CE407 SEPARATIONS

Lecture 12

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Various Distillation Topics

Distillation Column Specifications

- Specify the Feed – composition and quality
  - Binary $\mathbf{x_F}$ and $\mathbf{q}$ $\mathbf{x_F}$ refers to the light component
    - Mole fraction of heavy component = 1 – $\mathbf{x_F}$
  - Ternary $\mathbf{x_{1F}}$, $\mathbf{x_{2F}}$, and $\mathbf{q}$
    - $\mathbf{x_{3F}} = 1 - \mathbf{x_{1F}} - \mathbf{x_{2F}}$
  - Generic **N** components $\mathbf{x_{1F}}, \mathbf{x_{2F}}, \mathbf{x_{3F}}, \ldots \mathbf{x_{N\text{-}1F}}$, and $\mathbf{q}$
    - $x_{N,F} = 1 - \sum_1^{N-1} x_{i,F}$
- After specifying the feed you have 4 Degrees of Freedom (things you get to pick…)

Common Examples

(each case could be specified with different variables than these)

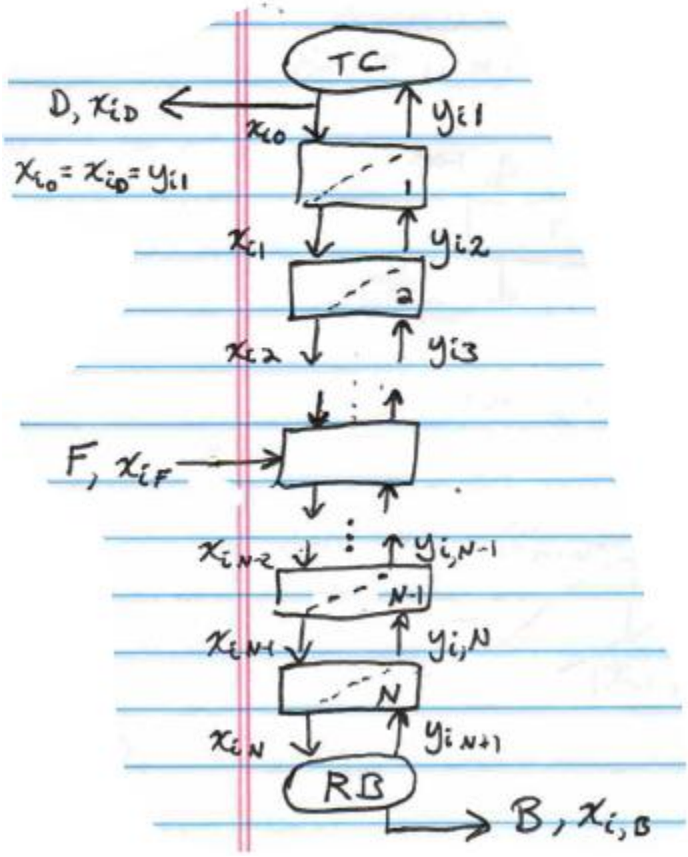
- Binary: $\mathbf{x_D}$, $\mathbf{x_B}$, **R**, Feed Location
- Ternary: $\mathbf{x_{1D}}$, $\mathbf{x_{1B}}$, $\mathbf{x_{3D}}$, **R**
- Once you specified four variables the entire process is determined

Degrees of Freedom

- Look at the Ternary Case
- If we specified $\mathbf{x_{1D}}$, $\mathbf{x_{1B}}$ and $\mathbf{x_{3D}}$
- Then $\mathbf{x_{2D}} = 1 - \mathbf{x_{1D}} - \mathbf{x_{3D}}$
- Now mass balances dictate that
  - $\mathbf{F = D + B}$
  - $\mathbf{Fx_{1F} = Dx_{1D} + Bx_{1B}}$
- That is 2 equations and 2 unknowns, therefore D and B are determined and we cannot choose them.
- Mass balance also dictates that
  - $\mathbf{Fx_{3F} = Dx_{3D} + Bx_{3B}}$
  - $\mathbf{x_{3B}}$ is the only unknown, so it is now determined
- We now have flow rates and composition of Feed, Distillate, and Bottoms
- **We must not over-specify the system or it may not be solvable!**

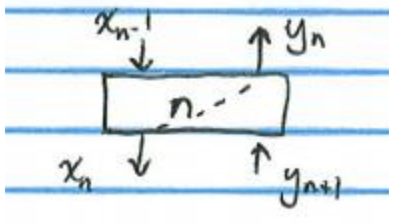
Multi-component Distillation

- Notice that now all of the mole fractions carry a subscript designating which component as well as what stage that flow is leaving
- A mass balance can be done for each component leading to a collection of Operating Lines
- $y_{i,n+1} = \frac{R}{R+1} x_{i,n} + \frac{x_{i,D}}{R+1}$ Rectifying Line for Component i
  - $R = {}^{L}/_{D}$
- $y_{i,n+1} = \frac{S+1}{S} x_{i,n} - \frac{x_{i,B}}{S}$ Stripping Line for Component i
  - $\mathbf{S} = {}^{\overline{V}}/_{B} = \frac{D}{B}(R+q) + q - 1$
- We will limit our discussion to constant molar flows

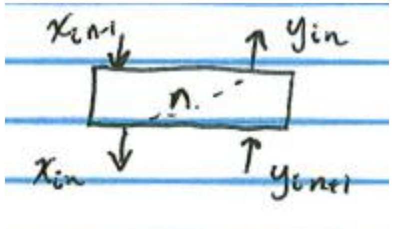


Detailed Multi-component Distillation Calculations

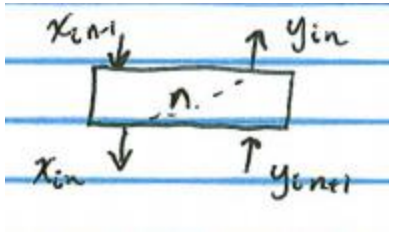
- We will start by comparing Binary calculations to Multi-component
- Binary
  - Calculate one set of Operating Lines
  - $\mathbf{x_n}$ is related to $\mathbf{y_{n+1}}$ via OP Line
  - $\mathbf{x_n}$ is related to $\mathbf{y_n}$ via Equilibrium Curve



- Multi-component
  - We need to include subscripts for the component
  - Calculate a set of Operating Lines for each component
  - $\mathbf{x_{i,n}}$ is related to $\mathbf{y_{i,n+1}}$ via OP Line for specific component
  - Equilibria Calculations – must include all components in calculation
    - When working downward in column use DEW POINT calculation to determine $\mathbf{x_{i,n}}$ from $\mathbf{y_{i,n}}$
    - When working upward in column use BUBBLE POINT calculation to determine $\mathbf{y_{i,n}}$ from $\mathbf{x_{i,n}}$



Bubble and Dew Point Calculations



- Working Down the Column
- We know $\mathbf{x_{i,o}}$ and $\mathbf{y_{i,1}}$ from $\mathbf{x_{i,D}}$
- We use Dew Point calculation to get $\mathbf{x_{i,1}}$ from $\mathbf{y_{i,1}}$
- $\sum_{i=1}^{N} x_{i,n} = \sum_{i=1}^{N} \frac{y_{i,n}}{k_{i,n}} = 1.0$
- $k_{i,n}$ is $\frac{P_i^{sat}}{P}$ for ideal case
- Then the rectifying OP Lines are used to relate $\mathbf{y_{i,n+1}}$ to $\mathbf{x_{i,n}}$
- Continue Down step by step
- Working Up the Column
- We use Bubble Point calculation to get $\mathbf{y_{i,n}}$ from $\mathbf{x_{i,n}}$
- $\sum_{i=1}^{N} y_{i,n} = \sum_{i=1}^{N} k_{i,n}\, x_{i,n} = 1.0$
- Then the stripping OP Lines are used to relate $\mathbf{x_{i,n-1}}$ to $\mathbf{y_{i,n}}$
- Continue Upward step by step

Detailed Multi-Component Distillation Calculations

- The reason we work from both ends is that we do not where the feed is going to enter (we don't know how many stages we have) and therefore we do not know when to switch from R OP Line to S OP Line
- The two series of calculations will eventually cross one another. That is the feed stage
- We can add the number of R stages and S stages at the crossing point to get the total number of stages
- Practically we use commercial Process Simulation Software
- Unisim is really amazing and you will learn to use it in CE 408!

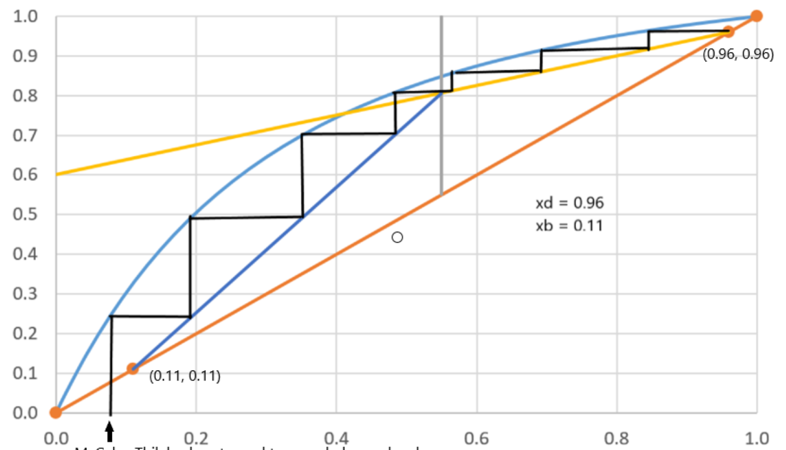
Performance Calculations

- We have been doing design models so far
  - We have a desired separation and are determining what number of stages, reflux ratio, and feed location are required to achieve it
- What if we have a column and want to know what it will do?
  - Given a certain feed, # of stages, reflux ratio, feed location plus one more (S or D, perhaps) what will the compositions of D and B actually be?
- Unisim will calculate flows, compositions, temperatures
  - It won’t work if you over-specify the system

Performance Calculations

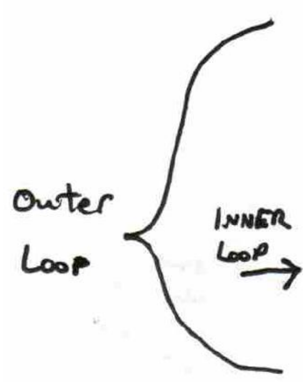
Binary Example

- Guess a value for x_D
- Use mass balance based on the feed and this x_D to determine what x_B would be
- Calculate operating lines from R, x_D, and feed information
- Draw in the number of stages that the column actually has
- Determine $x_{B\ obtained}$ from McCabe-Thiele
- If $x_{B\ obtained}$ does not match x_B from material balance then the x_D was incorrect
- Adjust x_D and iterate until $x_{B\ obtained}$ matches x_B
- You want them to match quite well because we are trying to determine what the compositions are that we will get
  - Before we were trying to determine how many stages we need to meet or beat a purity specification



Performance Calculations

Multi-Component Example



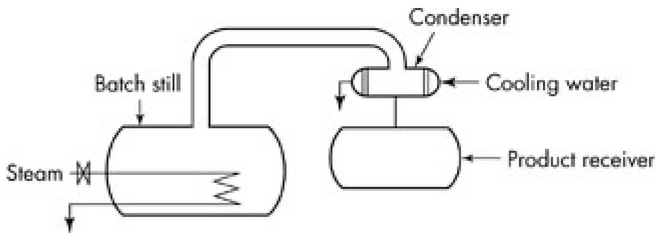
- Program chooses values for $\mathbf{x_{iD}}$
- Mass balance calculations based on this $\mathbf{x_{iD}}$ to determine what $\mathbf{x_{iB}}$ would be
- Tray to tray calculations determine $\mathbf{x_{iB\ obtained}}$
- If $\mathbf{x_{iB\ obtained}}$ does not match $\mathbf{x_{iB}}$ from material balance then adjust $\mathbf{x_{iD}}$ and iterate until $\mathbf{x_{iB\ obtained}}$ matches $\mathbf{x_{iB}}$

- One can use a simulator to get a design by specifying column and then use inner and outer loop to determine outputs. Then one can make adjustments to the specifications until they are satisfied with the result

Extremely (Embarrassingly) Rough Multi-Component Short Cut

- If LK and HK represent a substantial portion of the feed…
- Perform a McCabe-Thiele analysis treating the case as a binary mixture of the LK and HK
- This gives an estimate of the number of stages and the feed location
- This estimate **MUST** be presented as a very low confidence starting point for the design

Binary Batch Distillation McSH pp 724-727



- Batch distillation is much simpler in terms of equipment, but the analysis is actually more involved…
- Define terms
  - A = light component
  - B = heavy component
  - **n** = total number of moles of liquid in the still pot
  - $\mathbf{n_A}$ = number of moles of liquid A in the still pot
  - x = mole fraction of component A in the still pot (liquid)
  - y = mole fraction of component A in the vapor coming off of the still pot

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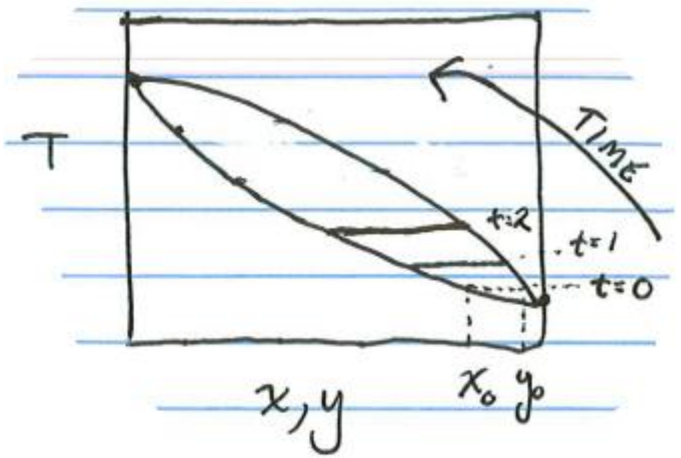
Binary Batch Distillation

Binary Batch Distillation

- **x** and **y** values at any given time will be in equilibrium with one another
- A, being the lighter component, has a higher mole fraction in the vapor phase than it does in the liquid phase
- As a result, component A is being depleted from the liquid and **x** will drop with time
  - Unfortunately that means the mole fraction, **y**, of the vapor being generated is also dropping
- What is the rate of change of number of moles of A in the still pot?
- First express the number of moles of A in the still pot as:



$$\boldsymbol{n_A = x\,n}$$

(# moles A = mole fraction A * total number of moles)

Binary Batch Distillation

- $dn_A = d(xn) = n\,dx + x\,dn$ when expressed in terms of liquid mole fraction
  - dn_A is rate of change of moles of A ***in the still pot***, the rate that moles of A are leaving when expressed in terms of liquid mole fraction
  - dx is rate of change of liquid mole fraction
  - dn is rate of change of total moles ***in still pot***
- $dn_A = y\,dn$ when expressed in terms of vapor mole fraction
- Note: the total moles leaving (dn) have a mole fraction **y** so we can express simply as $y\,dn$. The total moles leaving DO NOT have a mole fraction of **x** and therefore that expression is more complex.
- Obviously both expressions must equal one another

$$n\,dx + x\,dn = y\,dn$$

$$n\,dx = (y - x)\,dn$$

$$\frac{dx}{y - x} = \frac{dn}{n}$$

Binary Batch Distillation

$$\frac{dx}{y-x} = \frac{dn}{n}$$

- Integrate from time t_0 where $x = x_0$ and $n = n_0$ to an arbitrary time where $x = x$ and $n = n$
- To avoid mathematical confusion between the values x and n at the arbitrary time and the variables x and n as we integrate we will express the variables as x' and n'
- Express vapor mole fraction as $y = y(x')$ to explicitly indicate that the instantaneous value of y must be in equilibrium with the instantaneous value of x'
- $\int_{n_0}^{n} \frac{dn'}{n'} = \boxed{ln\left(\frac{n}{n_0}\right) = \int_{x_0}^{x} \frac{dx'}{y(x') - x'}}$ eq 21.86

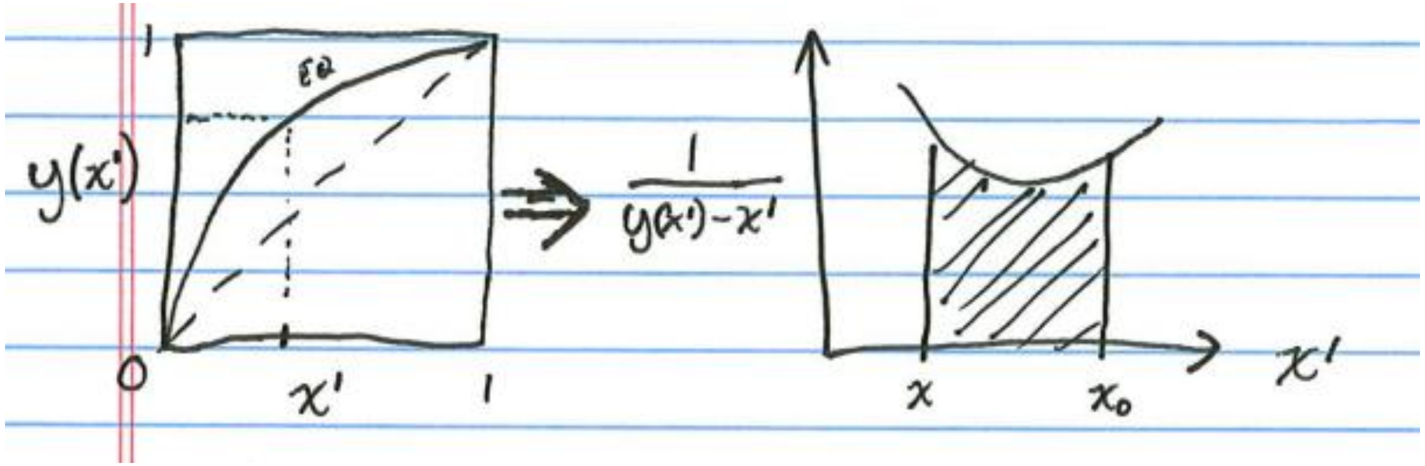
Rayleigh Equation

Rayleigh Equation

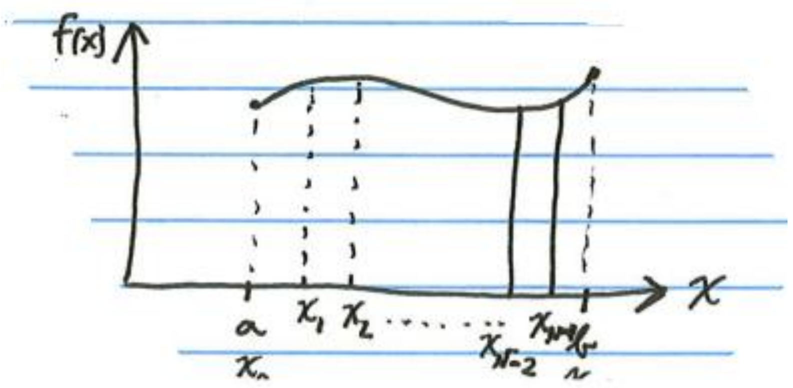
- The Rayleigh Equation gives the relationship between the total moles left in the still pot and the mole fraction of component A of the material left in the still pot

$$ln\left(\frac{n}{n_0}\right) = \int_{x_0}^{x} \frac{dx'}{y(x') - x'}$$

- We need to interpret the integral on the right hand side of this equation
  - For various values of x' from x to x_0 read off $y(x')$ and calculate $\frac{1}{y(x') - x'}$
- We can now approximate the integral



Use Trapezoid Rule



- $\int_a^b f(x)dx \cong \left[\frac{1}{2}f(x_0) + f(x_1) + \cdots + f(x_{N-1}) + \frac{1}{2}f(x_N)\right]h$
- Where we break the range into N equally spaced slices
- $h = \frac{b-a}{N}$ is the thickness of each slice
- The area of a slice, **n**, is approximately $A_n = \left(\frac{f(x_{n-1})+f(x_n)}{2}\right)h$
  - Average height in that range times the width of the range
- Add up the slices $\sum = \left(\frac{f(x_0)+f(x_1)}{2}\right)h + \left(\frac{f(x_1)+f(x_2)}{2}\right)h + \cdots + \left(\frac{f(x_{N-1})+f(x_N)}{2}\right)h$

$$= \left[\frac{f(x_0)}{2} + f(x_1) + \cdots + f(x_{N-1}) + \frac{f(x_N)}{2}\right]h$$

- The greater a number N is, the more accurate the approximation

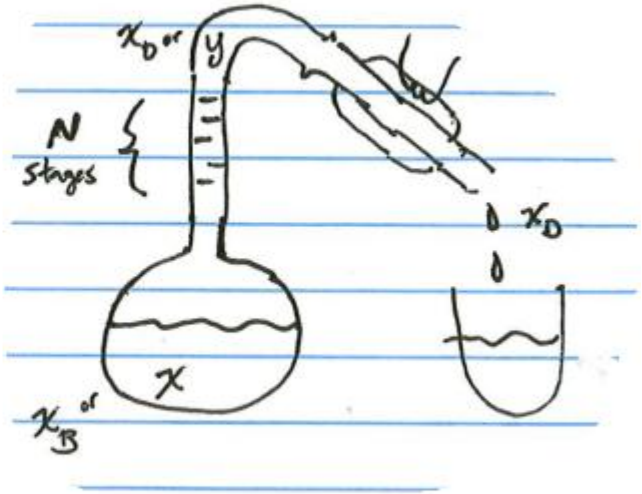
Binary Batch Distillation

- $ln\left(\frac{n}{n_0}\right) = \int_{x_0}^{x} \frac{dx'}{y(x') - x'}$
- Notice that our integral goes from x_0 to x and $x_0 > x$. Therefore our integral has a negative value.
- This makes sense because $\frac{n}{n_0} < 1$ and therefore *ln* ($\frac{n}{n_0}$) will be a negative number
- Our trapezoidal sum represents the absolute value of the integral, be sure to change the sign
- Method:
    - Pick a value of x and use trapezoidal approximation to estimate $\int_{x_0}^{x} \frac{dx'}{y(x') - x'}$
    - Calculate n using $ln\left(\frac{n}{n_0}\right) = \int_{x_0}^{x} \frac{dx'}{y(x') - x'}$
    - Repeat for various values of x. Create table of **x** and **n**
    - We now have the connection between # moles left in still pot and the mole fraction of the liquid left in the pot
    - Please see ***Binary Batch Distillation Examples*** in Notes for discussion of how we go from this knowledge of x vs n to an understanding of volume remaining and cumulative mole fraction of distillate

x	n
-	-
-	-
-	-

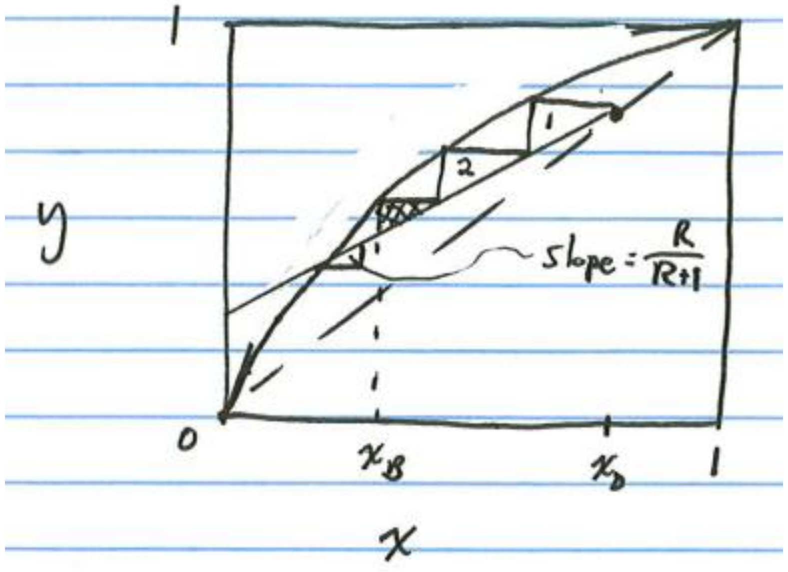
Batch Distillation with Reflux

- Improve separation by adding rectifying stages and reflux
- Improves purity of distilled product but not of the bottoms (material left in still pot)
- **y** refers to mole fraction above the **N** stages and is equal to $\mathbf{x_D}$, composition of condensed material leaving the still
- x refers to the mole fraction of liquid remaining in still pot and can be referred to as $\mathbf{x_B}$



Batch Distillation with Reflux

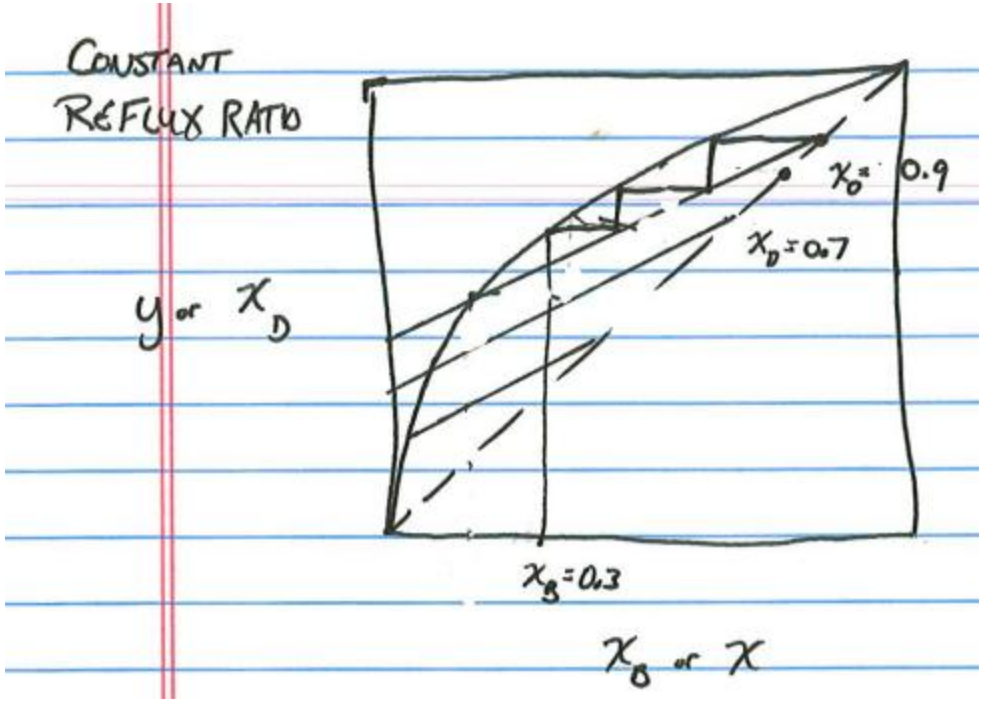
- Analyzing a still with a set number of stages
- Pick a value of $\mathbf{x_D}$ and determine the value of $\mathbf{x_B}$
- Draw rectifying line with slope = R/(R+1)
- In this example with 2 stages a third step is drawn in
  - This represents the step in the still pot itself (equivalent to the reboiler)
  - This allows you to determine what $\mathbf{x_B}$ will correspond to that value of $\mathbf{x_D}$
- Choose multiple values of $\mathbf{x_D}$ and get $\mathbf{x_B}$ for each
- Make a table of $\mathbf{x_B}$ vs $\mathbf{x_D}$
- Do the same steps w/Rayleigh equation, etc



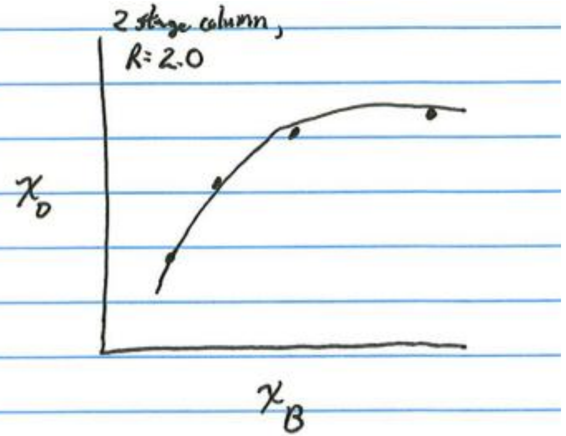
Batch Distillation with Reflux

Constant Reflux Rate

- $\mathbf{x_D}$ will change with time
- Draw multiple operating lines
  - All have same slope of R/(R+1)
- Step off # of steps corresponding to # of stages +1 for still pot
- Read off $\mathbf{x_B}$
- Generate table of $\mathbf{x_D}$ vs $\mathbf{x_B}$



x_D	x_B
0.95	0.63
0.9	0.33
0.8	0.12
0.7	0.08



Batch Distillation with Reflux

Variable Reflux Rate

- $\mathbf{x_D}$ will be constant with time
- Draw various operating lines all originating from ($\mathbf{x_D}$, $\mathbf{x_D}$) each having a different slope
- Value of R for each line can be obtained from intercept = $\frac{x_D}{R+1}$
- Walk off the appropriate # of steps (= # stages+1) for each line to determine what $\mathbf{x_B}$ will corresponds to that reflux ratio
- Plot **R** vs $\mathbf{x_B}$ to show what **R** will be required for each $\mathbf{x_B}$ in order to maintain the desired $\mathbf{x_D}$