Distillation

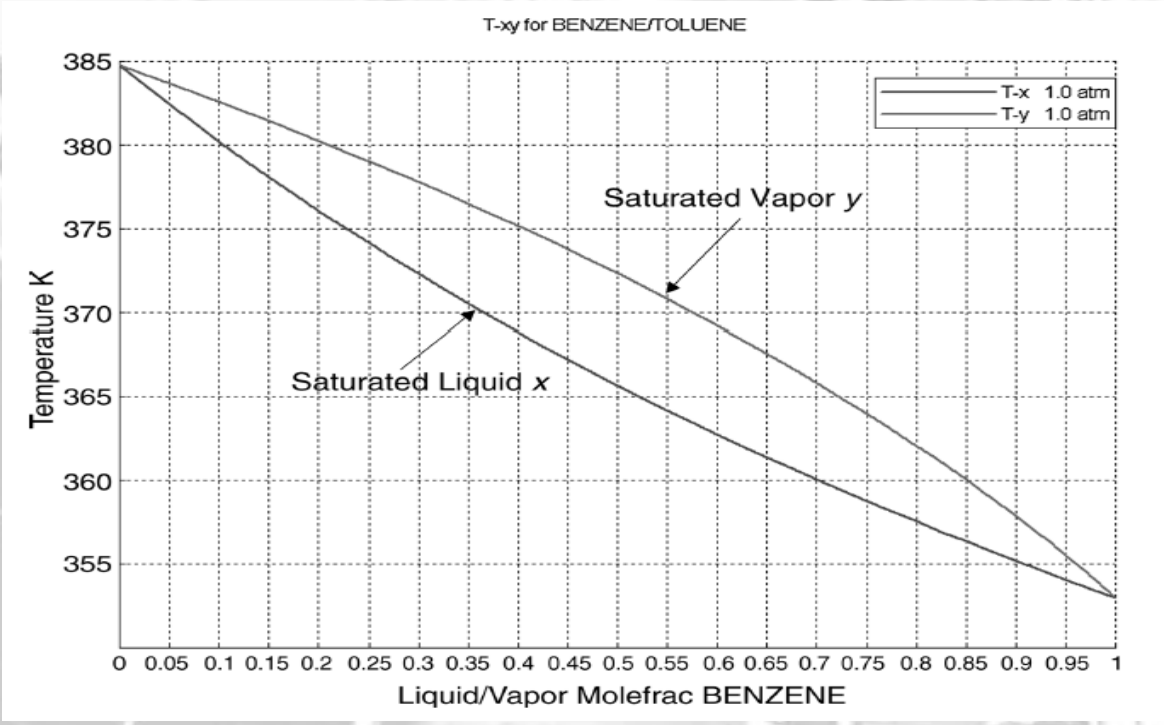
Background theory
Construction of Distillation Towers
Analysis of Distillation Towers
Issues with Distillation Towers

Background Theory

- Phase diagrams
- Vapour-Liquid Equilibrium Diagrams
- Relative Volatility

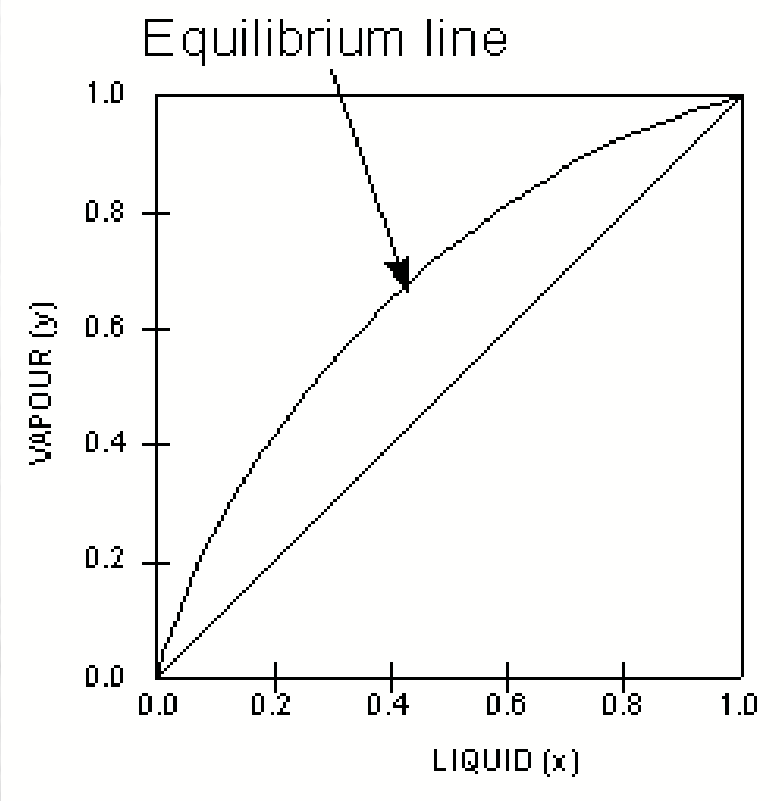
Phase Diagrams

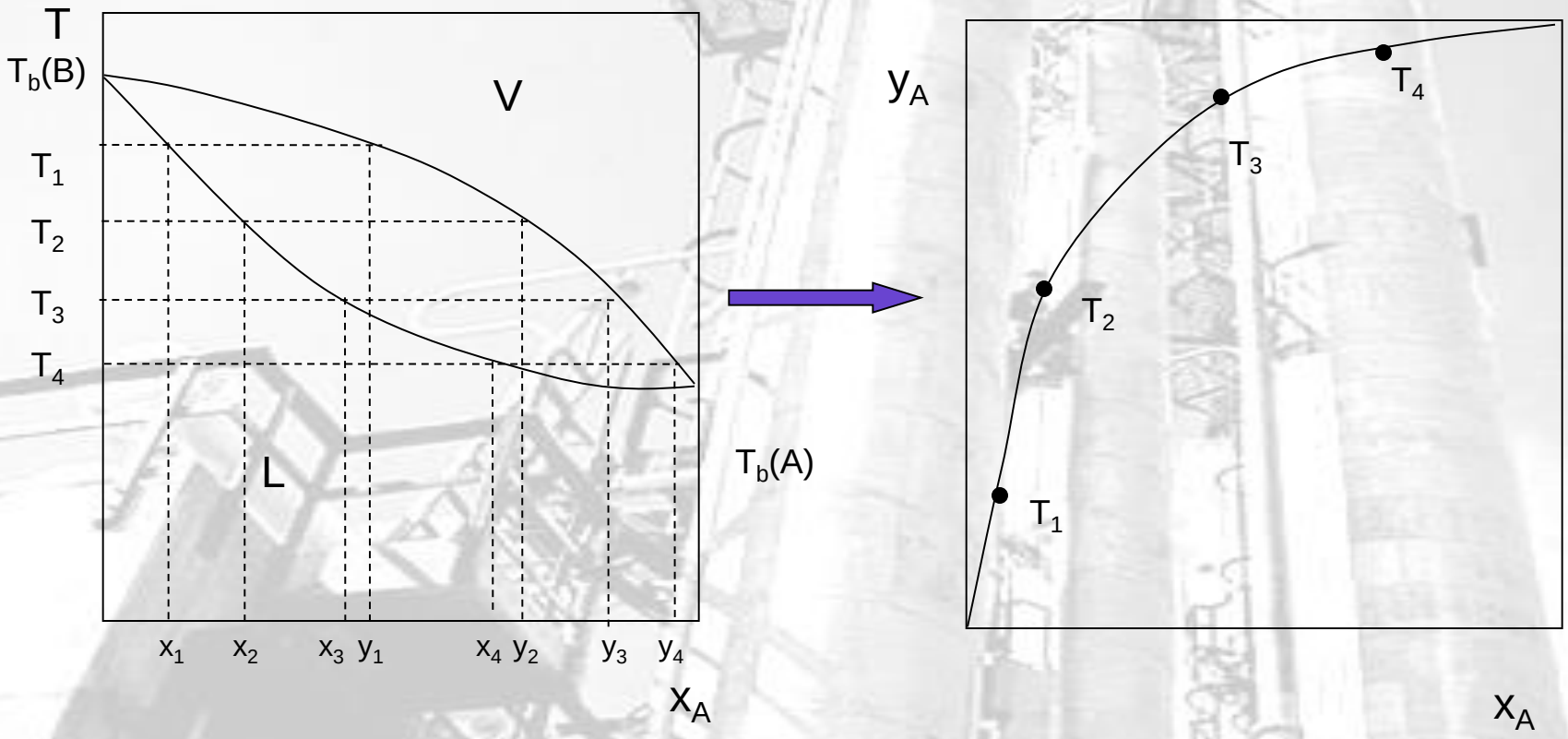
- Presents graph of boiling point as a function of composition
- Example below is phase diagram for benzene toluene at 101.3kPa



Vapour-Liquid Equilibrium Diagram

- Based on the phase diagram we can produce a vapour-liquid equilibrium diagram (VLE)
- Taken by noting the composition of the saturated liquid line and saturated vapour line at different temperatures




T
$T_b(B)$
V
T_1
T_2
T_3
T_4
L
x_1 x_2 x_3 y_1 x_4 y_2 y_3 y_4
x_A
$T_b(A)$
y_A
T_4
T_3
T_2
T_1
x_A

VLE Diagrams

- Note that the vapour composition is denoted y and goes on the y-axis, the liquid composition on x axis
- For the volatile component, the VLE curve is above 45^{o} line
- The further the VLE line from the 45^{0} line, the more different the two components and the easier the separation will be

Relative Volatility

- Measurement of how far from 45^0 line the VLE curve is
- Defined as:

$$\alpha_{AB} = \frac{y_A / x_A}{y_B / x_B}$$

- If system obeys Raoult's Law this becomes:

$$\alpha_{AB} = P_A / P_B$$

- α_{AB} doesn't tend to change much with temperature (see benzene toluene example overleaf)
- Don't do distillation unless α_{AB} >1.05
- Decreases with increasing pressure

Example: α_{AB} for Benzene Toluene

$$\alpha_{AB} = \frac{y_A / x_A}{y_B / x_B}$$

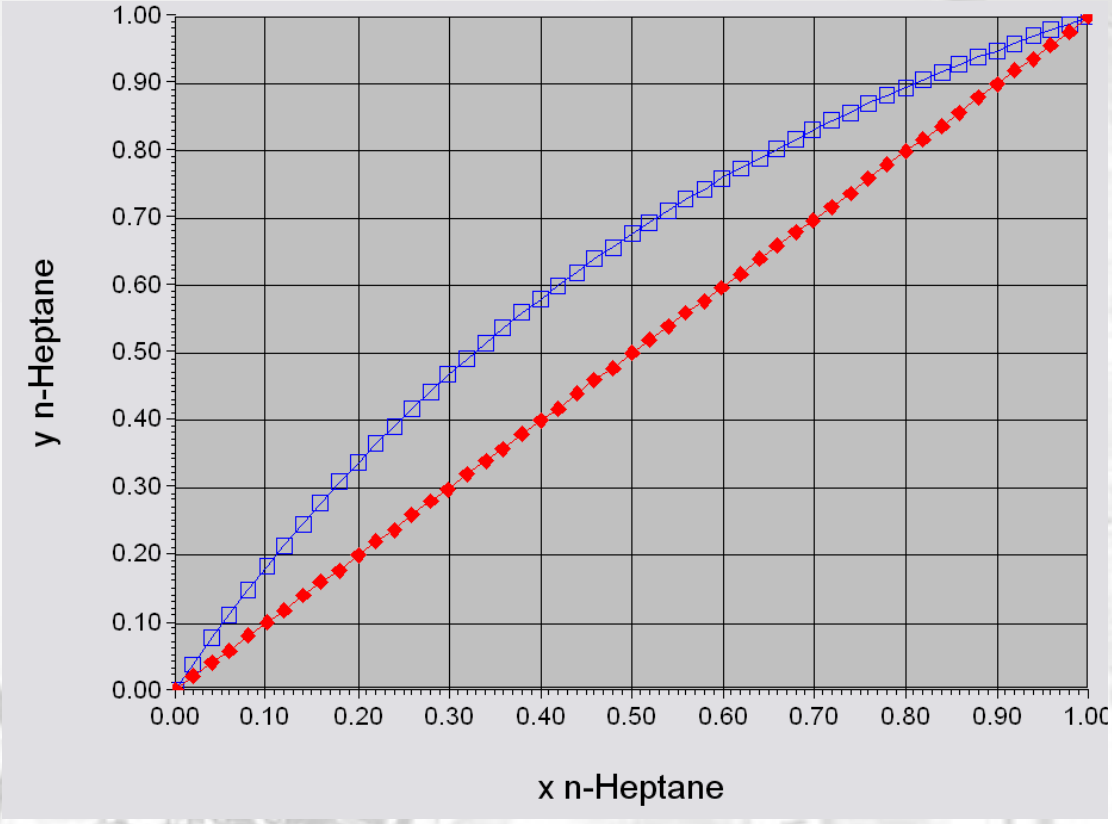
$$= \frac{y_A / x_A}{(1-y_A)/(1-x_A)}$$

$$= 2.27 at 362.2\text{K}$$

$$= 2.46 at 380.45 K$$

T, K	P, atm	X	Y
383.9	1	0.008	0.018
383.17	1	0.022	0.051
380.45	1	0.077	0.170
379.95	1	0.088	0.191
373.94	1	0.230	0.424
369.53	1	0.352	0.573
366.44	1	0.449	0.668
364.37	1	0.519	0.727
362.20	1	0.599	0.786

- http://www.cheric.org/research/kdb/hcvle/hcvle.php

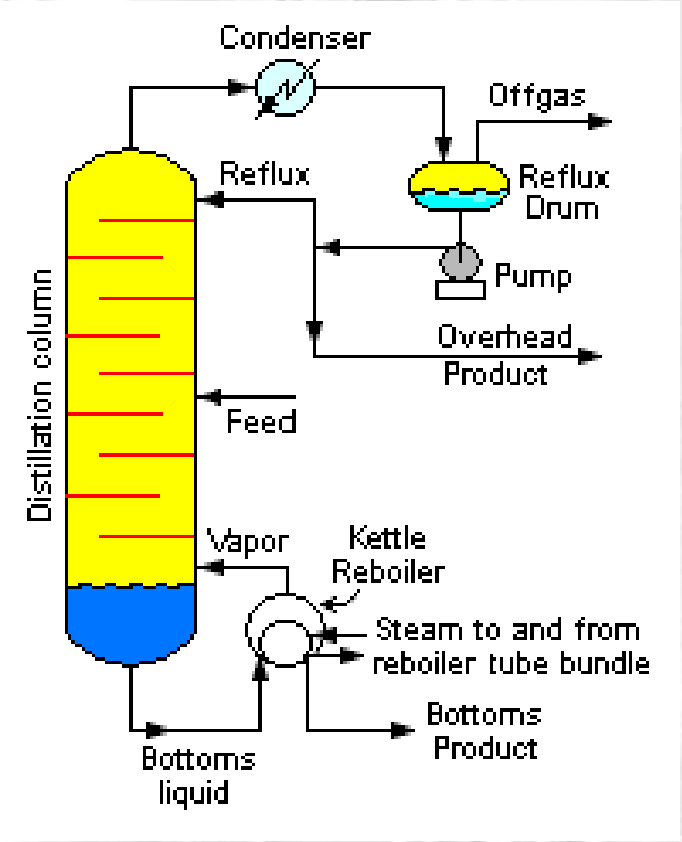


Above is a VLE diagram for n-Heptane and n-Octane
Calculate the relative volatility of this system when $x_{n\text{-Heptane}}$ is:

- 0.3
- 0.6
- 0.9

Distillation Tower Construction

- Schematic
  - Feed is input mixture
  - More volatile components in overhead, distillate
  - Less volatile in bottoms
  - Within tower there is a series of stages (called trays or plates)

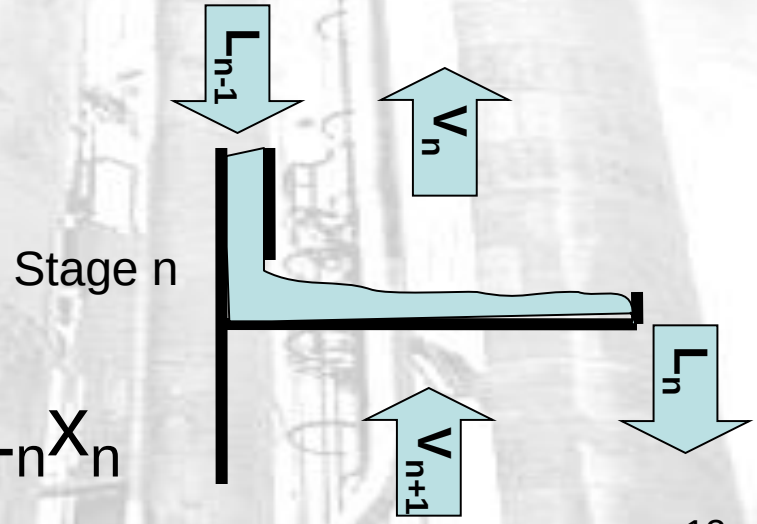


Distillation Tower Construction

- We'll discuss several aspects of construction
  - Overall principle
  - Feed conditions
  - Trays
  - Downcomers
  - Condensers
  - Reflux drum
  - Reboilers

Overall Principle

- Series of flash vaporisation stages
- Vapour and liquid flow counter-current to each other
- At each stage equilibrium is reached, for each component



$$V_{n+1} + L_{n-1} = V_n + L_n \text{ and}$$
$$V_{n+1}y_{n+1} + L_{n-1}x_{n-1} = V_n y_n + L_n x_n$$

Feed Conditions

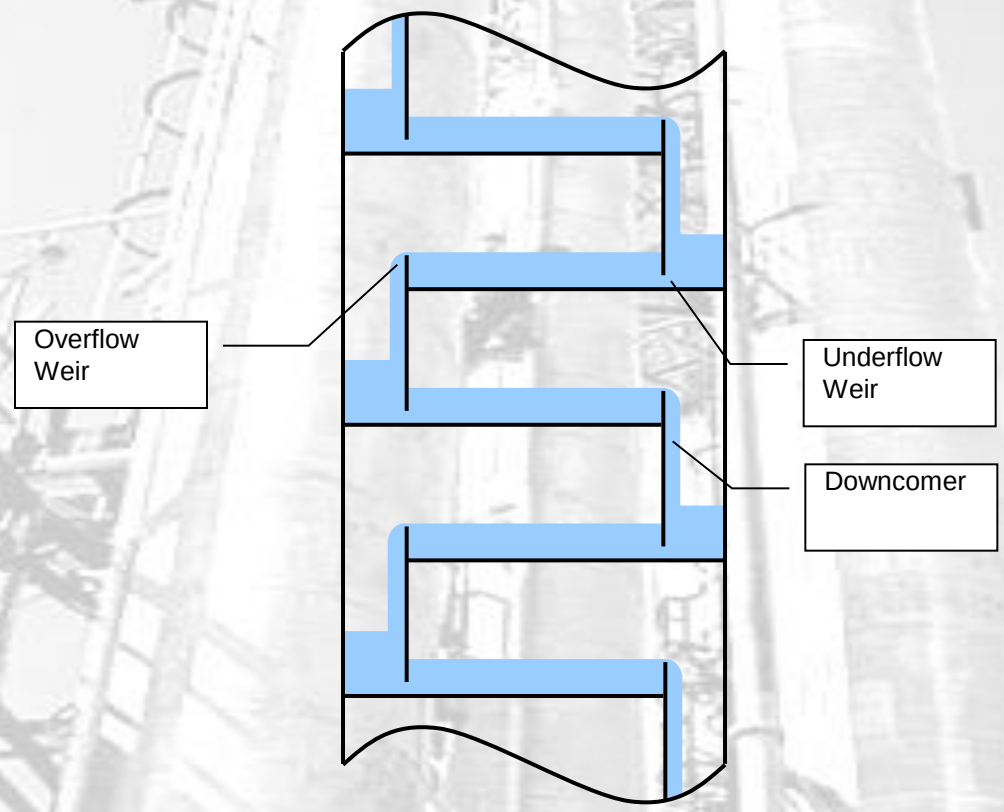
- Important parameters are:
  - State of feed (vapour fraction)
  - Composition
- These dictate choice of feed tray
- May have several possible feed trays to budget for variations in feed

Trays

- Trays or plates form stages of column
- Different types
  - Sieve
    - simply metal plates with holes in them. Vapour passes straight up through the liquid on the plate.
  - Bubble cap
    - Each hole fitted with a riser covered by a cap. Space around cap lets vapour escape.
  - Valve
    - Each hole fitted with a valve, pushed open by vapour flow.
- At edge of each tray there is a weir
  - Maintains liquid on tray, called hold-up
  - Height of weir enough to cover bubble caps
- Pressure drop of ~1kPa per tray

Downcomers

- Feed liquid down to lower trays
- Need to be wide enough to avoid flooding



Condensers

- Temperature = boiling point of light component
  = dew point of mixture
- Heat exchangers recover energy from system
- Often integrated with reboiler for energy conservation
- Chosen pressure important
  - Like to be able to cool with water

Reflux Drum

- Holds condensed vapour from top of column so that liquid reflux can be recycled back to column
- Reflux ratio
  - proportion of condensed vapour returned to top of column
  - R = L/D
  - Ensures sufficient traffic of vapour and liquid in column
- Reflux ratio important parameter in functioning of tower
- Holds condensed vapour from top of column so that liquid reflux can be recycled back to column

Reboilers

- Temperature = boiling point of heavy component
  = bubble point of mixture
- Heat exchangers injecting energy into system
- Often integrated with condenser for energy conservation
- Pressure choice important
  - Make sure temperature is such that products do not decompose

Analysis of Distillation Towers

- McCabe Thiele Method
  - Graphical method based on VLE
- Fenske – Underwood – Gilliland (FUG) method
  - Set of equations
  - We won't look at this
- Stuff we want to know
  - Number of stages
  - Position of feed tray
  - Reflux ratio
- Also would like
  - Size, diameter of trays
  - Separation of trays
  - Tray efficiency
  - Pressure drop

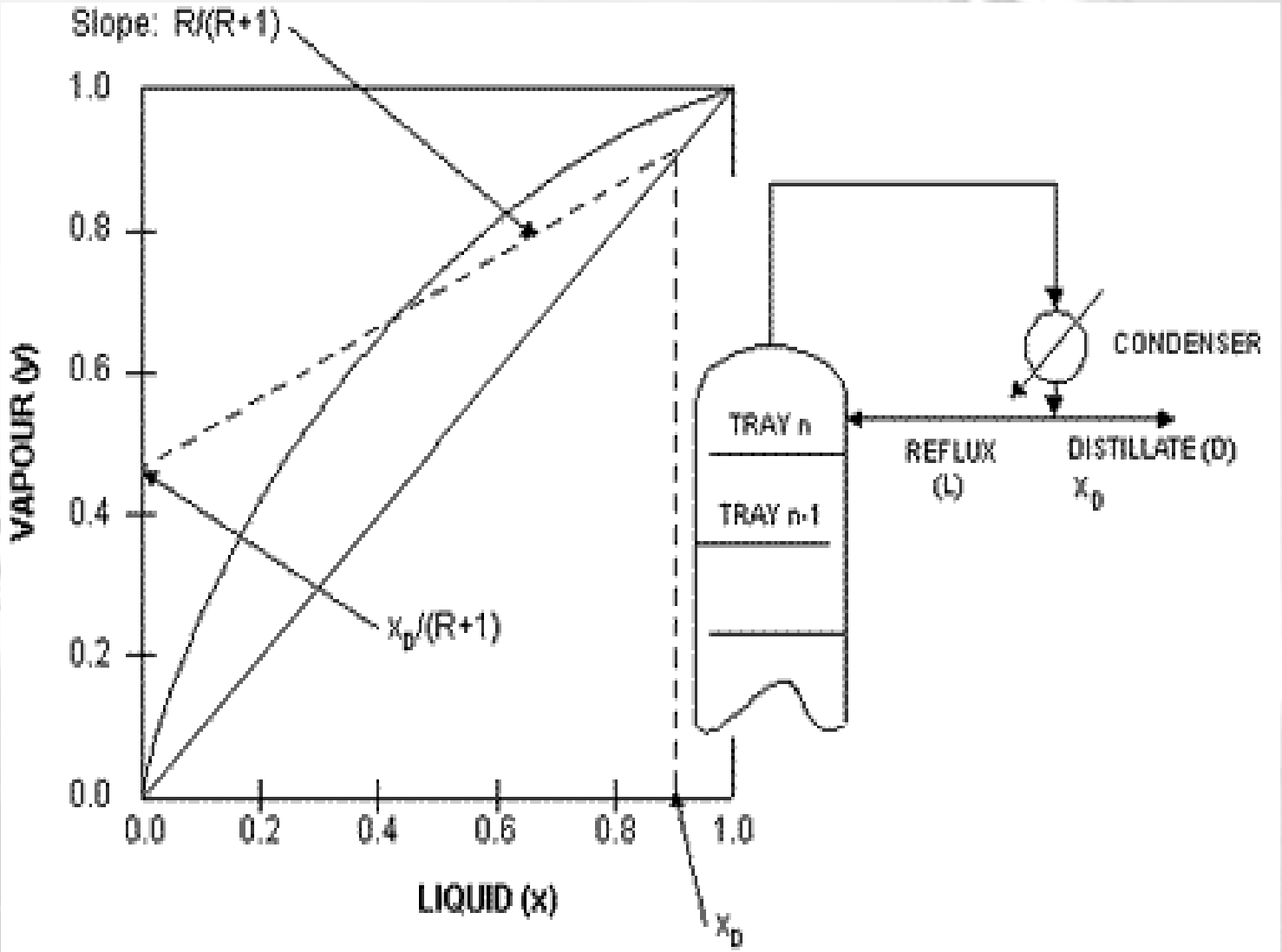
McCabe Thiele Design Method

- Graphical approach
- Uses VLE plot to determine the theoretical number of stages required to effect the separation of a binary mixture
- Assumes constant molar overflow
  - Means amount of liquid and amount of vapour at each stage is the same across the column
  - molal heats of vaporisation of the components are roughly the same
  - heat effects (heats of solution, heat losses to and from column, etc.) are negligible
  - for every mole of vapour condensed, 1 mole of liquid is vaporised
- Only for binary mixtures
- Sometimes need trial and error to get correct conditions

- Start with VLE
- Draw operating lines
  - define the mass balance relationships between the liquid and vapour phases in the column.
  - There is one operating line for the bottom (stripping) section of the column, and on for the top (rectification or enriching) section of the column.
  - Use of the constant molar overflow assumption also ensures the the operating lines are straight lines.

Operating Line for the Rectification Section

- The operating line for the rectification section is constructed as follows.
  - First the desired top product composition is located on the VLE diagram, and a vertical line produced until it intersects the diagonal line that splits the VLE plot in half.
  - A line with slope R/(R+1) (= L/V for this section) is then drawn from this instersection point as shown in the diagram below.
  - R is the ratio of reflux flow (L) to distillate flow (D) and is called the reflux ratio and is a measure of how much of the material going up the top of the column is returned back to the column as reflux.
  - R = L/D

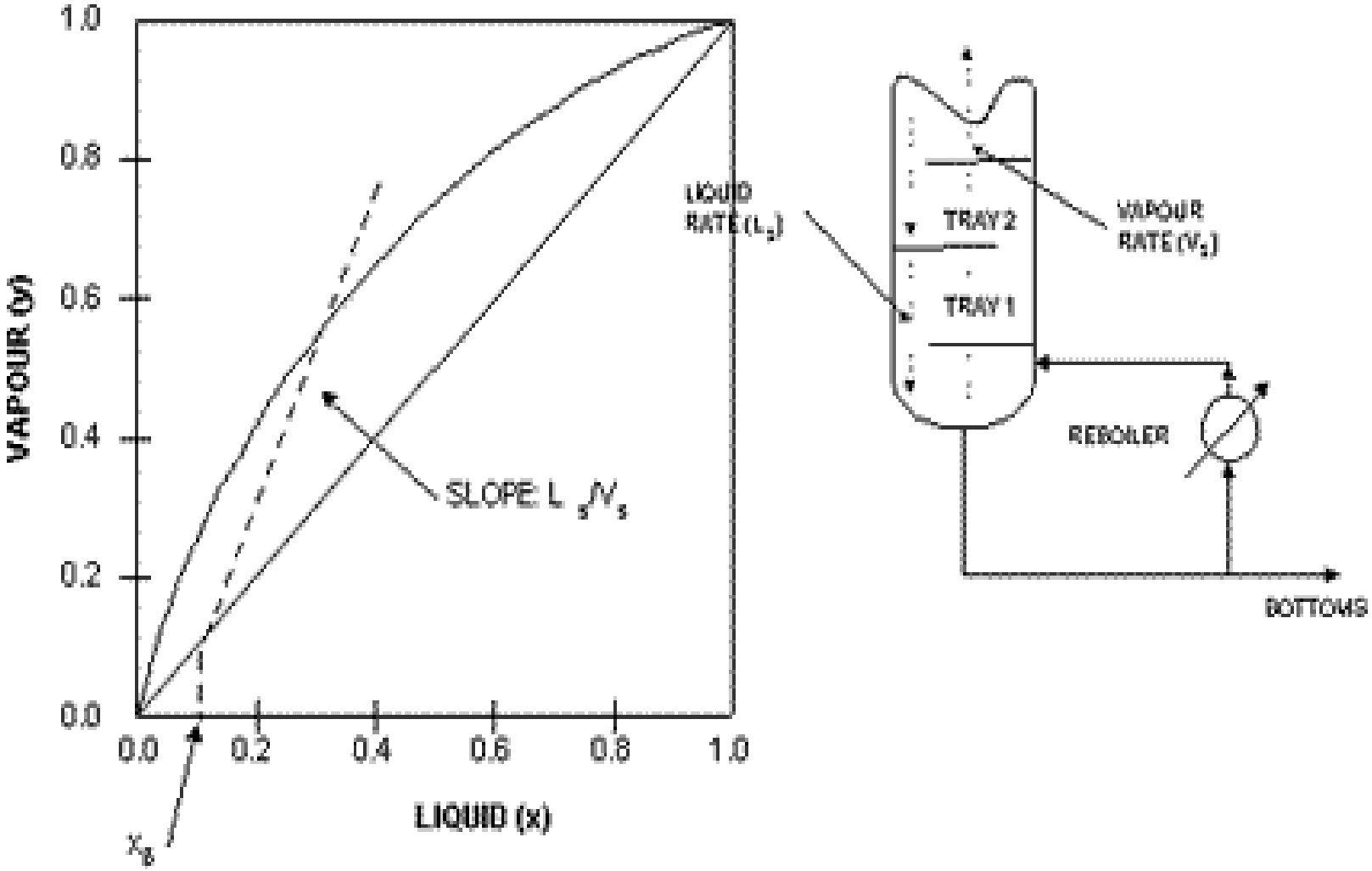

Slope: R/(R+1)
1.0
0.8
0.6
0.4
0.2
0.0
VAPOUR (y)
0.0
0.2
0.4
0.6
0.8
1.0
LIQUID (x)
$x_D/(R+1)$
x_D
TRAY n
TRAY n-1
CONDENSER
REFLUX
(L)
DISTILLATE (D)
x_D

Operating Line for the Stripping Section

- The operating line for the stripping section is constructed in a similar manner.
  - However, the starting point is the desired bottom product composition.
  - A vertical line is drawn from this point to the diagonal line, and a line of slope L_s/V_s is drawn as illustrated in the diagram on next slide.
  - L_s is the liquid rate down the stripping section of the column

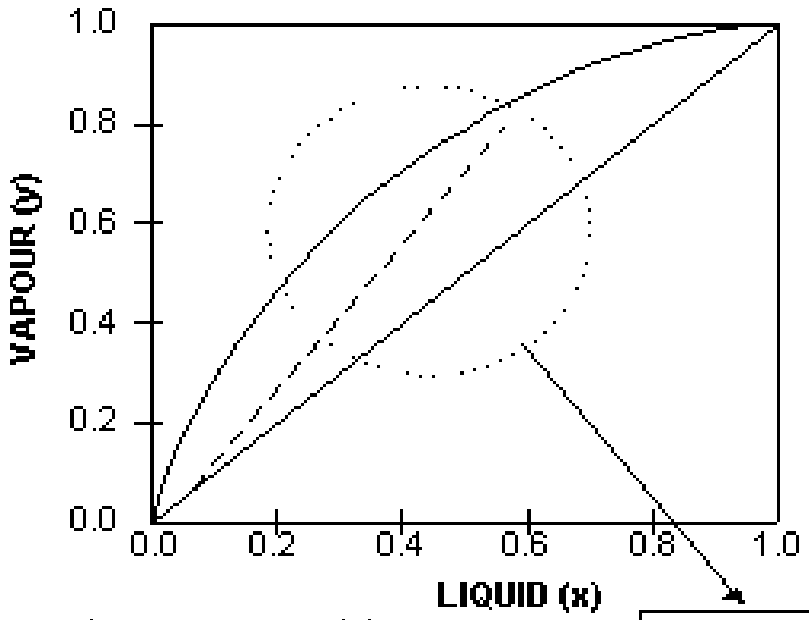
    = (L above feed) + qFeed
  - while V_s is the vapour rate up the stripping section of the column

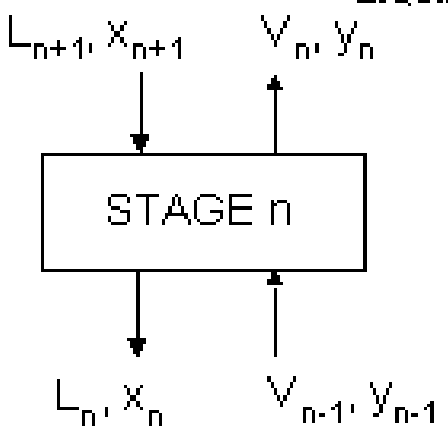
    = (V above feed) – (1-q)Feed
  - Thus the slope of the operating line for the stripping section is a ratio between the liquid and vapour flows in that part of the column.

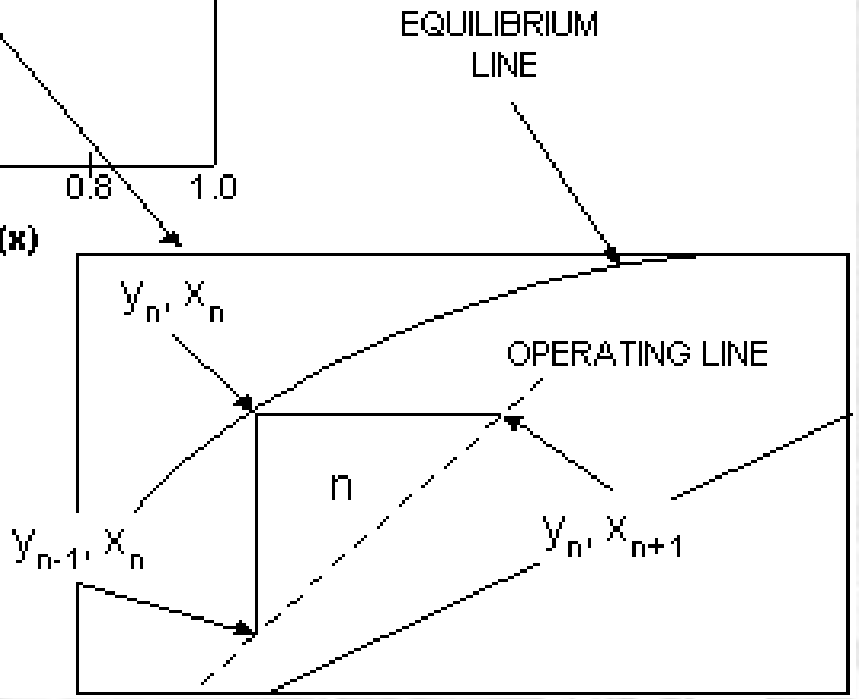

1.0
0.8
0.6
0.4
0.2
0.0
VAPOUR (y)
0.0
0.2
0.4
0.6
0.8
1.0
LIQUID (x)
SLOPE L s/V s
LIQUID RATE (L s)
VAPOUR RATE (V s)
TRAY 2
TRAY 1
REBOILER
BOTTOMS

Equilibrium and Operating Lines

- The McCabe-Thiele method assumes that the liquid on a tray and the vapour above it are in equilibrium
- How this is related to the VLE plot and the operating lines is depicted graphically in the diagram on the right.
- A magnified section of the operating line for the stripping section is shown in relation to the corresponding n'th stage in the column
  - L's are the liquid flows while V's are the vapour flows.
  - x and y denote liquid and vapour compositions and the subscripts denote the origin of the flows or compositions. That is 'n-1' will mean from the stage below stage 'n' while 'n+1' will mean from the stage above stage 'n'.
  - The liquid in stage 'n' and the vapour above it are in equilibrium, therefore, x_n and y_n lie on the equilibrium line.
  - Since the vapour is carried to the tray above without changing composition, this is depicted as a horizontal line on the VLE plot.
  - Its intersection with the operating line will give the composition of the liquid on tray 'n+1' as the operating line defines the material balance on the trays.
  - The composition of the vapour above the 'n+1' tray is obtained from the intersection of the vertical line from this point to the equilibrium line.

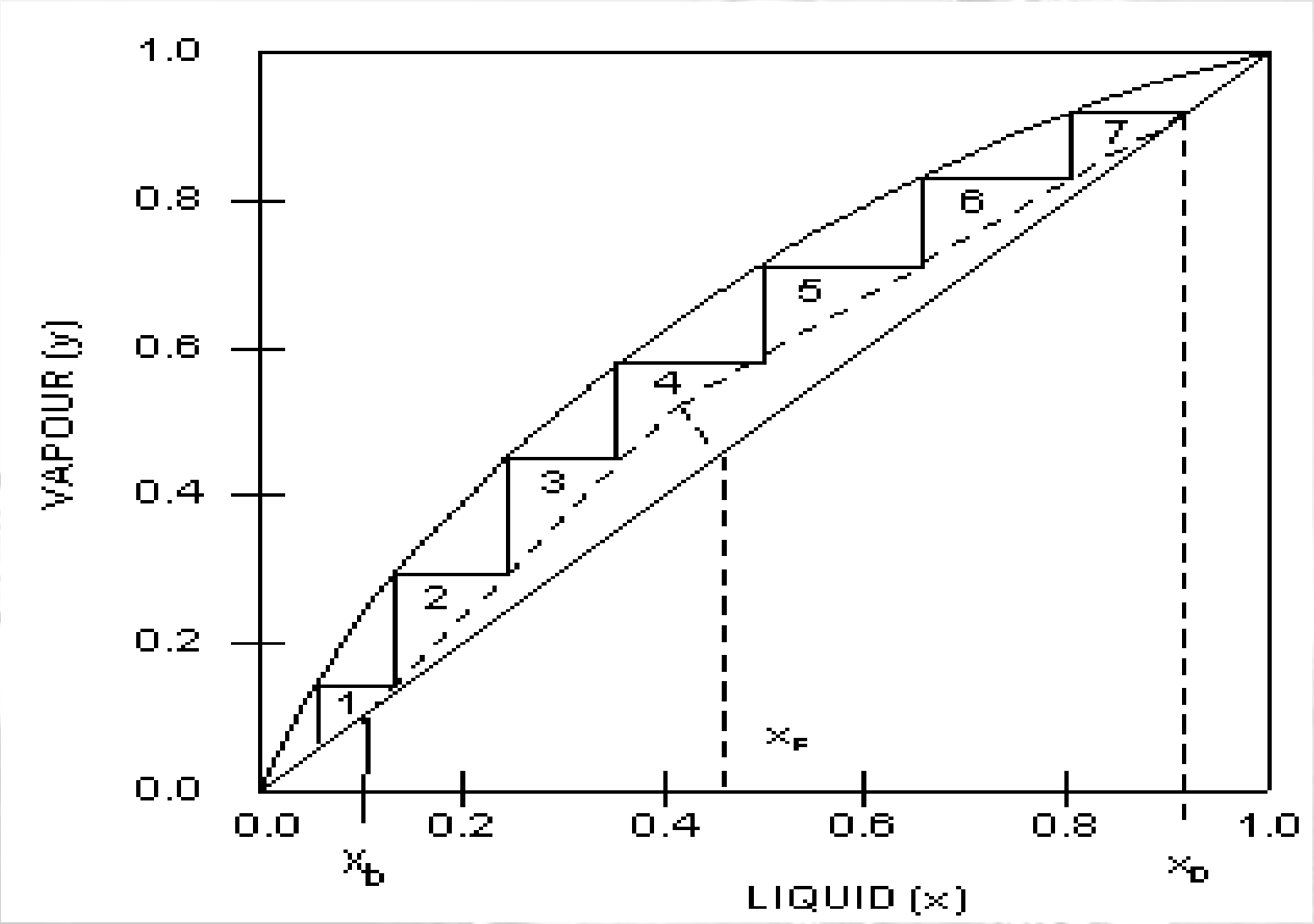

1.0
0.8
0.6
0.4
0.2
0.0
VAPOUR (y)
0.0
0.2
0.4
0.6
0.8
1.0
LIQUID (x)


L_{n+1}, x_{n+1}
V_n, y_n
STAGE n
L_n, x_n
V_{n-1}, y_{n-1}


EQUILIBRIUM
LINE
y_n, x_n
OPERATING LINE
n
y_{n-1}, x_n
y_n, x_{n+1}

Number of Stages and Trays

- Doing the graphical construction repeatedly will give rise to a number of 'corner' sections, and each section will be equivalent to a stage of the distillation.
- This is the basis of sizing distillation columns using the McCabe-Thiele graphical design methodology as shown in the diagram on the next page.
- Given the operating lines for both stripping and rectification sections, the graphical construction described above was applied. This particular example shows that 7 theoretical stages are required to achieve the desired separation.
- The required number of trays (as opposed to stages) is one less than the number of stages since the graphical construction includes the contribution of the reboiler in carrying out the separation.


1.0
0.8
0.6
0.4
0.2
0.0
VAPOUR (y)
0.0
0.2
0.4
0.6
0.8
1.0
LIQUID (x)
1
2
3
4
5
6
7
x_F
x_b
x_D

- The actual number of trays required is given by the formula:

  trays = (number of theoretical trays)/(tray efficiency)

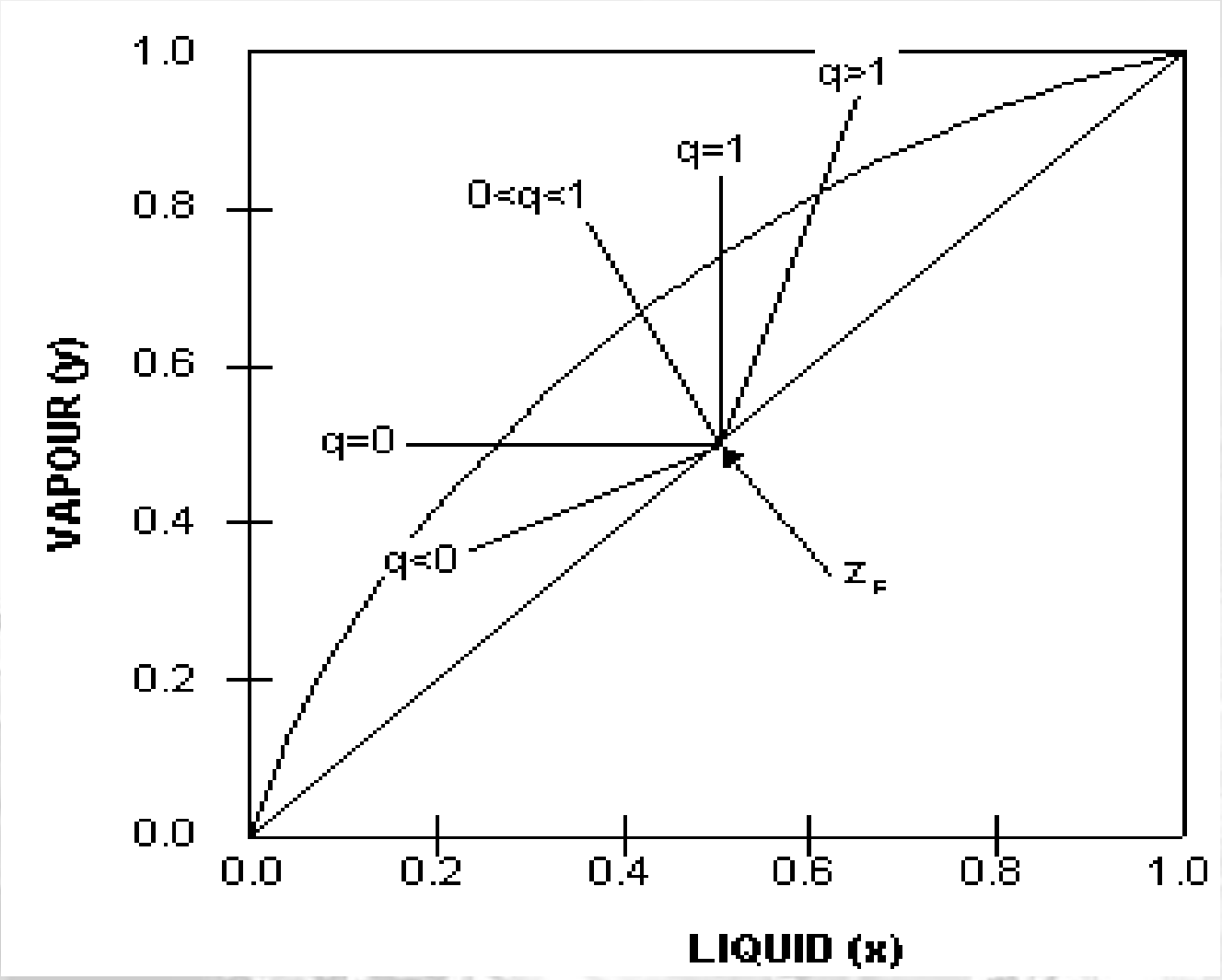
- Typical values for tray efficiency ranges from 0.5 to 0.7 and depends on a number of factors, such as the type of trays being used, and internal liquid and vapour flow conditions. Sometimes, additional trays are added (up to 10%) to accomodate the possibility that the column may be under-designed.

The Feed Line – q line

- The diagram above also shows that the binary feed should be introduced at the 4'th stage.
- However, if the feed composition is such that it does not coincide with the intersection of the operating lines, this means that the feed is not a saturated liquid.
- The condition of the feed can be deduced by the slope of the feed line or q-line.
- The q-line is that drawn between the intersection of the operating lines, and where the feed composition lies on the diagonal line.

Slope of q Line

- Depends on the state of the feed
- q is fraction of feed that is liquid
  - q = 0 (saturated vapour)
  - q = 1 (saturated liquid)
  - 0 < q < 1 (mix of liquid and vapour)
  - q > 1 (subcooled liquid)
  - q < 0 (superheated vapour)
- The q-lines for the various feed conditions are shown in the diagram below



- The slope of the line is given by: $slope = \frac{-q}{1-q}$
- Remember q line can't go below 45^0 line

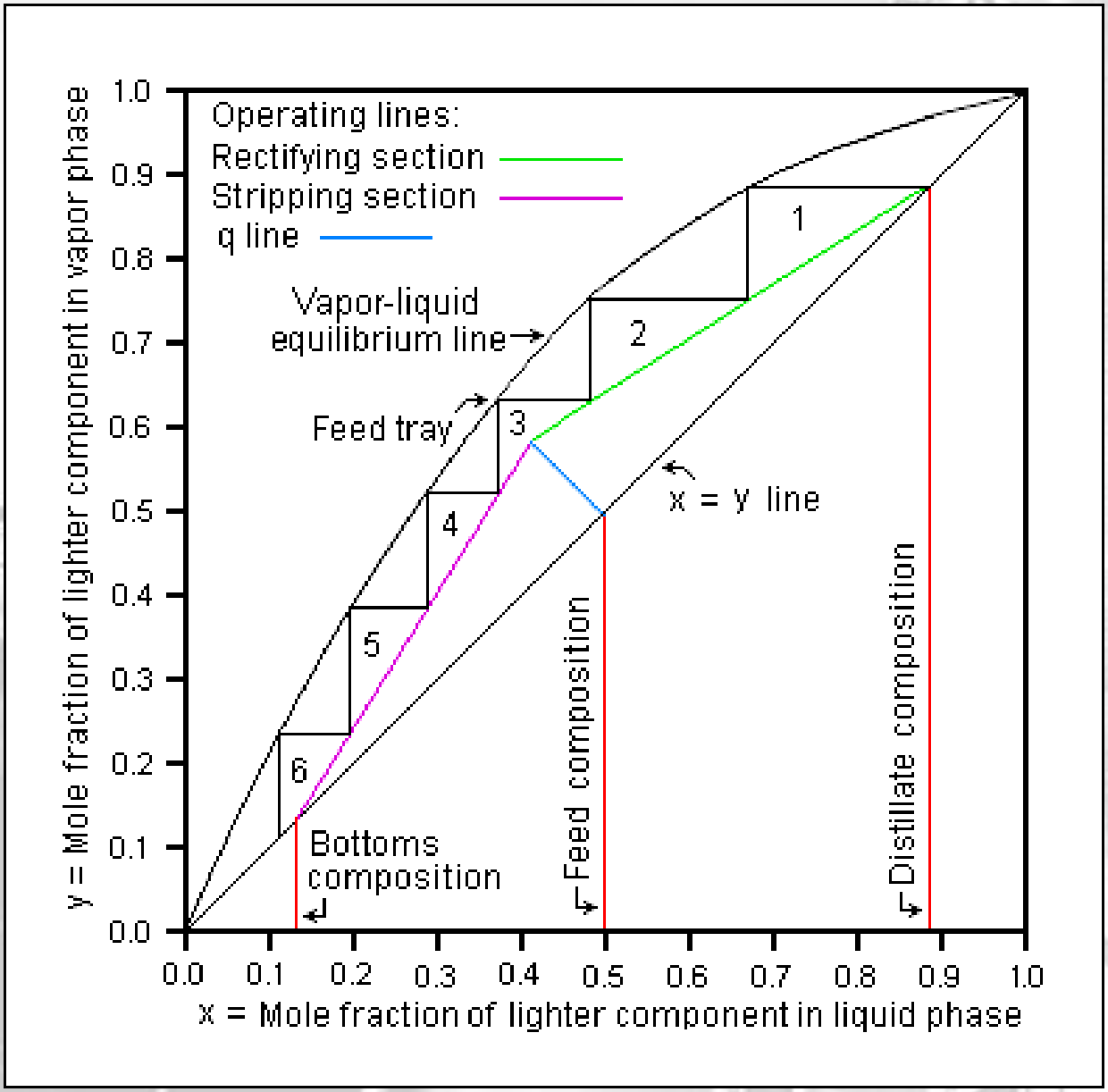
Using Operating Lines and the Feed Line in McCabe-Thiele Design

- If we have information about the condition of the feed mixture, then we can construct the q-line and use it in the McCabe-Thiele design. However, excluding the equilibrium line, only two other pairs of lines can be used in the McCabe-Thiele procedure. These are:
  - feed-line and rectification section operating line
  - feed-line and stripping section operating line
  - stripping and rectification operating lines
- This is because these pairs of lines determine the third.

- Very nice flash demonstration of McCabe Thiele at:
- Lorien.ncl.ac.uk/ming/distil/dist-tut.htm

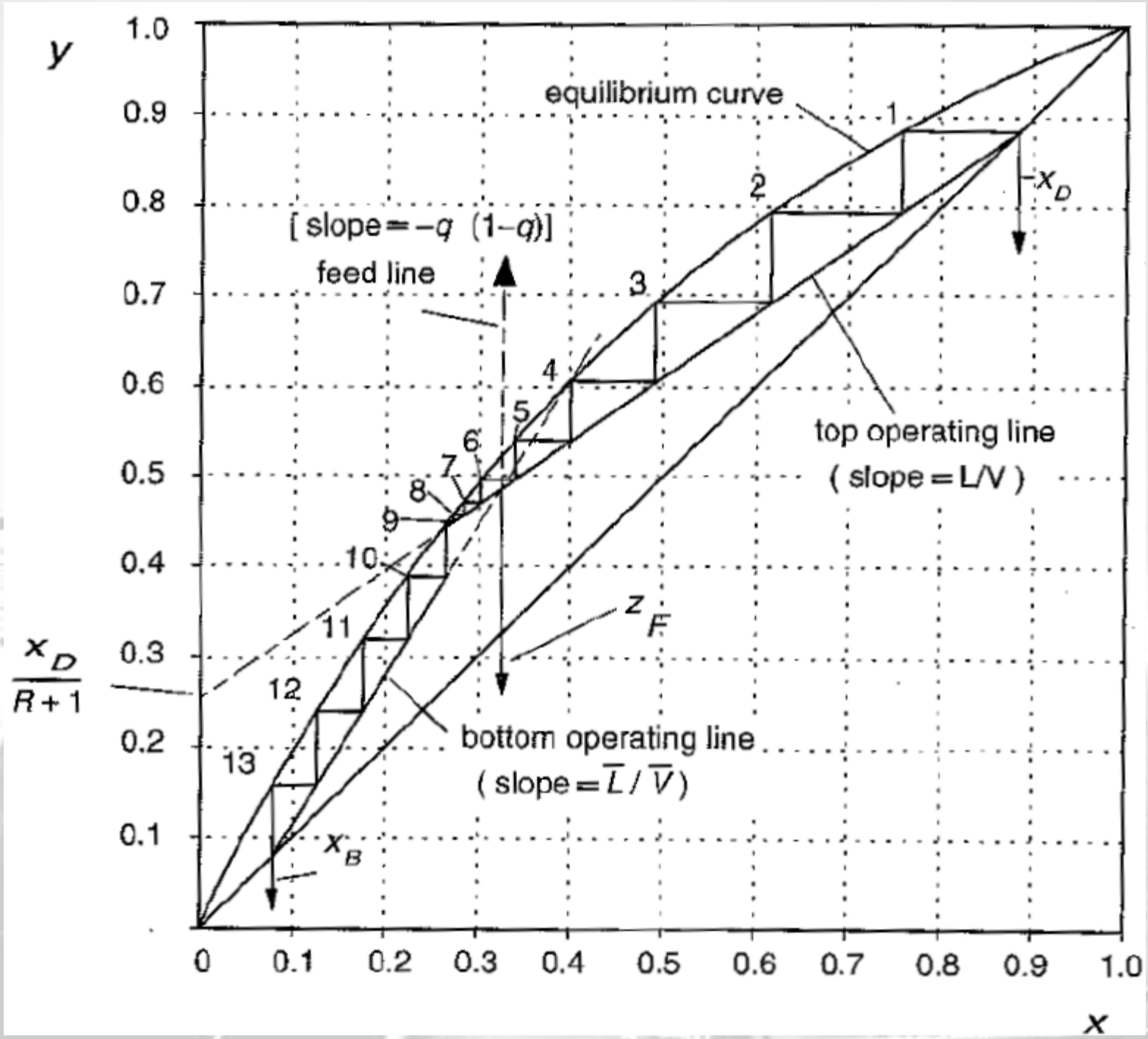
http://lorien.ncl.ac.uk/ming/distil/dist-tut.htm

- Written by Jon Lee of Newcastle University


Operating lines:
Rectifying section
Stripping section
q line
Vapor-liquid equilibrium line
Feed tray
x = y line
Feed composition
Distillate composition
Bottoms composition
1
2
3
4
5
6
y = Mole fraction of lighter component in vapor phase
x = Mole fraction of lighter component in liquid phase
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0

Example Problem

- Benzene Toluene mixture
- Liquid feed stream at 32.1% benzene and 100kmol/hr
- Top product of distillate stream of 88.5% benzene at 30kmol/hr (82.7% of benzene recovered)
- The reflux ratio is 2.38


y
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0
x
equilibrium curve
[slope = −q (1−q)]
feed line
top operating line
(slope = L/V)
bottom operating line
(slope = L̄ / V̄)
x_D
$\frac{x_D}{R+1}$
z_F
x_B
1
2
3
4
5
6
7
8
9
10
11
12
13

How to Solve this Problem

- Rectifying line
  - Line between points (x_D, y_D) and (0, x_D/(R+1))
- Draw feed line until it hits the rectifying line
  - Vertical line from feed composition
- Calculate bottoms composition from material balance
- Stripping line by joining central point at feed composition to ($x_{\mathbf{B}}$, y_B)
- Construct staircase

Bottoms Composition from Material Balance

- $F = D + B$ overall material balance
- $F x_F = Dx_D + Bx_B$ Benzene balance
  - x_F, x_D, and x_B refer to Benzene concentrations

Using 1. we get B = 100 – 30 = 70kmjol/hr

Using 2. we get $x_B = (F x_F = Dx_D)/B$

$=(100*0.321 – 30*0.885)/70$

$= 0.0793$

Means bottoms composition in Benzene is 7.93%

Issues with Column Construction

- stable column operation
  - flooding
  - weeping
  - entrainment
- tray efficiency
- pressure drop across each tray
- tray diameter
- tray separation

Conditions for Stable Operation

- vapor should flow only through the open regions of the tray between the downcomers
- liquid should flow only through the downcomers
  - liquid should not weep through tray perforations
  - liquid should not be carried up the column entrained in the vapor
- vapor should not be carried down the column in the liquid
- vapor should not bubble up through the downcomers

Flooding

- liquid backing up onto the tray
- happens if downcomer area too small for flow rate of liquid
- increasing downcomer size increase cost of column

Weeping

- liquid seeping through the holes in the plates
- shouldn't happen if trays designed correctly

Entrainment

- droplets of liquid being carried up the column in the vapour stream

Tray Efficiency

- column operation assumes vapour liquid equilibrium
- don't get this across whole tray
  - worst beside downcomer
  - best beside weir
- Typical efficiencies are about 50%
- actual number of stages in a column = theoretical number / efficiency

Tray Diameter

- depends on liquid and vapour flow rates through the column
- usually specify it to be 10% to 20% above that needed for normal operation
- need to be able to handle upsets

Tray Separation / Column Height

- typically 0.6m for wide columns
  - space needed for service
  - can be closer for narrow columns
- ~3m of disengaging space at top and bottom
- bottom of tower must be big enough to contain hold-up (say five minutes)
- towers rarely more than 50m tall, rarely tall and skinny
  - can design and split process to give two towers