Lab #1 – Separation and Purification of Caffeine

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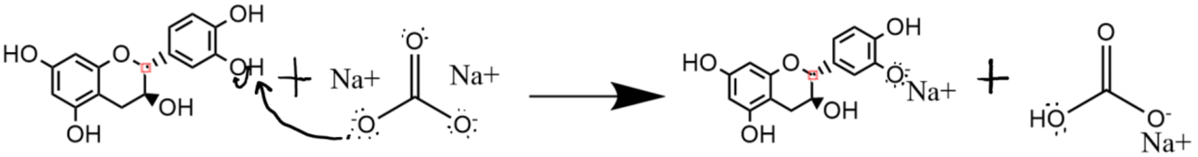
CHM2123 C

Introduction

Isolating and purifying a compound is an important skill to have in an organic chemistry laboratory. To effectively perform this, extraction, thin layer chromatography and sublimation techniques can be used.

The goal of this experiment is to isolate and purify caffeine from steeped tea. In a cup of steeped tea, caffeine and tannins like catechin are dissolved in the water, however plant materials are not. Both catechin and caffeine are soluble in the dichloromethane. To transform catechin into a compound that will instead be soluble only in the aqueous phase Na2CO3 can be added to the solution. A deprotonation reaction takes place and the catechin becomes an ionic salt. If caffeine and the deprotonated catechin are in different immiscible phases, the bottom organic phase containing caffeine can be extracted using an extraction funnel.

Figure 1: Deprotonation of catechin



Even after an extraction of the organic phase, there can still be traces of water in a solution, a drying agent like Na2SO4 can be added to remove the water. The solid can then be filtered out. The solvent, dichloromethane can then be evaporated, with crude caffeine left behind. To purify the extracted caffeine, sublimation with a cold finger can be done. Sublimation is when a solid goes directly into the gas phase. Caffeine can be sublimated if it is heated in a low pressure environment. The caffeine in gas phase can then condense back into solid form on a very cold test tube filled with dry ice called a cold finger.

To verify the purity of the isolated product, thin layer chromatography (TLC) and melting point tests can be done. Chromatography is a technique used to separate the components of a mixture based on their polarity. A mobile phase (99:1 or 80:20 acetone:acetic acid) carries the spotted materials including crude caffeine, sublimed caffeine and a caffeine reference solution up a silica gel strip. Once a strip has developed, it can be viewed using ultra-violet light. The less a spot travels up the silica gel strip, the more polar it is because it interacts with the polar silica gel. Rf values or retention factor values can be assigned to the spots and the components of the substances compared. Comparing the theoretical melting point of a compound with it’s experimental melting range, it is possible to gauge purity. The theoretical melting point of caffeine is 238. A pure product's melting point range is often experimentally less than 2 °C. The range will broaden with the presence of any impurities due to the deformation of the crystalline lattice belonging to the compound. Often, when impurities are present, less energy will be required to break the intermolecular interactions in the solid lattice.

Table of Reagents

Reagent	Mol mass (g/mol)	quantity	Density (g/mL)	Mmol	equivalents	Melting point (°C)
Na_2CO_3	105.99	2.0g	n/a	18.87	53	851
dichloromethane	84.93	45 mL	1.325		n/a	n/a
Na_2SO_4	142.04	One scoop	n/a	n/a		884
caffeine	194.19	n/a	n/a	n/a		238
Catechin	290.27	n/a	n/a	n/a		175-177

Experimental Procedure

Refer to CHM2123 lab manual, pages 6-8

Note: Data for melting point and TLC was obtained from two other groups

Melting point: Group with Tara Snyder and Carlas Barbery

TLC: Group with Ammar Salvini and Dylan Vincent

Observations

Key step	Observation
Steep two tea bags in 60 mL boiling water for five minutes	Solution becomes brown and slightly "milky" looking
Add Na_2CO_3 to steeped tea	Solution becomes no longer milky looking, (less cloudy) Solution is still brown
Extract the organic phase using separatory funnel by adding dichloromethane to solution	-Top layer is dark brown -Bottom layer is transparent with no colour -Between the two layers is a layer with large clear droplets -As the funnel was vented some of the brown layer got pushed out of the hole which meant that later on there were small brown droplets in the clear extracted organic layer
Add scoop of Na_2SO_4	-Salt clumps into bigger chunks at bottom of beaker
Once solids have been filtered out boil off solvent	-White solid forms at bottom of beaker
Sublimate the caffeine onto the cold finger	-the suction was too strong therefore sublimated white solid deposited on the entrance to the side arm and in the second flask -ice, along with a small amount of the white solid, formed on the cold finger

Table of Results

Compound	Mol. Mass (g/mol)	Quantity (g)	Mmol	% Yield	Melting Point °C)
Caffeine	194.19	0.01	0.05	5.23	237.5-239.4

<u>Calculations</u>

Mass of Tea in two bags = 4.78g
(Assume tea contained 4% caffeine for theoretical value)
Theoretical mass of purified caffeine = 4.78g x 0.04 = 0.1912g
Experimental mass of purified caffeine = 0.01g

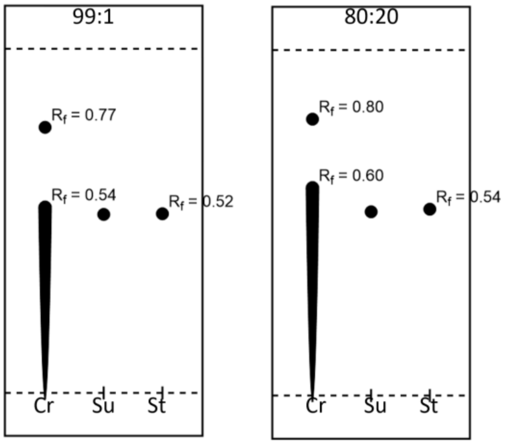
$$\% \text{ Yield} = \left(\frac{\text{Experimental mass of purified caffeine}}{\text{Theoretical mass of purified caffeine}} \right) \text{ x } 100\%$$

$$= \left(\frac{0.01g}{0.1912g} \right) \text{ x } 100\%$$

$$= 5.2301255\%$$

$$\approx 5.23\ \%$$

<u>TLC plates</u>



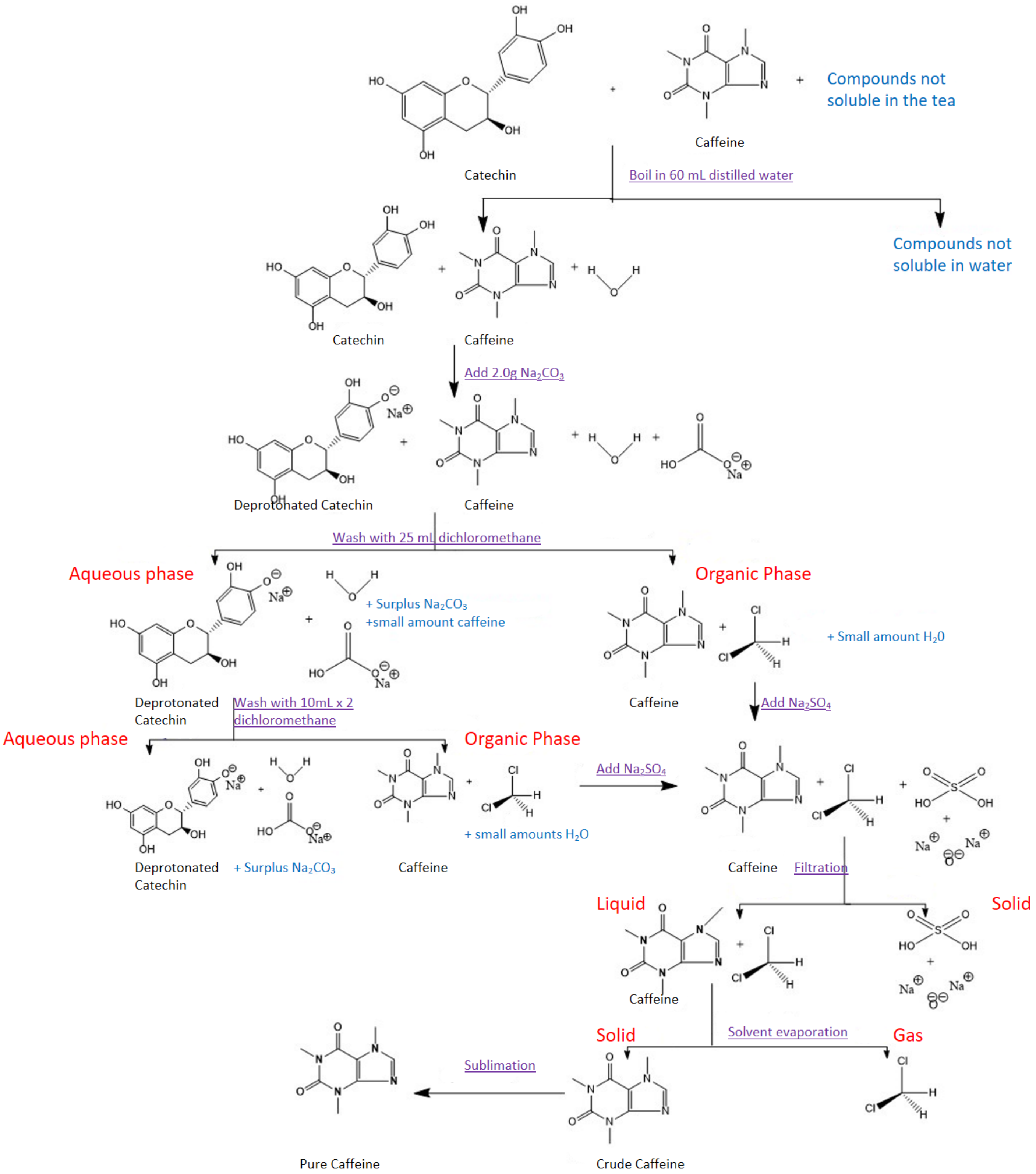
Cr: Crude Caffeine dissolved in DCM

Su: Sublimated product dissolved in..

St: Caffeine reference solution

Both mobile phases are acetone:acetic acid but one is 99:1 and the other is 80:20

Flow Chart



Catechin
Caffeine
+ Compounds not soluble in the tea
Boil in 60 mL distilled water
Compounds not soluble in water
Catechin
Caffeine
Add 2.0g Na_2CO_3
Deprotonated Catechin
Caffeine
Wash with 25 mL dichloromethane
Aqueous phase
+ Surplus Na_2CO_3
+small amount caffeine
Deprotonated Catechin
Organic Phase
+ Small amount H_20
Caffeine
Add Na_2SO_4
Wash with 10mL x 2 dichloromethane
Aqueous phase
Organic Phase
Deprotonated Catechin
+ Surplus Na_2CO_3
Caffeine
+ small amounts H_2O
Add Na_2SO_4
Caffeine
Filtration
Liquid
Caffeine
Solid
Solvent evaporation
Solid
Gas
Sublimation
Pure Caffeine
Crude Caffeine

Discussion

The goal of this experiment was to isolate and purify caffeine from steeped tea. Na_2CO_3 was added to the solution to convert catechin into an ionic salt so it would be soluble in water. Dichloromethane was added and caffeine was able to dissolve in it. The clear organic layer containing caffeine was extracted using an extraction funnel. Na_2So_4 was added to the organic layer to remove any traces of water, the hydrated solid was filtered off. Then, the dichloromethane was boiled off until crude caffeine solid remained. The crude caffeine heated and sublimated in a low-pressure environment and condensed. The product did not condense on the cold finger but rather other parts of the apparatus due to suction error. Due to lost product, the % yield was calculated to be 5.23%.

The TLC results from the group of Dylan Vincent and Ammar Salvini, as well as the melting point results from the group of Tara Snyder and Carlas Barbery confirmed that the isolated caffeine was indeed pure. The Rf values for the sublimated product was the same as that of the reference solution in both the mobile phases (99:1 and 80:20 acetone:acetic acid). This shows that the sublimated product was pure. Acetic acid is more polar than acetone which is why the 80:20 solvent system gave higher Rf values. The Rf of caffeine was 0.52 in the 99:1 system and 0.54 in the 80:20 system. The experimental melting range was determined to be 237.5-239.4 °C. The theoretical melting point was 238 °C and the experimental range was less than 2°C so it can be determined that the product was pure.

Sources of error:

The suction technique is not consistent enough to give an ideal pressure. In fact, a significant error was that the suction was too strong which resulted in the condensed product appearing at the entrance to the side-arm and in the second flask. It was not possible to remove all the condensed solid from the apparatus therefore the measured mass of the product was significantly lower than the actual mass of the product. This resulted in a very low yield.

The hot plate is also not a consistent enough technique to provide an ideal temperature at which to sublimate. The solid at the bottom of the sublimation flask became dark in colour. It is possible it got heated too much. This could have reduced the yield.

Caffeine is partially soluble in water and a small amount may have been lost to the aqueous phase during extraction. Using more DCM to wash could help with the end yield.

There may have been water present in the sublimation flask because ice formed on the cold finger. The flask and test tube could have been put in a drying oven beforehand to remove any water and then cooled before use. Water may have dissolved part of the purified caffeine.

During the filtering of the hydrated Na2SO4, some of the liquid containing caffeine may have been absorbed by the filter paper. The paper could have been saturated with DCM before use and washes with DCM could have been done to improve yield.

A large smudge was seen in the Crude Caffeine lane of both TLCs, the spotted solution could be more diluted with dichloromethane to reduce this.

Works Cited

Magnan, F. Exp. XXX. In *CHM2123 Laboratory of Organic Chemistry Manual*, University of Ottawa: Ottawa, 2019; pp 1-8.