

Sep-tek.
Ch.11 (continued)
Distillation (Multistage with reflux)
Sigurd Skogestad

Distillation

- Separation of liquid mixtures by repeated evaporation
 - multi-stage with reflux
 - Old name: "Rectification"
- Basis: Components have different boiling points (different volatility)
- Most common separation process in industry
 - Chemicals
 - Oil and gas
- "Separating agent" = heat (thermal energy)
- 3% of world energy consumption is for distillation

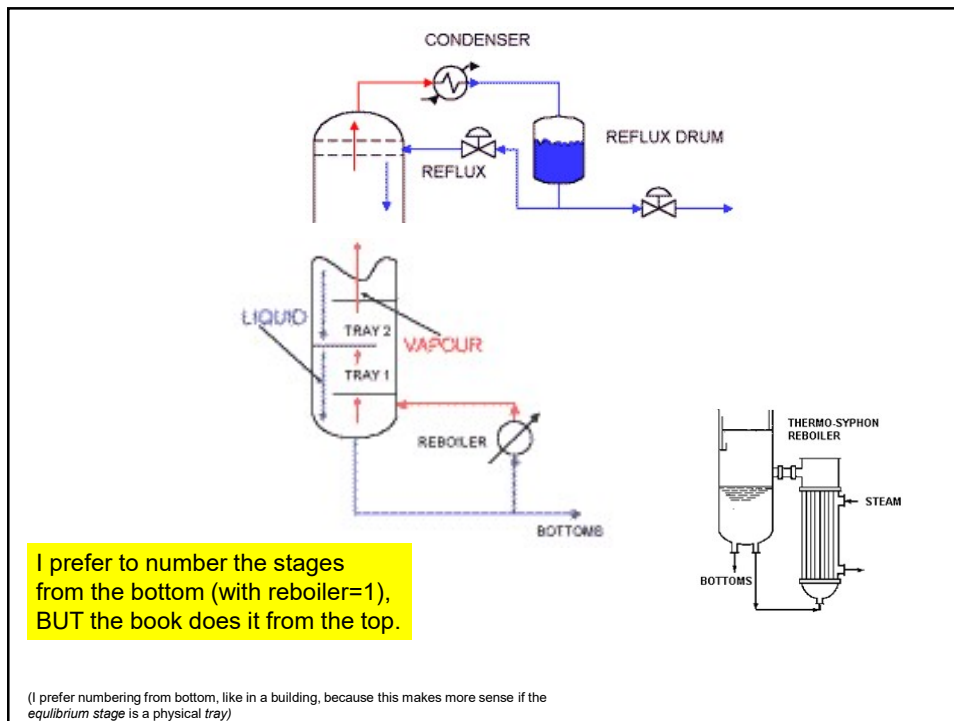
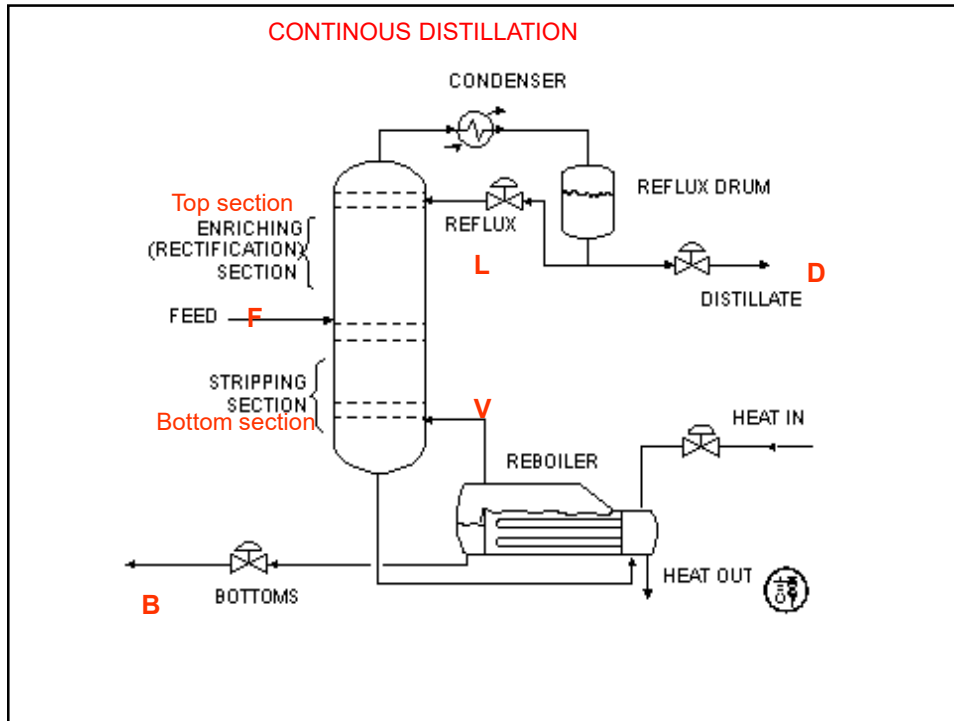


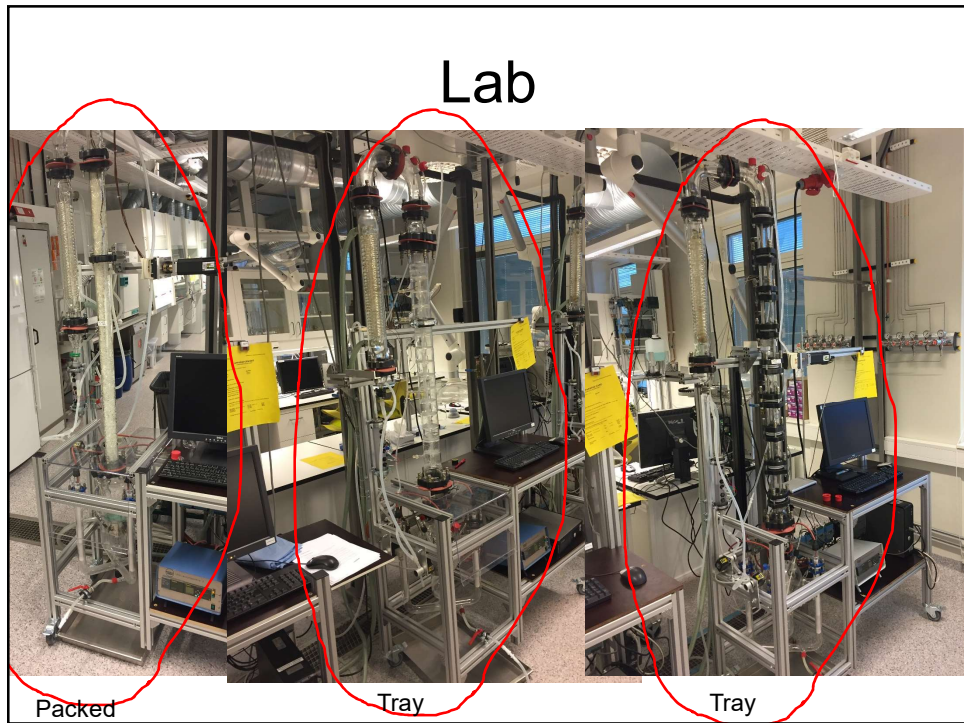
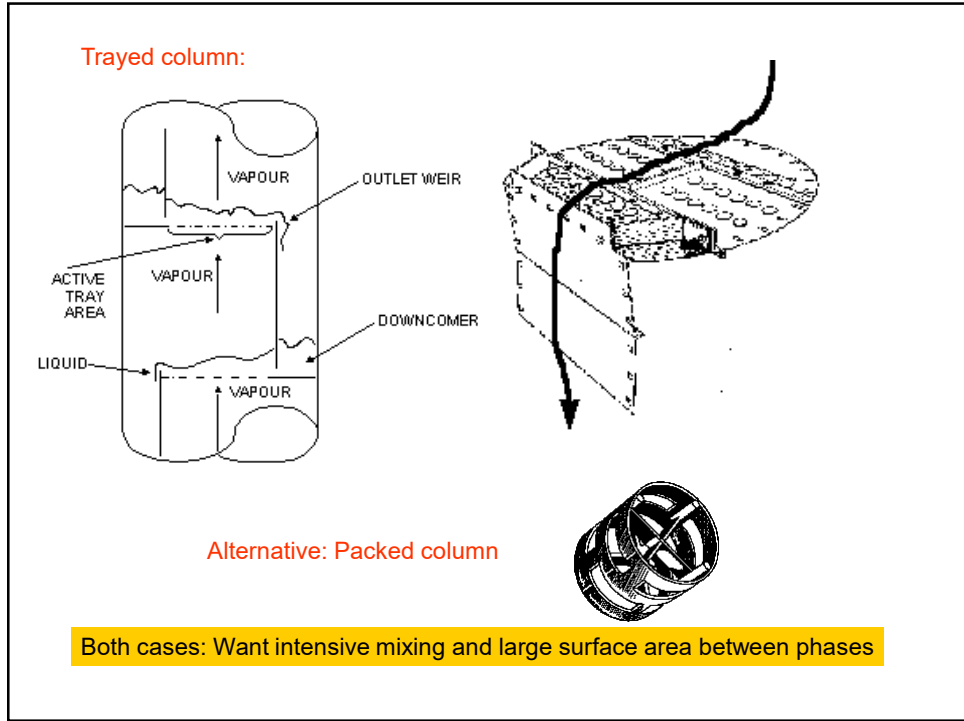
When use distillation?

- Liquid mixtures (with difference in boiling point)
- Unbeatable for high-purity separations because
 - Essentially same energy usage independent of (im)purity!
 - » Going from 1% to 0.0001% (1 ppm) impurity in one product increases energy usage only by about 1%
 - Number of stages increases only as log of impurity!
 - » Going from 1% to 0.0001% (1 ppm) impurity in one product increases required number of stages only by factor 3 (Proof using Fenske: $\ln(1.e-6)/\ln(1.e-2)=3$)
- Well suited for scale-up
 - » Columns with diameters over 18 m
- Examples of unlikely uses of distillation:
 - » High-purity silicon for computers (via SiCl_3 distillation)
 - » Water – heavy-water separation (boiling point difference only 1.4C)

Sigurd and distillation

- 1980-83 (Norsk Hydro): Steady-state design and simulation
 - Crude oil, Mongstad refinery
 - Petrochemicals, VCM
 - Methanol-water, formic acid-water
 - Mostly thermodynamics
- 1983-rest of life: Control and dynamics
- 1995-2000: Optimal batch (multivessel)
- 1997-present: Optimal integrated continuous distillation columns (Petlyuk, Kaibel)
- <http://www.nt.ntnu.no/users/skoge/distillation/>

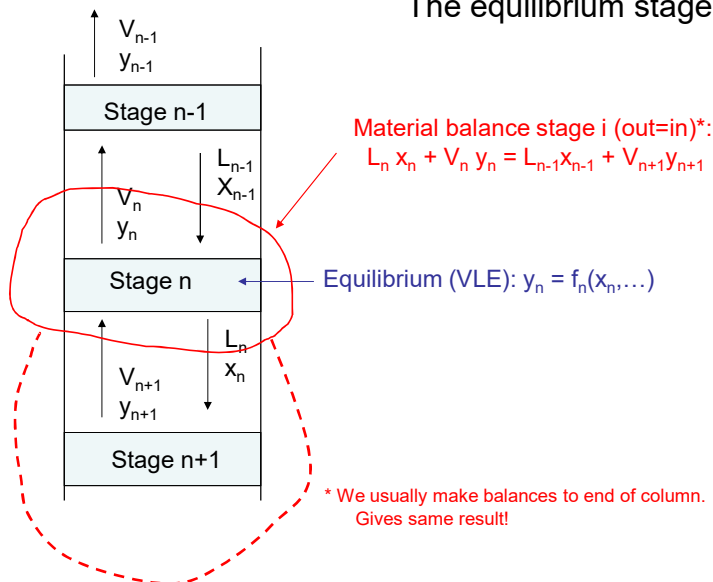




Distillation column modeling

- Equilibrium stage concept: Many flashes on top of each other!
 - Each stage is an adiabatic flash:
 - Component balance
 - Energy balance
 - Vapor-liquid equilibrium (VLE), $y = K x$
1. Detailed numerical solution is straightforward but requires computer
 - Commercial: Unisim / Hysys, Aspen
 - Free software (very nice!): ChemSep.org
 2. Analytical solution
 - Possible in certain cases
 - Most common assumption: **Constant molar flows** (simplified energy balance)
 - Constant K (VLE) + **constant molar flows**: Kremser-equations
 - Constant relative volatility (VLE) + **constant molar flows**: Fenske, Underwood.
 3. Graphical solution
 - Graphical solution of component balances + VLE
 - Any VLE + **Constant molar flows**: McCabe-Thiele

The equilibrium stage concept



Note: Numbering is here from top (as in book).

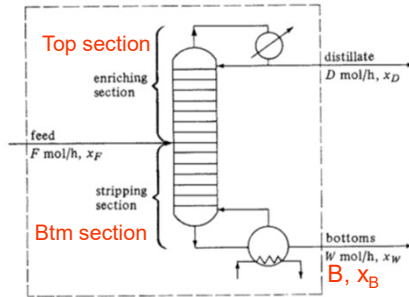


FIGURE 11.4-3. Distillation column showing material-balance sections for McCabe-Thiele method.

1. Material balances overall column ("In=Out").
Total

$$F = D + B$$

Component

$$Fx_F = Dx_D + Bx_B$$

Top operating line:
y and x
between stages
in top section

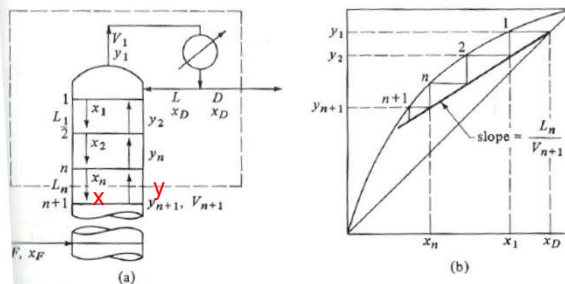


FIGURE 11.4-4. Material balance and operating line for enriching section: (a) schematic of tower, (b) operating and equilibrium lines.

2. Material balances top section ("In=Out").

Total:

$$V_{n+1} = L_T + D$$

Component:

$$V_{n+1}y_{n+1} = L_n x_n + Dx_D \Rightarrow y_{n+1} = \frac{L_n}{V_{n+1}} x_n + \frac{Dx_D}{V_{n+1}}$$

Top operating line. Goes through point (x_D, x_D) and has slope $L_T/V_T < 1$.

Proof: $x = x_D$ gives: $y = (L_T + D)x_D/V_T = x_D$

Bottom operating line:
y and x
between stages
in bottom section

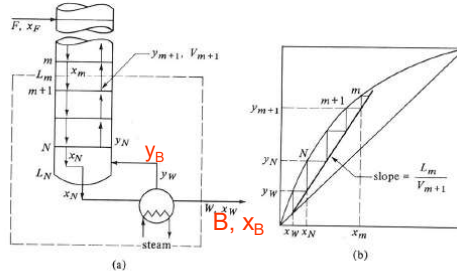


FIGURE 11.4-5. Material balance and operating line for stripping section: (a) schematic of tower, (b) operating and equilibrium lines.

3. Material balances bottom section ("In=Out").

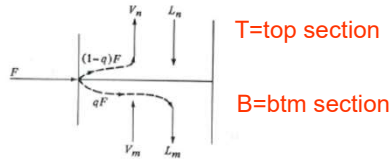
Total: $L_m = V_{m+1} + B$

Component: $L_m x_m = V_{m+1} y_{m+1} + B x_B \Rightarrow y_{m+1} = \frac{L_m}{V_{m+1}} x_m - \frac{B x_B}{V_{m+1}}$

Bottom operating line. Goes through point (x_B, x_B) and has slope $L_B/V_B > 1$.

y and x for
intersection of
operating lines

FIGURE 11.4-6. Relationship between flows above and below the feed entrance.



$$L_B = L_T + qF$$

$$V_T = V_B + (1-q)F$$

q = Fraction of liquid in feed

4. Feed line (crossing of operating lines):

$$y = \frac{q}{q-1} x - \frac{x_F}{q-1}$$

Proof:

Top: $V_T y = L_T x + D x_D$

Bottom: $V_B y = L_B x - B x_B$

At crossing of these lines, y and x are same. Subtract:

$$y(V_T - V_B) = x(L_T - L_B) + F x_F$$

$$\Rightarrow y = \frac{-q}{1-q} x + \frac{x_F}{1-q} \text{ (feed line)}$$

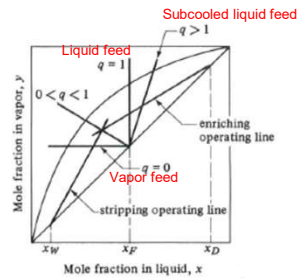


FIGURE 11.4-7. Location of the q line for various feed conditions: liquid below boiling point ($q > 1$), liquid at boiling point ($q = 0$), liquid + vapor ($0 < q < 1$), saturated vapor ($q = 0$).

Usually: Constant molar flows assumption

- Each section: Constant molar flows of liquid L and vapor V:
 - $V_T = V_1 = V_2 = \dots = V_n = V_{n+1}$
 - $L_T = L_1 = L_2 = \dots = L_n = L_{n+1} = L$
 - $V_B = V_N = V_m = V_{m+1}$
 - $L_B = L_N = L_m = L_{m+1}$
- Replaces energy balance
 - Holds for components with similar heat of vaporization

Summary operating lines for Constant molar flows

"Operating line" = Relationship between y and x between stages (from material balance).

Top section: $y = \frac{L_T}{V_T}x + \frac{D}{V_T}x_D$

Goes through $(y,x)=(x_D,x_D)$ + $(y,x)=(Dx_D/V_T,0)=(x_D/(R+1),0)$

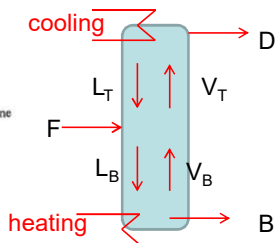
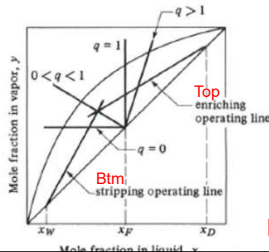
Btm section: $y = \frac{L_B}{V_B}x - \frac{B}{V_B}x_B$

Goes through $(y,x)=(x_B,x_B)$ + crossing of top/feed-lines

Crossing (feed line): $y = \frac{q}{q-1}x - \frac{x_F}{q-1}$

Goes through $(y,x)=(x_F,x_F)$ + has slope $q/(q-1)$

Total balance at feed: $L_B = L_T + qF$, $V_T = V_B + (1-q)F$



q = Fraction liquid in feed

McCabe-Thiele graphical method

Example: Benzene-Toluene continuous distillation

- $F = 100$ kmol/h
 - $x_F = 0.45$, $q_F = 1.19$ (subcooled liquid)
 - Desired products: $x_D = 0.95$, $x_B = 0.1$
 - Given: $R = L_T/D = 4$
 - How many stages are required?
-
- x, y – mole fractions benzene (light component)

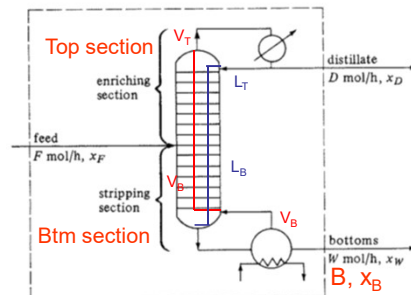


FIGURE 11.4-3. Distillation column showing material-balance sections for McCabe-Thiele method.

Material balances overall column ("In=Out").

$$\text{Total: } F = D + B$$

$$\text{Component: } Fx_F = Dx_D + Bx_B$$

$$\Rightarrow 100 \cdot 0.45 = D \cdot 0.95 + (100 - D) \cdot 0.1$$

$$\Rightarrow D = 41.17 \text{ [kmol/h]}$$

Other flows:

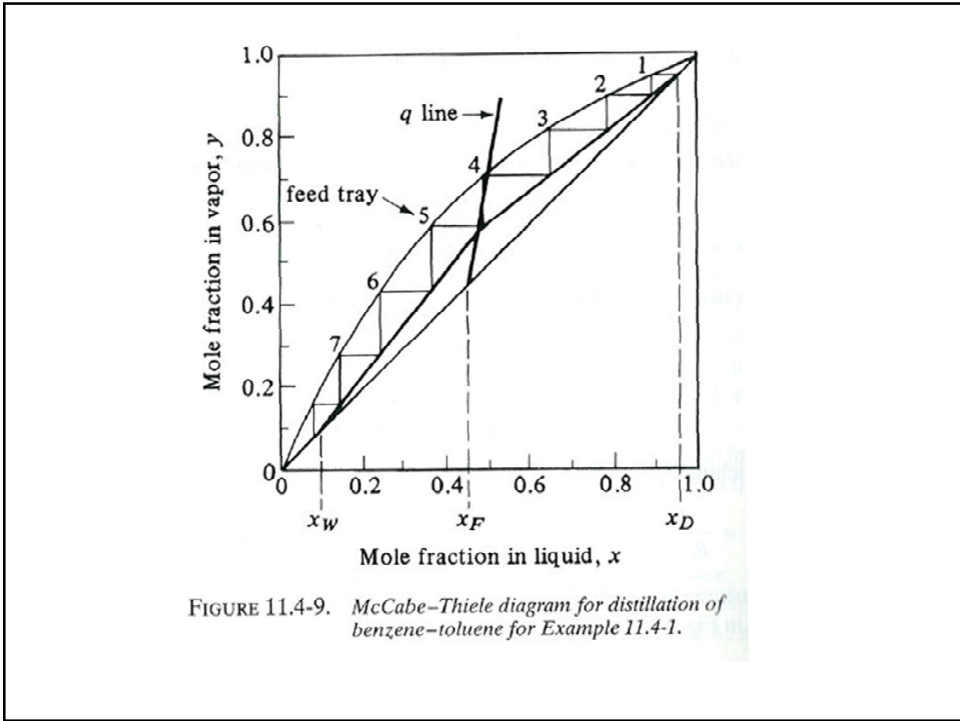
$$B = F - D = 100 - 41.17 = 58.83 \text{ [kmol/h]}$$

$$L_T = R \cdot D = 4 \cdot 41.17 = 164.68$$

$$V_T = L_T + D = 205.85$$

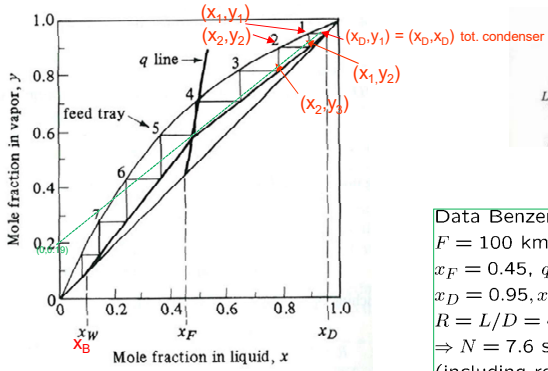
$$L_B = L_T + q \cdot F = 164.68 + 1.19 \cdot 100 = 283.68$$

$$V_B = L_B - B = 224.85$$



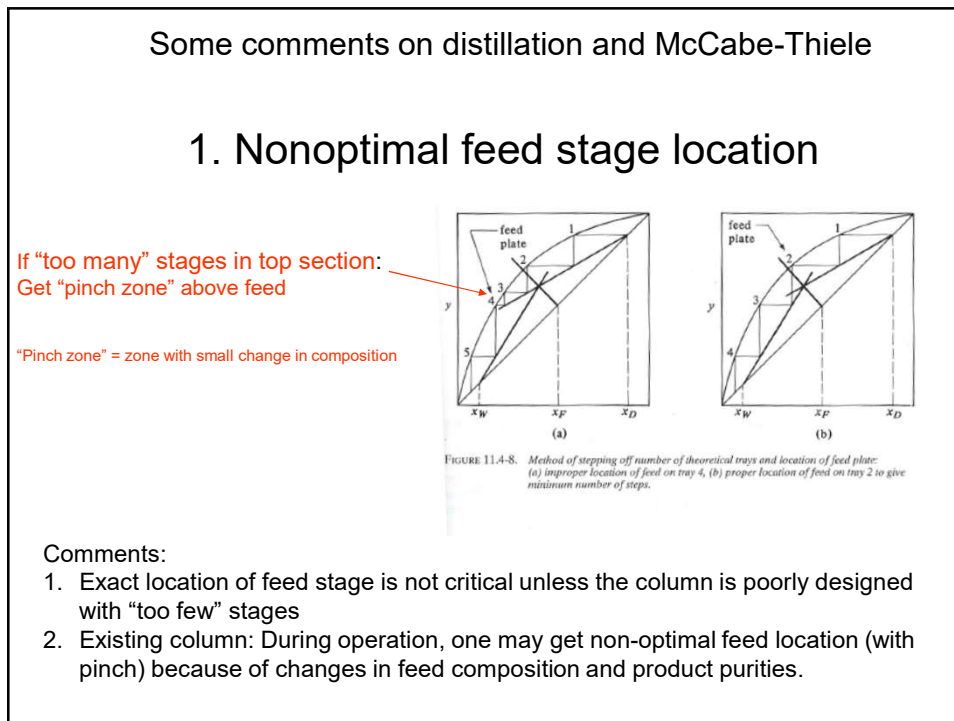
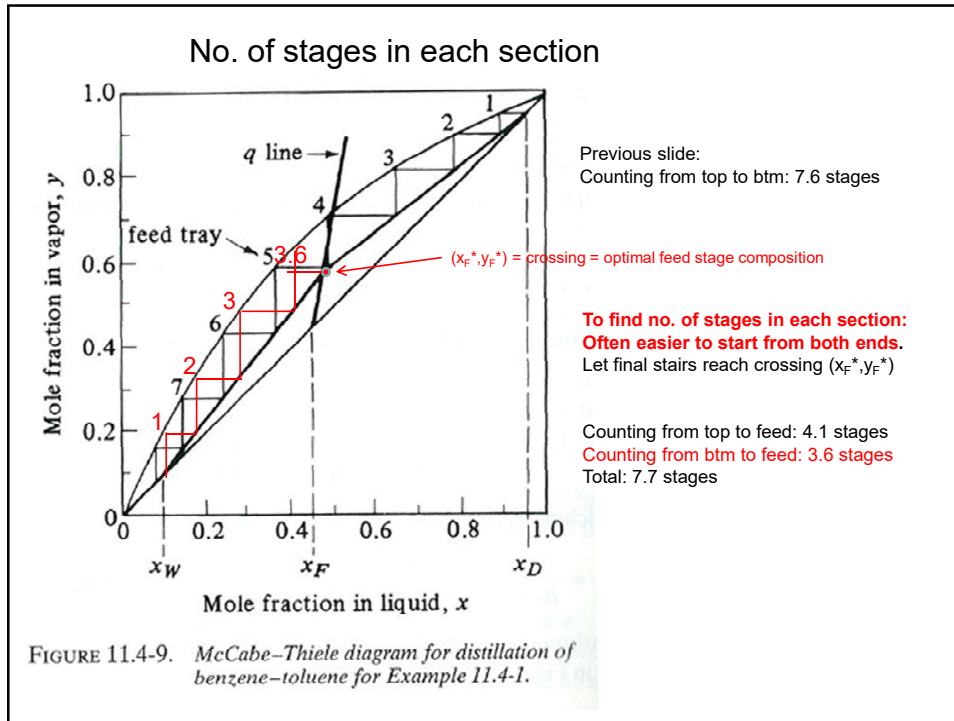
McCabe-Thiele graphical solution

0. Use overall mass balances to find product compositions, product flows and flows inside column.
1. Draw equilibrium curve: (x,y) on stages.
2. Draw upper operating line: (x,y) between stages. Starts in (x_D, x_D) and has slope $L/V_1 = R/(R+1)$ where $R=L/D$. Goes through $(x,y)=(0, x_D/(R+1))=(0, 0.19)$
3. Draw feed lines (goes through (x_F, x_F) and has slope $-q/(1-q)$.
 - Feed sat. liquid ($q=1$): Vertical.
 - Feed sat. vapor ($q=0$): Horizontal
 - Partly vapor ($0 < q < 1$): Between (negative slope)
 - Feed subcooled ($q > 1$): Positive slope (see Figure below)
4. Draw lower operating line: (x,y) between stages. Starts in (x_B, x_B) and goes through crossing of feed line and upper operating line
5. Graphical solution to find number of stages: Start from product and switch between on stages (x_n, y_n) and between stages (x_{n+1}, y_{n+1}) by making staircase
6. Optimal feed location (design): Switch between operating lines at feed line (=crossing of operating lines)
7. Given N and N_p (more difficult): Need to guess product composition and iterate to fit in N stages



Data Benzene-Toluene:
 $F = 100 \text{ kmol/h}$
 $x_F = 0.45, q = 1.19$
 $x_D = 0.95, x_B = 0.1$
 $R = L/D = 4$
 $\Rightarrow N = 7.6 \text{ stages}$
 (including reboiler)

FIGURE 11.4-9. McCabe-Thiele diagram for distillation of benzene-toluene for Example 11.4-1.
 Note: This feed (q) line is for subcooled liquid feed ($q > 1$)



2. Reboiler and condenser duty

- Reflux: $L = L_T$
- Assuming constant molar flows:

$$L_B = L + qF$$

$$V_B = L_B - B = L + qF - B$$

$$V_T = V_B + (1-q)F = L + D$$
- Reboiler and condenser duty
 - $Q_B \text{ [J/s]} = V_B \text{ [mol/s]} \cdot \Delta H_B^{\text{vap}} \text{ [J/mol]}$
 - $Q_C \text{ [J/s]} = V_T \text{ [mol/s]} \cdot \Delta H_D^{\text{vap}} \text{ [J/mol]}$ (total condenser)
 - Note: If feed is liquid & total condenser: $Q_B \approx Q_C$.

Example: $L=L_T=(L/D)*D=4*41.17 = 164.68 \text{ kmol/h}$,
 $V_B = L + qF - B = 164.68 + 1.19*100 - 58.83 = 224.85 \text{ kmol/h}$
 $Q_B = V_B \cdot \Delta H_B^{\text{vap}} = 224.85*(1000/3600)*32.1*1000 \text{ J/s} = 2.0 \text{ e6 J/s} = 2.0 \text{ MW}$

3. Minimum number of stages, N_{\min} (with total reflux, that is, infinite energy)

A. Graphical

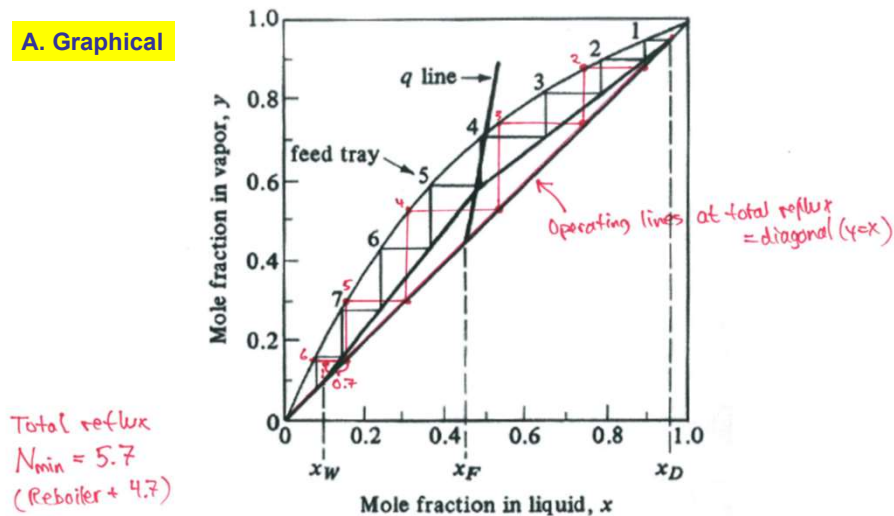
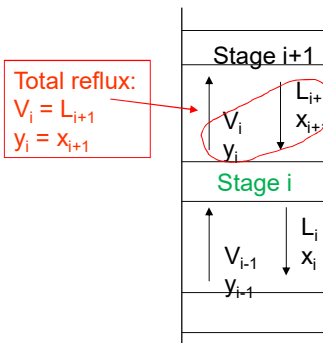


FIGURE 11.4-9. McCabe-Thiele diagram for distillation of benzene-toluene for Example 11.4-1.

3. Minimum no. of stages, N_{\min} (with total reflux)

B. Analytical (constant α)



- Assumption VLE: Constant relative volatility.

Stage i (L=light component, H = heavy component):

$$\alpha = \frac{y_{L,i}/x_{L,i}}{y_{H,i}/x_{H,i}} = \frac{x_{L,i+1}/x_{L,i}}{x_{H,i+1}/x_{H,i}} = \frac{x_{L,i+1}/x_{H,i+1}}{x_{L,i}/x_{H,i}}$$

- Repeat for all N stages

$$\alpha^N = \frac{(x_L/x_H)_D}{(x_L/x_H)_B} \equiv S \text{ (Separation factor)}$$

- Fenske's formula for minimum no. of stages

$$N_{\min} = \ln S / \ln \alpha$$

- Applies also to column sections
- Example. Benzene-Toluene.

$$\alpha = \sqrt{2.38 \cdot 2.54} = 2.46.$$

$$N_{\min} = \frac{\ln \frac{0.95/0.05}{0.1/0.9}}{\ln 2.46} = \frac{5.14}{0.90} = 5.71$$

(almost same as found graphically!)

Number of stages increases only as log of impurity!

$$N_{\min} = \ln S / \ln \alpha \quad S = \frac{(x_L/x_H)_{\text{top}}}{(x_L/x_H)_{\text{btm}}}$$

- Example:** $\alpha=2$. Binary separation with purities: 90% light in top and 90% heavy in bottom:

$$S = \frac{0.9/0.1}{0.1/0.9} = \frac{0.9 \cdot 0.9}{0.1 \cdot 0.1} = 81; \ln S = 4.4$$

$$\Rightarrow N_{\min} = 4.4 / \ln 2 = 6.3$$

- Example:** $\alpha=2$. Binary separation with purities: 99.9% light in top and 98% heavy in bottom:

$$S = \frac{0.999/0.001}{0.02/0.98} = 48951; \ln S = 10.8$$

$$\Rightarrow N_{\min} = 10.8 / \ln 2 = 15.6$$

Comments

Number of stages

- Reboiler ("partial reboiler"): Gives 1 theoretical stage
- Total condenser: 0 stage
- Partial condenser: 1 theoretical stage
- N is usually (e.g., in Fenske's formula, $N = \ln S / \ln \alpha$) the total no. of theoretical stages including reboiler and partial condenser
- Thus: $N_{\text{stages inside column}} = N - 1$ (or $N - 2$ for partial condenser)

"Total reflux" vs. "Total condenser"

We use "total" with (at least) two different meanings:

1. "Total condenser" = all is condensed (so D is liquid)
2. "Total reflux" = (all is condensed and) all is sent back as reflux (so $D=0$ or more generally $L \gg D$)

4. Minimum reflux (minimum energy) (with infinite number of stages)

A. Graphical

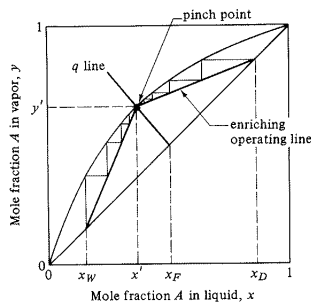
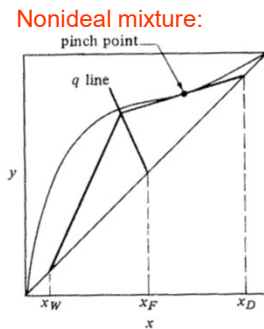


FIGURE 11.4-11. Minimum reflux ratio and infinite number of trays by McCabe-Thiele method.

FIGURE 11.4-12. Minimum reflux ratio and infinite number of trays when operating line is tangent to equilibrium line.



Nonideal mixture:

Find upper (enriching) line that gives pinch:

$$\text{Slope top oper. line} = \left(\frac{L}{V}\right)_{\min} = \frac{L_{\min}}{L_{\min} + D} = \frac{R_{\min}}{R_{\min} + 1}$$

$$\text{or } R_{\min} = \frac{L_{\min}}{D} = \frac{(L/V)_{\min}}{1 - (L/V)_{\min}}$$

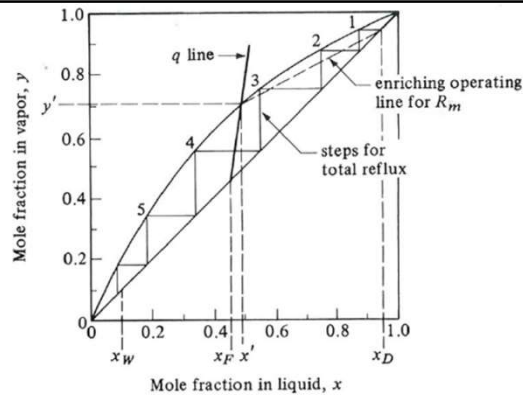


FIGURE 11.4-13. Graphical solution for minimum reflux ratio R_m and total reflux for Example 11.4-2.

$$\text{Slope} = \left(\frac{L}{V}\right)_{\min} = \frac{x_D - y'}{x_D - x'} = \frac{0.95 - 0.702}{0.95 - 0.49} = 0.539$$

$$R_{\min} = \frac{L_{\min}}{D} = \frac{0.539}{1 - 0.539} = 1.17$$

$$L_{\min} = 1.17 \cdot D = 1.17 \cdot 41.17 = 48.16 \text{ kmol/h}$$

$$\text{Minimum boilup: } V_{\min} = L_{\min} + qF - B \text{ (assuming constant molar flows)}$$

$$= 48.16 + 1.193 \cdot 100 - 58.82 = 108.65 \text{ kmol/h}$$

$$\text{Minimum energy: } Q_{\min} = V_{\min} \cdot \Delta H^{\text{vap}} = 108.65 \text{ kmol/h} \cdot 32.099 \text{ kJ/mol} = 3487 \text{ MJ/h} = 0.97 \text{ MW}$$

NOT IN BOOK

4. Minimum reflux (minimum energy) (with infinite number of stages)

B. Analytical (constant α)

- King's formula, assuming pinch at feed (not in book):

$$L_{\min} = \frac{(r_L^D - r_H^D)}{\alpha - 1} \cdot F + (1 - q)B$$

$$r_L^D = Dx_{D,L} / Fz_{F,L} \text{ - recovery of L in top } (\approx 1)$$

$$r_H^D = Dx_{D,H} / Fz_{F,H} \text{ - recovery of H in top } (\approx 0)$$

Example benzene-toluene

$$r_L^D = 39.11/45 = 0.869$$

$$r_H^D = 2.06/55 = 0.037$$

$$L_{\min} = \frac{0.869 - 0.037}{2.46 - 1} \cdot 100 + 58.82(1 - 1.19) =$$

$$56.99 - 11.35 = 45.634 \text{ kmol/h}$$

- Note: Essentially same energy usage independent of (im)purity!

Pure products:

$$L_{\min} = \frac{F}{\alpha - 1} + (1 - q)B$$

$$V_{\min} = \frac{F}{\alpha - 1} + qD$$

Example benzene-toluene

$$L_{\min} = \frac{100}{2.46 - 1} + (1 - 1.19) \cdot 58.82 = 57.31$$

$$V_{\min} = \frac{100}{2.46 - 1} + 1.19 \cdot 41.18 = 117.5 \text{ kmol/h}$$

Easy to remember!

Proof of last formula is just algebra.

From mass balance: $\frac{D}{F} = \frac{z_F - x_B}{x_D - x_B}$

Minimum flows: $\frac{L}{V} = \frac{x_D - y'}{x_D - x'}$

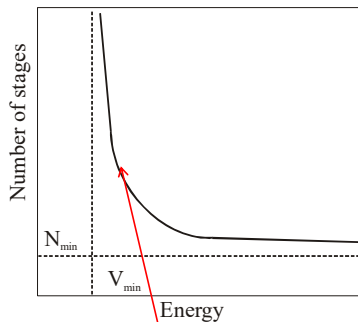
Gives: $\frac{L}{D} = \frac{L}{V-L} = \frac{L/V}{1-L/V} = \frac{x_D - y'}{y' - x'}$

Introducing pure products ($x_D = 1, x_B = 0$) and feed liquid:

$z_F = x_F, x' = x_F, y' = y_F = \frac{\alpha x_F}{1 + (\alpha - 1)x_F}$

gives: $\frac{L}{F} = \frac{L}{D} \cdot \frac{D}{F} = \frac{1 - y_F}{y_F - x_F} x_F = \frac{1 - y_F}{(y_F/x_F) - 1} = \frac{1}{\alpha - 1}$

5. Design: "Optimum" reflux or How many stages (N)?



5A. Energy (V) vs. number of stages (N)

- Trade-off between number of stages and energy

- Actual V approaches V_{\min} for N approximately $2 \times N_{\min}$ or larger, typically:

$$N = 2N_{\min} \rightarrow V_{\min} + 25\%$$

$$N = 3N_{\min} \rightarrow V_{\min} + 3\%$$

$$N = 4N_{\min} \rightarrow V_{\min} + 0.3\%$$

Conclusion:

Recommend $N = 2.5 N_{\min}$ (use higher value to save energy and simplify operation).

Gives actual boilup (energy) $V \approx 1.1 V_{\min}$

10.6B Equipment for absorption and distillation

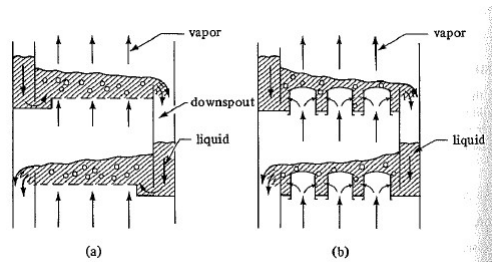


FIGURE 10.6-1. Tray contacting devices: (a) detail of sieve-tray tower, (b) detail of valve-tray tower.

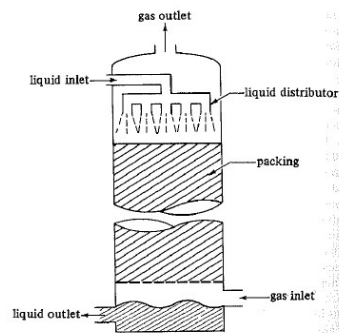


FIGURE 10.6-3. Packed tower flows and characteristics for absorption.

Random/dumped packings

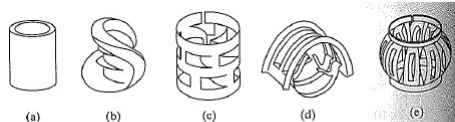


FIGURE 10.6-4. Typical random or dumped tower packings: (a) Raschig ring; (b) Berl saddle; (c) Pall ring; (d) Intalox metal, IMTP; (e) Jaeger Metal-Ti-Pack.



Structured packing (1970's)

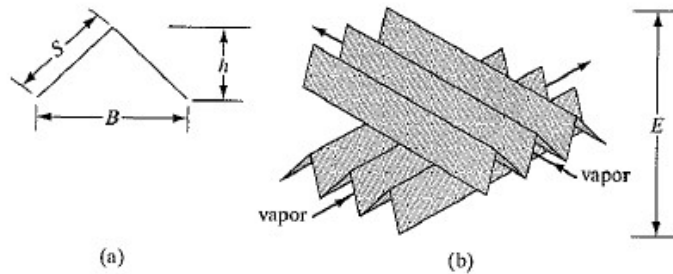


FIGURE 10.6-2. Typical corrugated structured packing: (a) triangular cross section of flow channel; (b) flow-channel arrangement, with vapor flowing upward, indicated by arrows, and liquid downward. [From J. R. Fair and J. L. Bravo, Chem. Eng. Progr., 86, (Jan.), 19 (1990). With permission.]

Structured packings (today)



Koch-Glitsch



Mellapak (Sulzer)

6. Height of real columns (section 11.5)

- **N = no. of theoretical stages (total)**
- N_{inside} = no. of theoretical stages inside column
 - = N - 1 (total condenser, assuming reboiler gives 1 theoretical stage)
 - = N - 2 (partial condenser)
- **Tray columns:**
 - N_{trays} = Actual number of trays inside column
 - E_o = overall tray efficiency (typical 0.7)
 - $N_{\text{trays}} = N_{\text{inside}} / E_o$
 - Comment: Sigurd does not recommend using Murphy or Point efficiency
- **Packed columns:**
 - H = height of packings [m] = $N_{\text{inside}} * \text{HETP}$
 - HETP = height equivalent to 1 theoretical plate
 - Estimation of HETP:
 1. Mass transfer model
 $\text{HETP} \approx H_{OG} * f = f * V / (K_y a S)$ (10.6-55) (not very reliable, $f = \ln(1/A)/(1/A-1)$)
 2. **Rule of thumb (recommended in practice)**
 - **Random packing: $\text{HETP [m]} = 18 D_p \text{ [m]}$ (equation 11.5-11)**
 - D_p = diameter of each packing element
 3. **(Best) Data from packing manufacturer**

7. Column operation and diameter

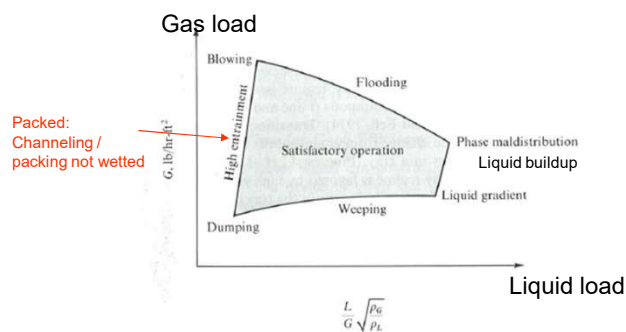


Figure 12-9 Effects of vapor and liquid loadings on sieve-tray performance. (From *Bolles and Fair, 1963, p. 556; used by permission.*)

Column diameter

- In many cases the diameter is determined by the (superficial) gas velocity v [m/s]

$$v[\text{m/s}] = V[\text{m}^3/\text{s}] / A[\text{m}^2]$$

$$\text{Where for ideal gas: } V[\text{m}^3/\text{s}] = V[\text{mol/s}] \cdot RT/p$$

- Example.** Column at 2 bar with $V = 50 \text{ mol/s}$ and given $v_{\text{max}} = 1.5 \text{ m/s}$.
Temperature bottom (highest) = 400K.
 - Then $V [\text{m}^3/\text{s}] = 50 \text{ mol/s} \cdot 8.31 \text{ J/K, mol} \cdot 400\text{K} / 2 \cdot 10^5 \text{ Pa} = 0.83 \text{ m}^3/\text{s}$
 - $A [\text{m}^2] = V[\text{m}^3/\text{s}] / v_{\text{max}} [\text{m/s}] = 0.83 / 1.5 = 0.553 \text{ m}^2$
 - Column area $A = (\pi/4) D^2$
 - Conclusion: Diameter $D = \sqrt{(4A/\pi)} = \sqrt{(4 \cdot 0.553 / 3.14)} = 0.84 \text{ m}$

Trayed column

- Maximum vapor velocity is limited by entrainment
- Tray column. Fair correlation:

$$v_{\text{max}} = K_V \left(\frac{\sigma}{20} \right)^{0.2} \sqrt{\frac{\rho_L - \rho_V}{\rho_V}}$$

σ = surface tension [dyn = mN/m].

Organic liquids: $\sigma \approx 20$ and σ -factor drops out

Typical value: $v_{\text{max}} = 3 \text{ m/s}$

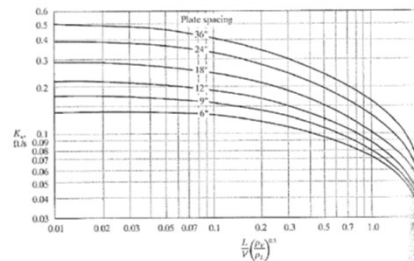
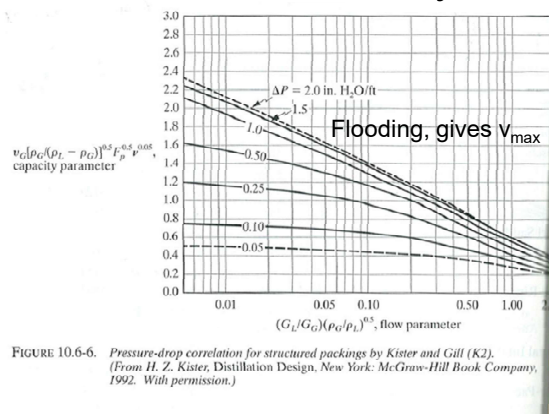


FIGURE 11.5-3. Estimation of K_V value for allowable vapor velocity. (From Fair, J. R., *Petroleum Ref.*, 33(10), 45 (1961). With permission.)

Packed column

- Maximum gas load limited by flooding



8. Trays vs. packings

- Packings:
 - + Much smaller pressure drop (typically 1/10)
 - + Usually: More stages for given column height
 - Problems with liquid distribution in larger columns (can use structured packings, but more expensive)
- Trays:
 - + More easy to clean
 - + Better for large capacity columns
 - + Larger holdup (typically, 2 times larger): Advantage for control (“have more time”)
 - Can have inverse response in bottom of column (λ - effect - difficult to predict)
- Overall: Differences are surprisingly small – also for process control

9. Special cases of distillation

1. Stripping column (no top part)
2. Enriching column (no btm part)
3. Stripper / absorber (no condenser or reboiler)
4. "Direct" steam injection (instead of heat)
5. Column with side stream
6. Partial condenser (gives 1 extra eq. stage)
7. More than one feed

- All cases: Exactly same principles (can use McCabe-Thiele)
- Start with material balances to derive operating lines
- Need equilibrium data!

10. Multicomponent mixtures

- Most of the derivations apply to the multicomponent case
- Identify "key components" (light and heavy, L-H) for split
- McCabe-Thiele: Plot xy-diagram for pseudo-binary, $x = x_L / (x_L + x_H)$
- Fenske's formula OK: $N_{\min} = \ln S / \ln \alpha$.
 - α = relative volatility between L and H
 - S = separation factor between L and H

11. Shortcut: "How to design a distillation column in 5 min"

1. Multicomponent: Identify key components (LH)
2. Estimate relative volatility α . Can use Sigurd's formula*
3. Find split (D/F) from desired product purities
4. Stages (height of column): Find Separation factor S and $N_{\min} = \ln S / \ln \alpha$. Select $N = 2.5 N_{\min}$
5. Feed stage: Can use another Sigurd-formula (last slide)
6. Reflux / Energy usage: Find $L_{\min} = F/(\alpha-1)$ (feed liquid) and V_{\min}/F . Real V is about 10% higher
7. Diameter: $A = (V_{[\text{mol/s}]RT/p}) / v_{\max}$. $D = \sqrt{(4A/\pi)}$. Typical value: $v_{\max} = 2\text{m/s}$.

*Sigurd's formula

$$\ln \alpha \approx \frac{\Delta H^{\text{vap}}}{RT_B} \cdot \frac{\Delta T_B}{T_B}$$

typical: $\approx 9-15$

IDEAL VLE (constant α)

Example: "5 min column design"

- Design a column for separating air
- Feed: 80 mol-% N_2 (L) and 20% O_2 (H)
- Products: Distillate is 99% N_2 and bottoms is 99.998% O_2
- Component data
 - Nitrogen: $T_b = 77.4\text{ K}$, $\Delta H^{\text{vap}} = 5.57\text{ kJ/mol}$
 - Oxygen: $T_b = 90.2\text{ K}$, $\Delta H^{\text{vap}} = 6.82\text{ kJ/mol}$
- **Problem:** 1) Estimate α . 2) Find split D/F. 3) Stages: Find N_{\min} and 4) suggest values for N and N_F . 5) Energy usage: Find V_{\min}/F for a) vapor feed and b) liquid feed.
- Given: For vapor feed and sharp sep. of binary mixture: $V_{\min}/F = 1/(\alpha-1)$

IDEAL VLE (constant α)

Solution “5-min design”

Also see paper (“Theory of distillation”)

1. Relative volatility: The mixture is relatively ideal and we will assume constant relative volatility. The estimated relative volatility at 1 atm based on the boiling points is $\ln \alpha \approx (\Delta \hat{H}^{vap}/RT_b) [(T_{bH} - T_{bL})/T_b]$ where $\Delta \hat{H}^{vap} = \sqrt{5.57 \cdot 6.82} = 6.16 \text{ kJ mol}^{-1}$, $T_b = \sqrt{T_{bH}T_{bL}} = 83.6 \text{ K}$ and $T_H - T_L = 90.2 - 77.7 = 18.8$. This gives $(\Delta \hat{H}^{vap})/(RT_b) = 8.87$ and we find $\alpha \approx 3.89$ (however, it is generally recommended to obtain α from experimental VLE data).
2. Product split: From the overall material balance we get $D/F = (z - x_B)/(x_D - x_B) = (0.8 - 0.00002)/(0.99 - 0.00002) = 0.808$.

IDEAL VLE (constant α)

3. Number of stages: The separation factor is $S = (0.99 \times 0.99998)/(0.01 \times 0.00002) = 4950000$, i.e. $\ln S = 15.4$. The minimum number of stages required for the separation is $N_{min} = \ln S / \ln \alpha = 11.35$ and we select the actual number of stages as $N = 23$ ($\approx 2N_{min}$).
4. Feed-stage location: With an optimal feed location we have at the feed stage ($q = 0$) that $y_F = z_F = 0.8$ and $x_F = y_F/(\alpha - (\alpha - 1)y_F) = 0.507$. Skogestad's approximate formula for the feed-stage location gives:

$$\begin{aligned} N_T - N_B &= \ln \left(\left[\frac{(1 - y_F)}{x_F} \right] \left[\frac{x_B}{(1 - x_D)} \right] \right) / (\ln \alpha) \\ &= \ln \left(\left[\frac{0.2}{0.507} \right] \times \left[\frac{0.00002}{0.01} \right] \right) / 1.358 \\ &= -5.27 \end{aligned}$$

corresponding to the feed stage $N_F = [N + 1 - (N_T - N_B)]/2 = (23 + 1 + 5.27)/2 = 14.6$.

IDEAL VLE (constant α)

5. Energy usage: The minimum energy usage for a vapour feed (assuming sharp separation) is $V_{\min}/F = 1/(\alpha - 1) = 1/2.89 = 0.346$. With the choice $N = 2N_{\min}$, the actual energy usage (V) is then typically about 10% above the minimum (V_{\min}), i.e. V/F is about 0.38.

Remark 1 The actual minimum energy usage is slightly lower since we do not have sharp separations. The recovery of the two components in the bottom product is $r_L = (x_{L,B})/(z_{FL}F) = 0.9596$ and $r_H = (x_{H,B})/(z_{FH}F) \approx 0$, so from the formulas given earlier the exact value for nonsharp separations is $V_{\min}/F = (0.9596 - 0.0 \times 3.89)/(3.89 - 1) = 0.332$.

Remark 2 For a liquid feed we would have to use more energy, and for a sharp separation:

$$V_{\min}/F = 1/(\alpha - 1) + D/F = 0.346 + 0.808 = 1.154$$

Remark 3 We can check the results with exact stage-by-stage calculations. With $N = 23$, $N_F = 15$ and $\alpha = 3.89$ (constant), we find $V/F = 0.374$, which is about 13% higher than $V_{\min} = 0.332$.

Remark 4 A simulation with more rigorous VLE computations, using the Soave-Redlich-Kwong (SRK) equation of state, has been carried out using the HYSYS (Hypnotech Ltd.) simulation package. The result is a slightly lower vapour flow due to a higher relative volatility (α in the range 3.99–4.26 with an average of 4.14). More precisely, a simulation with $N = 23$, $N_F = 15$ gave $V/F = 0.291$, which is about 11% higher than the minimum value $V_{\min} = 0.263$ found with a very large number of stages (increasing $N > 60$ did not give any significant energy reduction below V_{\min}). The optimal feed stage (with $N = 23$) was indeed found to be $N_F = 15$.

Thus, the results from HYSYS confirm that a column design based on the very simple short-cut methods is very close to results from much more rigorous computations.

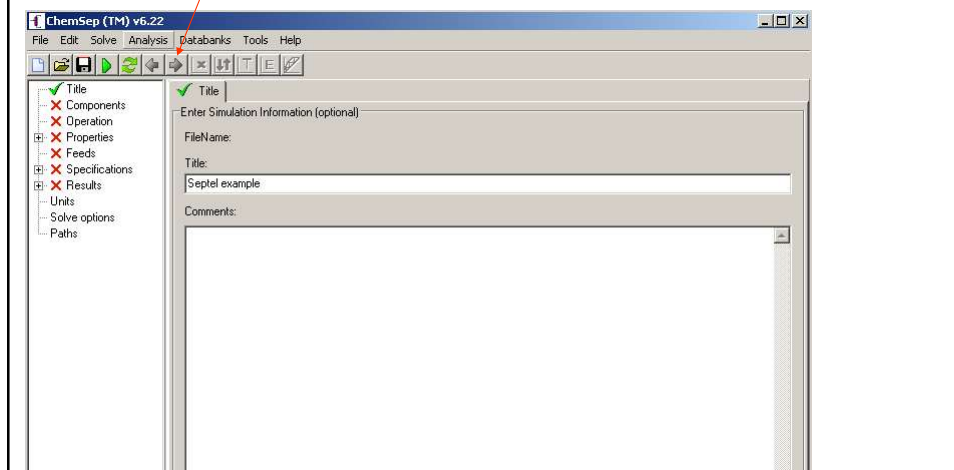
11. Distillation design in practice: Numerical solution using “simulators”.

Example using Chemsep

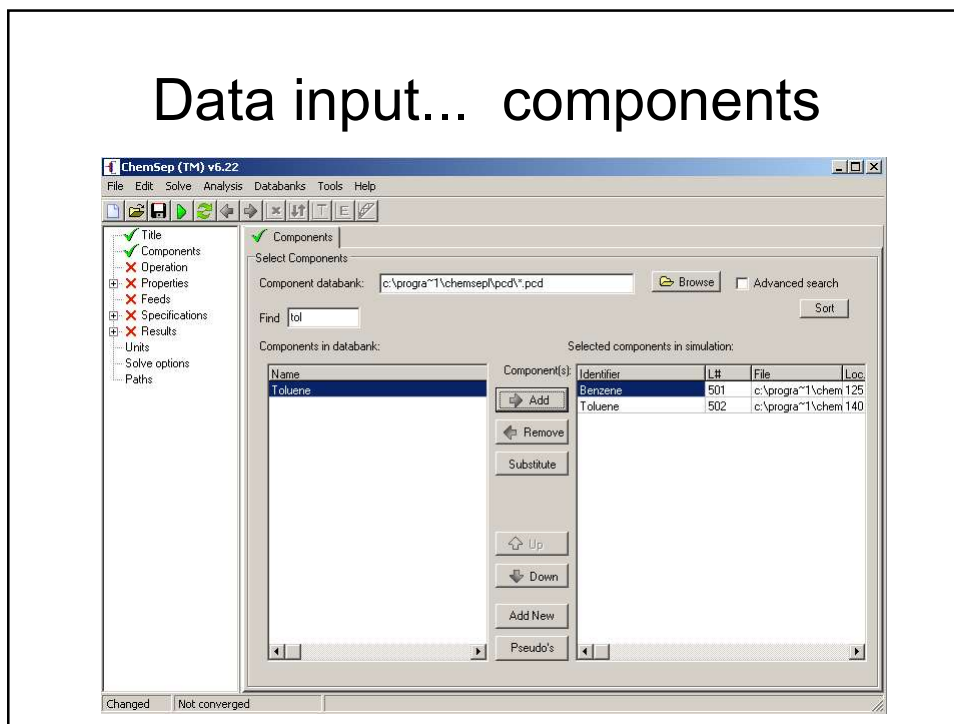
- <http://www.chemsep.org/>
- Written by Ross Taylor, Clarkson University
- Lite version:
 - max 50 stages and 5 components
 - free and extremely simple to use
- Example 11.4-2: Benzene-Toluene
 - Specify 8 stages, feed on stage 5 from top
 - Specify product compositions, $x_D=0.95$, $x_B=0.10$
 - Find: Reflux ratio L/D (was 4 but then we needed only 7.6 stages)

Enter title....

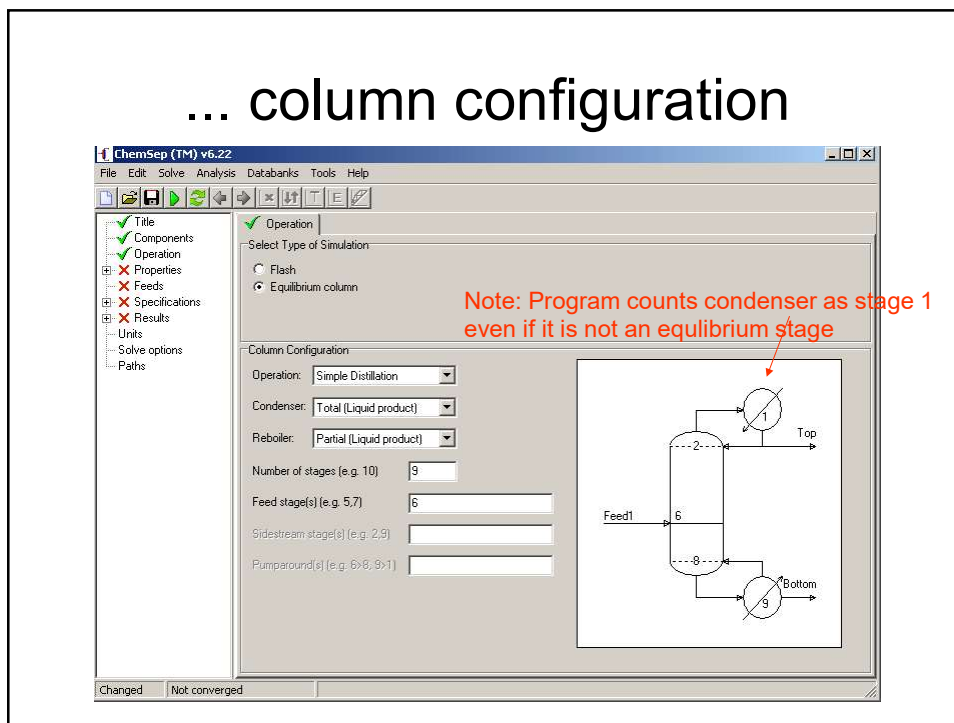
Use arrow to go through menus



Data input... components



... column configuration



... thermodynamics

The screenshot shows the 'Thermodynamics' settings window in ChemSep (TM) v6.22. The 'Select Thermodynamic Models' section is configured as follows:

- K-Value: Raoult's law
- Equation of state: Ideal gas law
- Activity coefficient: Ideal solution
- Vapour pressure: Antoine
- Enthalpy: Ideal

The 'Enter Thermodynamic Model Parameters (when required)' section shows 'No input of thermodynamic model parameters required'.

Below the window, red text provides additional context:

Here: Raoult's law + ideal enthalpy
Normally use:
- Soave-RK equation of state (EOS) for relatively ideal mixtures like hydrocarbons
- Gamma-Phi (UNIQUAC, SRK) for non-ideal components

... feed data

The screenshot shows the 'Feeds' settings window in ChemSep (TM) - benzene-toluene.sep. The 'Feed Stream(s) Specifications' table is as follows:

Feed	1
Name	Feed1
Stage	S
Two-phase feed	Not split
State	T & p
Pressure (N/m ²)	101325
Vapour fraction (-)	
Temperature (K)	320.000
Flowrates (kmol/s)	
Benzene	45.0000
Toluene	55.0000
Total flowrate	100.000

Red text annotations provide additional context:

- Normally choose "Split", but does not matter here since the feed is subcooled
- Can write 1atm and program converts automatically
- Note: Use 55 mol/s (instead of 55 kmol/h)

Analysis | Pressures | Heaters/Coolers | Efficiencies | Column specifications

Column Pressure Specifications

Condenser pressure: 101325 (N/m²)
 Column pressure: Constant pressure
 Top pressure: 101325 (N/m²)
 Pressure drop per stage: (N/m²)
 Bottom pressure: (N/m²)

Assume constant pressure (1 atm) in column
(Normally the pressure drop is about 0.1 bar from the bottom to the top)

Analysis | Pressures | Heaters/Coolers | Efficiencies | Column specifications

Column and Stage Heat Duties

Column heat loss: 0 (J/s)
 Name column duty: Qcolumn

Stage heat exchangers:

Assume no heat loss

Analysis | Pressures | Heaters/Coolers | Efficiencies | Column specifications

Specify Stage Efficiencies

Default stage efficiency: 1.00000 (-)

Assume 100% stage efficiency

TOP: Specify $x_D=0.95$
 BTM: Specify $x_B=0.1$

ChemSep (TM) v6.22

File Edit Solve Analysis Databanks Tools Help

Title | Components | Operation | Properties | Thermodynamic: | Physical properti | Reactions | Feeds | Specifications | Analysis | Pressures | Heaters/Coolers | Efficiencies | Column specifications | Results | Units | Solve options | Paths

Analysis | Pressures | Heaters/Coolers | Efficiencies | Column specifications

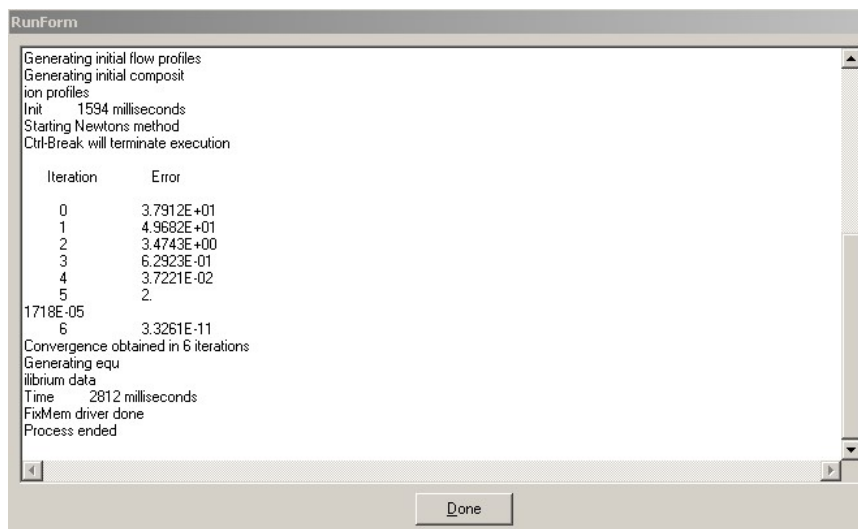
Column Product Specifications

Top product name: Top Condenser duty name: Qcondenser
 Top specification: Mole fraction of a component = 0.950000 (-)
 Benzene

Bottom product name: Bottom Reboiler duty name: Qreboiler
 Bottom specification: Mole fraction of a component = 0.10 (-)
 Benzene

Product Guesses (optional)
 Use guesses for initialization

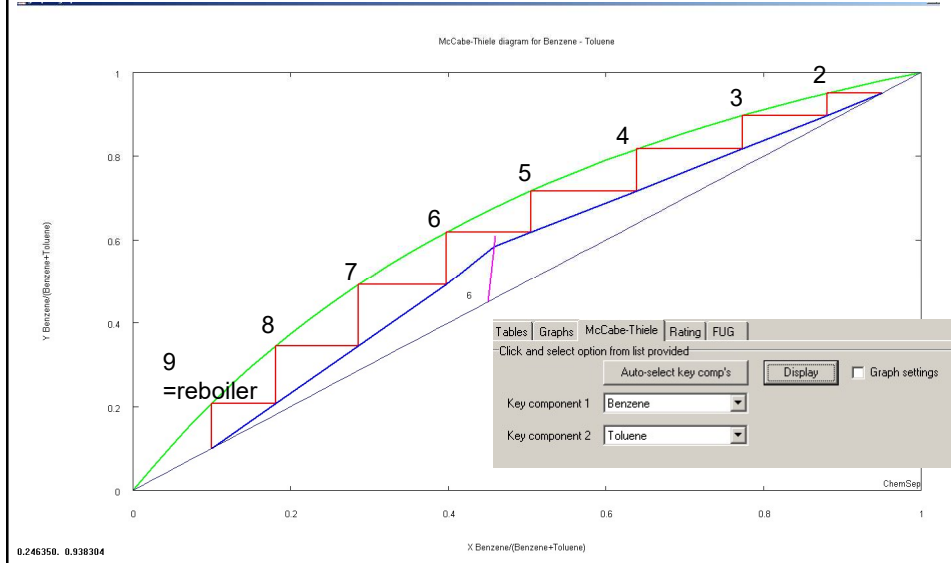
Press solve...



... results... Streams

Stream	Feed1	Top	Bottom
Stage	6	1	9
Pressure (N/m ²)	101325	101325	101325
Vapour fraction (-)	0.000000	0.000000	0.000000
Temperature (K)	320.000	354.324	379.111
Enthalpy (J/kmol)	-3.283E+07	-2.576E+07	-2.164E+07
Entropy (J/kmol/K)	-96660.1	-69806.8	-56702.3
Mole flows (kmol/s)			
Benzene	45.0000	39.1176	5.88235
Toluene	55.0000	2.05882	52.9412
Total molar flow	100.000	41.1765	58.8235
Mole fractions (-)			
Benzene	0.450000	0.950000	0.100000
Toluene	0.550000	0.050000	0.900000
Mass flows (kg/s)			
Benzene	3515.13	3055.64	459.494
Toluene	5067.75	189.702	4878.05
Total mass flow	8582.88	3245.34	5337.55
Mass fractions (-)			
Benzene	0.409551	0.941546	0.0860871
Toluene	0.590449	0.0584537	0.913913
Vapour:			
Mole weight (kg/kmol)			
Density (kg/m ³)			
Viscosity (N/m ² .s)			
Heat capacity (J/kmol/K)			
Thermal cond. (J/s/m ² /K)			
Liquid:			
Mole weight (kg/kmol)	85.8288	78.8153	90.7383
Density (kg/m ³)	845.987	811.463	785.444
Viscosity (N/m ² .s)	4.4675E-04	3.1339E-04	2.4727E-04
Heat capacity (J/kmol/K)	153293	150927	179584
Thermal cond. (J/s/m ² /K)	0.129008	0.123105	0.111220
Surface tension (N/m)	0.0253779	0.0210133	0.0185499

McCabe-Thiele diagram



Profiles

Tables | Graphs | McCabe-Thiele | Rating | FUG

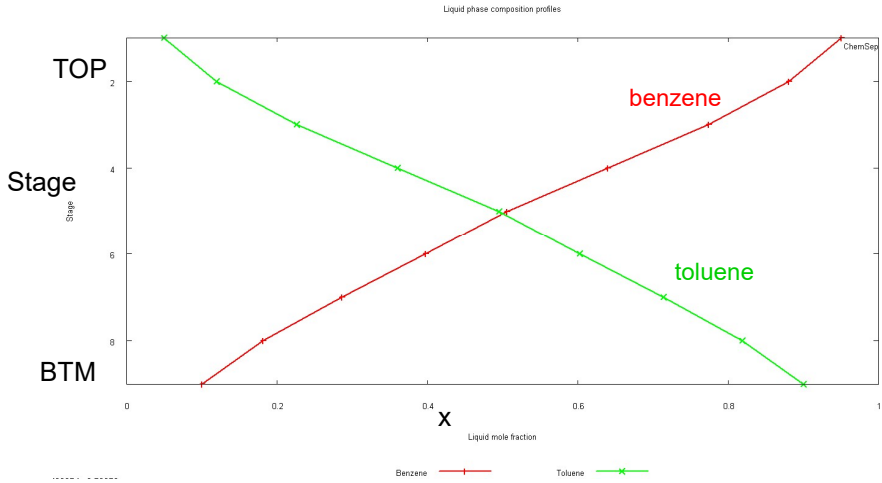
Tables

Select table: **T/P/Flow profiles**

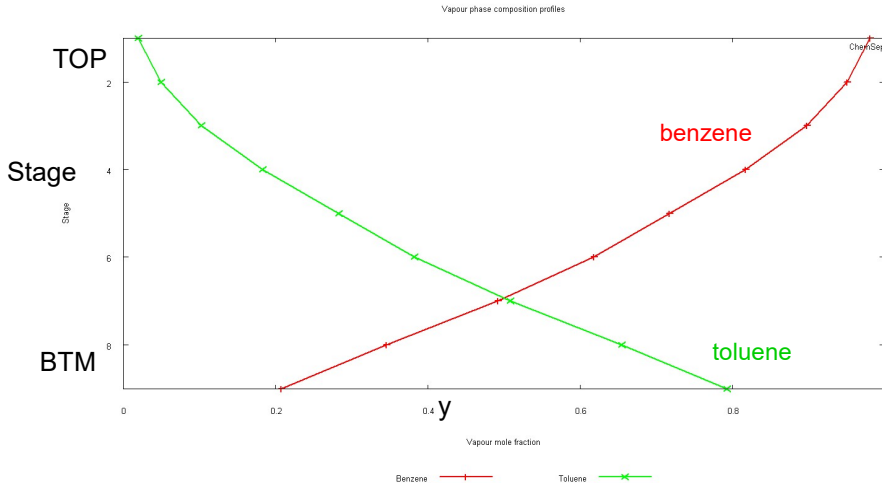
Stage	Temperatur (K)	Pressure (N/m ²)	Flow rates (kmol/s)		Feed	Product
			Liquid	Vapour		
1	354.324	101325	128.905	RR=3.13055		41.1765 L
2	355.809	101325	127.319	170.082		
3	358.220	101325	125.216	168.495		
4	361.523	101325	123.061	166.393		
5	365.176	101325	121.408	164.238		
6	368.414	101325	243.557	162.584	100.000	
7	372.082	101325	242.304	184.733		
8	375.883	101325	241.865	183.480		
9	379.111	101325	BR=3.1117	183.041		58.8235 L

Before R= 4. Now we need less reflux (and less energy) because we have more stages (increased from 7.6 to 8)

Liquid phase composition

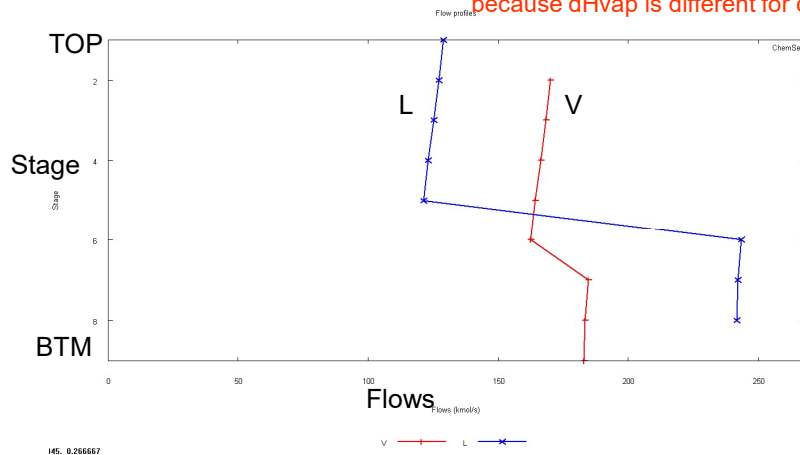


Vapor phase composition

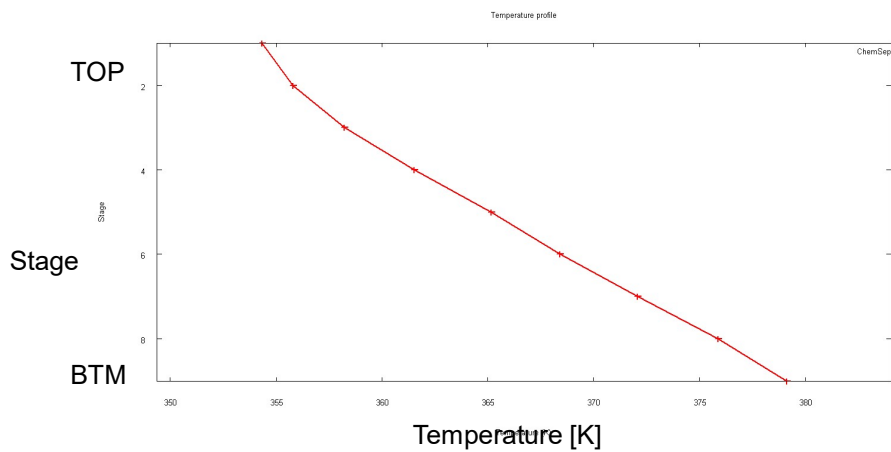


Flow profiles

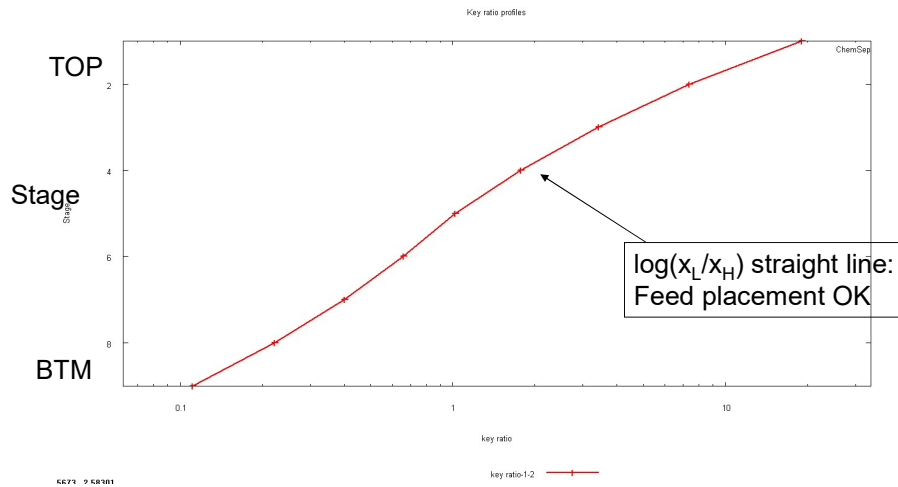
Note: Not quite constant molar flows because dH_{vap} is different for components



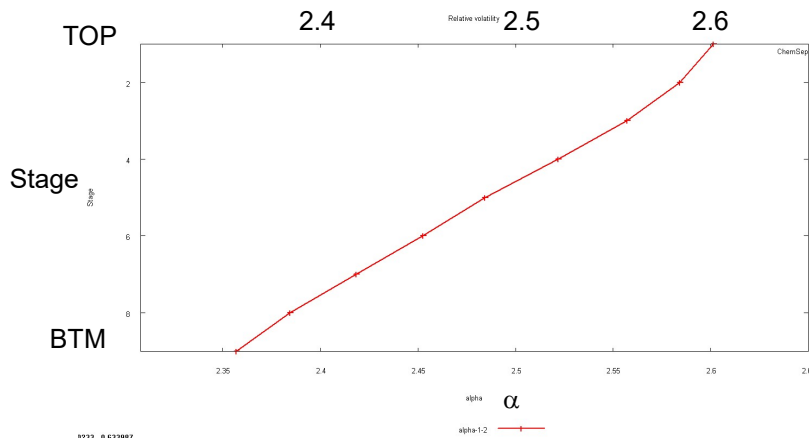
Temperature profile



Log (x_L/x_H)-plot (“key ratio profile”): Use to check feed location



Relative volatility



1. More literature on distillation

- King (Wiley, 1980) on distillation design
- Shinskey (McGraw-Hill, 1984) on distillation control
- Kister (McGraw-Hill, 1990) on distillation operation
- General info: <http://lorien.ncl.ac.uk/ming/distil/distil0.htm>
- I.J. Halvorsen and S. Skogestad, "Distillation Theory", In: Encyclopedia of Separation Science. Ian D. Wilson (Editor-in-chief), Academic Press, 2000, pp. 1117-1134.
- S. Skogestad, [Dynamics and control of distillation columns - A tutorial introduction](#), Trans IChemE (UK), Vol. 75, Part A, Sept. 1997, 539-562 (Presented at *Distillation and Absorption 97*, Maastricht, Netherlands, 8-10 Sept. 1997).
- More: see home page Sigurd Skogestad <http://www.nt.ntnu.no/users/skoge/>
<http://www.nt.ntnu.no/users/skoge/distillation>
- Free steady-state distillation software with thermo package :
<http://www.chemsep.org/>

Extra slides

IDEAL VLE (constant α)

Simple formula for feed stage location (Skogestad, 1987)

$$N_T - N_B \approx \frac{\ln\left(\frac{z_{FH} \cdot x_{BL}}{z_{FL} \cdot x_{DH}}\right)}{\ln \alpha}$$

Example. C3-splitter. $z_{FL}=0.65$, $x_{DH}=0.005$, $x_{BL}=0.1$, $\alpha=1.12$.

$$N_T - N_B \approx \frac{\ln\left(\frac{0.35 \cdot 0.1}{0.65 \cdot 0.005}\right)}{\ln 1.12} = 21.0$$

Feed stage (from btm.) with $N = N_T + N_B = 110$ stages:

$$N_F = N_B = \frac{N - (N_T - N_B)}{2} = \frac{110 - 21}{2} = 45$$