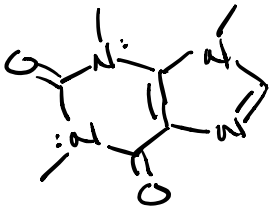
Extraction of Caffeine from Tea

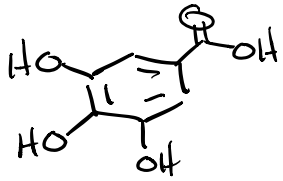


Caffeine

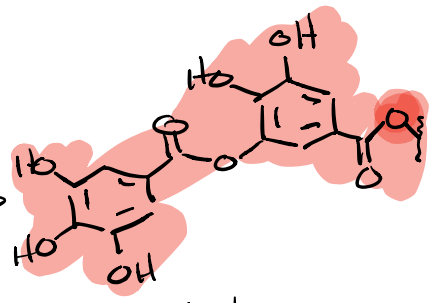
Mostly non-polar

Tannins

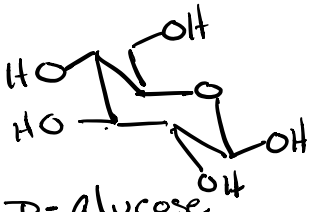
2



Gallic Acid

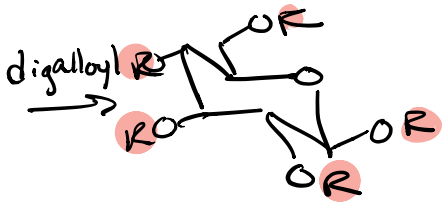


digalloyl group



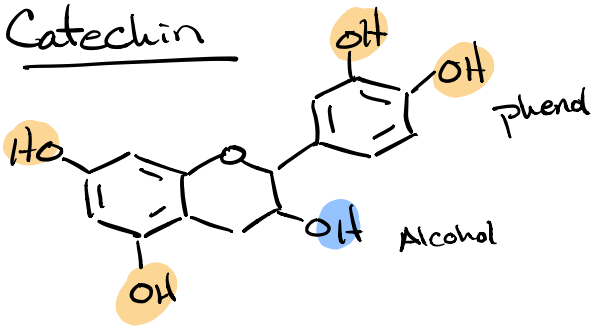
D-glucose

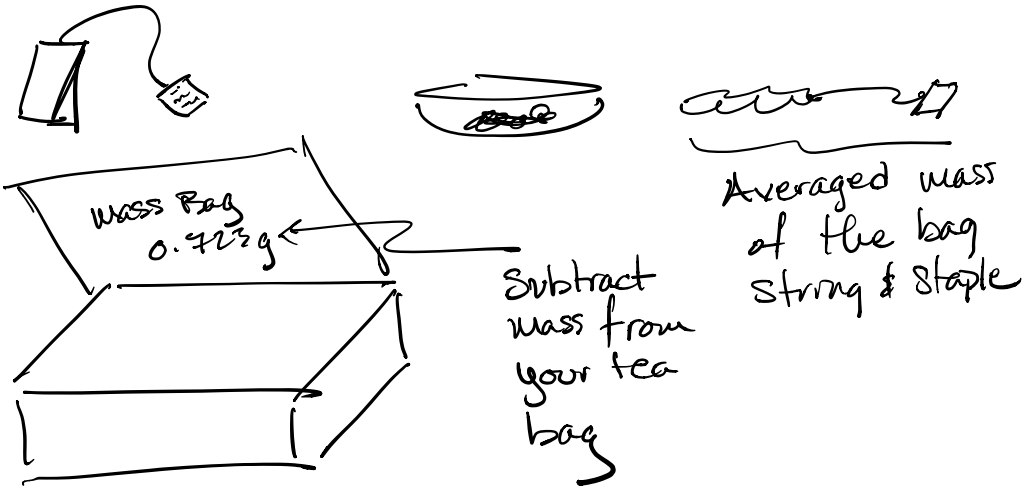
digalloyl

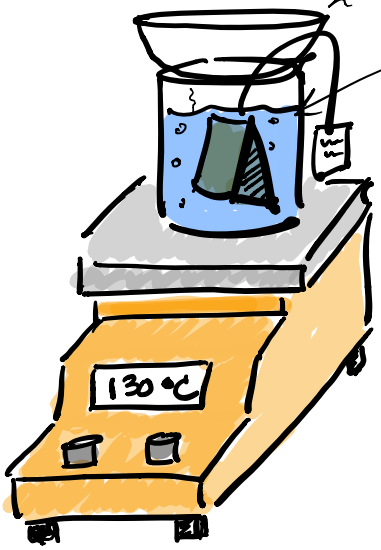


Tannin

Catechin





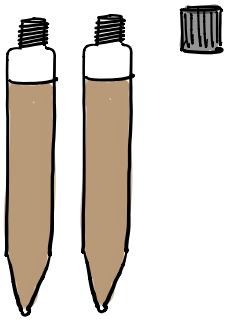


① watch glass

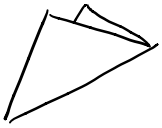
50-mL Beaker w/ 20 mL DI H_2O

boil for 15 min gently

Replacing H_2O as needed



Na_2CO_3



② Cool to room temp

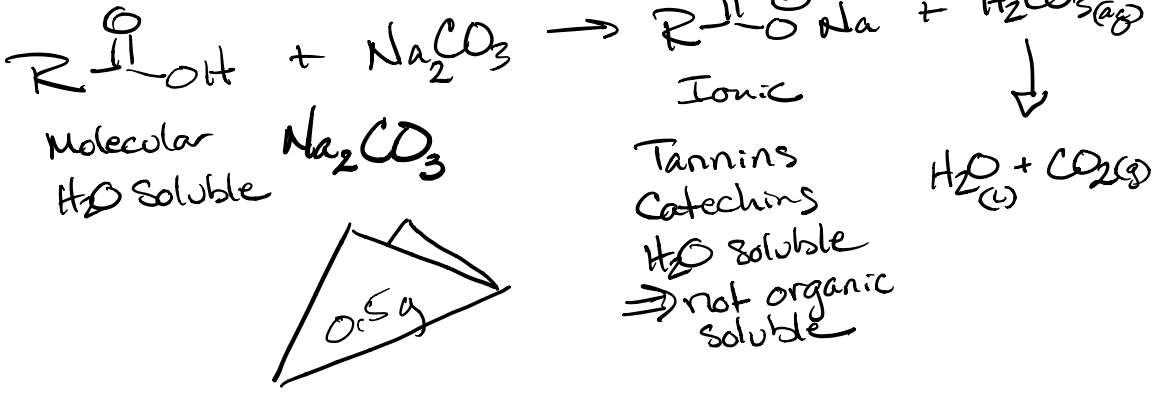
③ Split the extract between 2 centrifuge tubes

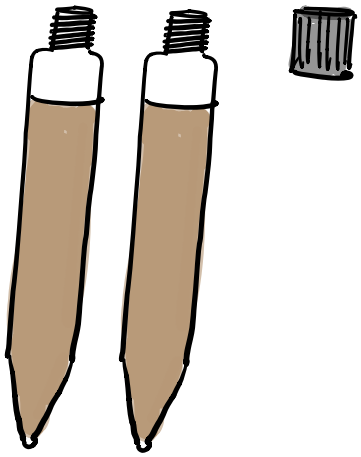
④ Rinse Teabag w/ 2mL DI H_2O and gently roll out using a test tube

Add additional extract to centrifuge tubes.

⑤ While still a little warm add 0.5g Na_2CO_3 to each centrifuge tube.

↑ Base

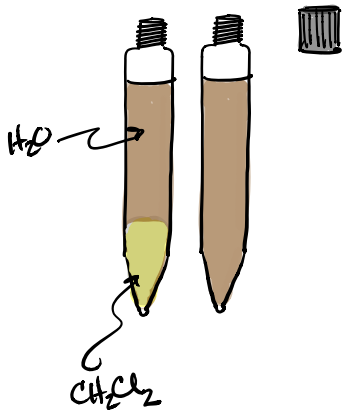




- Cap & dissolve Na_2CO_3

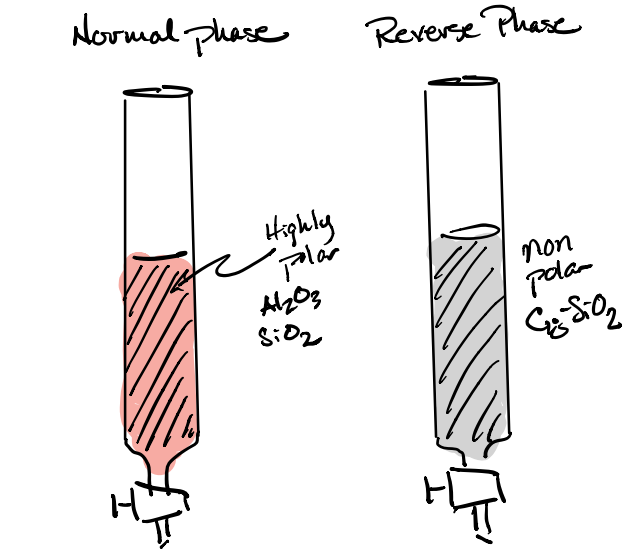
Traditional process

CH_2Cl_2 Extraction



Green Process

Reverse Phase Chromatography
Solid phase extraction



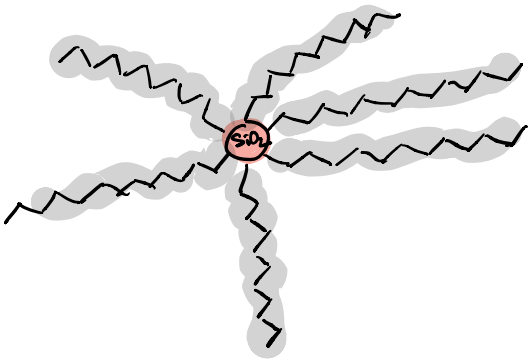
Elution order

Polar last out

non-Polar last out

non Polar 1st out

Polar 1st out

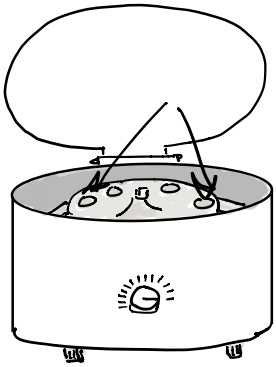


Traditional CH_2Cl_2 Extraction

After Step 5

6. Add 3 mL CH_2Cl_2 to each tube
7. Shake & vent ~ 30 seconds
   - Emulsion develops

8. Balance tubes for mass
   - weigh tubes w/ Lids & add H_2O to the lighter tube to match ± 0.1 g



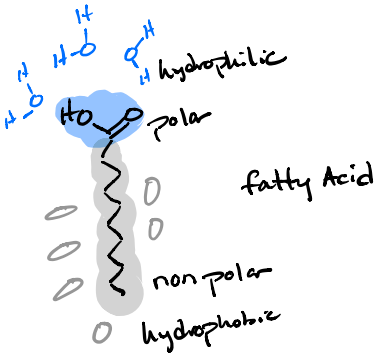
9. Centrifuge tubes for ~5 min
   Make sure tubes go across from each other

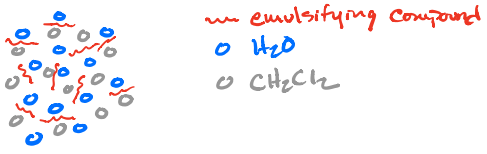
10. Extract CH_2Cl_2 layer & place in 25-mL Erlenmeyer flask

11. Add 3mL CH_2Cl_2 to each Centrifuge tube & repeat extraction & Centrifuge

12. 

13. Dry w/ $MgSO_4$





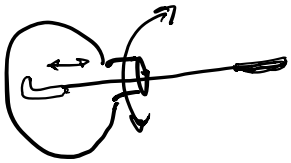
(14) Tare a RB fask 25-mL

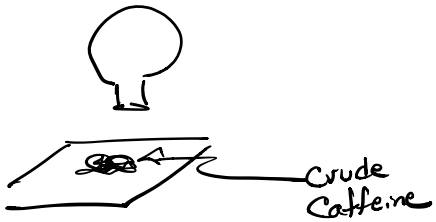
(15) decant CH_2Cl_2 into RB flask

(16) Rotovap

(17) Crude mass of Caffeine
Reweigh RB flask

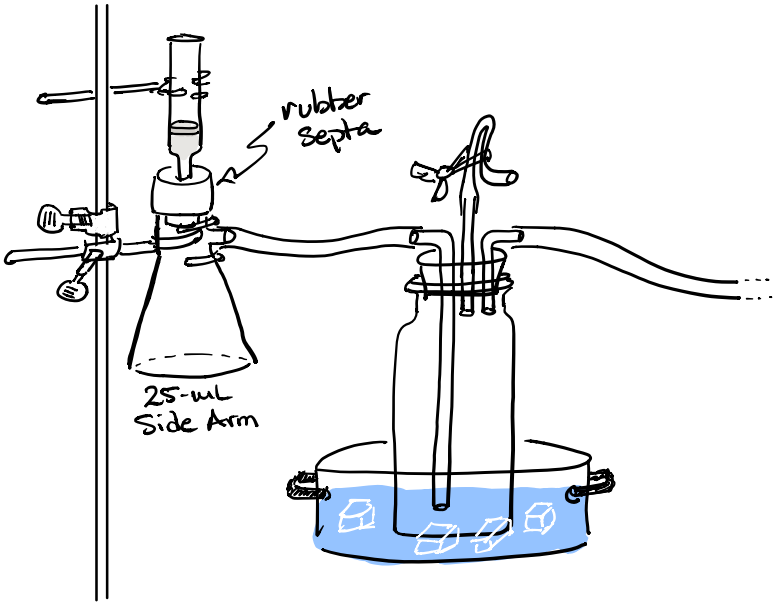
(18) Remove caffeine

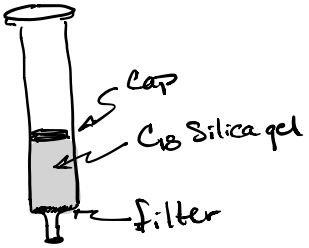




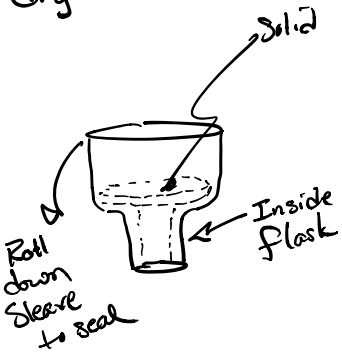
Solid Phase (SPE) Extraction

② Set up filtration apparatus





All pre made dry

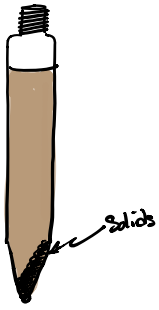


③ Condition Column

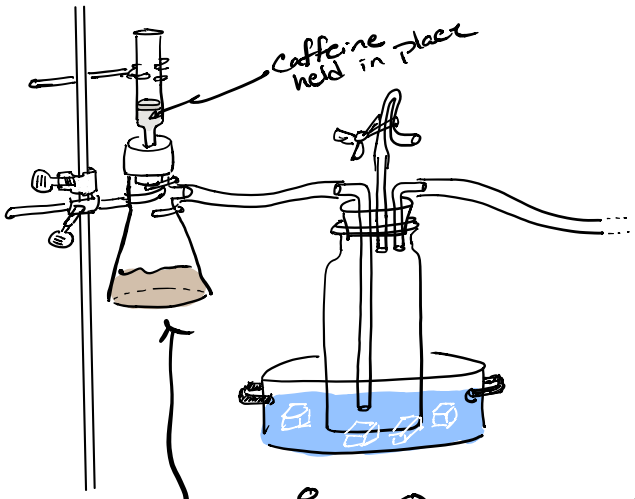
- Turn on vacuum
- Add 2mL MeOH
- Add 2mL DI H_2O

④ Without letting the column dry out filter both tea extracts - 1 mL at a time being careful not to add solids.

① * Tea Extract must be centrifuged before adding to SPE column to remove solids.



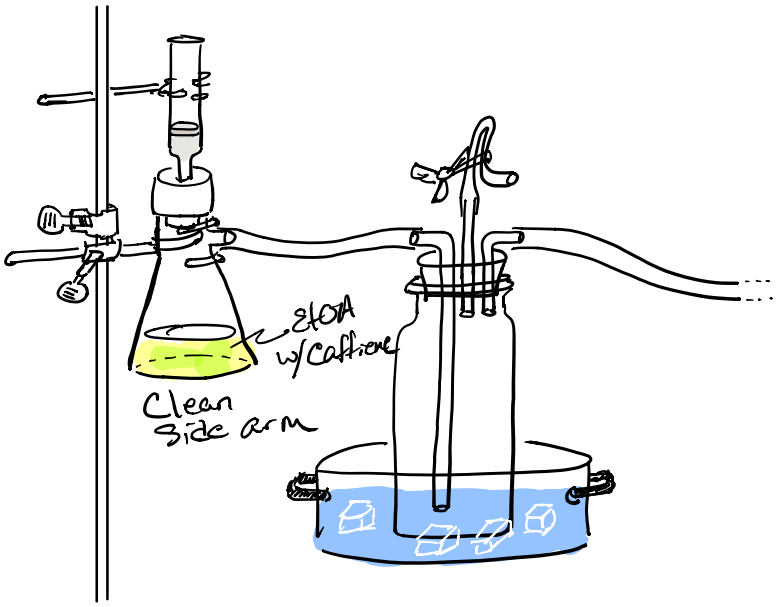
(5) - Disassymbol Side arm flask.
Haz out the liquid into aq waste.
- Clean flask & Reassymbol.



tannins
Catachins
H_2O
MeOH
Polar Compounds

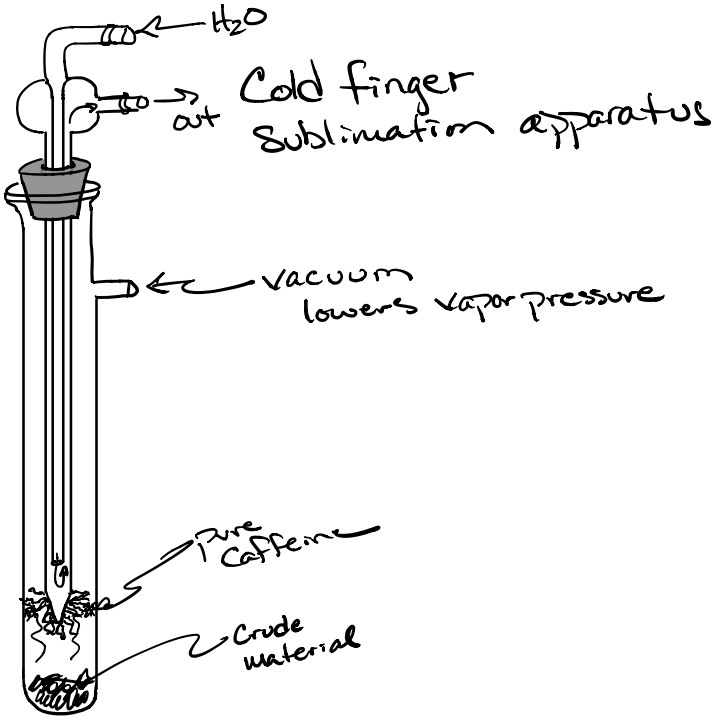
} aq waste

(6) Add 7mL Ethyl acetate to Column



7. Dry EtOAc w/ $MgSO_4$
8. Tare RB flask
9. Decant into RB
10. Rotovap
11. Reweigh & get Crude Mass Caffeine

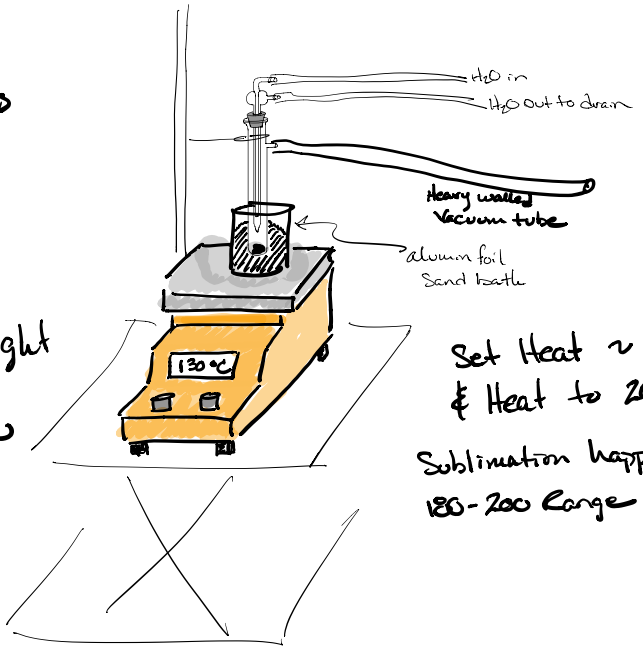
Sublimation of Caffeine



1. Heat & Sublime
2. Cool to RT
3. Extract Crystals
4. Wt
5. Mass & Calc
   % recovery
   from dry weight

$$= \frac{\text{mass caffiene}}{\text{mass tea}} \times 100$$

6. Solid FTIR

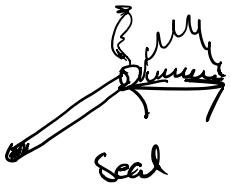


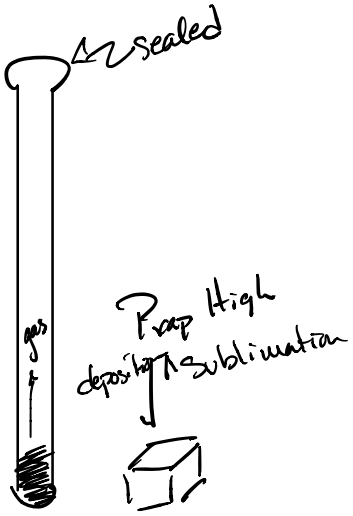
Set Heat ~ 75%
& Heat to 200°C

Sublimation happens
180-200 Range

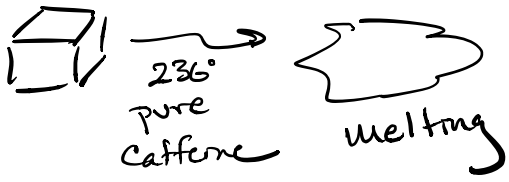
Caffeine(g) = P_{atm} ↓ Sublime by dropping P_{atm}

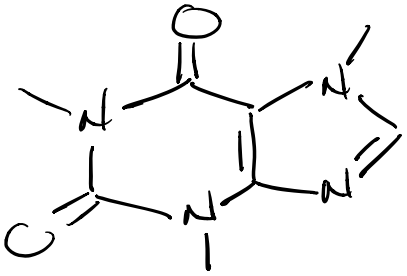
solid Caffeine(s)





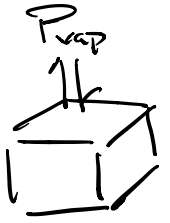
↑ Sublimation P_{vap} = P_{atm} on system ↑

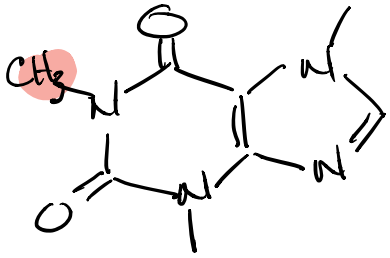




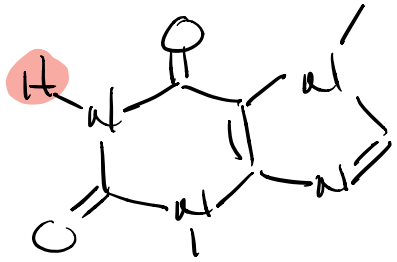
Aromatic
all sp^2

no H-bonding
dipole-dipole ⟷ weak
π-stacking





Caffeine



Theobromine
Chocholate

Coffee	20-30 mg	tea	4-20 mg
Instant Coffee	8-20 mg	Coke	3.75 mg
Espresso 2oz	50-70 mg		
Decaf	0.4-1 mg		

Leathal dose ~ 10g 80-100 $\mu g/mL$