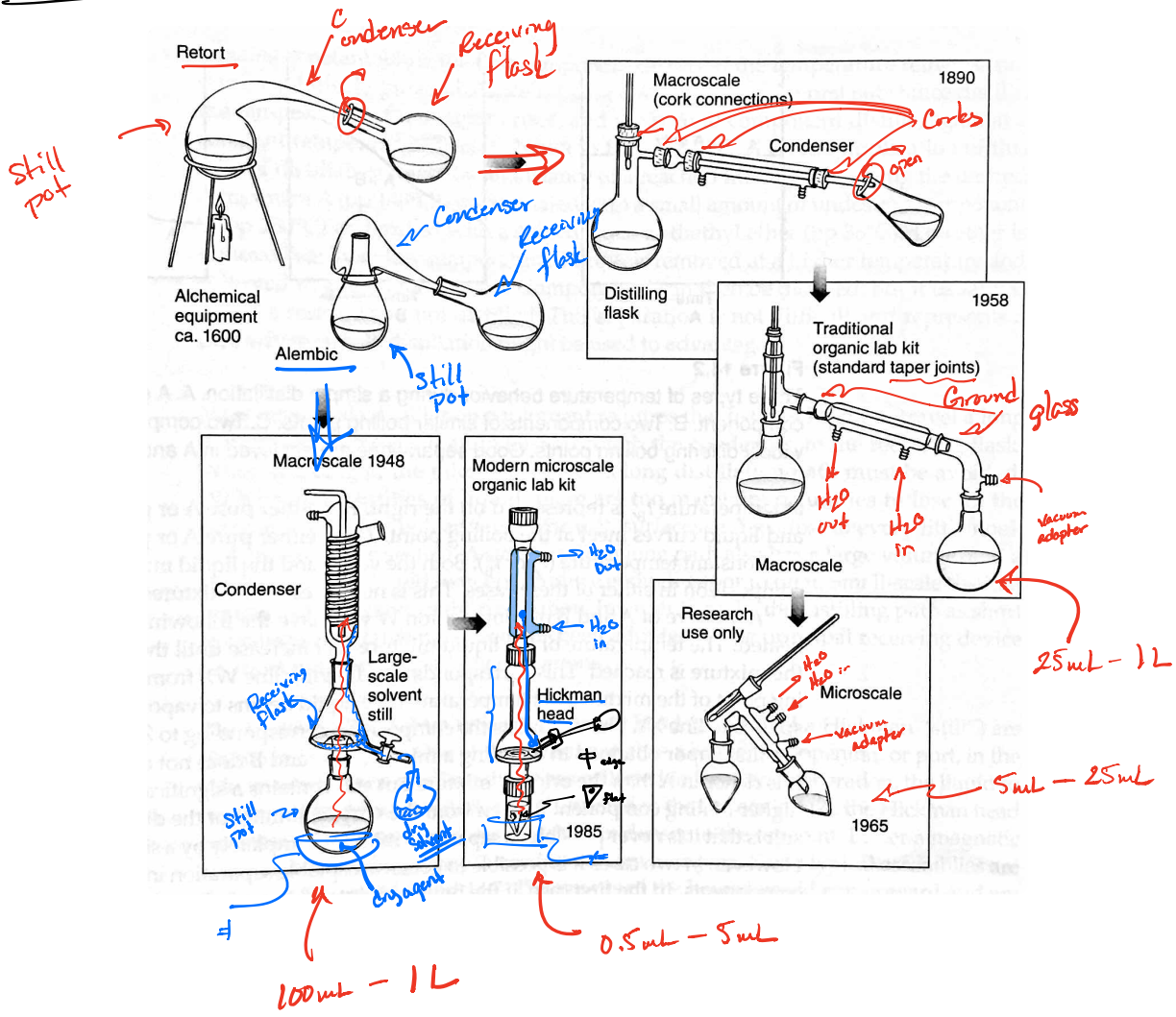
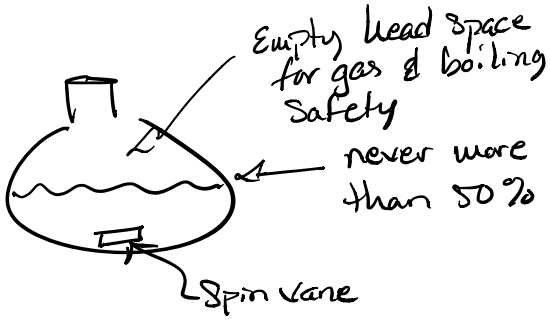
Distillation Lab



25 mL of Solvent
⇒ 50 mL flask

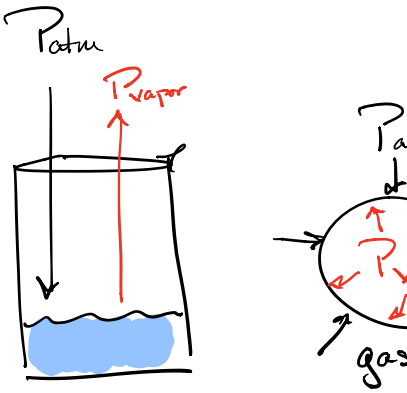


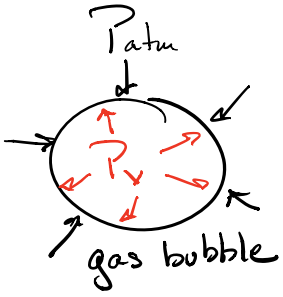
Distillation ⇒ purification of liquids

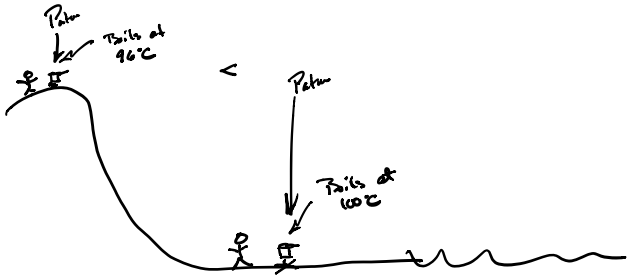
Recrystallization – purification of solids
75% – 90% range starting purity

Extraction – purification of solids (solutes)
Dependent on functional group
Acid/Base Extraction
Neutral compound from Acid/Base impurity
Any starting purity

Boiling Point







$P_{atm} > P_v$ No bubble ⇒ Evaporation

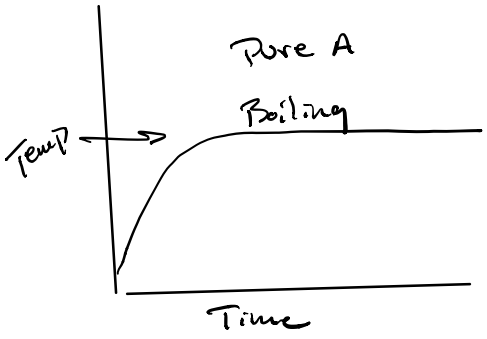
$P_{atm} = P_v$ Boiling Point

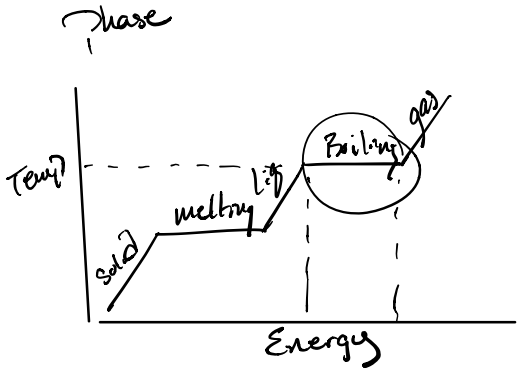
$P_{atm} < P_v$ Bumping Explosive Boiling

Boiling pt is the temp at which the P_{vap} is equal to the atmospheric pressure

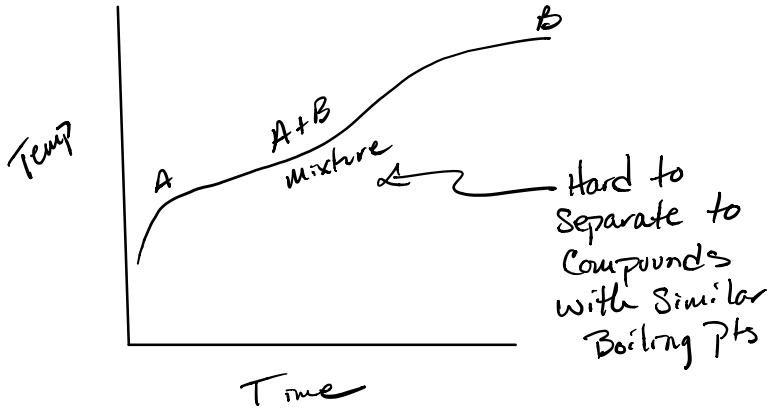
Temp vs Time Behavior of Systems while Boiling

① Single Component → Pure material "A"

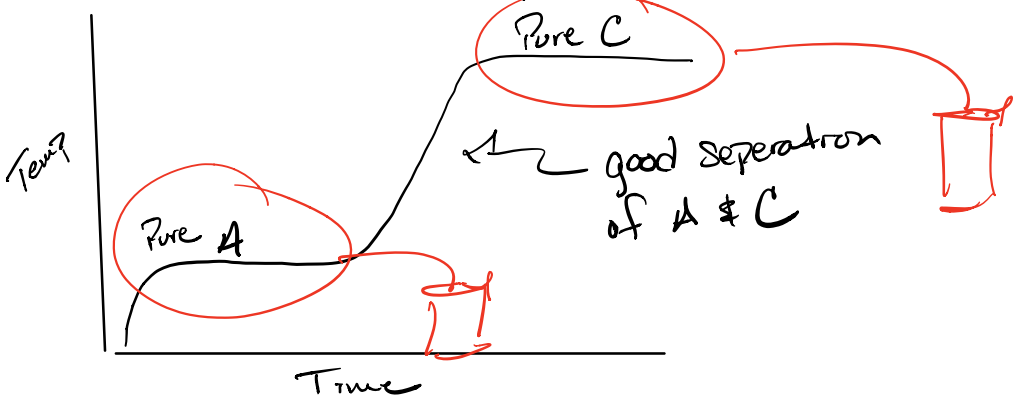




② Two Component Mixture of A & B with Close Boiling Pts.

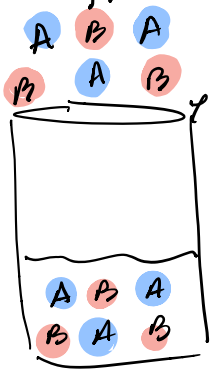


③ Two Component Mixture A+C w/ different Boiling Points



Vapor Composition as compared to liquid Composition in a 2 Component Mixture (Multi Component)

① Boiling Pts of A & B Same



Boiling Pts Same

∴ Vapor pressure Same

$PV = nRT$

$P_{vap} \propto n$ directly proportional

$P_{vap_A} = P^{\circ}_{A} N_A$

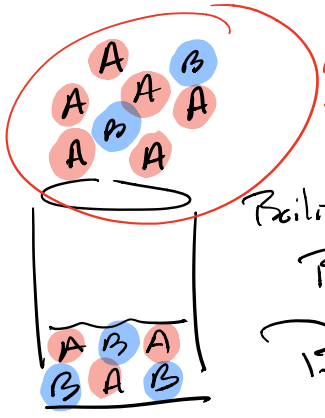
N_A = mole fraction

$N_A = \frac{\text{mole A}}{\text{mole A + mole B}}$

P_{vap_A} ↑ Vapor pressure in Mixture

P°_{A} ↑ Pure Vapor Pressure

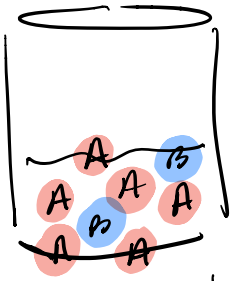
② Condenser



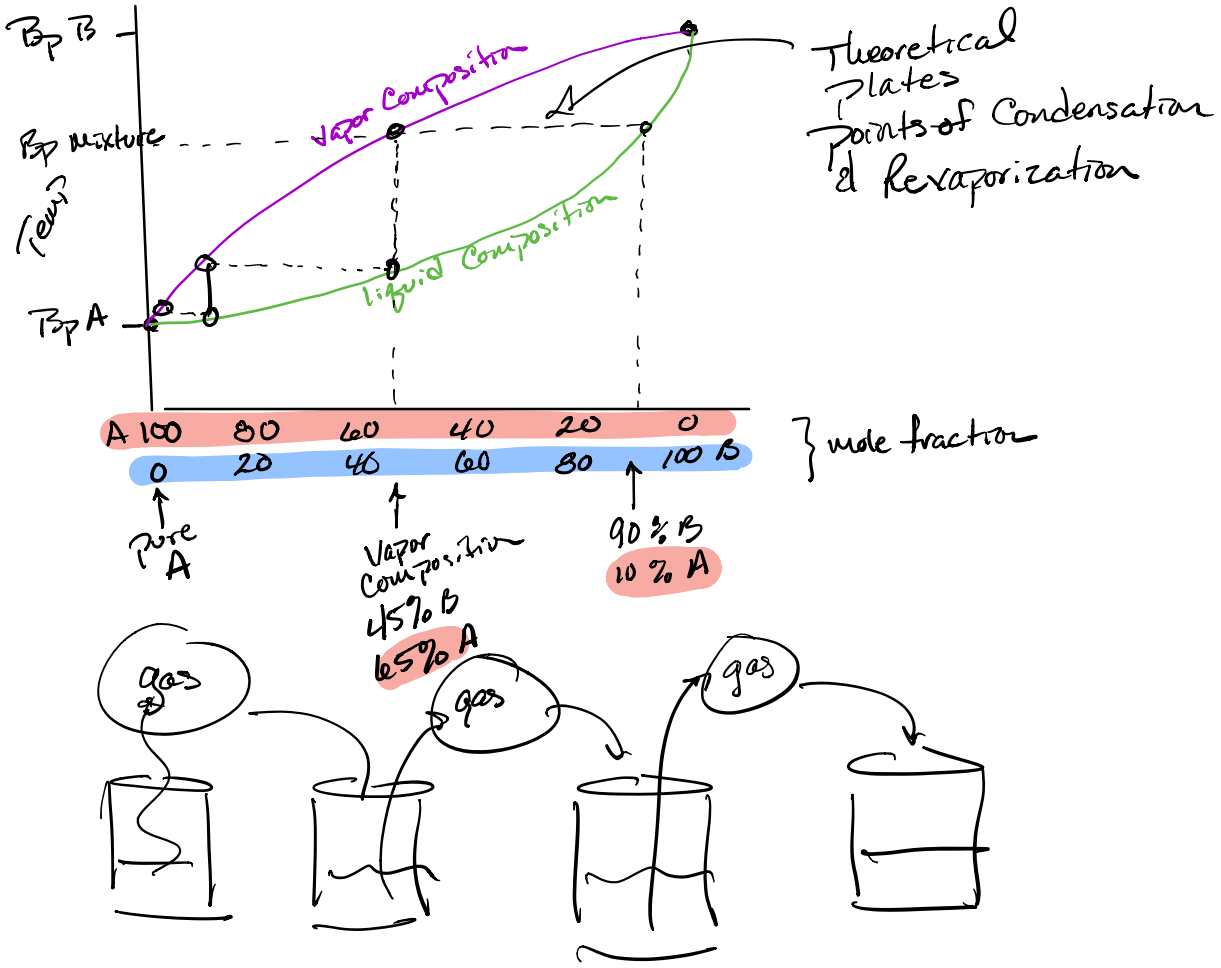
Boiling A less than Boiling pt B

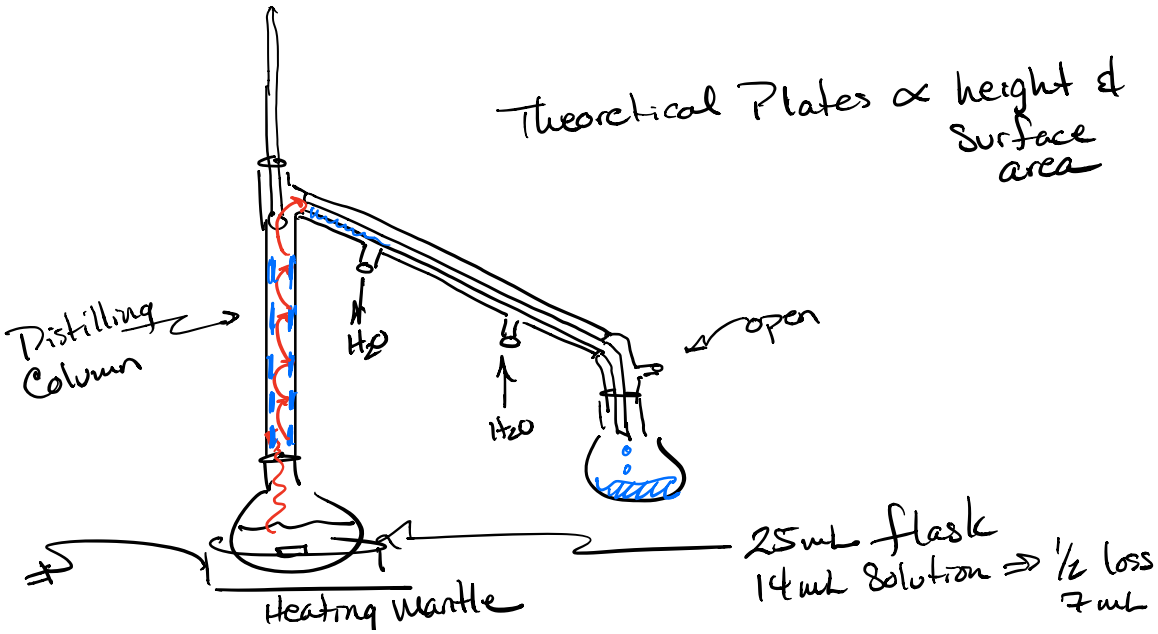
$P_{vap\,A} > P_{vap\,B}$

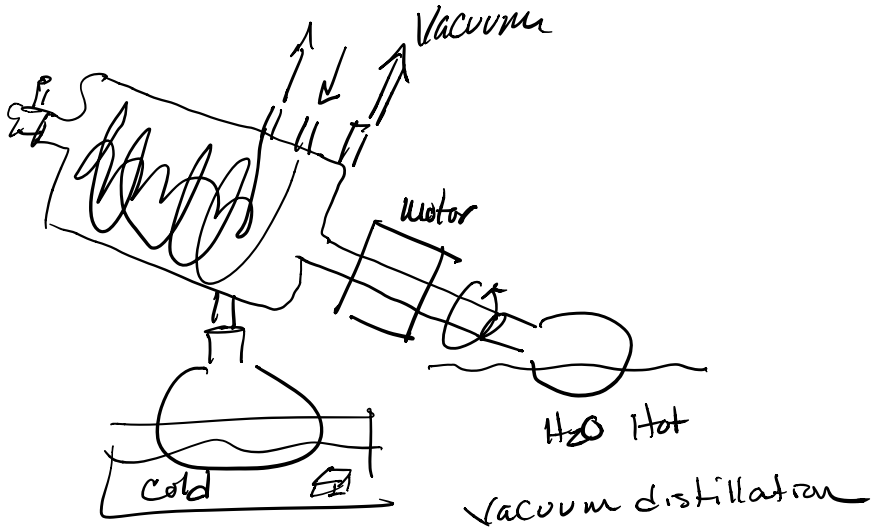
$N_A > N_B$



new Solution enriched in A


Bp B
Bp Mixture
(ew)
Bp A
Vapor Composition
Liquid Composition
Theoretical Plates
Points of Condensation
& Revaporization
A 100 80 60 40 20 0
0 20 40 60 80 100 B
} mole fraction
Pure A
Vapor Composition
45% B
65% A
90% B
10% A
gas
gas
gas

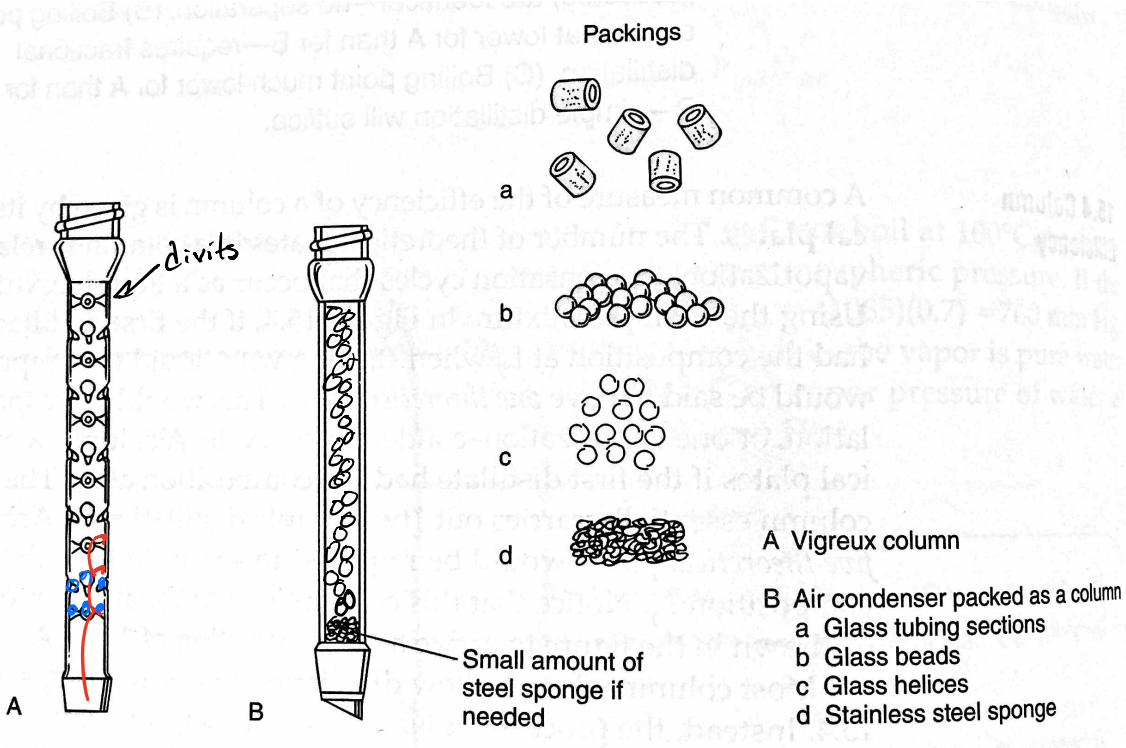

Theoretical Plates ∝ height & surface area
Distilling Column
H2O
H2O
open
Heating Mantle
25mL flask
14mL Solution ⇒ ½ loss
7mL



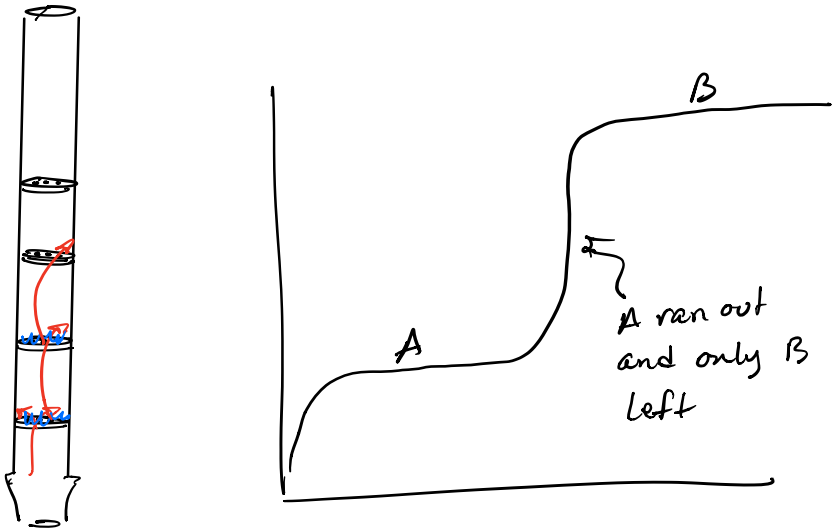
Vacuum distillation

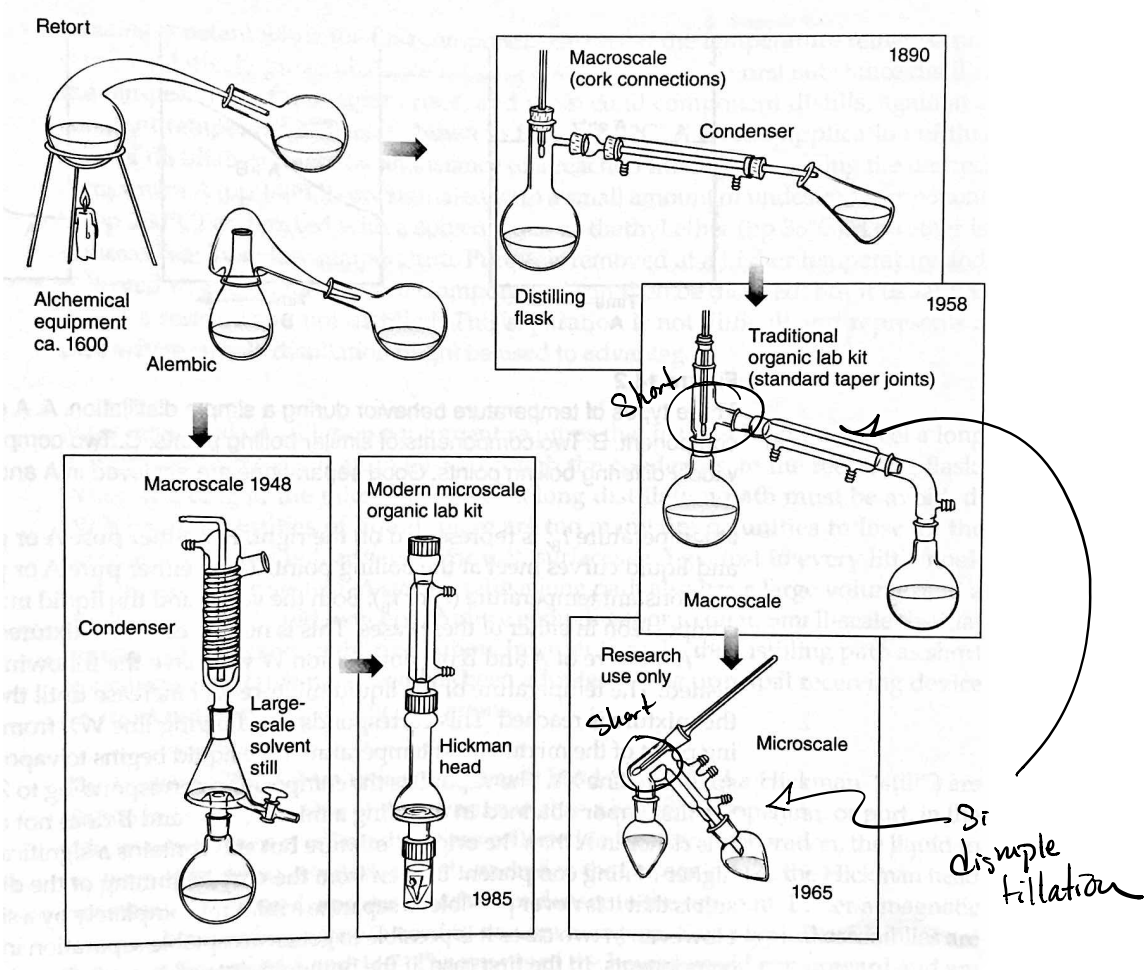
2 Component Mixture with ΔB_p vs # theoretical plates need

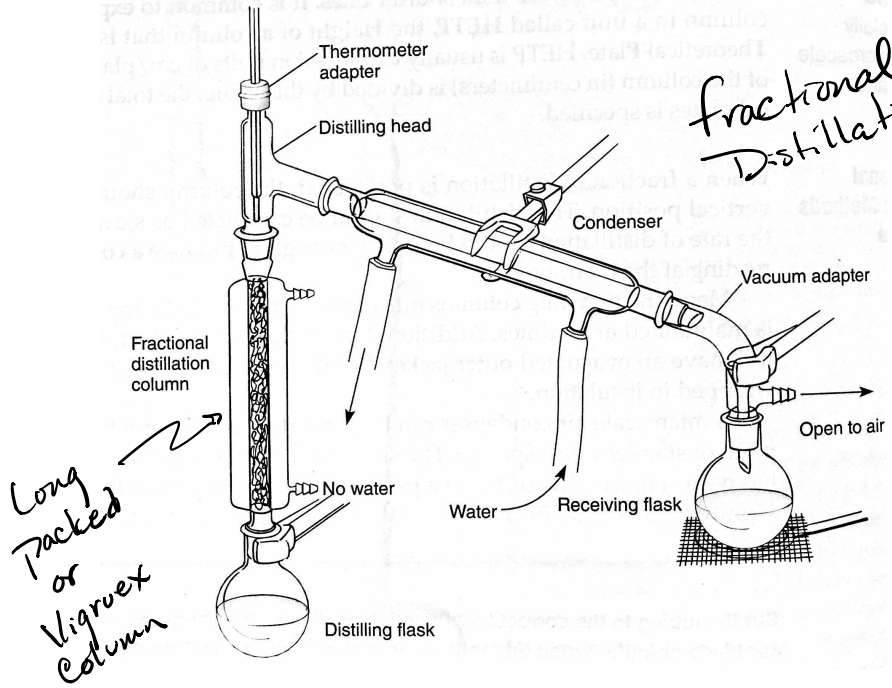
ΔBP	# of Theoretical Plates	
108°	1	Simple Column
72°	2	
54°	3	
43°	4	
36°	5	
20°	10	Fractional Column
10°	20	
7°	30	
4°	50	
2°	100	

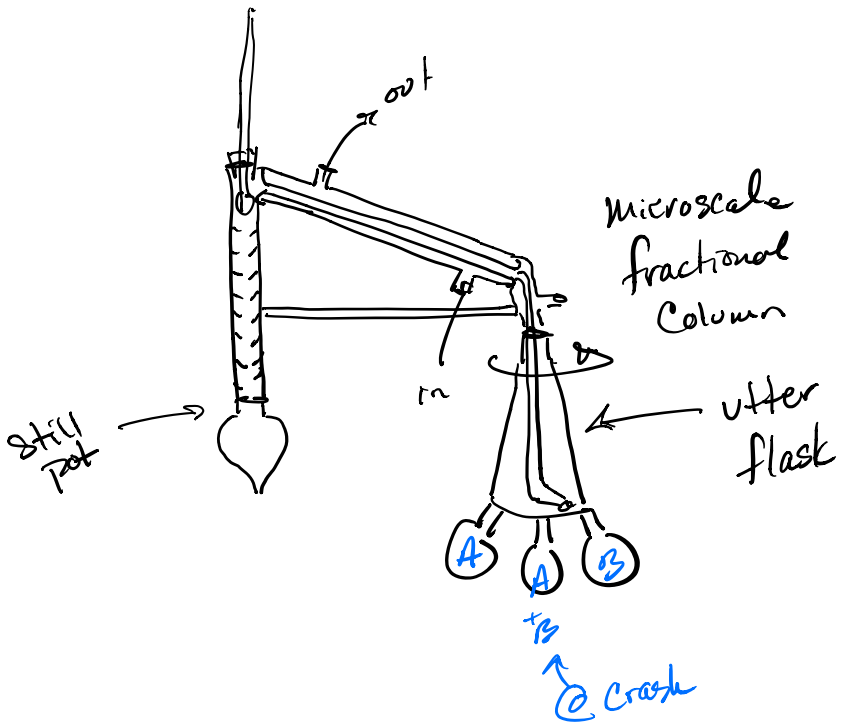


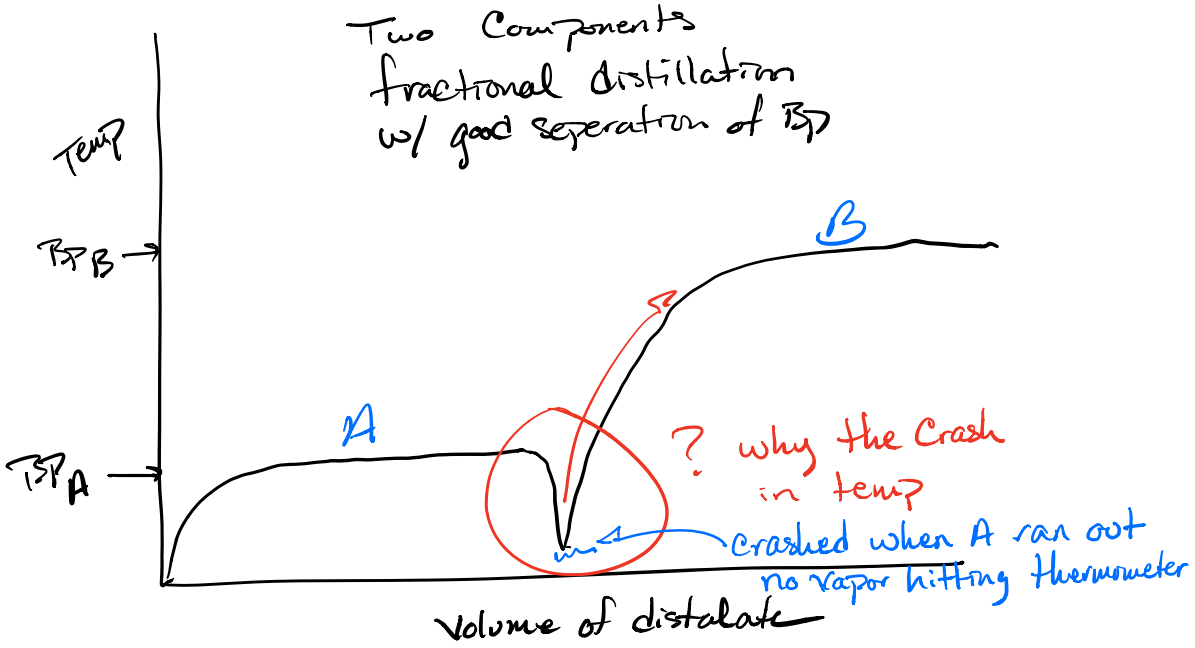
Vigreux Column

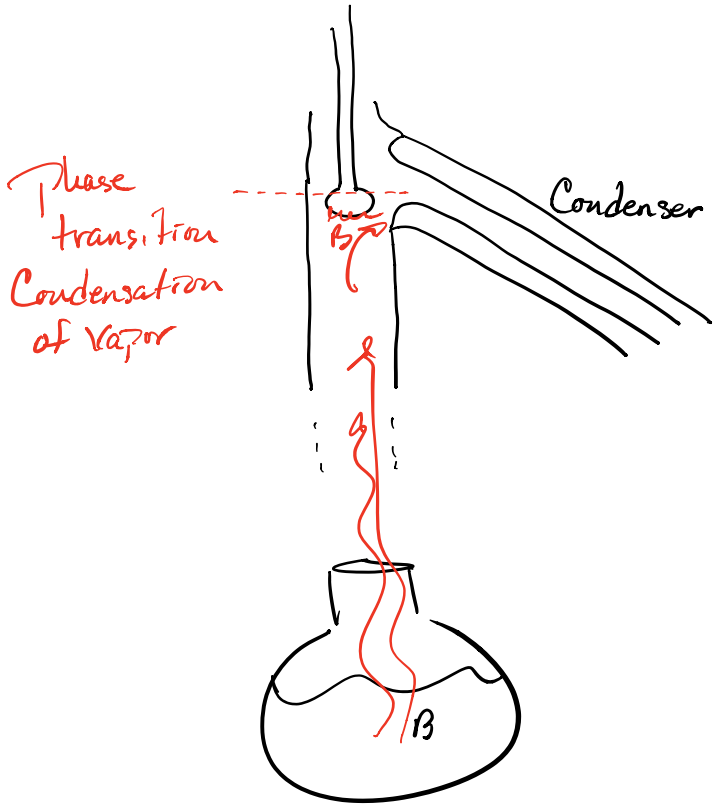


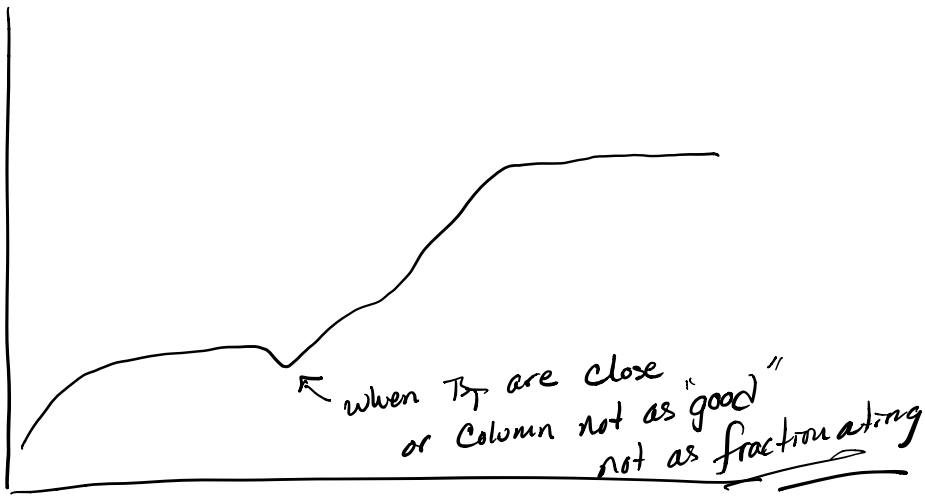

Retort
Alchemical equipment ca. 1600
Alembic
Macroscale (cork connections)
1890
Condenser
Distilling flask
1958
Traditional organic lab kit (standard taper joints)
Short
Macroscale
Macroscale 1948
Condenser
Large-scale solvent still
Modern microscale organic lab kit
Hickman head
1985
Research use only
Short
Microscale
1965
Simple distillation


Thermometer adapter
Distilling head
Condenser
Vacuum adapter
Fractional distillation column
Open to air
No water
Water
Receiving flask
Distilling flask
Long packed or Vigreux column
fractional Distillation


out
Microscale
fractional
Column
in
still
pot
utter
flask
A
A
B
+B
@ crash


Two Components
fractional distillation
w/ good seperation of BP
Temp
BP B
BP A
A
B
? why the Crash
in temp
crashed when A ran out
no vapor hitting thermometer
Volume of distalate


Phase transition Condensation of vapor
Condenser
B
B


when T_B are close
or column not as "good"
not as fractionating