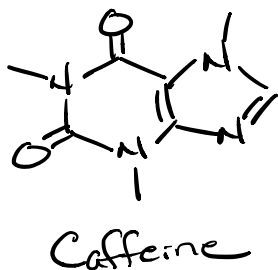
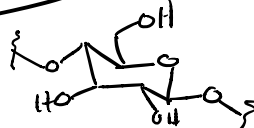


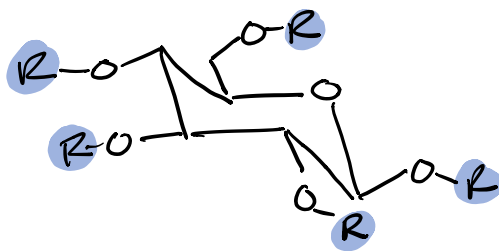
Isolation of Caffeine from Black Tea



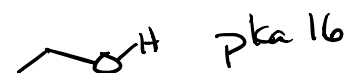
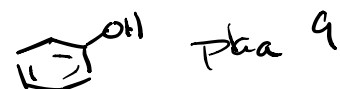
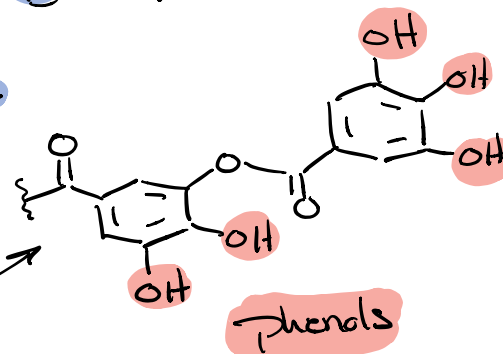
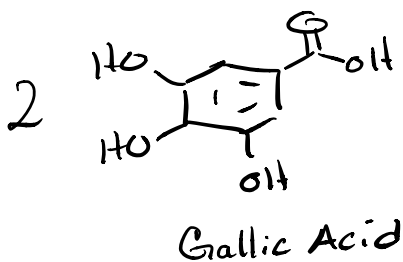
- Cellulose
- Pigments chlorophylls
- Tannins acidic polyhydroxyl compounds
- Catechins
- Caffeine



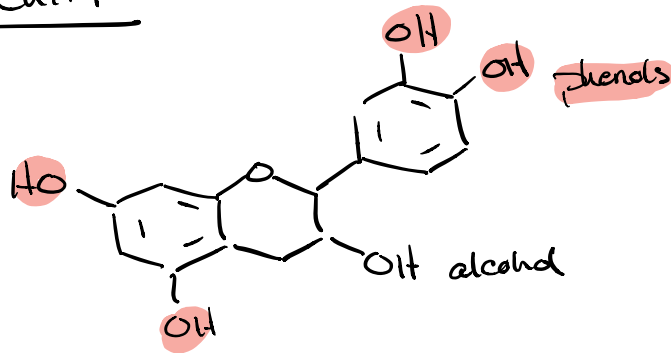
Tannins



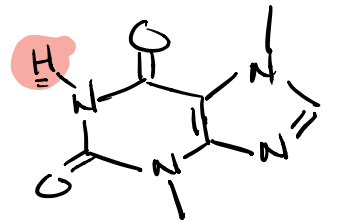
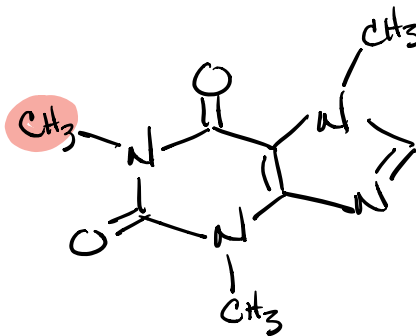
R = digallyl



Catechin



Caffeine



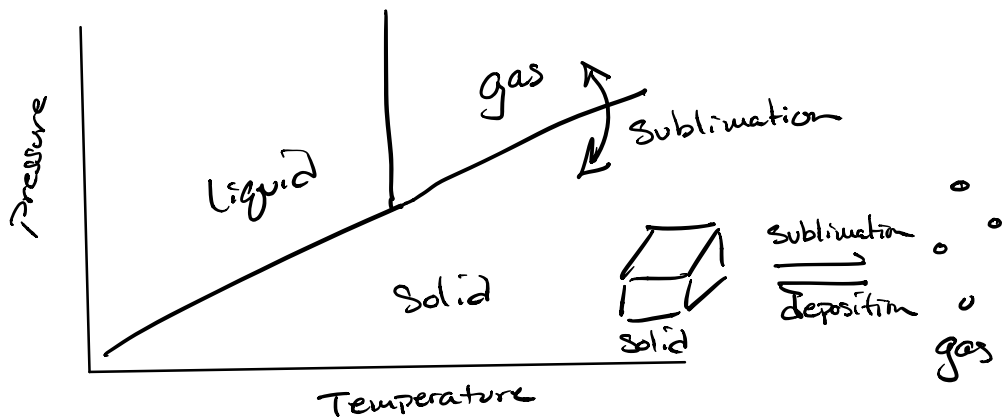
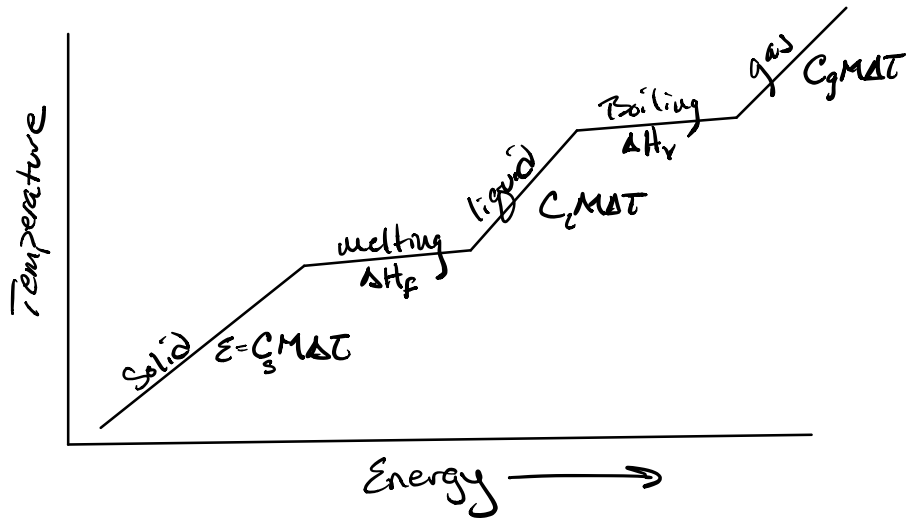
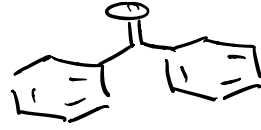
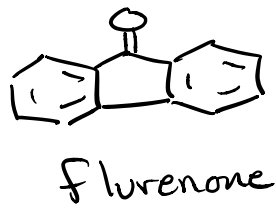
Theobromine
H-bonding

All sp^2
Aromatic
flat

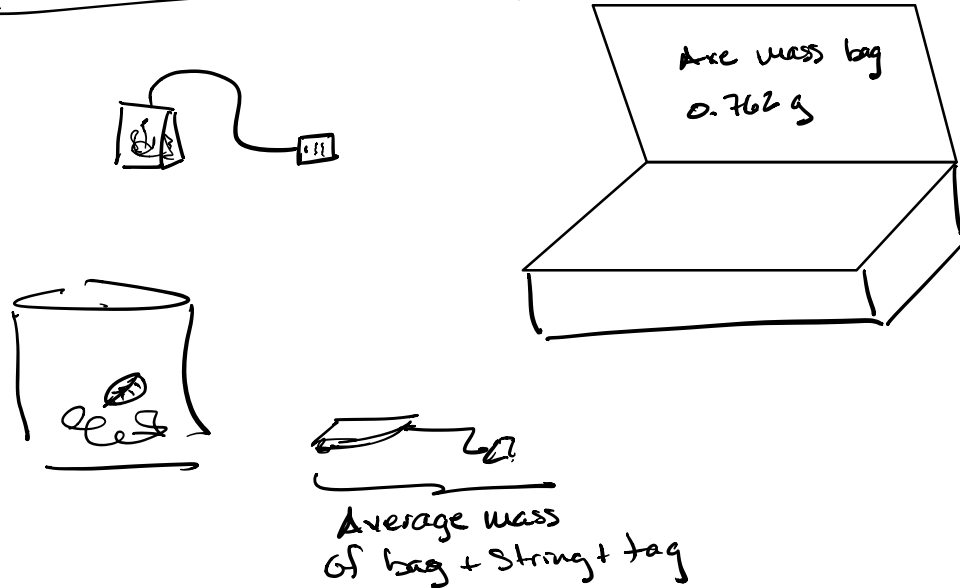
H₂O Soluble
CH₂Cl₂ Soluble

} Polar
dipole-dipole

Sublimes @ 180-200°C
100-200 torr



Extraction of Caffeine

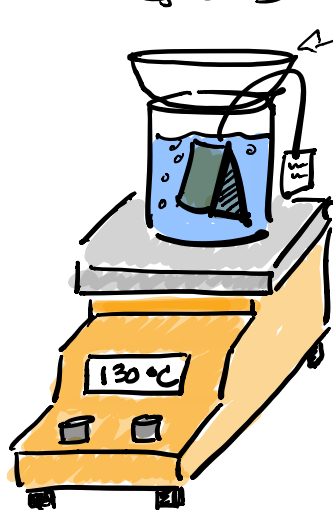


① Weigh tea bag

② Subtract Ave mass bag

→ Mass tea leaves ← Starting mass to base the % recovery on

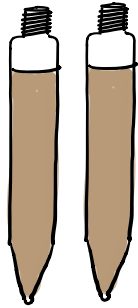
③ Boil tea gently in 20 mL DI for 15 min



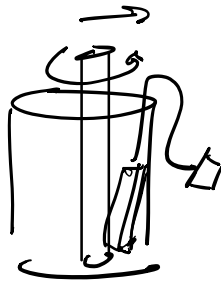
→ watch glass
- Replace H₂O as needed to maintain ~ 20 mL

④ Cool down so you can handle

⑤ divide tea in two centrifuge tubes

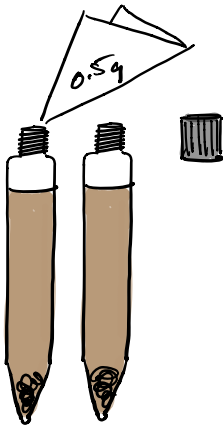


⑥ Rinse tea bag w/ 2ml DI & Squeeze out & Add to Centrifuge tubes.

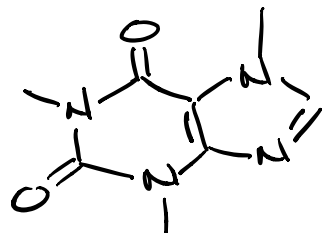
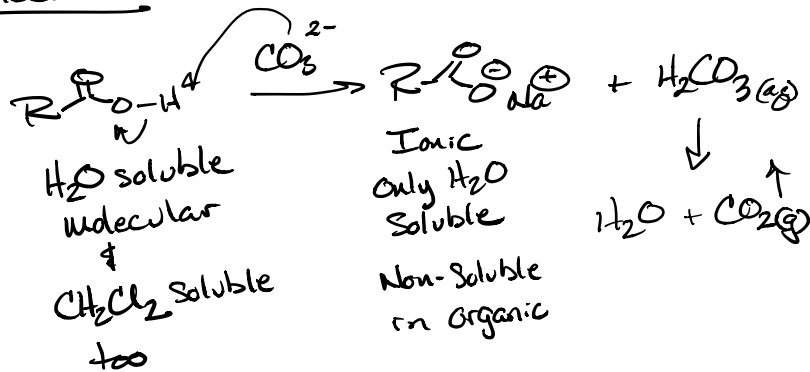


⑦ Add ~0.5g Na_2CO_3 to each centrifuge tube while warm. Cap, Shake, di.

Na_2CO_3



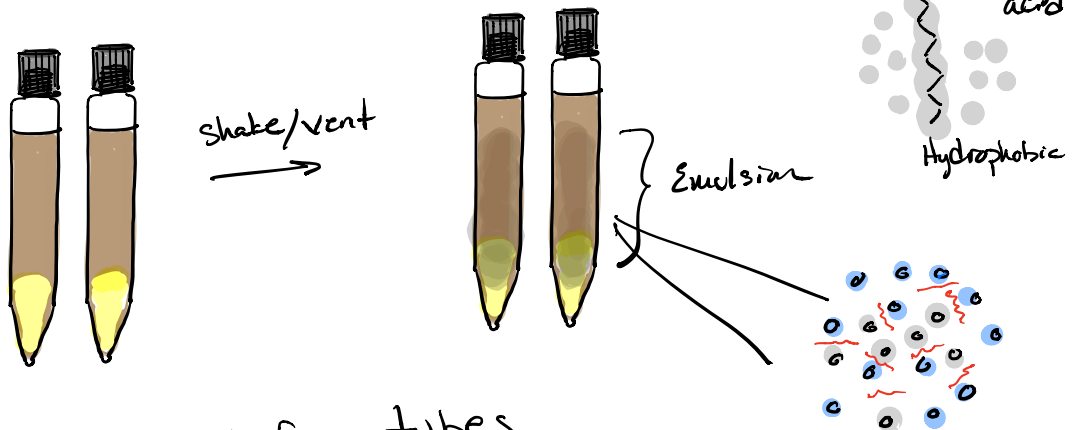
Tannins
Catechins



neither acid nor base

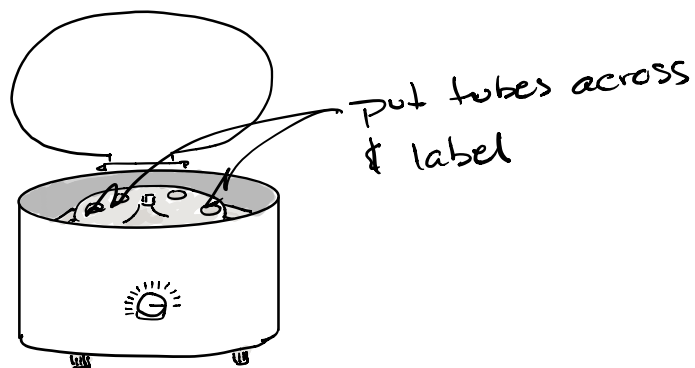
Methylene Chloride Extraction CH_2Cl_2

- ① Cool to room temp
- ② Add 2ml CH_2Cl_2 to each centrifuge tube
- ③ Cap / Shake / vent ~ 30 secs



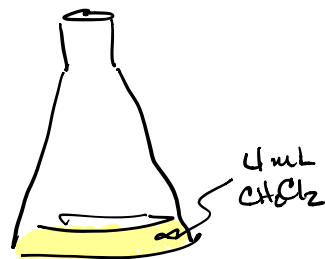
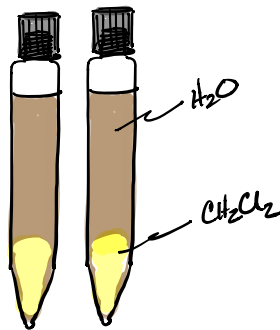
- ④ weigh centrifuge tubes & H_2O to the lighter tube to balance ± 0.1 g!

- ⑤ Centrifuge 5min



⑥ Extract lower organic layer & place in 25-ml Erlenmeyer flask

⑦ Repeat extraction (2-6) 2nd time w/ 2 new portions of CH_2Cl_2 (2 mL),



⑧ Dry CH_2Cl_2 (8 mL) with MgSO_4

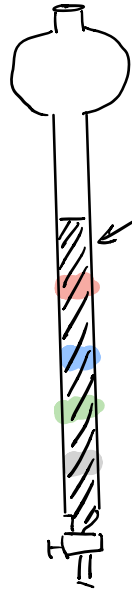
⑨ Tare 25-ml RB flask

⑩ decant CH_2Cl_2 into RB

⑪ Rotovap

⑫ Reweigh RB flask & find crude mass caffeine

Normal Chromatography



$$k = \frac{\sum \text{mobile}}{\sum \text{stationary}}$$

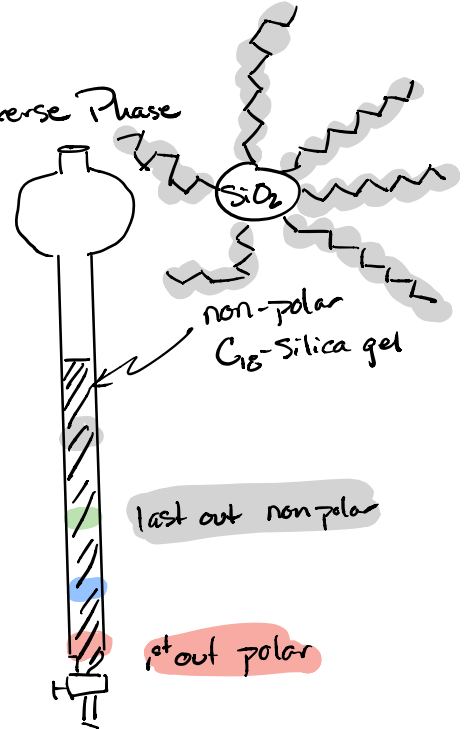
Silica gel SiO_2
Alumina Al_2O_3
Both Highly polar

last out polar compounds

↑ Increasing polarity

1st non-polar compounds

Reverse Phase

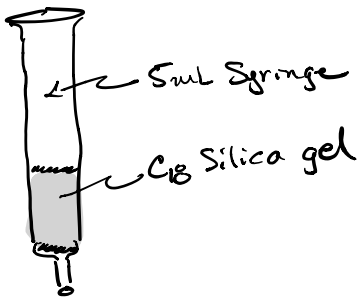


non-polar C_{18} -Silica gel

last out non-polar

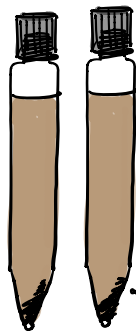
1st out polar

Solid Phase Extraction (SPE)



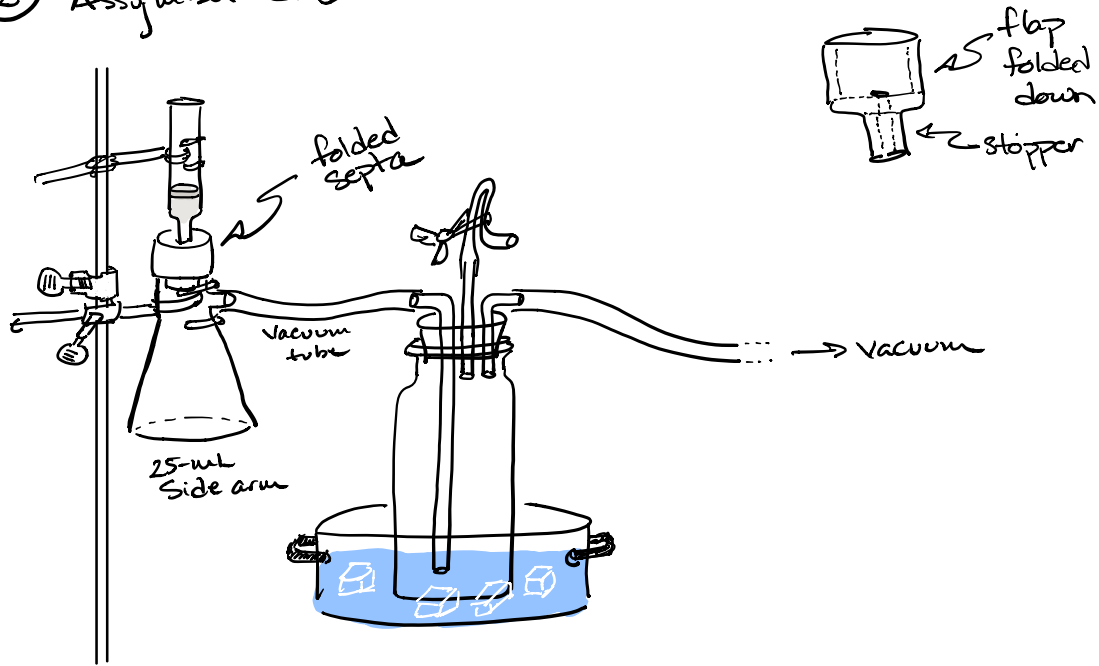
① Centrifuge the centrifuge tubes to pellet out solids

- Balance tubes $\pm 0.1g$
- Centrifuge 5-min



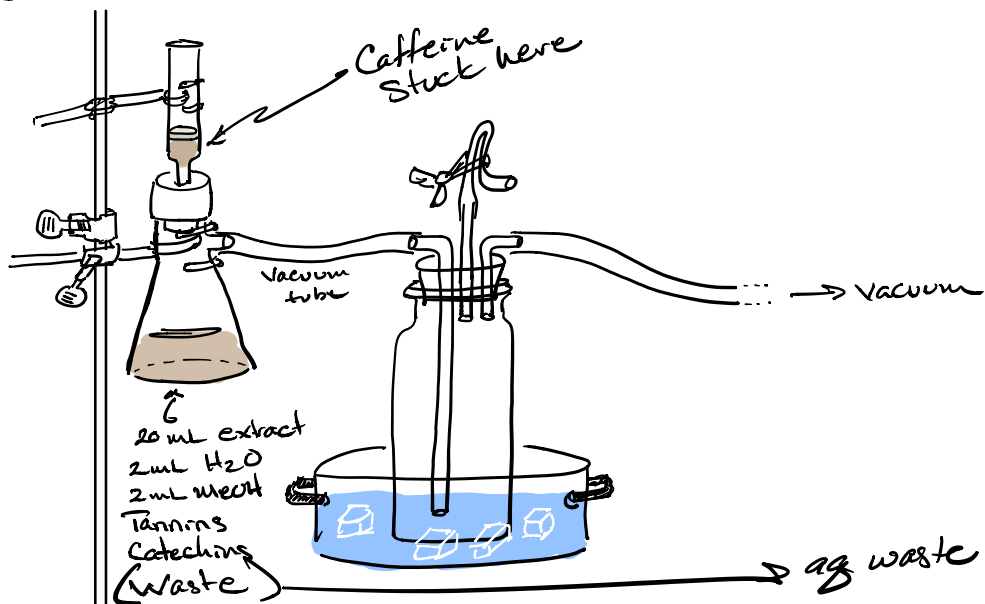
Solids
Cellulose

② Assemble SPE Column



- ③ Turn vacuum on
Add 2 mL of MeOH
Add 2 mL of DI H₂O

Quickly
Add 20 mL of tea extract in 1 mL portions

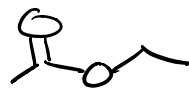


④ Dissassembled & hauled out as waste in side arm flask

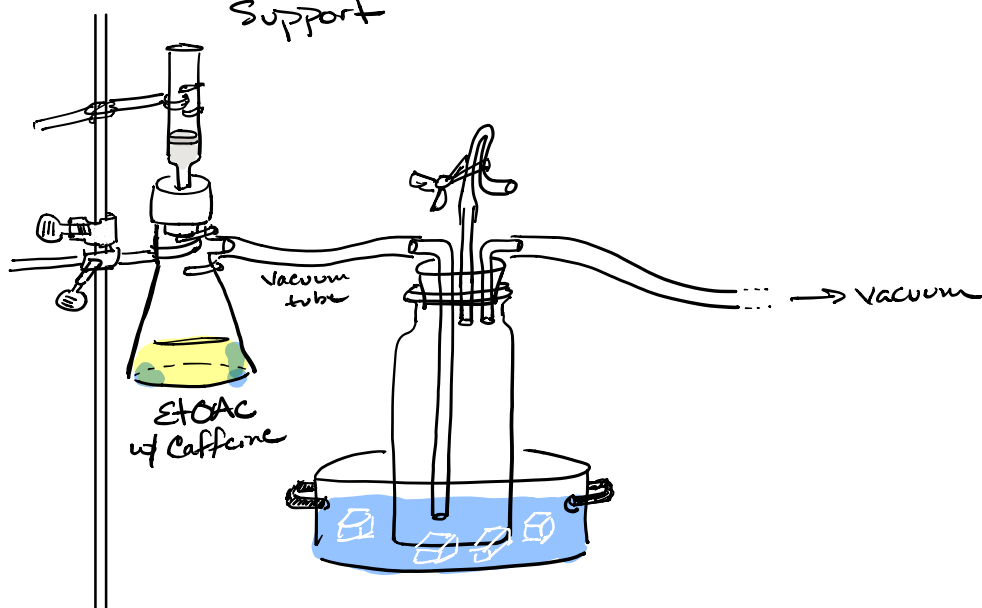
⑤ wash flask & re-assemble

⑥ Turn on vacuum

⑦ Add 7 mL Ethyl acetate



⇒ Release Caffeine from solid support



⑧ Dry EtOAc w/ $MgSO_4$

⑨ Tare RB

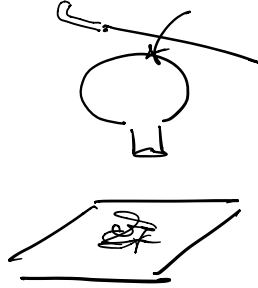
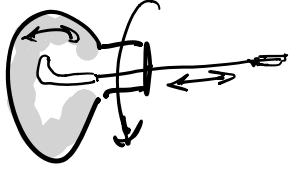
⑩ Decant in RB

⑪ Rotovap

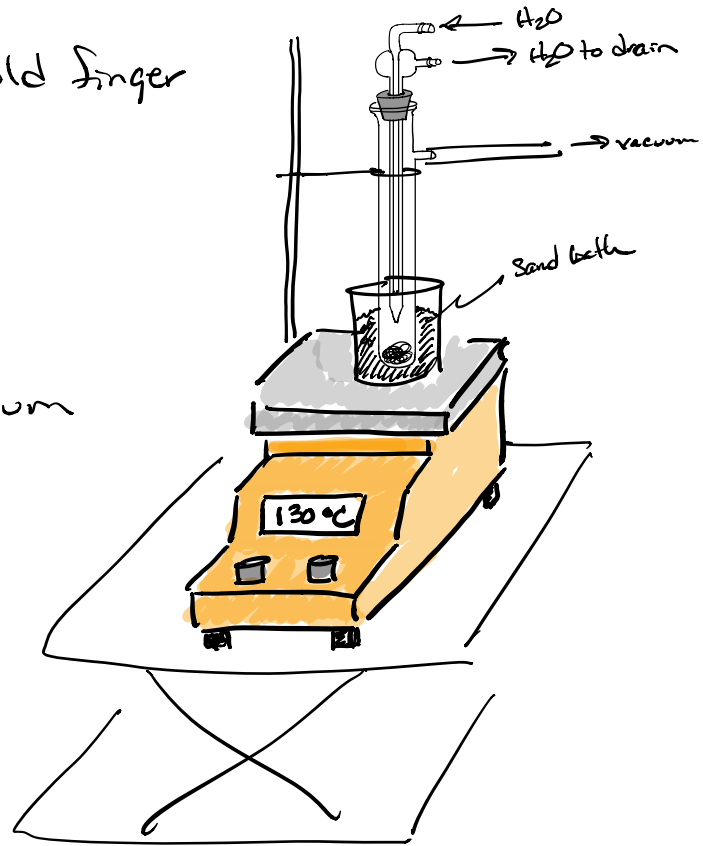
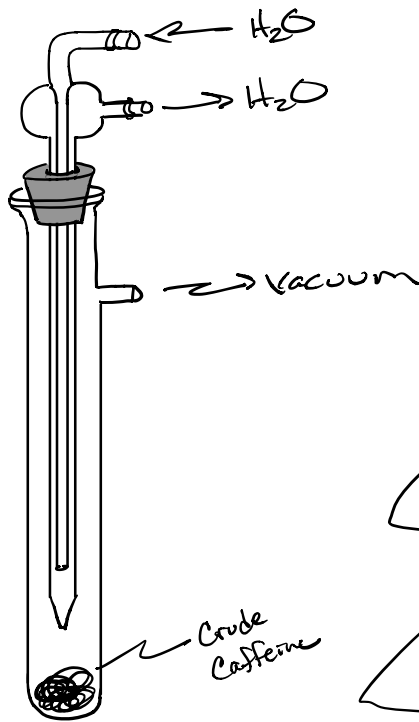
⑫ Reweigh & find mass of Crude Caffeine

Sublimation

① Remove crude Caffeine



② Set up Sublimation Cold Finger



③ Add Caffeine to Cold Finger

④ Turn on H₂O & Vacuum

⑤ Heat 180° - 200 °C

⑥ lower jackstand

⑦ cool to RT

⑧ Isolate
white
crystals

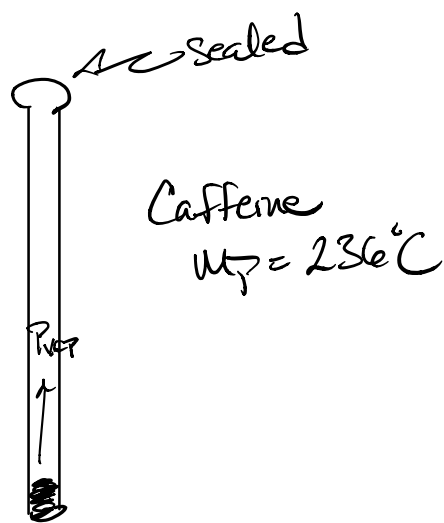
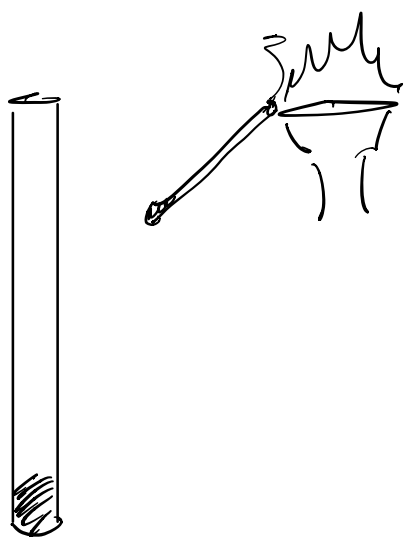
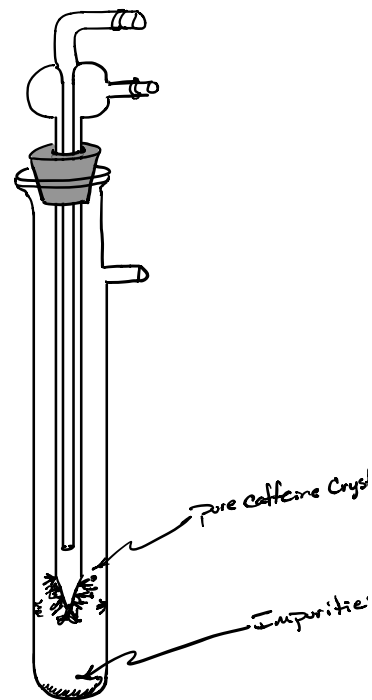
⑨ mass

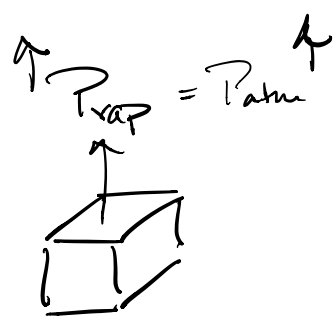
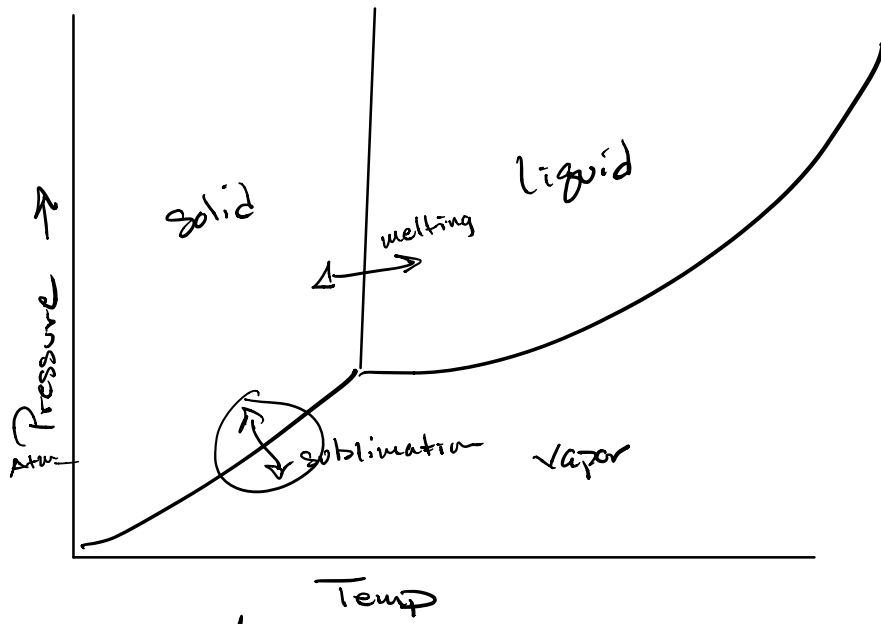
⑩ calc % recovery

$$\frac{\text{mass caffeine}}{\text{mass leaves}} \times 100\%$$

⑪ * Closed tube MP determination

⑫ KBr FTIR on Caffeine





Boiling Pt \uparrow $P_{vap} = P_{atm}$ \uparrow