caffeine is further purified through sublimation. Sublimation is the phase change from a solid state into a gas. The crude product is first heated, and the sublimed caffeine gas rises. Once the gaseous caffeine makes contact with the cold finger, it deposits onto the cold finger and recrystallizes, leaving a purified caffeine product. This isolates the product in the case where the impurities are non-volatile substances. In order for sublimation to work, a vacuum must be created to lower the air pressure inside, allowing caffeine to bypass the liquid phase and instead transition from solid to gas.

Thin layer chromatography is then used to identify the product formed. This is done by comparing the purified product to a reference of authentic caffeine. TLC plates consist of a stationary phase made of silica gel, which is highly polar. The plate is then spotted with the purified product in comparison to authentic caffeine. The developing solvent, or eluent, displaces these spots as the solvent rises up the plate through capillary action. Polar molecules can bind to the silica gel through hydrogen bonding and dipole-dipole interactions. Because of this, the more polar the compound is, the more it will interact with the silica gel, and the slower it will travel up the plate. Therefore, if the displacement of the spots, or the retention factor R_f, are equal, it can be determined that the purified product in question is in fact caffeine.

Melting point can also be used to determine purity of the product. Any impurities will cause defects within the crystal lattice structure, making the lattice unstable. As a result, the melting point of an impure product would have a lower melting point than the theoretical pure product, as the intermolecular interactions that hold the molecule together are weaker. Thus, it requires less energy to change phases. A melting point apparatus is used to measure the melting point of the purified product as another method of determining purity. However, this step is skipped due to time constraints.

PROCEDURE

Please refer to the CHM2123 lab documentation for experiment 1, part A, pages 8 – 10.

Modifications

- Organic solvent was boiled in step 15 using a water bath instead of a steam bath
- Cold finger was filled with dry ice in step 23 instead of crushed ice
- Did not determine melting point of the purified crystals, step 26 was skipped

RESULTS AND OBSERVATIONS

Table 1: Table of Reagents

Compound	Molar Mass (g/mol)	Quantity (g or mL)	Density (g/mL)	Moles (mmol)	Equivalents
Water	--	50 mL	--	--	--
Na_2CO_3	105.99	2.0003 g	--	18.873	--
Dichloromethane	--	35 mL	--	--	--
Na_2SO_4	--	--	--	--	--

Table 2: Observations

Key Step	Observation
Extraction of the organic phase	- Contains a few small droplets of the aqueous phase
Addition of Na_2SO_4 to organic phase	- White crystals become clumps at the bottom of the beaker, slightly brown
Boiling the organic phase	- Organic solvent evaporates, leaves an off-white powder at the bottom of the flask
Sublimation of caffeine	- Crude product quickly vaporizes, cold finger is coated with a white powder

Table 3: Results

	Mass (g)	Yield (%)	Height (cm)	R_f	Temp. (°C)
Tea Bags (2)	6.55	--	--	--	--
Empty Tea Bags (2)	0.24	--	--	--	--
Tea Leaves	6.31	--	--	--	--
Crude Caffeine	0.0086	--	1.6	0.36	--
Test Tube, Stopper, and 50 mL Beaker	64.5296	--	--	--	--
With Purified Caffeine	64.5328	--	--	--	--
Purified Caffeine	0.0032	1.27%	1.6	0.36	--
Authentic Caffeine	--	--	1.6	0.36	--
Solvent Front	--	--	4.5	--	--
Melting Point	--	--	--	--	239.9*

* Data taken from announcement on CHM2123 Blackboard Learn

Sample Calculations

$$Percent\ Yield = \frac{Experimental\ Yield}{Theoretical\ Yield}\ x\ 100\%$$

$$= \frac{0.0032\ g\ Caffeine}{(6.31\ g\ Tea\ Leaves)(0.04\ \frac{g\ Caffeine}{g\ Tea\ Leaves})}\ x\ 100\%$$

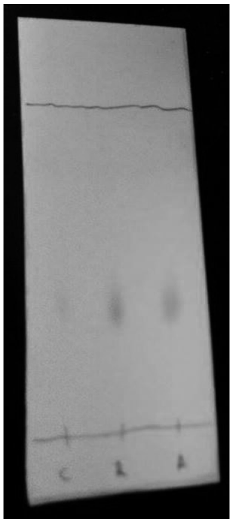
$$= 1.27\%$$

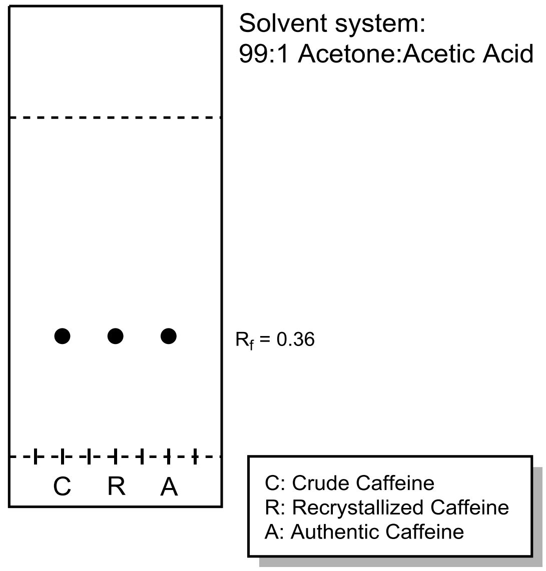
$$R_f = \frac{Height\ of\ Spot}{Height\ of\ Solvent\ Front}$$

$$= \frac{1.6\ cm}{4.5\ cm}$$

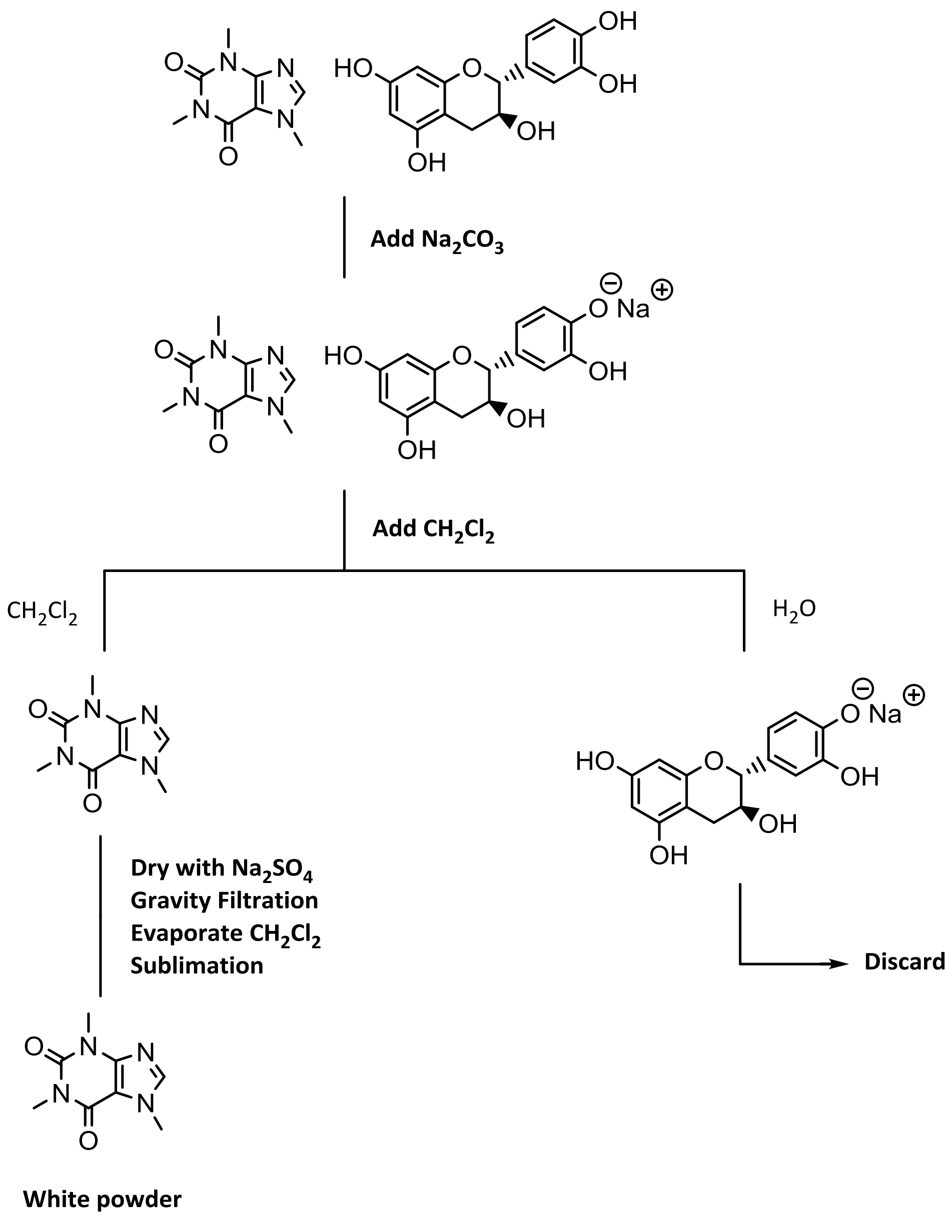
$$= 0.36$$

TLC Plate




Solvent system:
99:1 Acetone:Acetic Acid
R_f = 0.36
C R A
C: Crude Caffeine
R: Recrystallized Caffeine
A: Authentic Caffeine

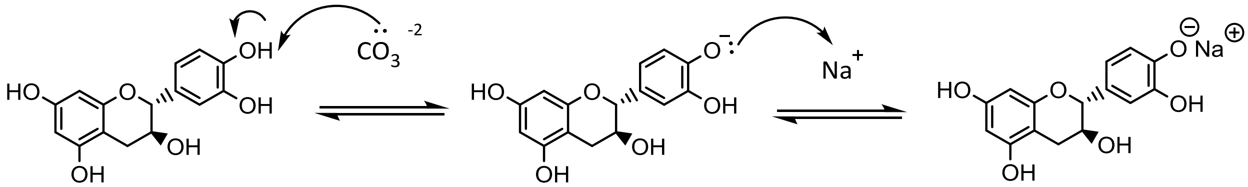
Flow Chart



Add Na_2CO_3
Add CH_2Cl_2
CH_2Cl_2
H_2O
Dry with Na_2SO_4
Gravity Filtration
Evaporate CH_2Cl_2
Sublimation
Discard
White powder

DISCUSSION

Simply doing a liquid-liquid extraction will not isolate caffeine. Tea contains many other organic compounds which would dissolve in the organic phase along with caffeine, leading to many impurities. This is remedied through the addition of Na_2CO_3, a base, into the steeped tea. It converts these other unwanted organic compounds in the tea, such as the weakly acidic tannins, into ionic salts through an acid-base reaction. The sodium salts formed are highly soluble in water as they are very polar, hence are insoluble in the organic solvent dichloromethane. Thus, during the extraction, these impurities will be dissolved in the aqueous phase and the organic phase that is extracted should only contain caffeine.



After extracting the organic phase, Na_2SO_4 is added as a drying agent. While the two liquids are immiscible, the organic phase is still capable of dissolving some water, especially when the separatory funnel is shaken. Furthermore, some of the aqueous phase will be extracted simply due to human error. Hence it is important to add a drying agent which will remove any trace amounts of water present. Because of this, sublimation is done for further purification of the caffeine crude product. To remove the hydrated salts, the organic solution was gravity filtered.

The experiment had a significantly low yield of approximately 1.27%. There are many factors that would contribute to this. While brewing the tea, of course not all the caffeine from the tea bags would be released. Some yield will also be lost from the tea bags itself, despite our attempts at drying them after brewing.

During the extraction, it may have been favourable to wash the aqueous solution with more dichloromethane to ensure the maximum amount of caffeine would be collected in the organic phase. Unfortunately, caffeine is also somewhat soluble in water, so it is very likely that some would remain in the aqueous phase. Prior to sublimation, the caffeine crude product had to be collected upon evaporating the organic solvent. This was very difficult, as it was contained in an Erlenmeyer flask, and the crude product was stuck to the bottom of the flask. It was clear that a significant portion of the product was lost as it simply could not be easily scraped out and retrieved.

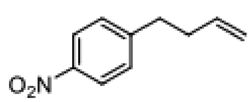
During sublimation, some yield may have also been lost as the caffeine vapour could possibly escape through the side arm flask, or for caffeine that did not sublimate and remained at the bottom of the flask. Furthermore, it is possible for the caffeine to decompose during this process, or react to create other products, due to the addition of heat, reducing the final yield.

Despite the low yield, through the TLC analysis, it was shown that our purified product was in fact caffeine, with the same R_f value of 0.36 as the reference, being authentic caffeine. This shows that the purified product had little impurities. Surprisingly, the crude caffeine product also had the same R_f value as the authentic caffeine. Essentially, this says that our crude product was actually very pure, as impurities should have resulted in a noticeable difference in polarity, and thus a different R_f value. Since the crude product was quite pure, it is possible that a significant portion of our yield was lost during sublimation through the potential issues stated above. This is a plausible explanation since a relatively significant amount of the crude product was lost during sublimation as the mass dropped below half, from 0.0086 g to 0.0032 g, yet the R_f value was identical to authentic caffeine. However, even if this was the case, the percent yield would still have been incredibly low.

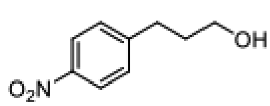
QUESTIONS

1. The addition of NaCl solution helps to break an emulsion in 2 ways. First of all, adding NaCl will increase the ionic strength of the aqueous layer, making it even more immiscible to the organic phase. Secondly, adding NaCl will increase the density of the aqueous phase. As the aqueous phase is the top layer in this case, it will further separate from the less dense organic phase.

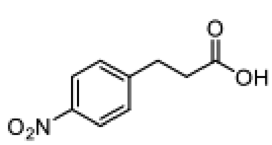
2. Catechin would have reacted with Na_2CO_3 to become an ionic salt, hence it would dissolve in the aqueous phase. After following the same steps up to extracting the organic phase, it would isolate it from caffeine and only the aqueous phase would remain in the separatory funnel. Add an acid, such as HCl, to the aqueous phase to protonate the catechin ionic salt back into catechin, as well as an organic solvent such as dichloromethane for catechin to be dissolved. Extract the organic phase, and add the drying agent Na_2SO_4 to the organic phase to remove any traces of water. Gravity filter to remove the drying agent, then evaporate the organic solvent to retrieve the catechin crude product.

3. 

Highest R_f: least polar compound, the alkene group lacks any capabilities to hydrogen bond or have dipole-dipole interactions with the silica gel, hence it travels up the TLC plate faster

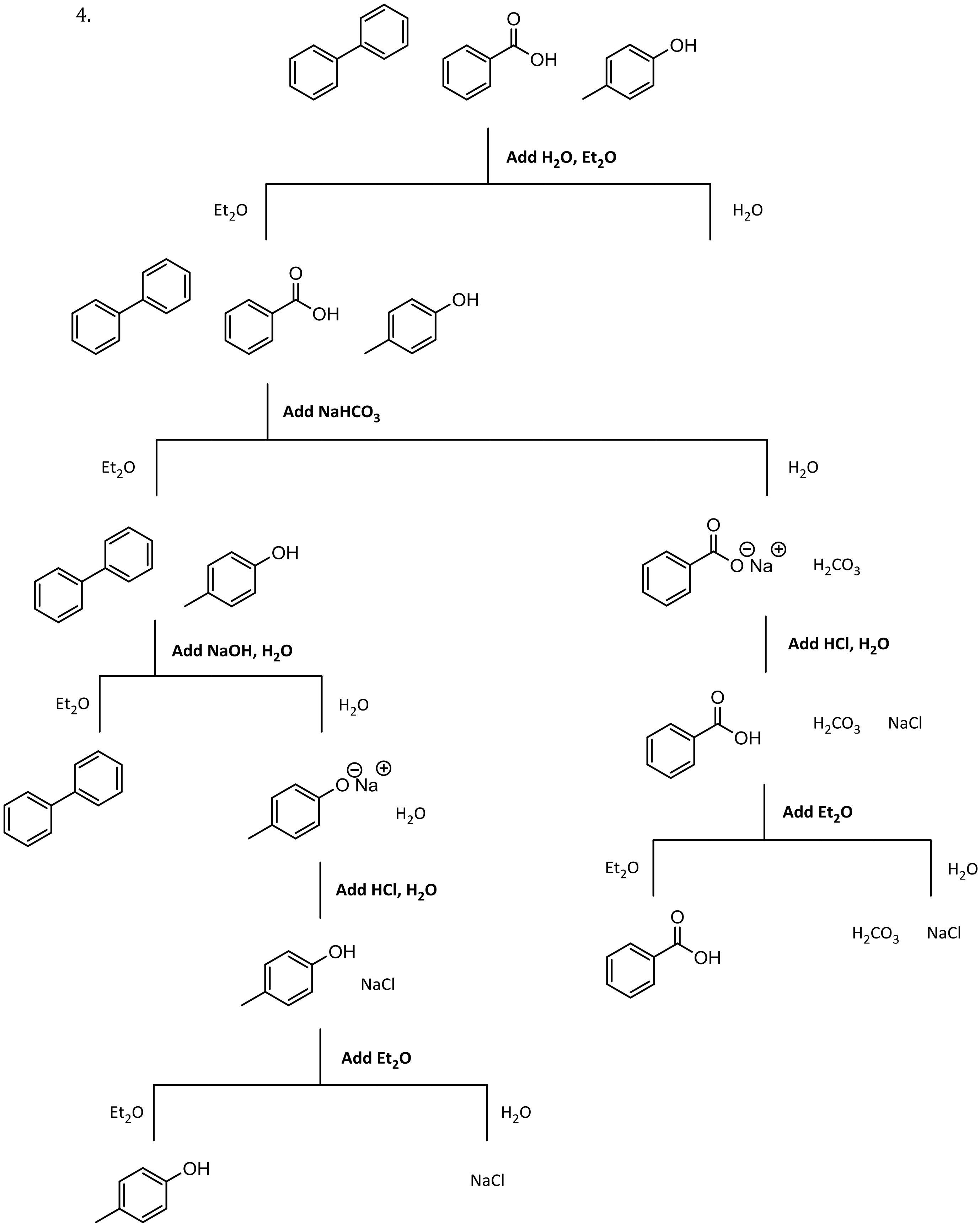


Alcohol group has fewer capabilities to hydrogen bond, and overall weaker dipole-dipole interactions than a carbonyl group. Hence it is more polar than above, but not the most polar compound, and will have an R_f in between these compounds.



Lowest R_f: most polar compound, the carbonyl group is best able to hydrogen bond with the polar silica gel with strong dipole-dipole interactions. Hence this compound will have the lowest R_f as it will travel up the TLC the slowest.

4.


OH
Add H2O, Et2O
Et2O
H2O
Add NaHCO3
NaOH, H2O
Add NaOH, H2O
O Na
H2CO3
Add HCl, H2O
NaCl
Add Et2O

5. Yes, placing the cold finger too far away from the base of the flask will affect his yield. Upon heating, the caffeine may decompose before it can even condense onto the cold finger and deposit as crystals. This will potentially lower the yield of the experiment. This is why it is important to have the cold finger roughly 1 cm away from the base – too close and the cold finger may be contaminated with impurities from contact or splatter.