

Distillation



Processos de Separação

LEQB

2023/2024

Summary

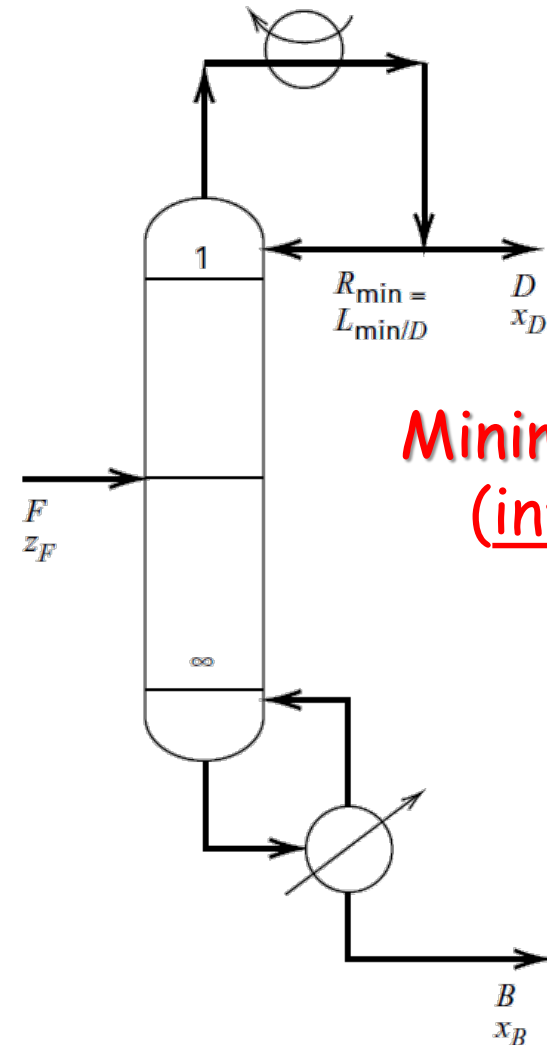
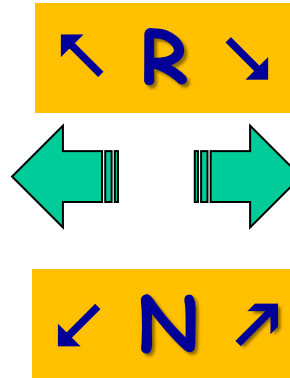
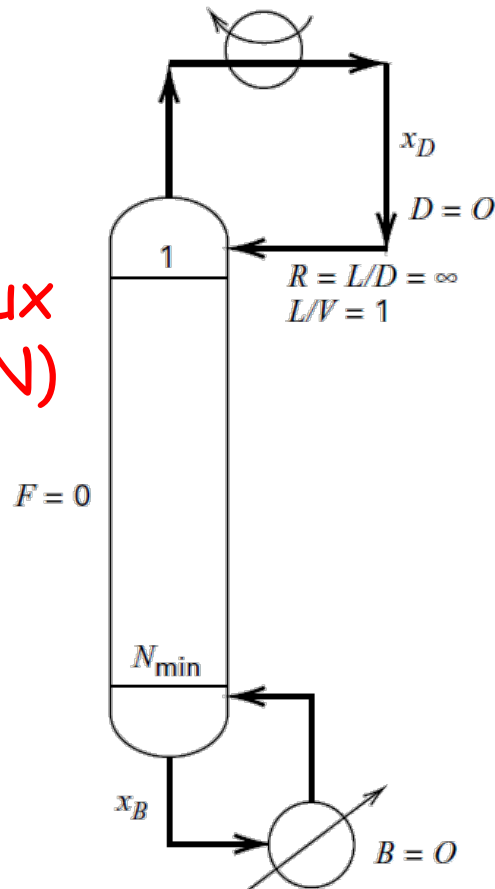
- Optimal reflux ratio
- Condenser and Boiler in a distillation column
- Energy Balances to a distillation column
- Stage efficiency
- Energetic balance to the feed stage

➤ **Optimal reflux ratio**

Reflux ratio, R

$$R = L/D$$

Total reflux
(minimum N)

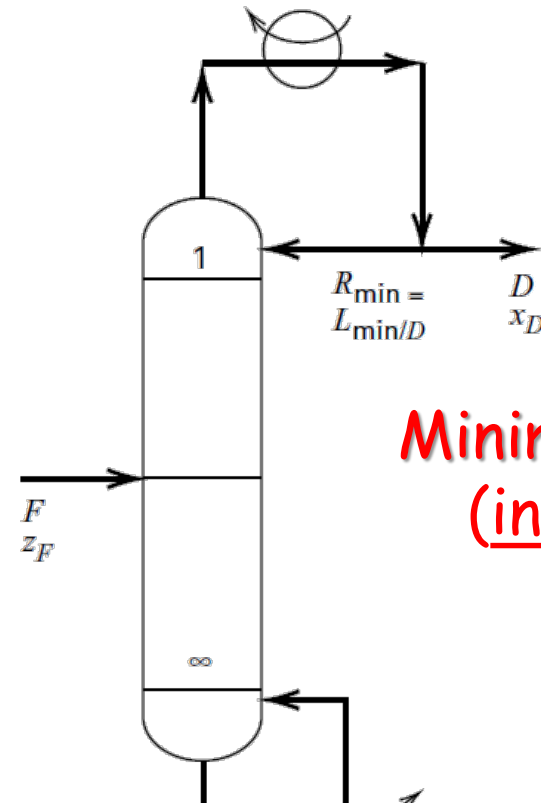
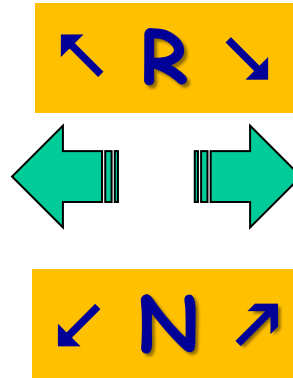
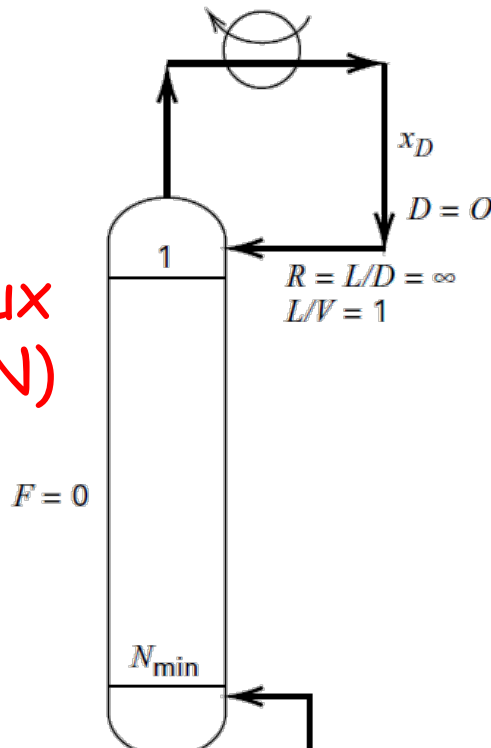


Minimum Reflux
(infinite N)

Reflux ratio, R

$$R = L/D$$

Total reflux
(minimum N)

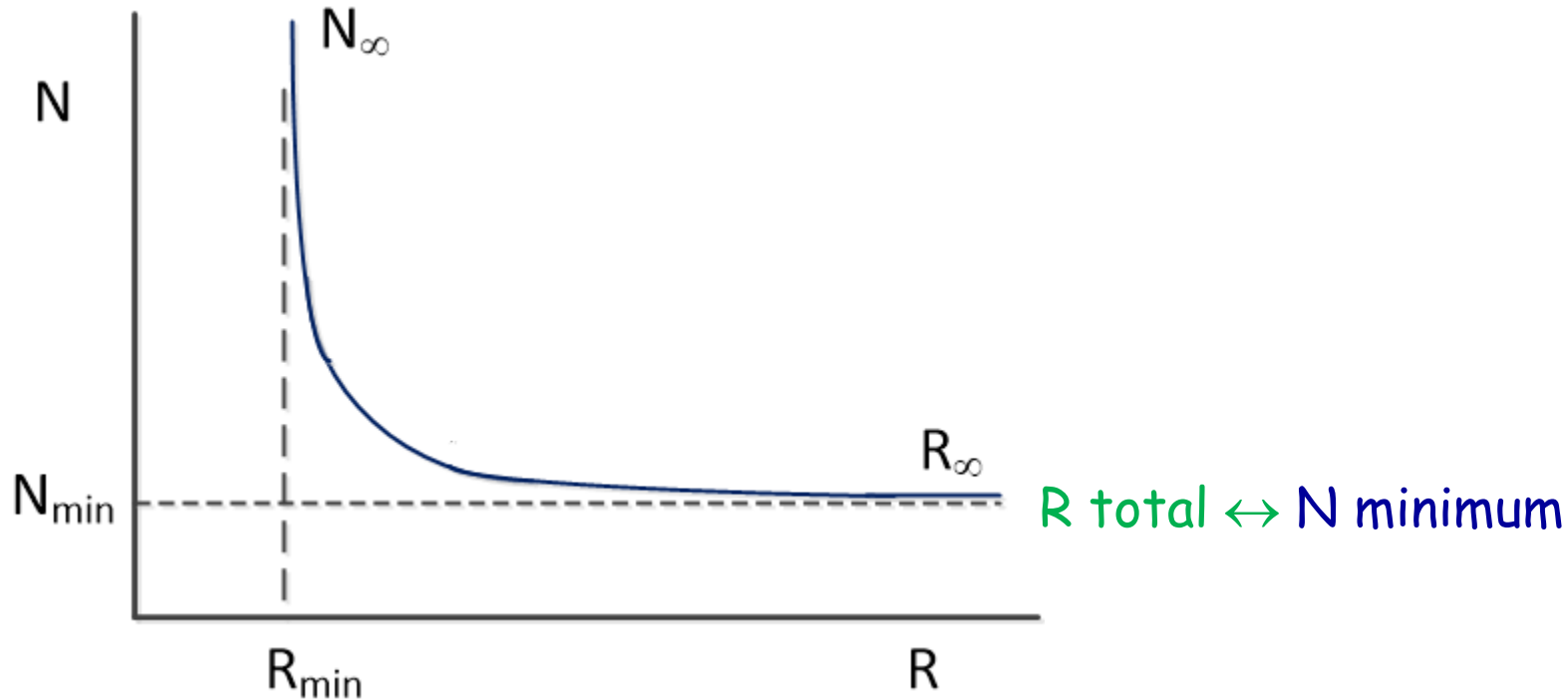


Minimum Reflux
(infinite N)

which value should we choose for the reflux ratio of a given distillation column, and how do we estimate that value ??

Reflux Ratio, $R \leftrightarrow$ Number of stages, N

$R_{\text{minimum}} \leftrightarrow N_{\text{infinite}}$

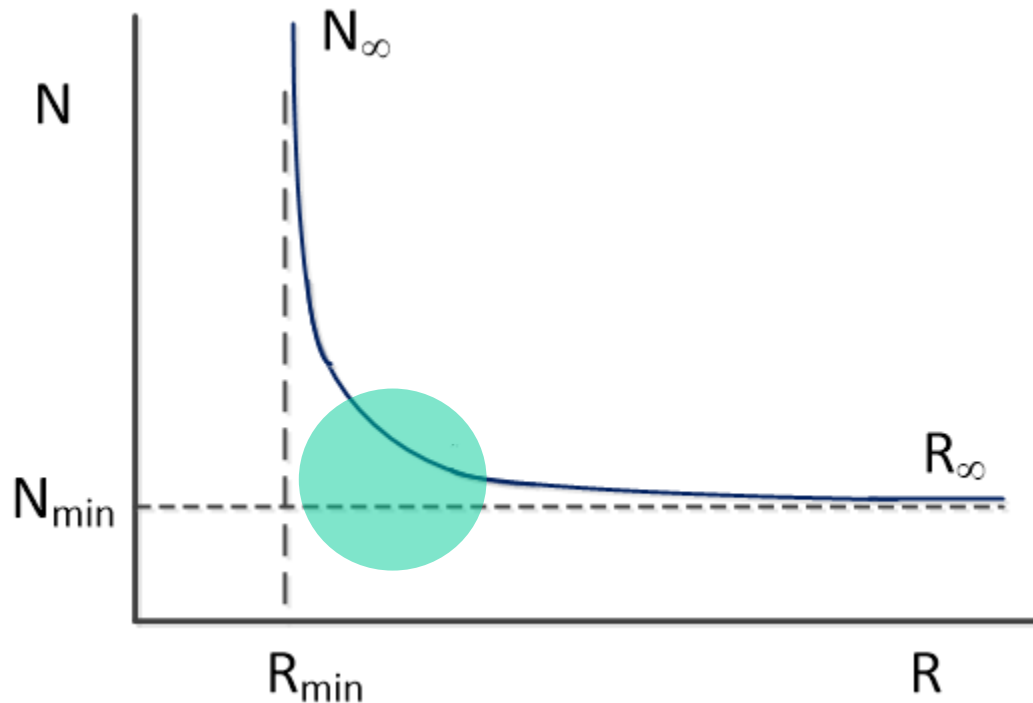


Reflux Ratio, $R \leftrightarrow$ Number of stages, N

Optimum Reflux



$$1.05 \text{ a } 1.5 \times R_{\min}$$



How?

- Material Balance
- Energy Balance
- Economic Analysis

Reflux Ratio, $R \leftrightarrow$ Number of stages, N

Effect of Reflux Ratio on Annualized Cost of a Distillation Operation

R/R_{\min}	Actual N
1.00	Infinite
1.05	29
1.14	21
1.23	18
1.32	16
1.49	14
1.75	13

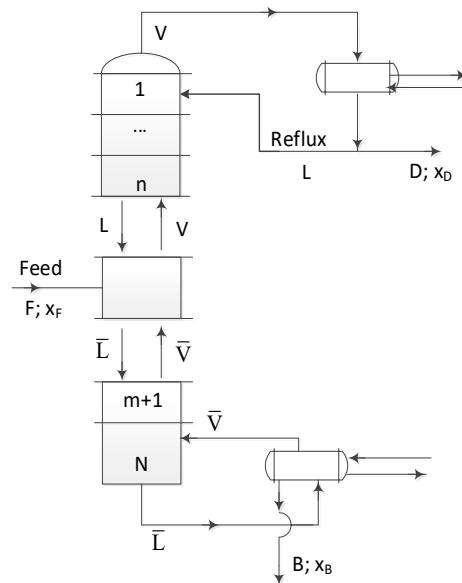
Values of F , x_F ,
 x_D , x_B and i are
constants

$$R = \frac{L}{D}$$

$$R \nearrow \Leftrightarrow L \nearrow$$

(Adapted from an example by Peters and Timmerhaus [6].)

System benzene + toluene



Reflux Ratio, $R \leftrightarrow$ Number of stages, N

Effect of Reflux Ratio on Annualized Cost of a Distillation Operation

R/R_{\min}	Actual N	Diam., ft
1.00	Infinite	6.7 (204 cm)
1.05	29	6.8
1.14	21	7.0
1.23	18	7.1
1.32	16	7.3
1.49	14	7.7
1.75	13	8.0 (243 cm)

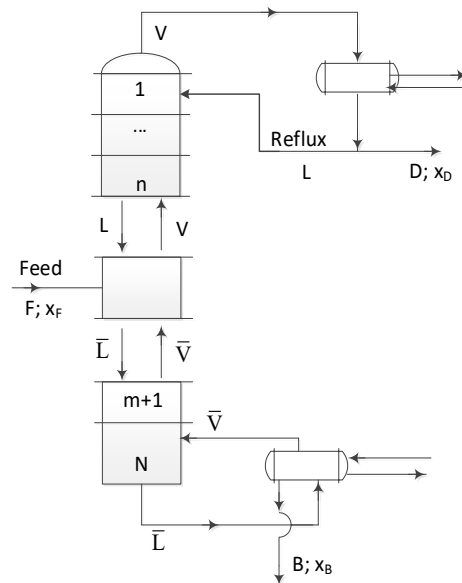
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System benzene + toluene



Reflux Ratio, $R \leftrightarrow$ Number of stages, N

Effect of Reflux Ratio on Annualized Cost of a Distillation Operation

R/R_{\min}	Actual N	Diam., ft	Reboiler Duty, Btu/h	Condenser Duty, Btu/h
1.00	Infinite	6.7	9,510,160	9,416,000
1.05	29	6.8	9,776,800	9,680,000
1.14	21	7.0	10,221,200	10,120,000
1.23	18	7.1	10,665,600	10,560,000
1.32	16	7.3	11,110,000	11,000,000
1.49	14	7.7	11,998,800	11,880,000
1.75	13	8.0	13,332,000	13,200,000

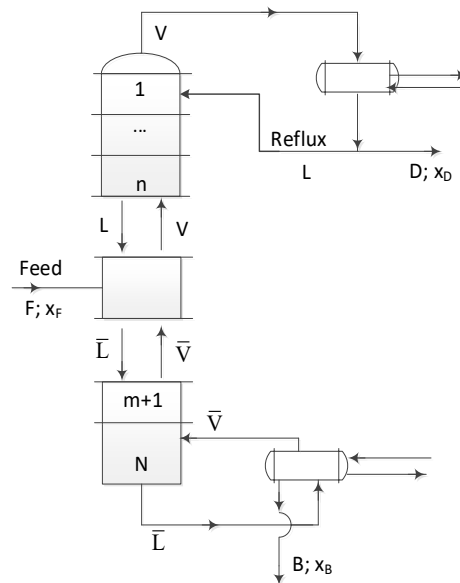
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R/R_{\min}	Actual N	Diam., ft	Reboiler Duty, Btu/h	Condenser Duty, Btu/h	Annualized Cost, \$/yr Equipment
1.00	Infinite	6.7	9,510,160	9,416,000	Infinite
1.05	29	6.8	9,776,800	9,680,000	44,640
1.14	21	7.0	10,221,200	10,120,000	38,100
1.23	18	7.1	10,665,600	10,560,000	36,480
1.32	16	7.3	11,110,000	11,000,000	35,640
1.49	14	7.7	11,998,800	11,880,000	35,940
1.75	13	8.0	13,332,000	13,200,000	36,870

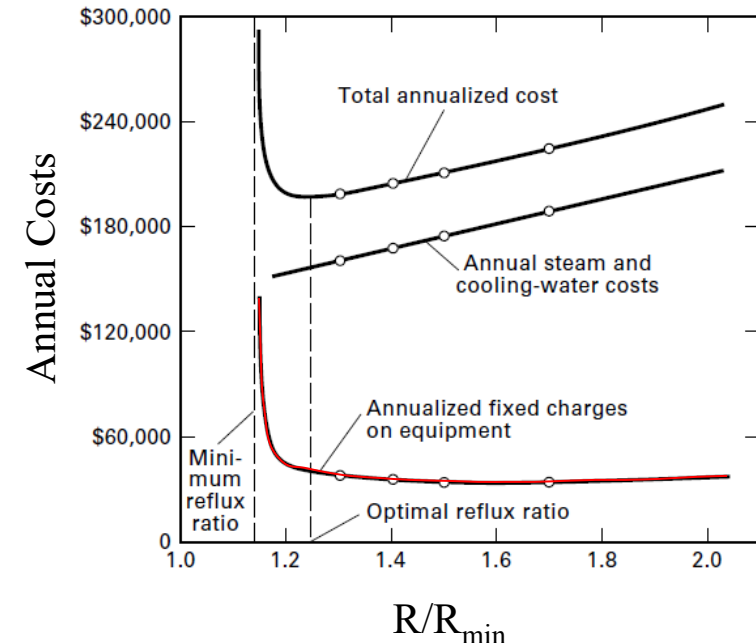
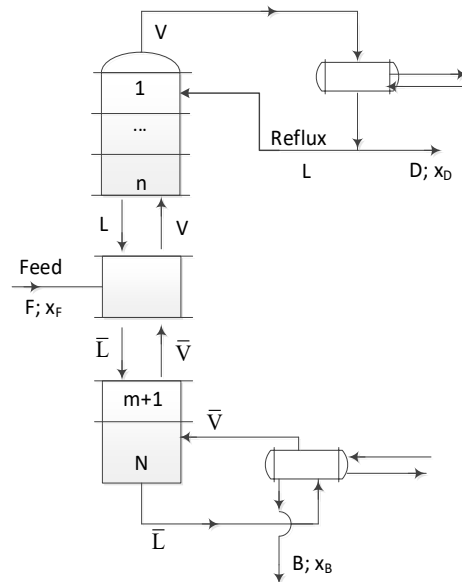
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					Equipment	Cooling Water	Steam
1.00	Infinite	6.7	9,510,160	9,416,000	Infinite	17,340	132,900
1.05	29	6.8	9,776,800	9,680,000	44,640	17,820	136,500
1.14	21	7.0	10,221,200	10,120,000	38,100	18,600	142,500
1.23	18	7.1	10,665,600	10,560,000	36,480	19,410	148,800
1.32	16	7.3	11,110,000	11,000,000	35,640	20,220	155,100
1.49	14	7.7	11,998,800	11,880,000	35,940	21,870	167,100
1.75	13	8.0	13,332,000	13,200,000	36,870	24,300	185,400

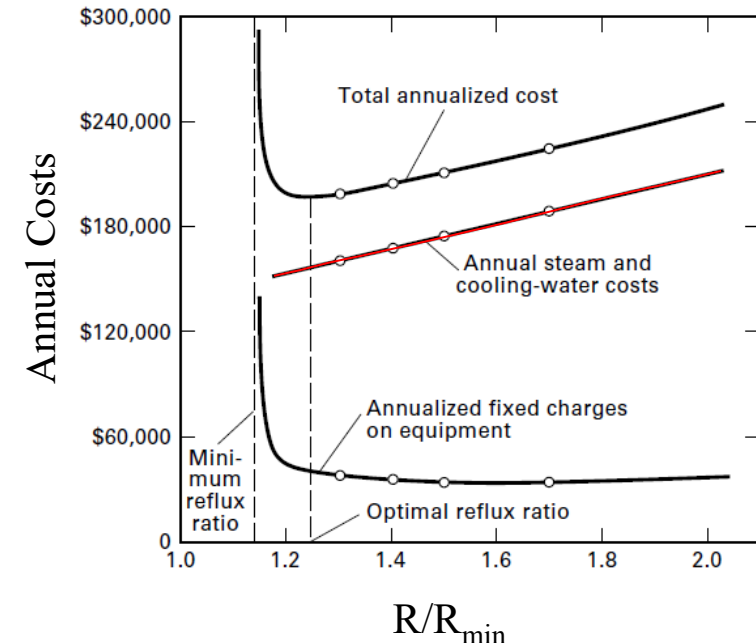
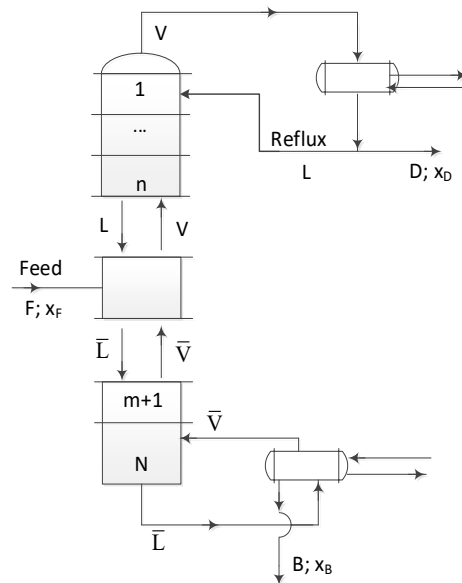
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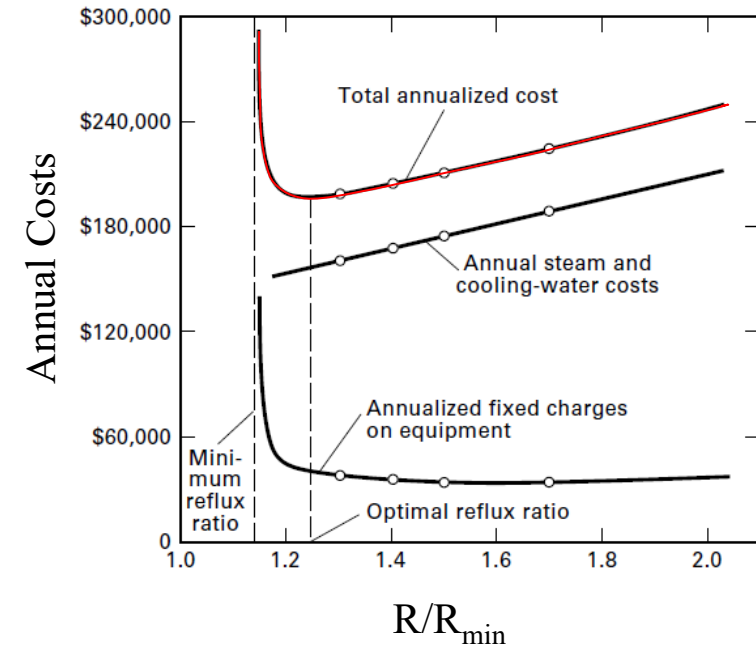
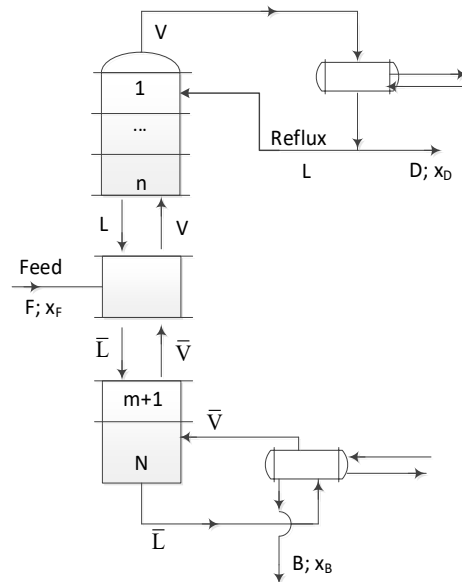
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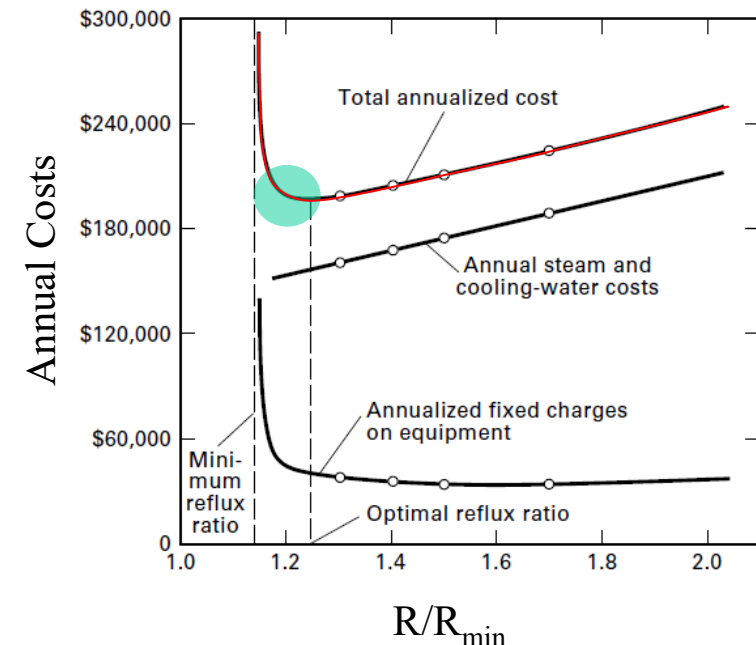
$$R = \frac{L}{D}$$

$$R \nearrow \Leftrightarrow L \nearrow$$

(Adapted from an example by Peters and Timmerhaus [6].)

System benzene + toluene

$$\left[\frac{R}{R_{\min}} \right]_{\text{optimum}} \cong 1.1$$



Reflux Ratio, $R \leftrightarrow$ Number of stages, N

Effect of Reflux Ratio on Annualized Cost of a Distillation Operation

R/R_{\min}	Actual N	Diam., ft	Reboiler Duty, Btu/h	Condenser Duty, Btu/h	Annualized Cost, \$/yr			Total Annualized Cost, \$/yr
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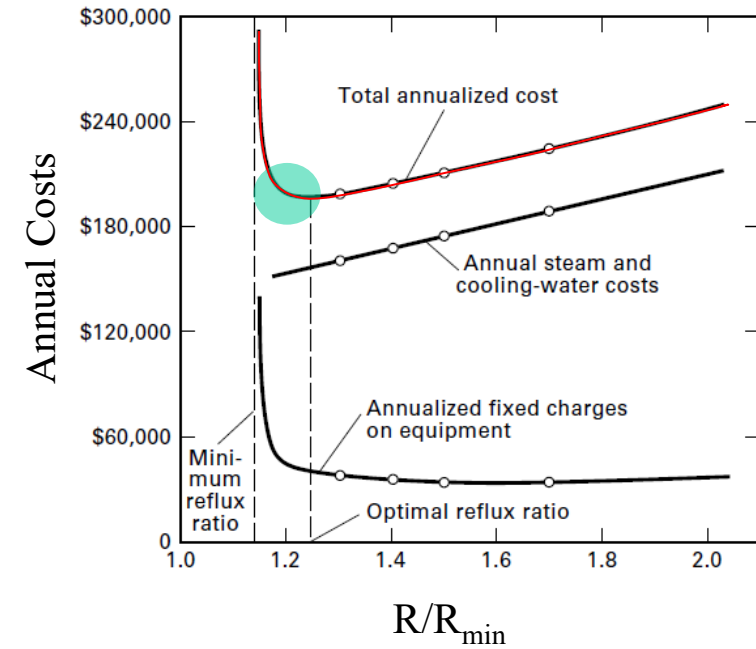
$$R \nearrow \Leftrightarrow L \nearrow$$

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System benzene + toluene

$$\left[\frac{R}{R_{\min}} \right]_{\text{optimum}} \approx 1.1$$

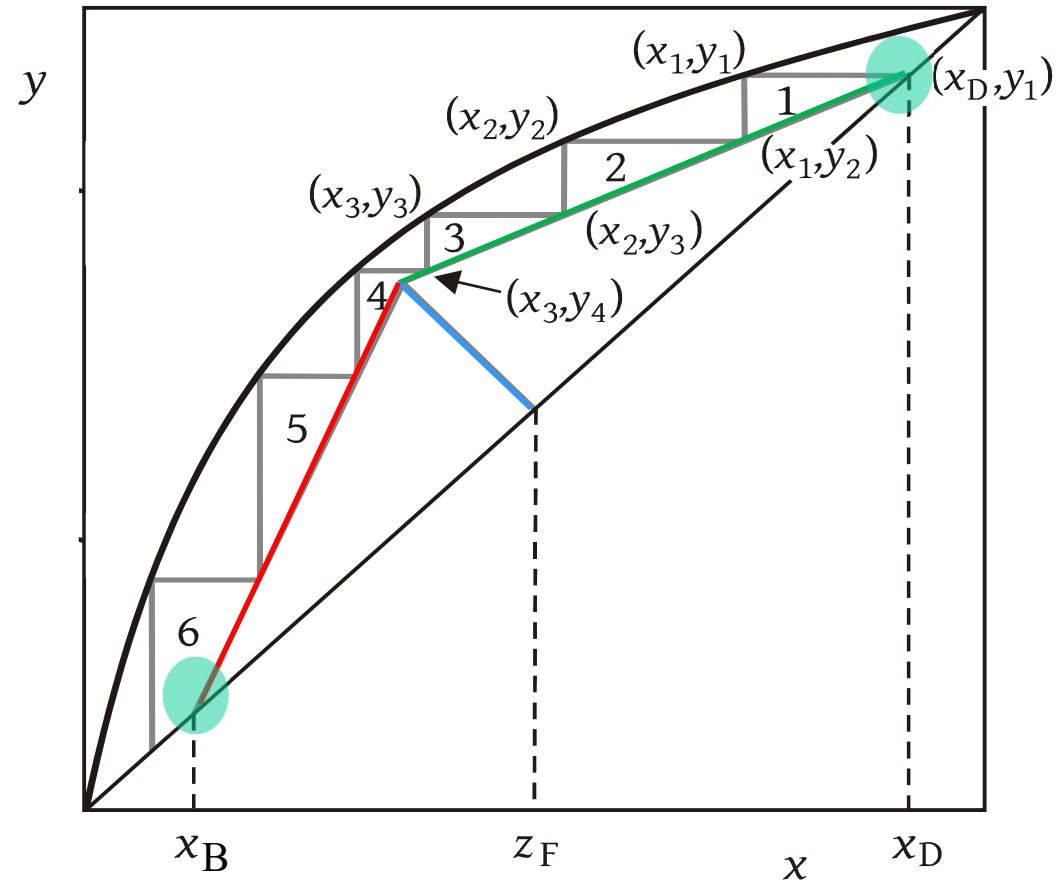
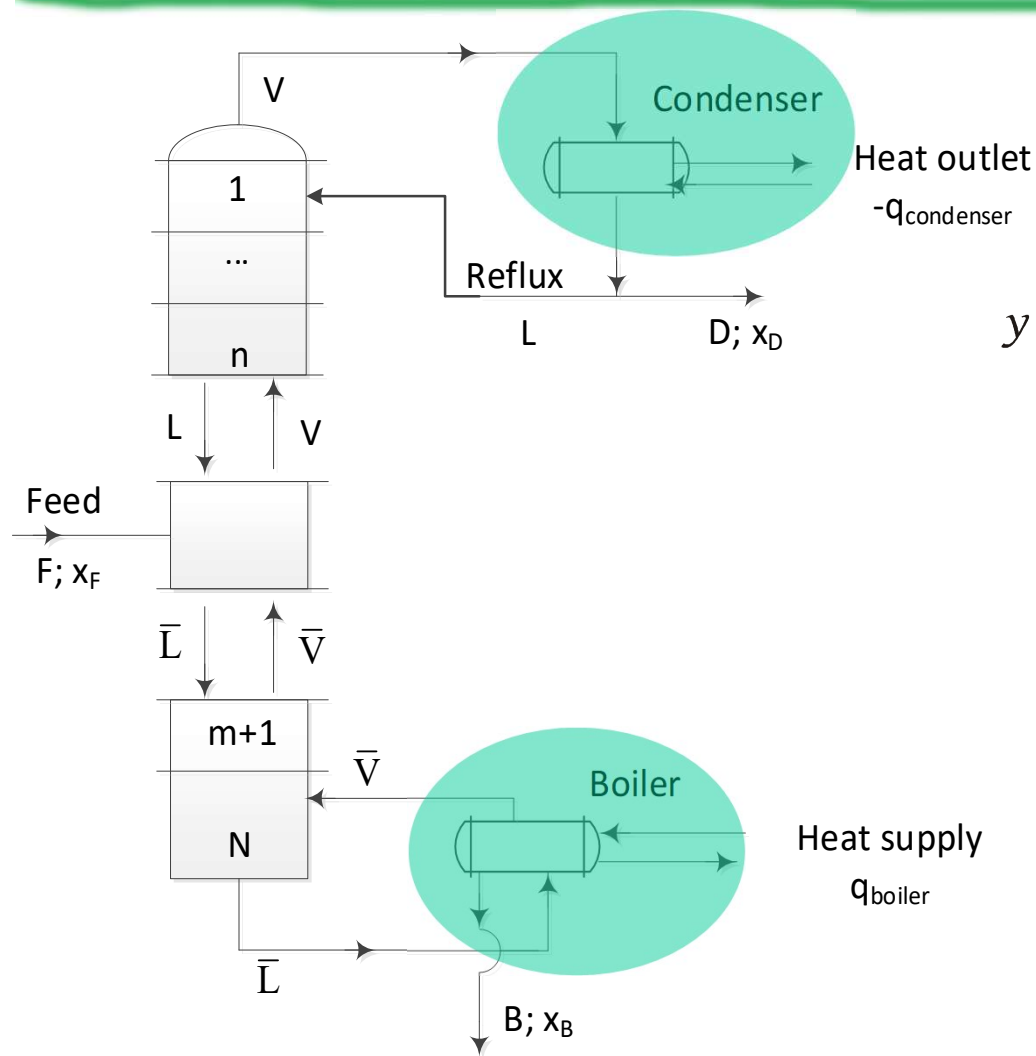
In this example!!!



Summary

- Influence of the reflux ratio on the efficiency of a distillation column
- Optimal reflux ratio
- **Condenser and Boiler in a distillation column**
- Energy Balances to a distillation column
- Energetic balance to the feed stage
- Stage efficiency

Distillation - McCabe - Thiele Method



Condenser and boiler

A distillation unit includes among others:

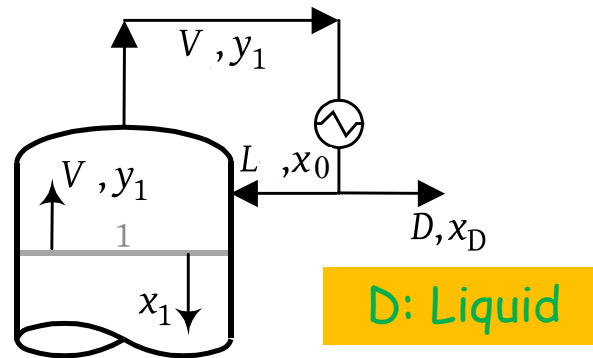
- 1) The distillation column
- 2) The condenser
- 3) The boiler

The McCabe-Thiele method calculates the total number of equilibrium stages of the distillation unit

Depending on the conditions, this number may include or not the boiler and the condenser

Condenser

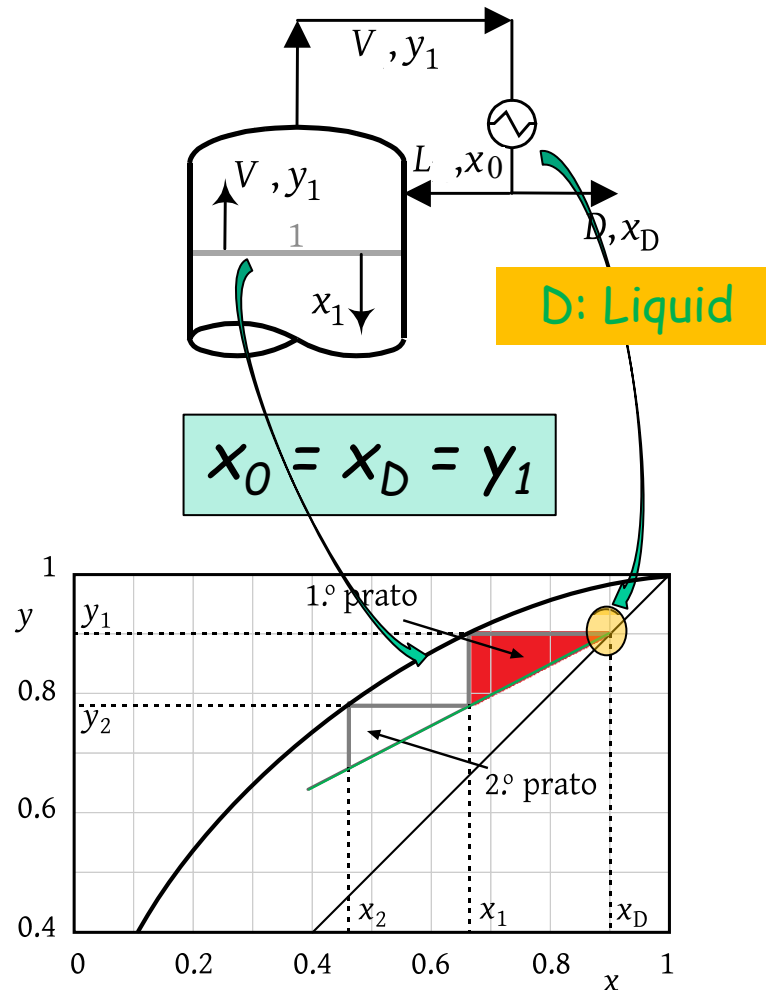
Total Condenser



$$x_0 = x_D = y_1$$

Condenser

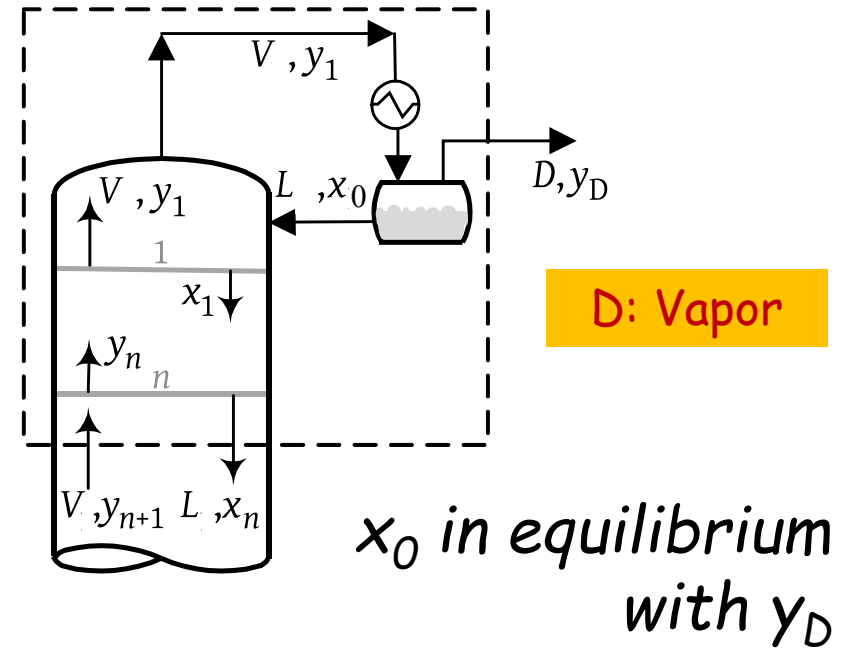
Total Condenser



The Condenser is not a equilibrium stage!

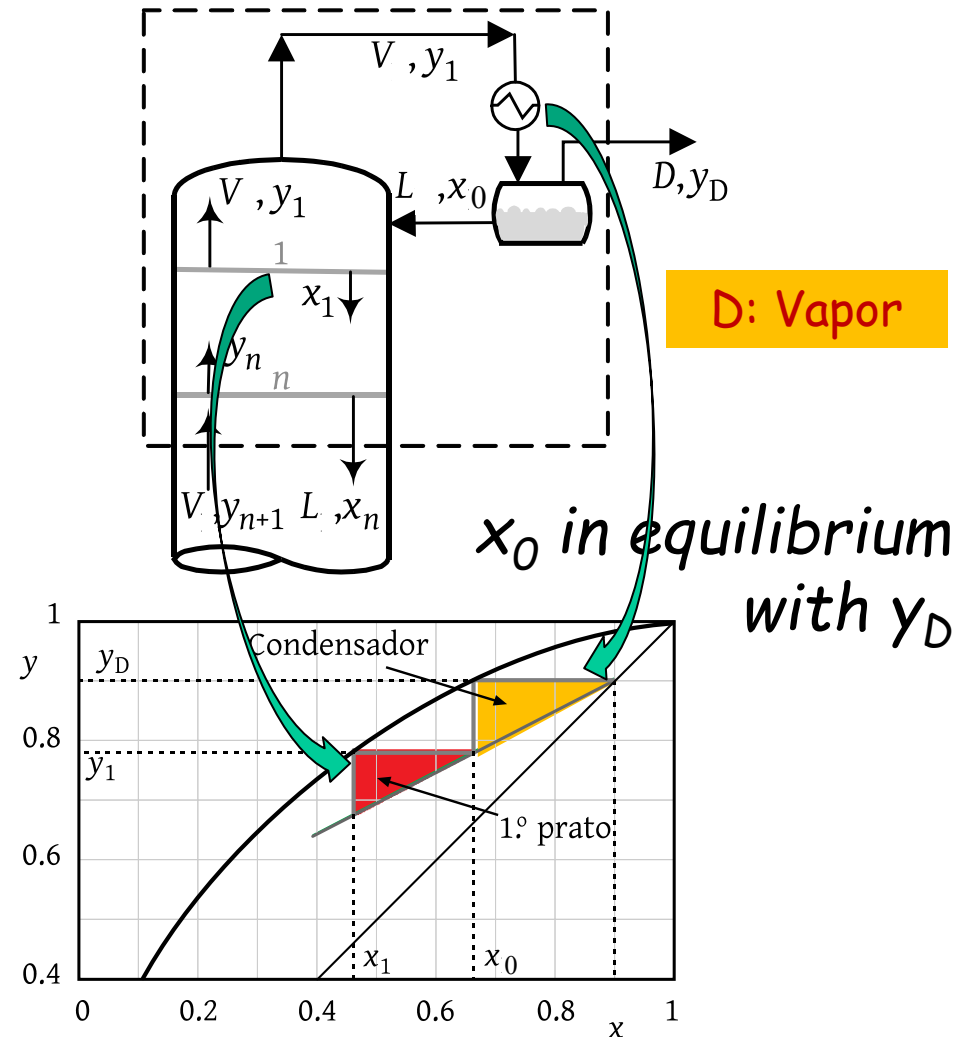
Condenser

Partial Condenser



Condenser

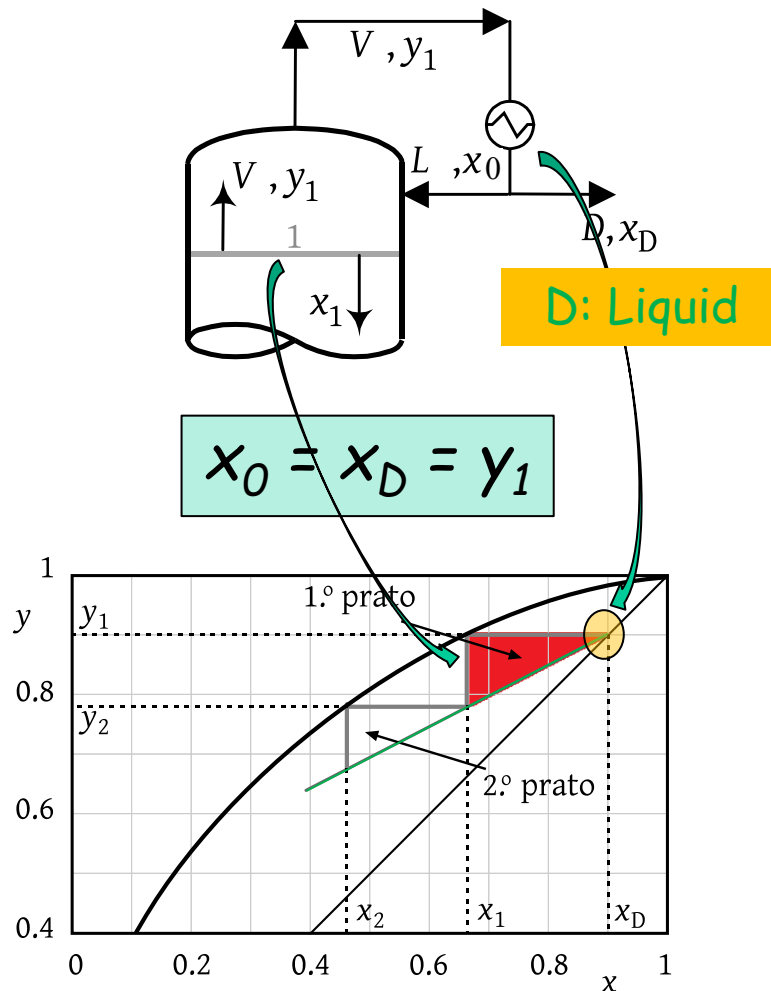
Partial Condenser



The Condenser is an equilibrium stage!

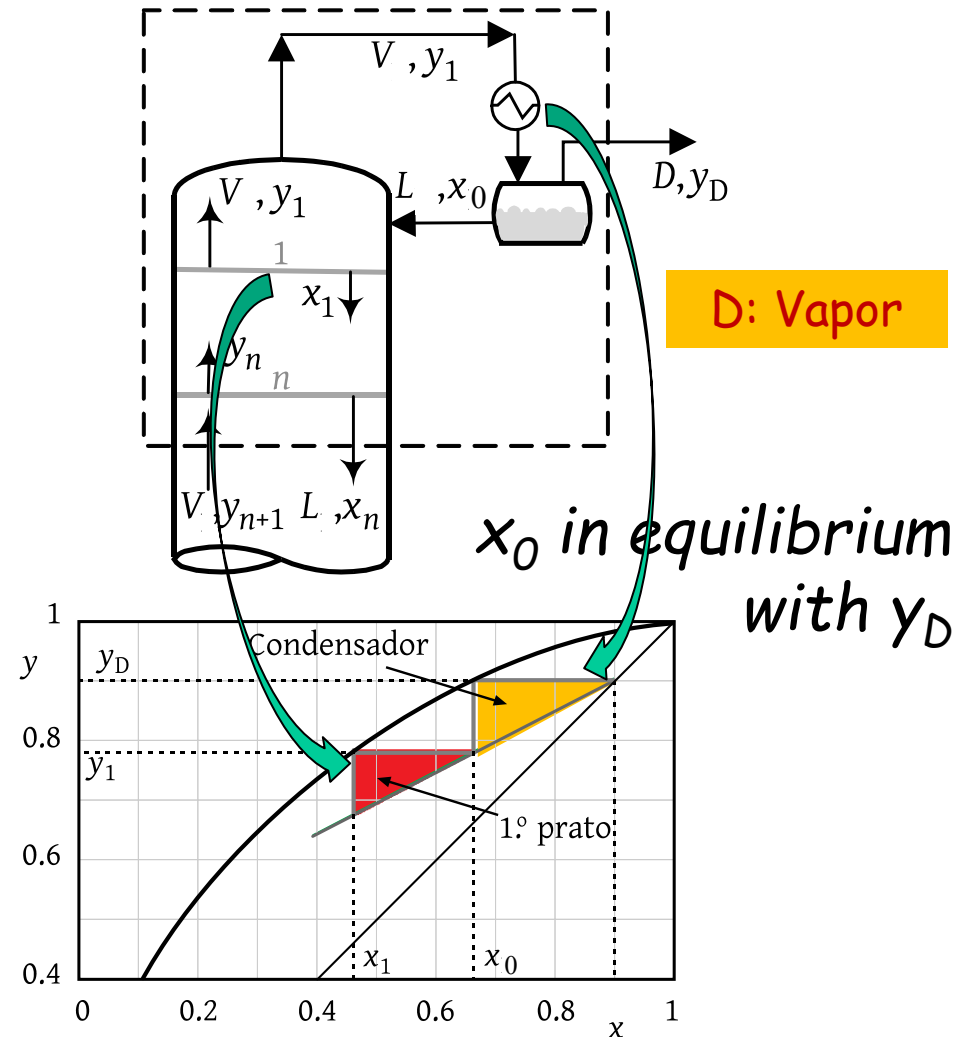
Condenser

Total Condenser



Condenser is not an equilibrium stage

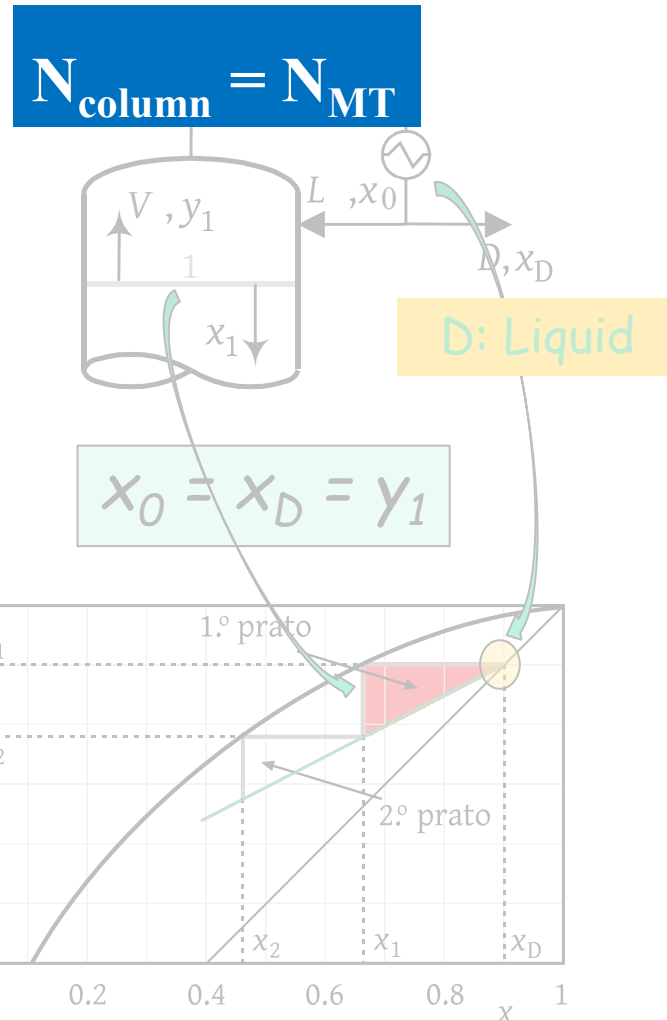
Partial Condenser



Condenser is an equilibrium stage

Condenser

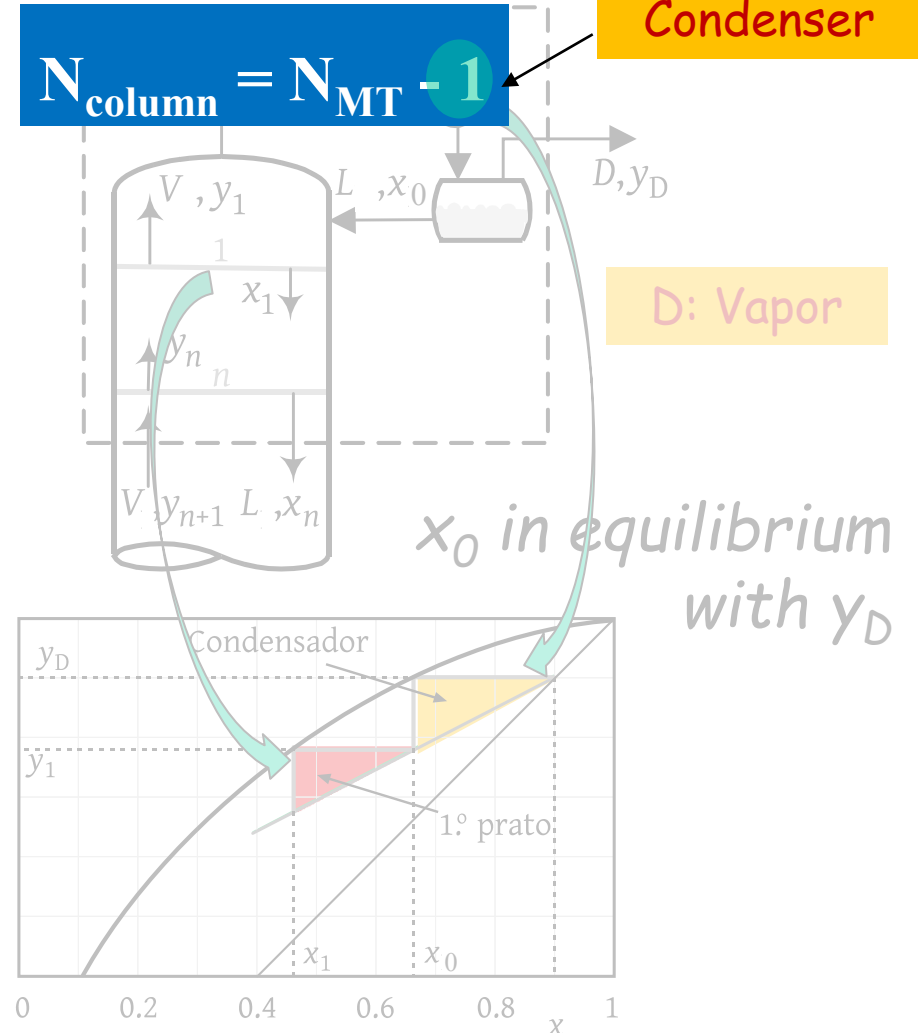
Total Condenser



MT: McCabe-Thiele

Condenser is not an equilibrium stage

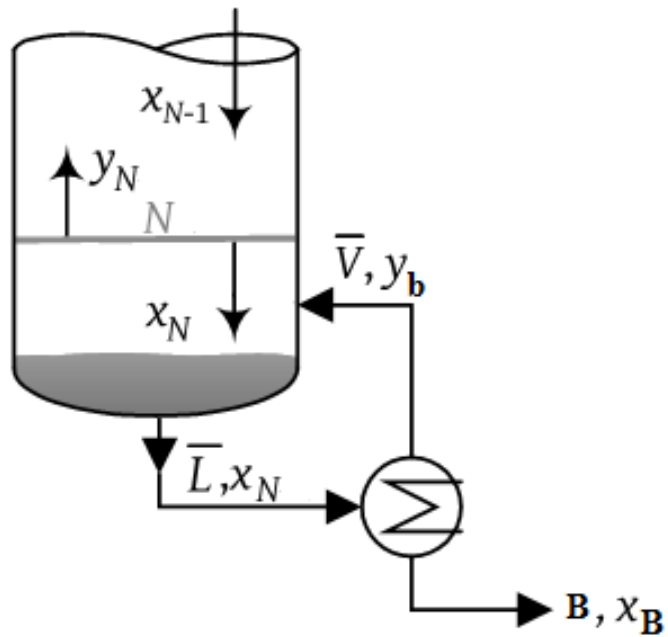
Partial Condenser



Condenser is an equilibrium stage

Boiler

Partial Boiler

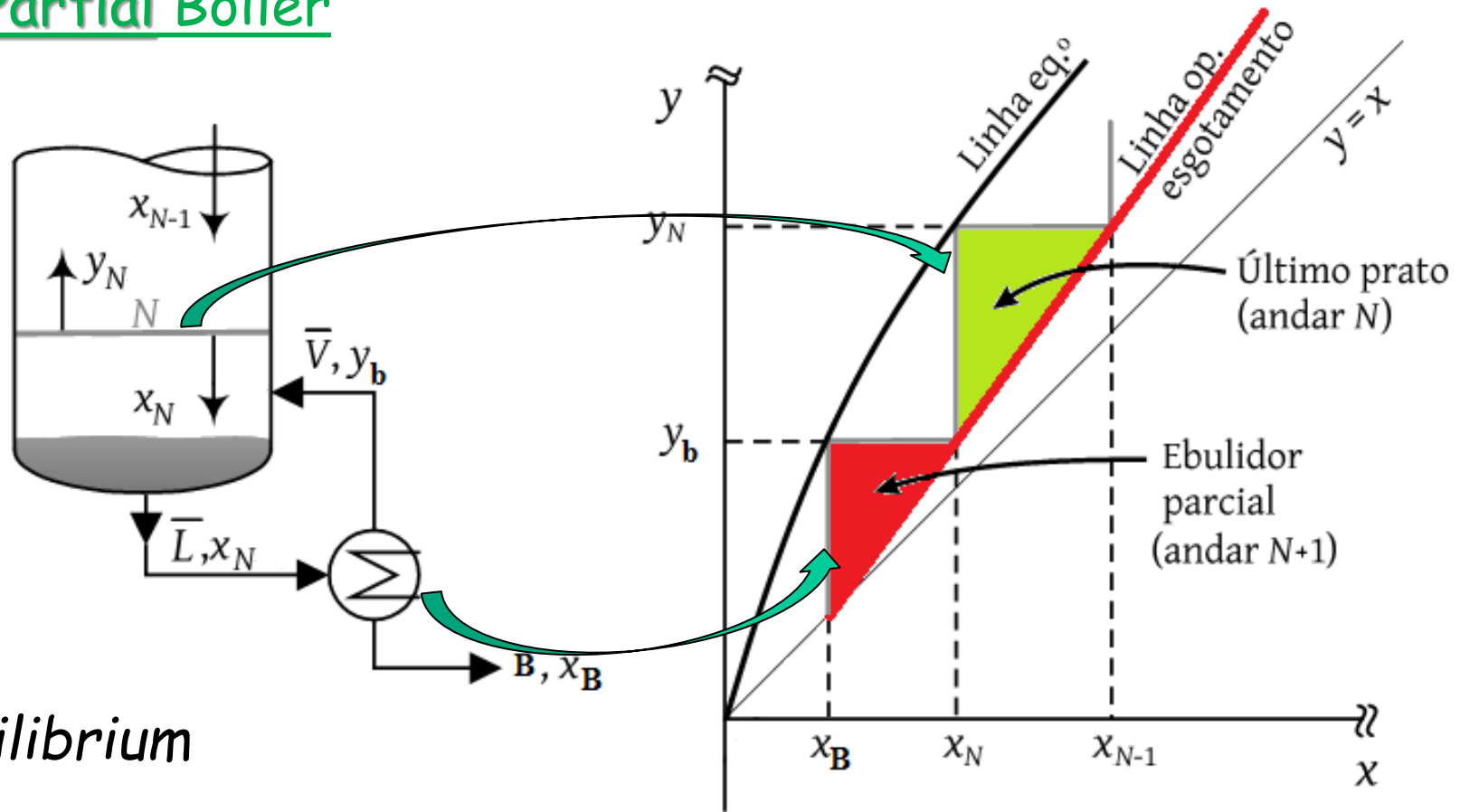


B: Liquid

x_B in equilibrium
with y_b

Boiler

Partial Boiler



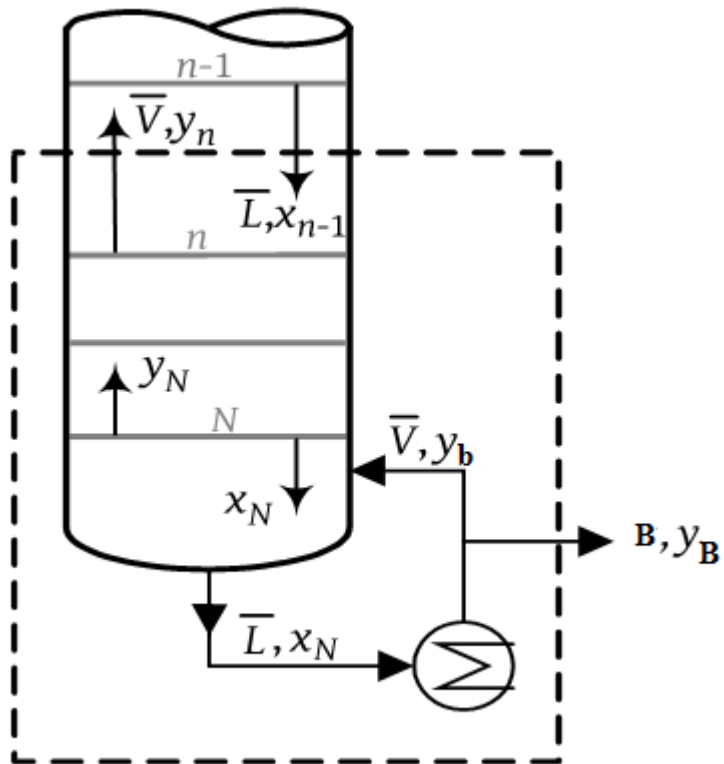
B: Liquid

x_B in equilibrium
with y_b

Boiler is an equilibrium stage

Boiler

Total Boiler

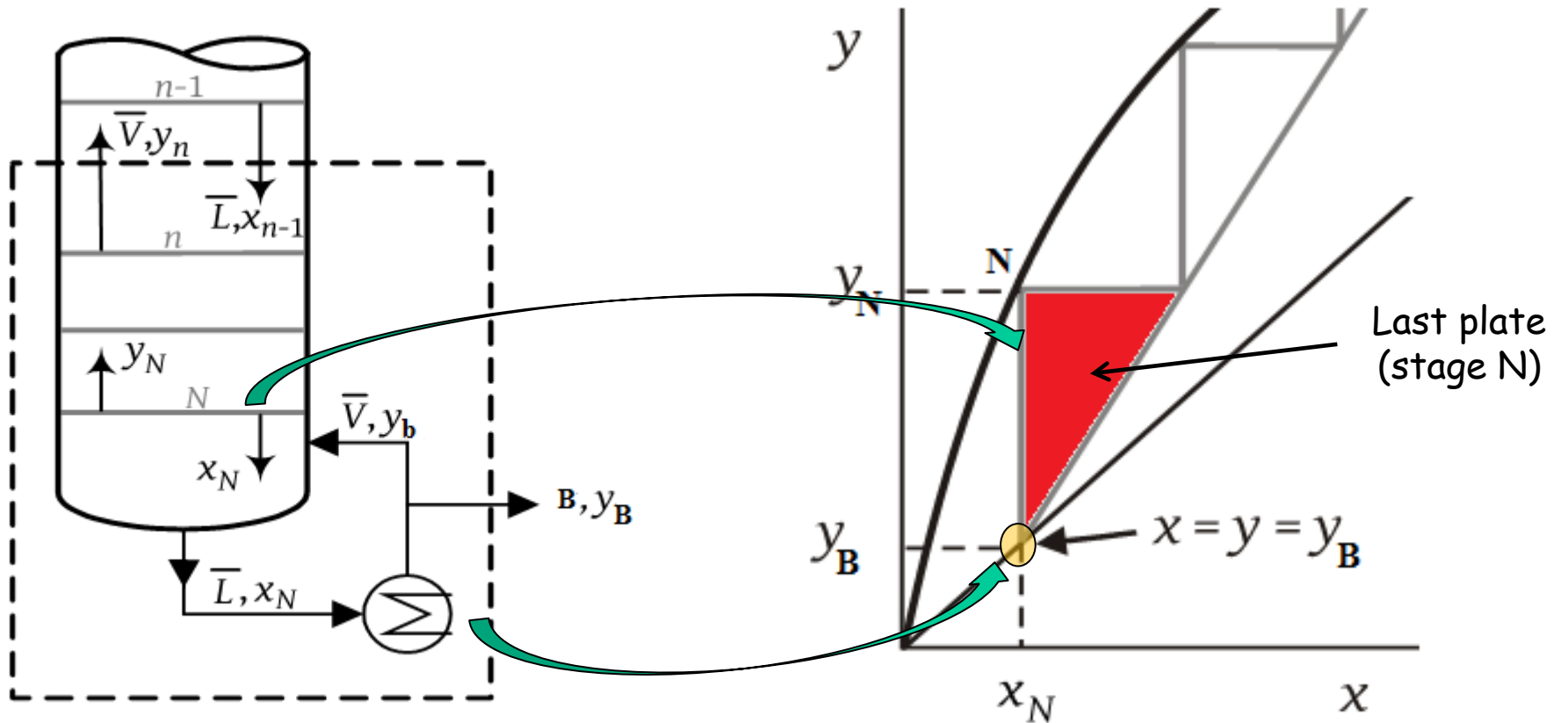


B: Vapor

$$x_N = y_B = y_b$$

Boiler

Total Boiler



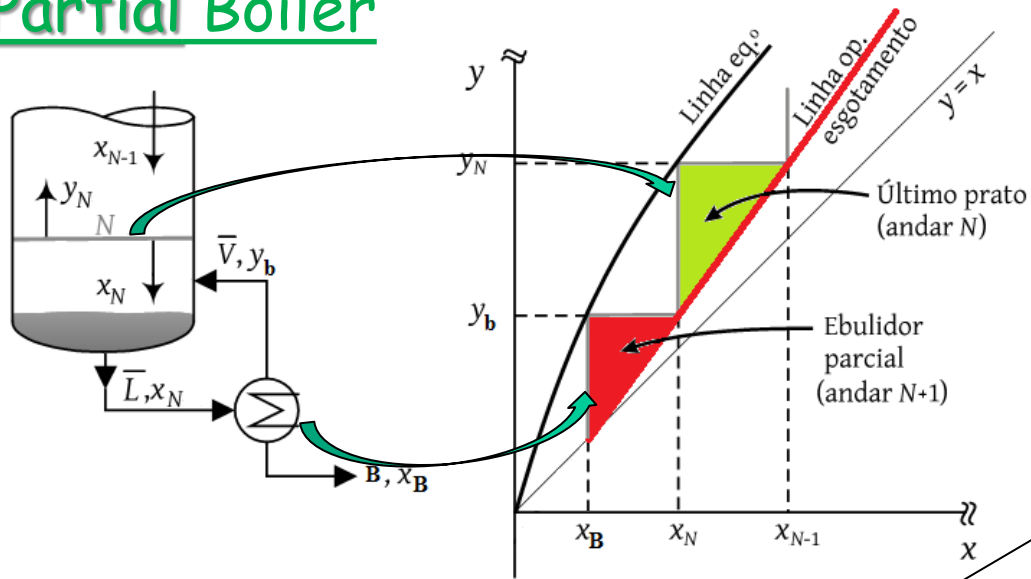
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$$x_N = y_B = y_b$$

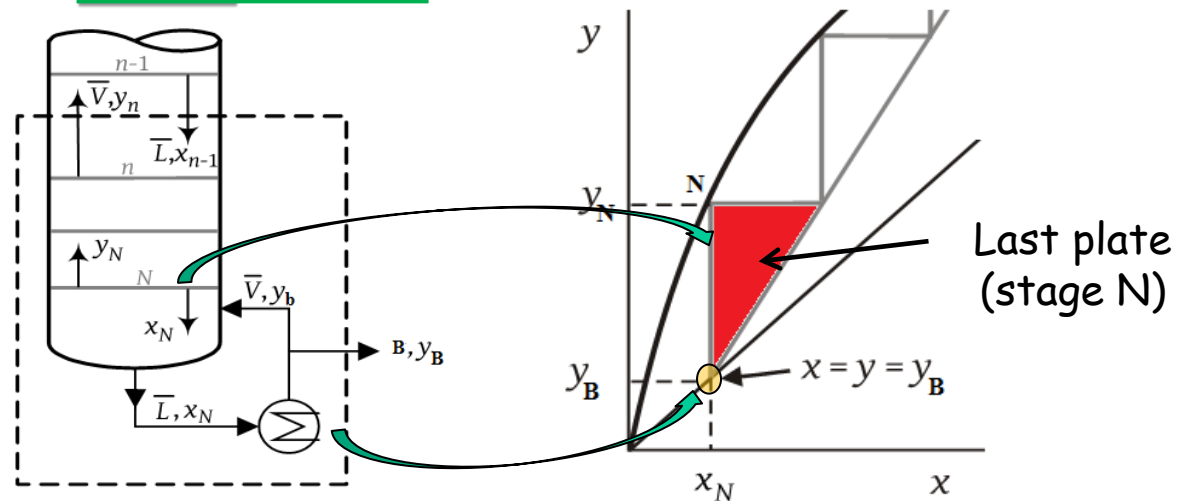
Boiler is not an equilibrium stage

Boiler

Partial Boiler

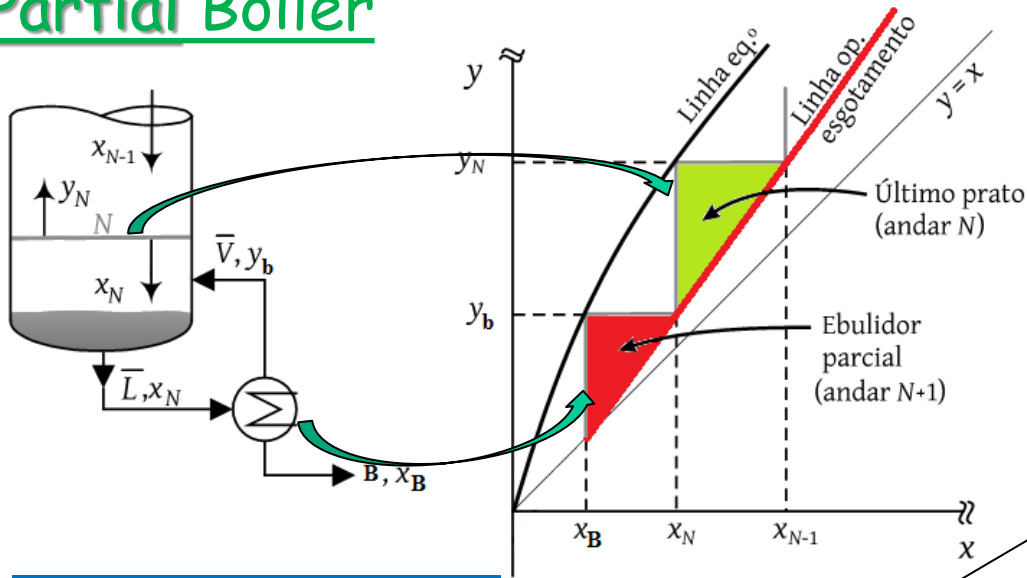


Total Boiler



Boiler

Partial Boiler



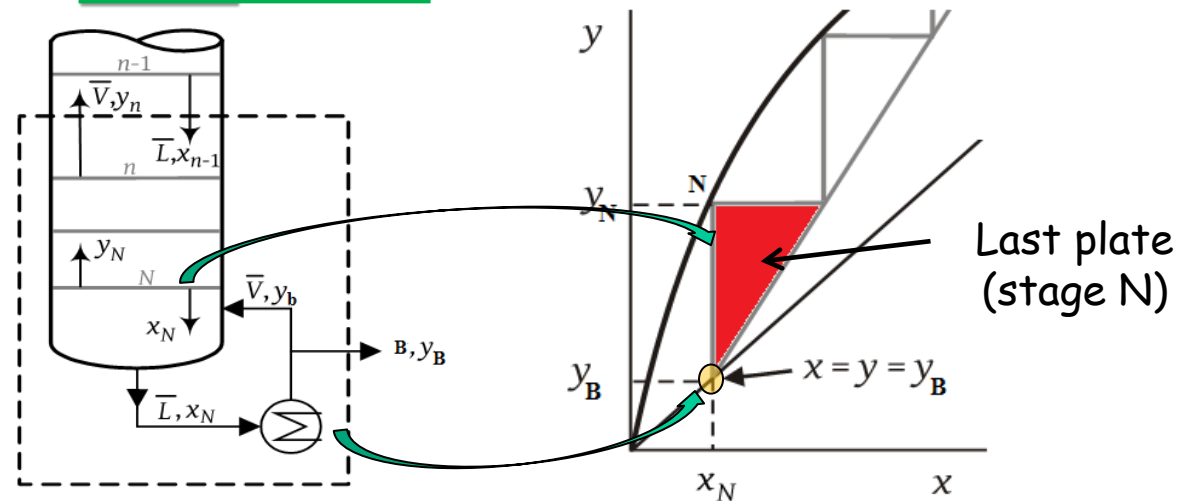
$$N_{\text{column}} = N_{\text{MT}} - 1$$

Boiler

MT: McCabe-Thiele

$$N_{\text{column}} = N_{\text{MT}}$$

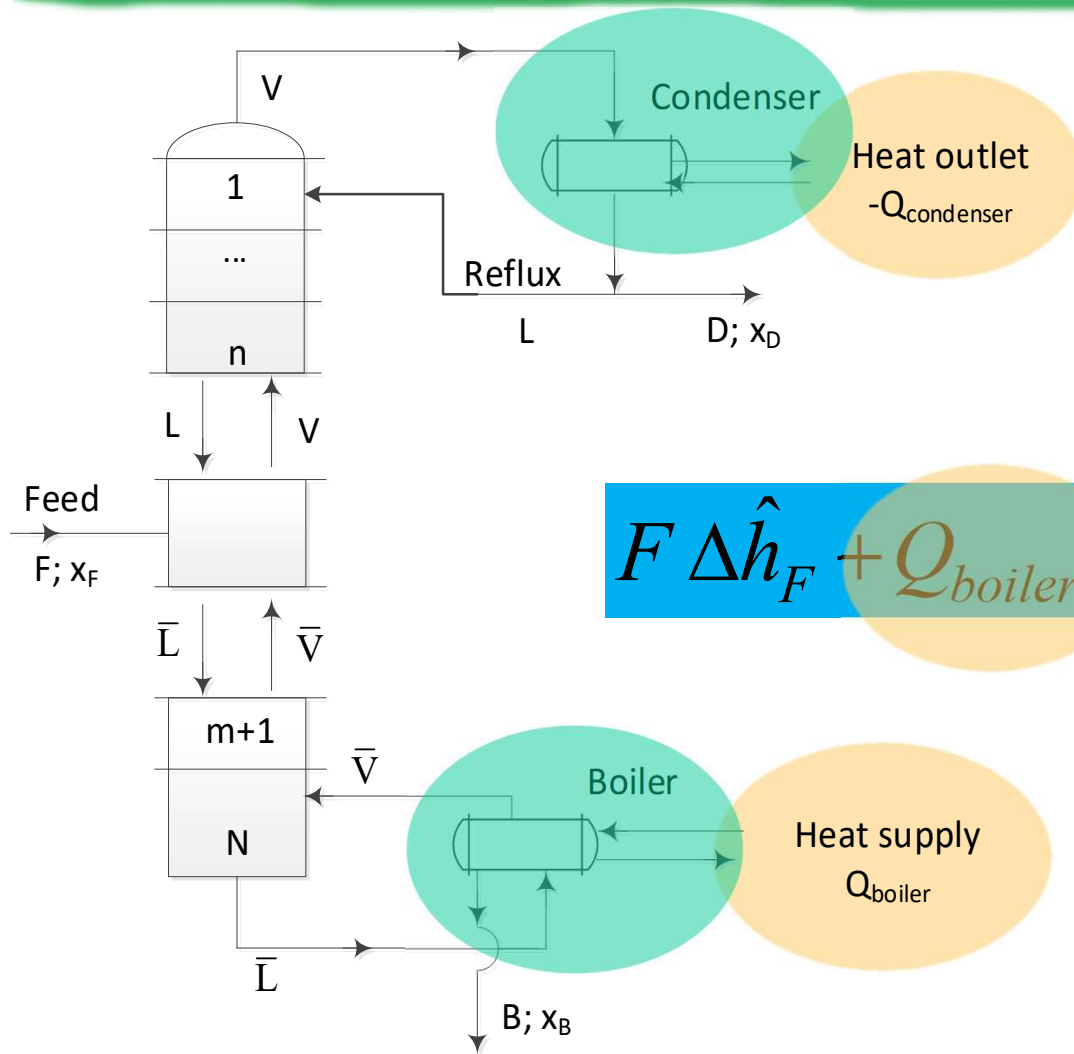
Total Boiler



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- Energetic balance to the feed stage
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Energy balances

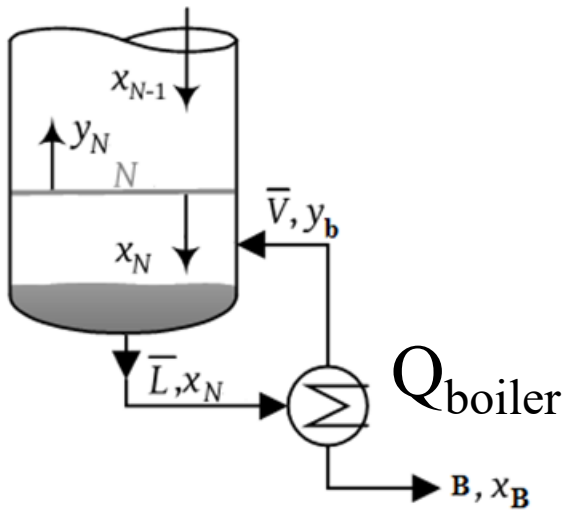


□ Overall balance

$$F \Delta \hat{h}_F + Q_{boiler} = B \Delta \hat{h}_B + D \Delta \hat{h}_D + Q_{condenser}$$

$\Delta \hat{h}_F, \Delta \hat{h}_D, \Delta \hat{h}_B$ [J/mol]: specific enthalpies of feed, distillate and residue

Heat required in the boiler, Q_{boiler}



$$Q_{\text{boiler}} = \bar{V} \cdot \Delta \hat{H}_{\text{vaporization}}$$

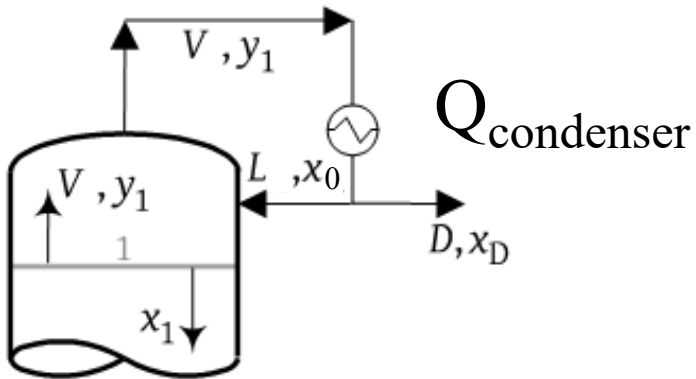
\bar{V} : molar rate of vapour produced in the boiler

$\Delta \hat{H}_{\text{vap}}$: molar enthalpy of vaporization of mixture

Partial boiler

(☞ considering sensible heats negligible!)

Heat to be removed from condenser, $Q_{\text{condenser}}$



$$Q_{\text{condenser}} = V \cdot \Delta \hat{H}_{\text{condenser}}$$

Total Condenser

V : molar rate of vapour fed to the condenser

$\Delta \hat{H}_{\text{cond}}$: molar enthalpy of condensation of mixture

Problem 1

A distillation column is used to separate 100 mol/h of a mixture made of two compounds A and C. The feed mixture consists of equal parts of saturated vapor and liquid, with a molar composition of 35% A. It is intended to obtain a distillate with a molar composition of 93% A and a residue with a molar composition of 97.8% C. The reflux ratio is equal to 4.

f) Calculate the heat involved in the boiler and in the condenser.

$$\Delta \hat{H}^{\text{vap}}_{(\text{mistura A+C})} = 31.2 \text{ kJ/mol}$$

Heat required in the boiler, Q_{boiler}

$$Q_{\text{boiler}} = \bar{V} \cdot \Delta \hat{H}_{\text{vaporization}}$$

\bar{V} : vapour molar rate produced in the boiler

Heat to be removed from condenser, $Q_{\text{condenser}}$

$$Q_{\text{condenser}} = V \cdot \Delta \hat{H}_{\text{condenser}}$$

V : molar rate of vapour fed to the condenser

Material balances - determine V, L, \bar{V}, \bar{L}

$$F = 100 \text{ mol/h}$$

$$D = 36.12 \text{ mol/h}$$

$$B = 63.88 \text{ mol/h}$$

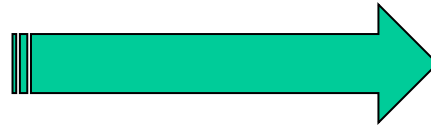
$$x_F = 0.35$$

$$x_D = 0.93$$

$$x_B = 0.022$$

$$R = 4$$

$$i = 0.5$$



Determine L, V
 \bar{L}, \bar{V}

$$R = L/D$$

$$i = \frac{\bar{L} - L}{F}$$

$$V = L + D$$

$$\bar{V} = \bar{L} - B$$

$$L = 144.5 \text{ mol/h}$$

$$V = 180.6 \text{ mol/h}$$

$$\bar{L} = 194.5 \text{ mol/h}$$

$$\bar{V} = 130.6 \text{ mol/h}$$

Heat required in the boiler, Q_{boiler}

$$Q_{\text{boiler}} = \bar{V} \cdot \Delta \hat{H}_{\text{vaporization}}$$

$$\Delta \hat{H}^{\text{vap}} = 31.2 \text{ kJ/mol}$$

$$\bar{V} = 130.6 \text{ mol/h}$$

$$Q_{\text{caldeira}} = 4,074.7 \text{ kJ/h}$$

Heat to be removed from condenser, $Q_{\text{condenser}}$

$$Q_{\text{condenser}} = V \cdot \Delta \hat{H}_{\text{condenser}}$$

$$\Delta \hat{H}^{\text{cond}} = -31.2 \text{ kJ/mol}$$

$$V = 180.6 \text{ mol/h}$$

$$Q_{\text{condensador}} = -5,634.7 \text{ kJ/h}$$

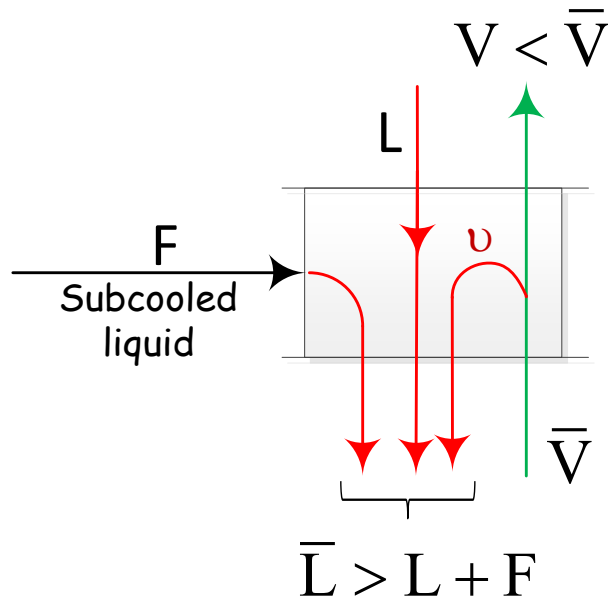
Summary

- Influence of the reflux ratio on the efficiency of a distillation column
- Optimal reflux ratio
- Condenser and Boiler in a distillation column
- Energy Balances to a distillation column
- **Energetic balance to the feed stage**
- Stage efficiency

Physical state of the Feed

$$T_F < T_L$$

a) Subcooled liquid



$$\bar{L} = L + F + v$$

$$i = \frac{\bar{L} - L}{F}$$

$$i = \frac{(L + F + v) - L}{F}$$

$$i = \frac{F + v}{F} > 1$$

Value of v : to be determined by an energy balance to feed plate

Example

A distillation column is used to separate a binary mixture A+C. We know that the feed enters the column at 17 °C. Estimate the slope of the feed operating line.

Feed mixture data:

- Specific heat: 225.4 J/(mol.K)
- Boiling point: 63 °C
- Enthalpy of vaporization: 25 900 J/mol

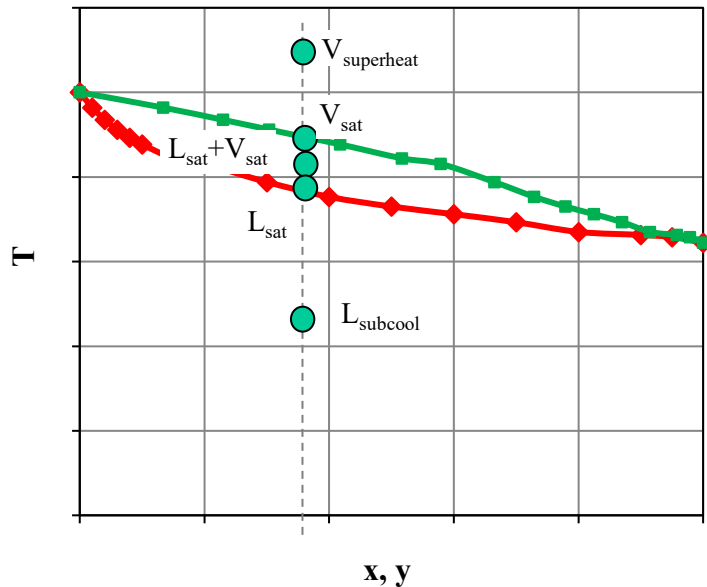
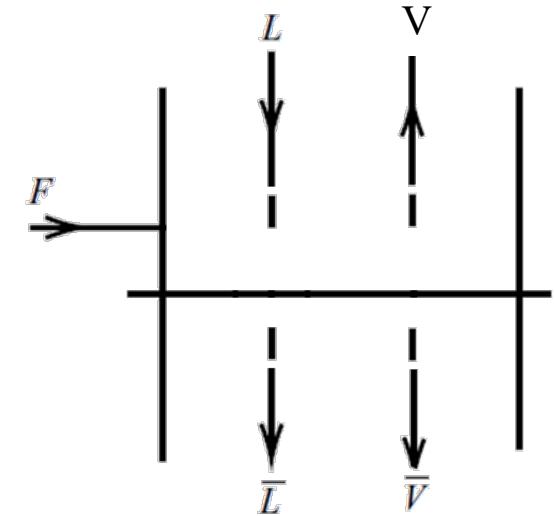
$$y_i = \frac{i}{i-1} x_i - \frac{x_F}{i-1}$$

③ Operating line of feed

$$y_i = \frac{i}{i-1} x_i - \frac{x_F}{i-1}$$

$$i = \frac{\bar{L} - L}{F}$$

- Temperature: 17 °C
- Specific heat: 225.4 J/(mol.K)
- Boiling point: 63 °C
- Enthalpy of vaporization: 25 900 J/mol

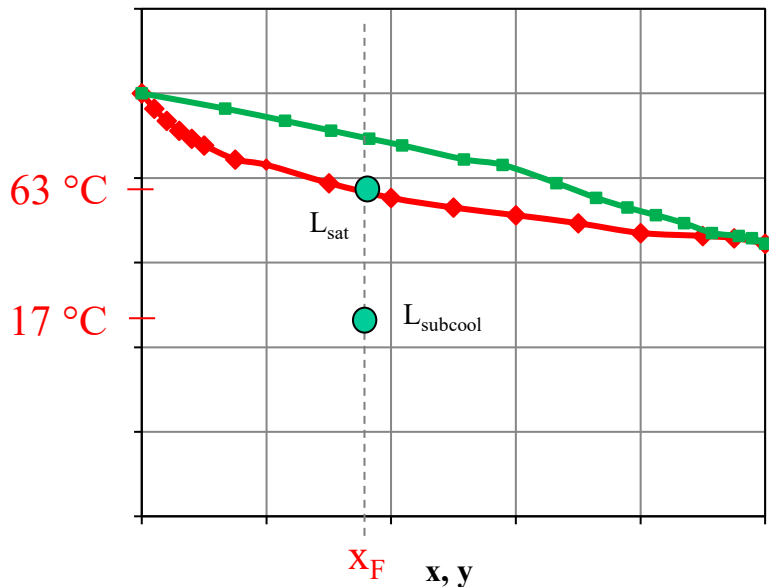
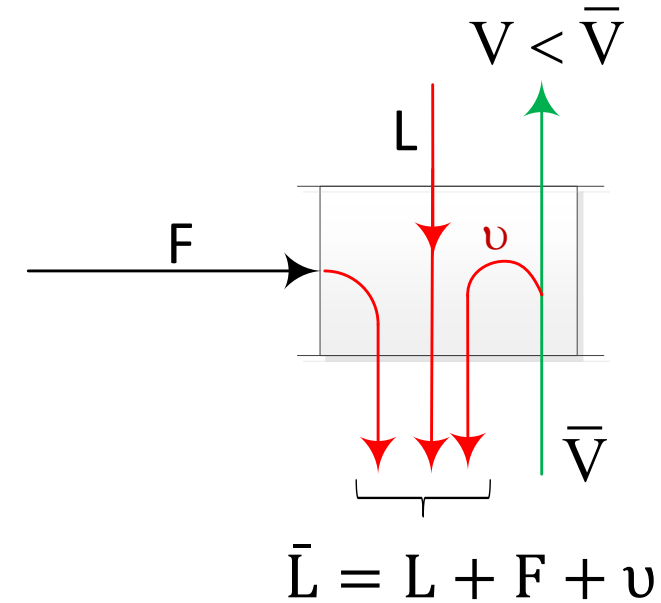


③ Operating line of feed

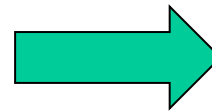
$$y_i = \frac{i}{i-1} x_i - \frac{x_F}{i-1}$$

$$i = \frac{\bar{L} - L}{F}$$

- Temperature: 17 °C
- Specific heat: 225.4 J/(mol.K)
- Boiling point: 63 °C
- Enthalpy of vaporization: 25 900 J/mol



$$T_{\text{feed}} < T_{\text{saturated liquid}}$$



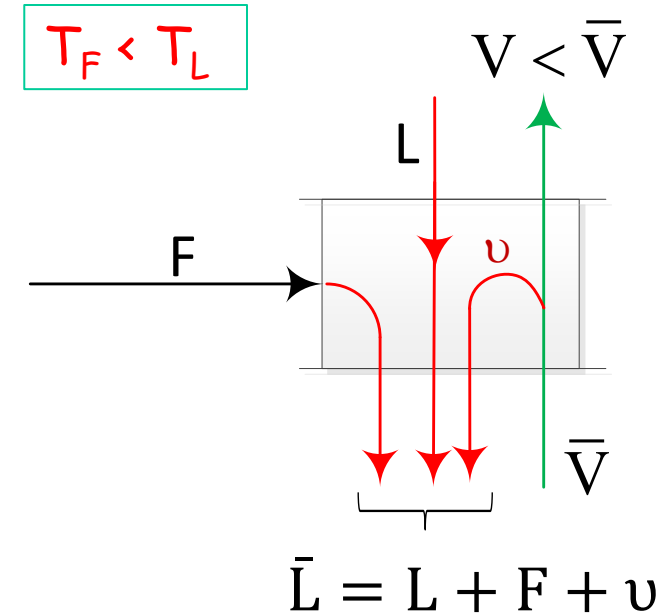
Subcooled liquid!

③ Operating line of feed

$$y_i = \frac{i}{i-1} x_i - \frac{x_F}{i-1}$$

$$i = \frac{\bar{L} - L}{F}$$

- Temperature: 17 °C
- Specific heat: 225.4 J/(mol.K)
- Boiling point: 63 °C
- Enthalpy of vaporization: 25 900 J/mol



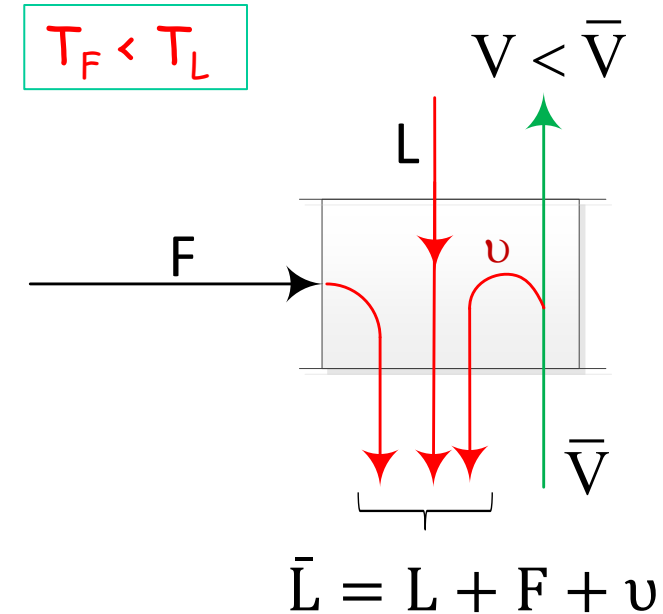
How to estimate v ?  Energy balance to the feed plate

③ Operating line of feed

$$y_i = \frac{i}{i-1} x_i - \frac{x_F}{i-1}$$

$$i = \frac{\bar{L} - L}{F}$$

- Temperature: 17 °C
- Specific heat: 225.4 J/(mol.K)
- Boiling point: 63 °C
- Enthalpy of vaporization: 25 900 J/mol



Energy balance to the feed plate

$$(\Delta T = T_L - T_F)$$

$T_{\text{eb, mixture}} =$

$$F \cdot c_p^{\text{liq}} \cdot \Delta T = v \cdot \Delta \hat{H}_{\text{condensation}}$$

Sensible heat to
warm F moles of
feed

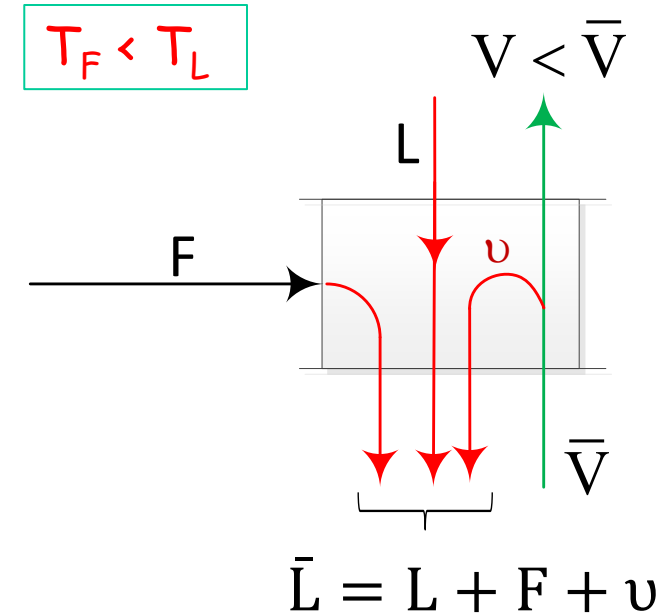
Latent heat resulting
from the condensation
of v moles of vapor

③ Operating line of feed

$$y_i = \frac{i}{i-1} x_i - \frac{x_F}{i-1}$$

$$i = \frac{\bar{L} - L}{F}$$

- Temperature: 17 °C
- Specific heat: 225.4 J/(mol.K)
- Boiling point: 63 °C
- Enthalpy of vaporization: 25 900 J/mol



Energy balance to the feed plate

$$F \cdot c_p^{liq} \cdot \Delta T = v \cdot \Delta \hat{H}_{condensation}$$

Sensible heat to
warm F moles of
feed

Latent heat resulting
from the condensation
of v moles of vapor

$$v = \frac{F \cdot c_p^{liq} \cdot \Delta T}{\Delta \hat{H}_{condensation}}$$

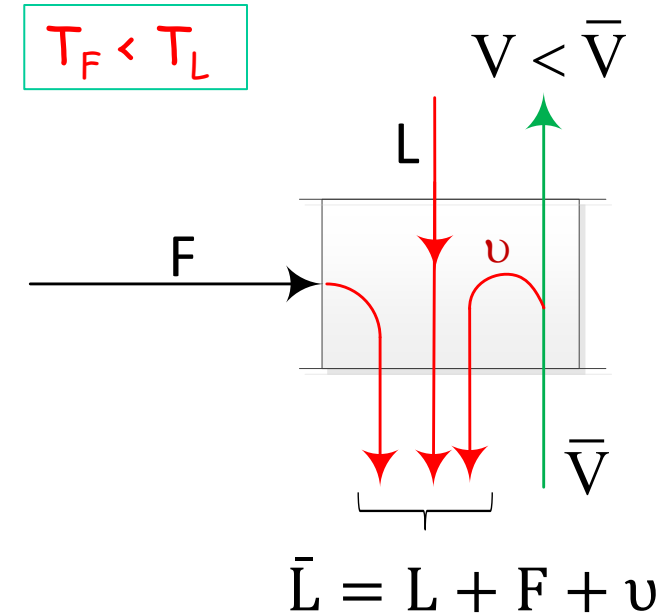
③ Operating line of feed

$$y_i = \frac{i}{i-1} x_i - \frac{x_F}{i-1}$$

$$i = \frac{\bar{L} - L}{F}$$

- Temperature: 17 °C
- Specific heat: 225.4 J/(mol.K)
- Boiling point: 63 °C
- Enthalpy of vaporization: 25 900 J/mol

$$v = \frac{F \cdot c_p^{liq} \cdot \Delta T}{\Delta \hat{H}_{condensation}}$$



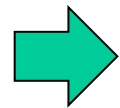
➡
$$v = \frac{225.4 \text{ Jmol}^{-1} \text{K}^{-1} (336 \text{ K} - 290 \text{ K})}{25900 \text{ Jmol}^{-1}} F = 0.4 F$$

③ Operating line of feed

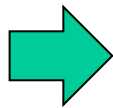
$$y_i = \frac{i}{i-1} x_i - \frac{x_F}{i-1}$$

$$i = \frac{\bar{L} - L}{F}$$

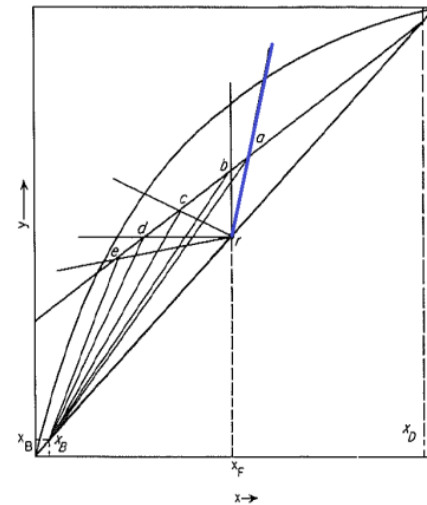
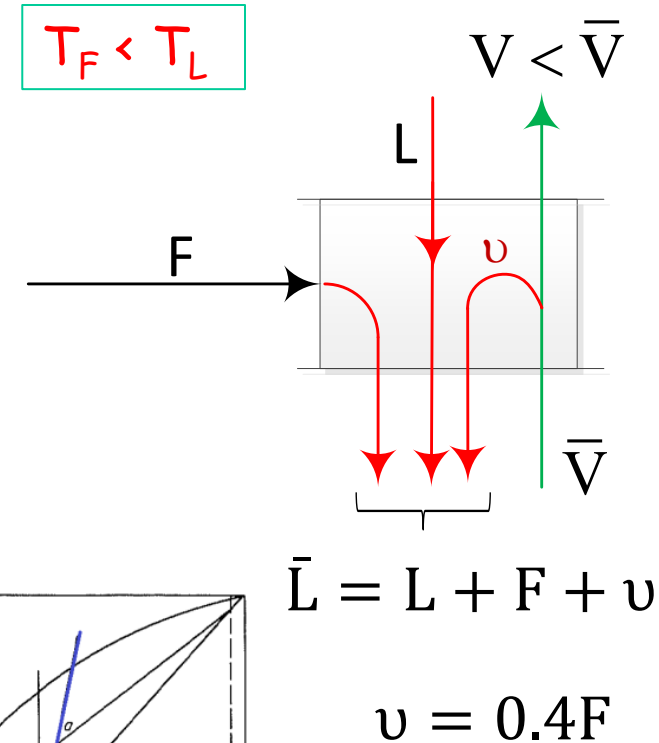
- Temperature: 17 °C
- Specific heat: 225.4 J/(mol.K)
- Boiling point: 63 °C
- Enthalpy of vaporization: 25 900 J/mol



$$i = \frac{\bar{L} - L}{F} = \frac{(L + F + 0.4F) - L}{F} = 1.4$$



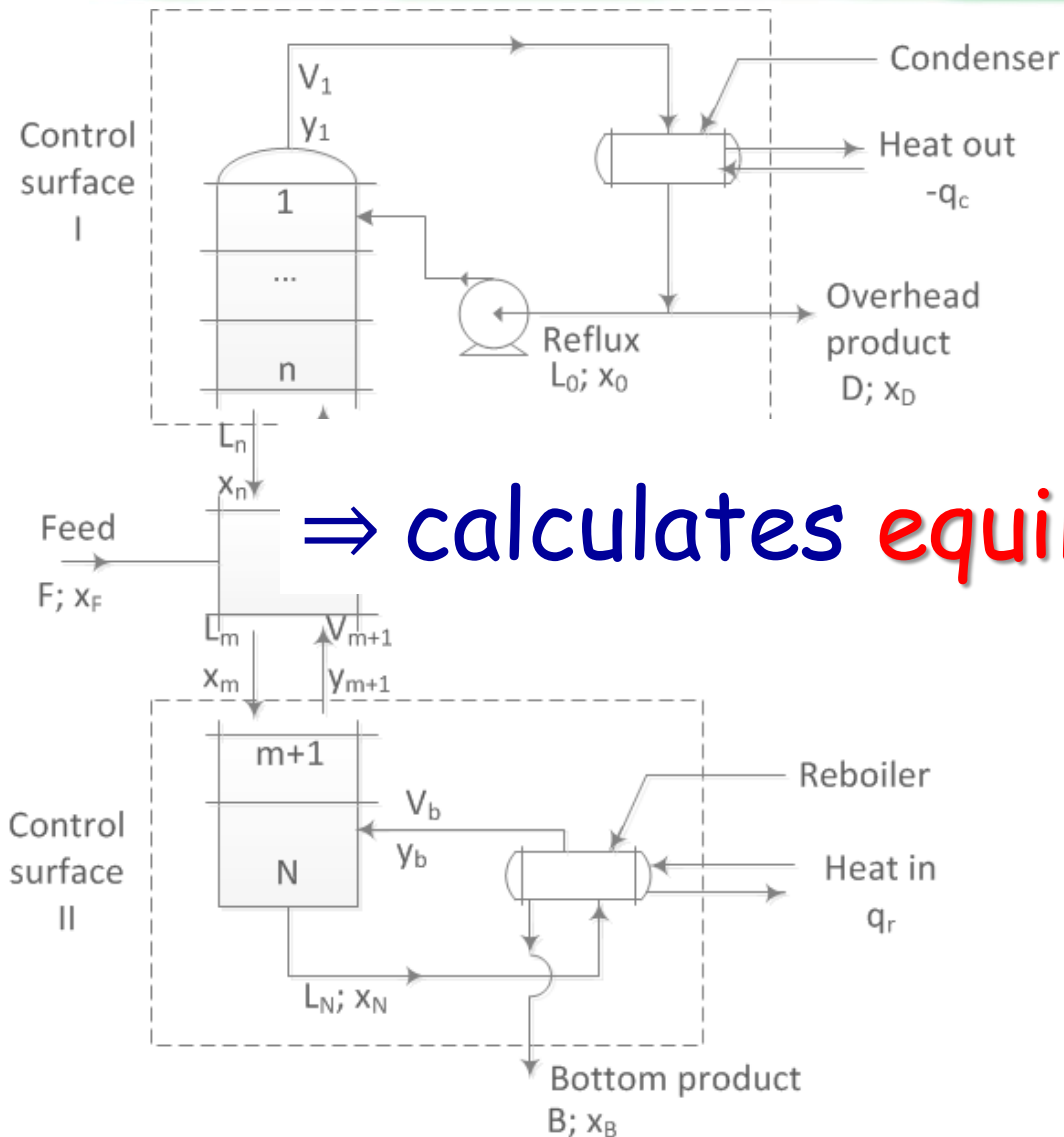
$$y_i = \frac{i}{i-1} x_i - \frac{x_F}{i-1} = 3.5x_i - 0.685$$



Summary

- Influence of the reflux ratio on the efficiency of a distillation column
- Optimal reflux ratio
- Condenser and Boiler in a distillation column
- Energy Balances to a distillation column
- Energetic balance to the feed stage
- **Stage efficiency**

McCabe - Thiele Method



⇒ calculates equilibrium stages!

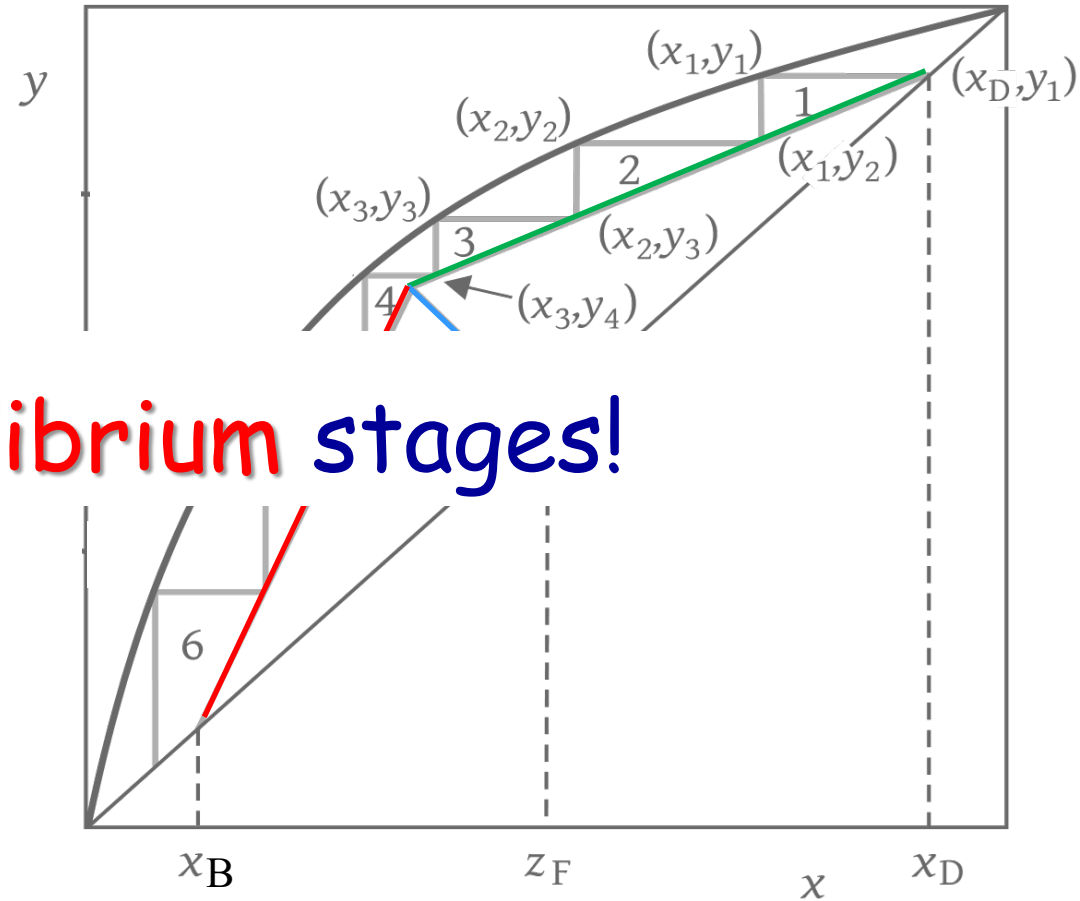


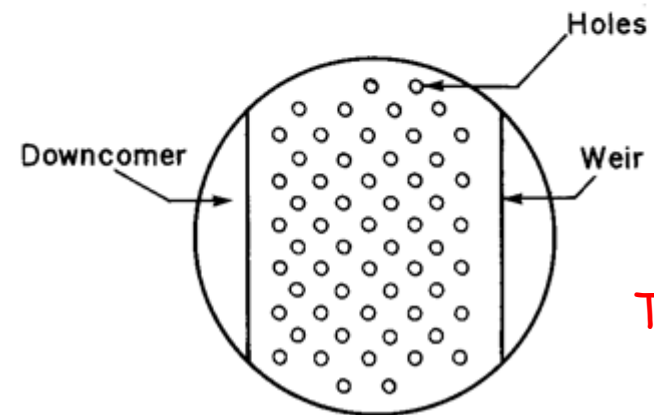
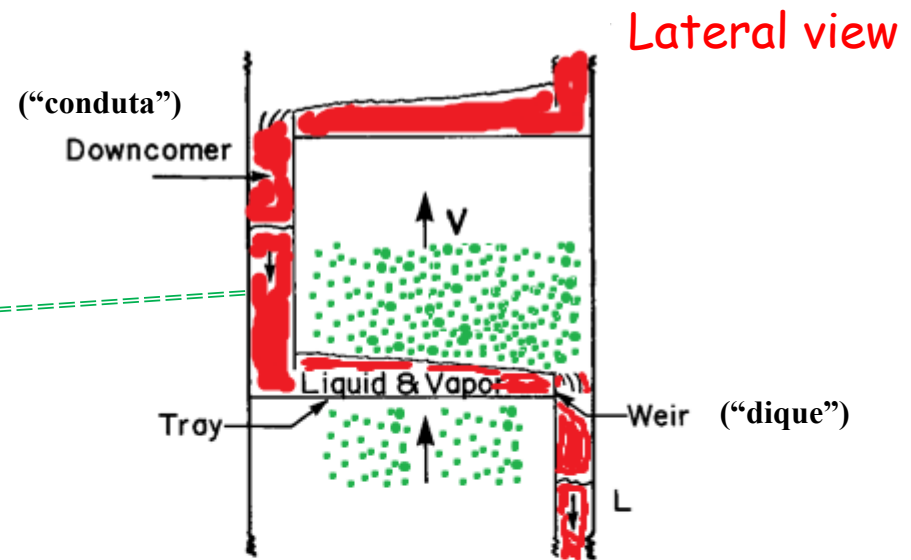
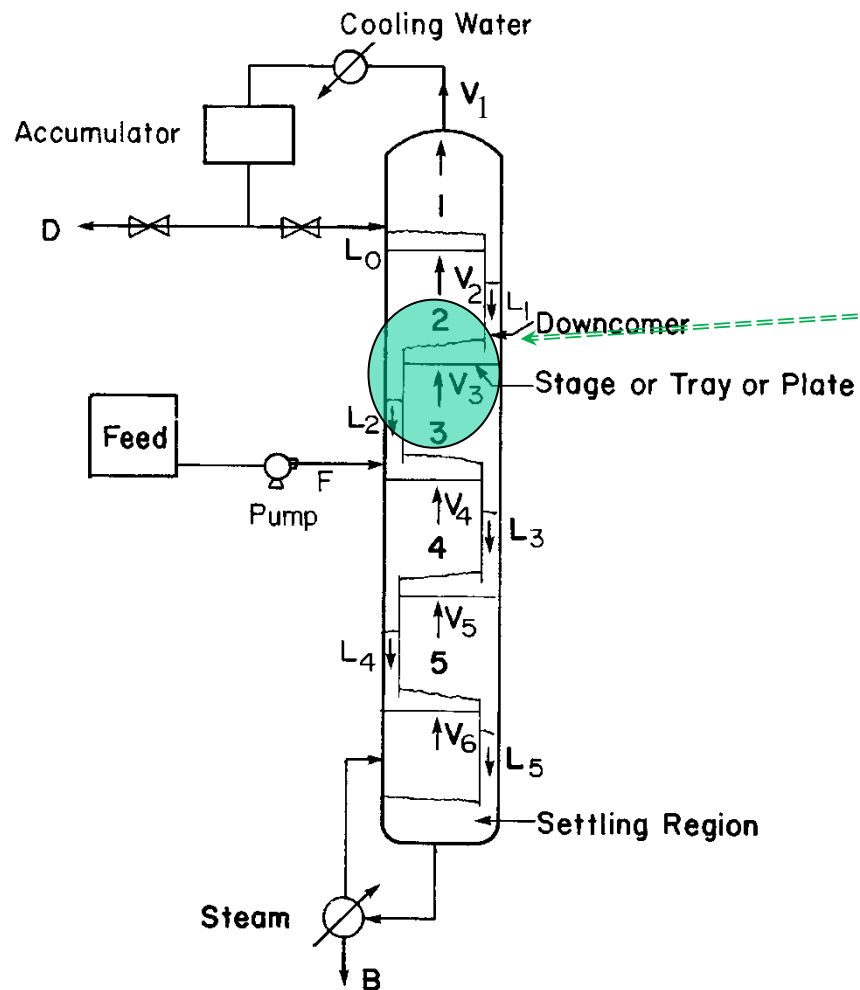
Plate efficiency

In practice, a real plate is **not** an equilibrium plate

Due to, among others...

- Insufficient time of contact between the two phases
- Deficient mixing of the two phases in the plate

⇒ The liquid and vapour streams exiting the plate are not in equilibrium!



Total efficiency (of the column...)

$$E_0 = \frac{N_{\text{equilibrium stages}}}{N_{\text{real stages}}} \leq 1$$

Depends on:

- Physical properties of currents L and V (ρ , μ)
- Flow conditions (V/L)
- Mass and heat transfer (Re, Pr, Sc, Nu, Sh)

It is determined by:

- comparison with performance data from industrial columns
- use of empirical efficiency equations derived from data on industrial columns
- use of semi theoretical models based on mass- and heat-transfer rates
- scale-up from laboratory or pilot plant columns

Total efficiency (of the column...)

$$E_0 = \frac{N_{\text{equilibrium stages}}}{N_{\text{real stages}}} \leq 1$$

For distillation processes where the compounds are close to their boiling points and the viscosity of the liquid phases is low, the efficiency of tray columns is often over 70% and can even reach 100%

- 
- Energetic efficiency on distillation columns

Energetic efficiency

Distillation - intensive energy operation

Optimization of distillation \Rightarrow \diamond optimize operating conditions
 \diamond minimize energy consumption

Practical example: $R \nearrow \Rightarrow$

- Energy consumption \nearrow
- $N_{\text{stages}} \searrow$

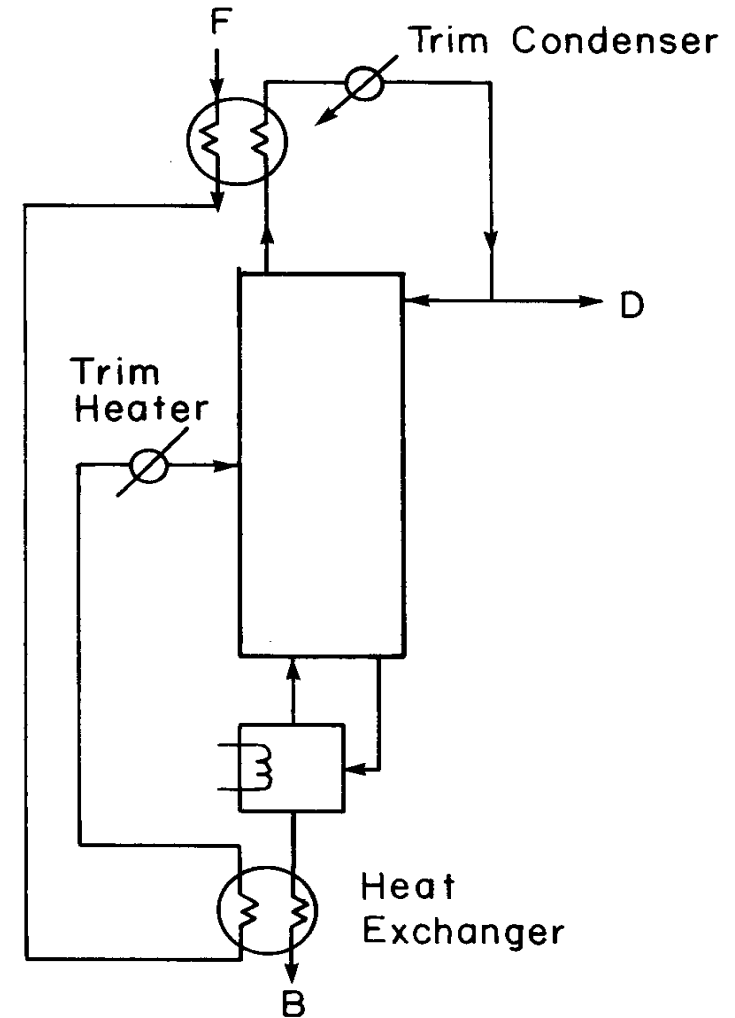
That is, a compromise must be established between equipment costs (i.e. sieve trays) and operational costs (i.e. energy costs)!

Energetic efficiency

How to optimize the energy demanding of a distillation unit?

Energetic integration

Hot streams are used to warm cold streams

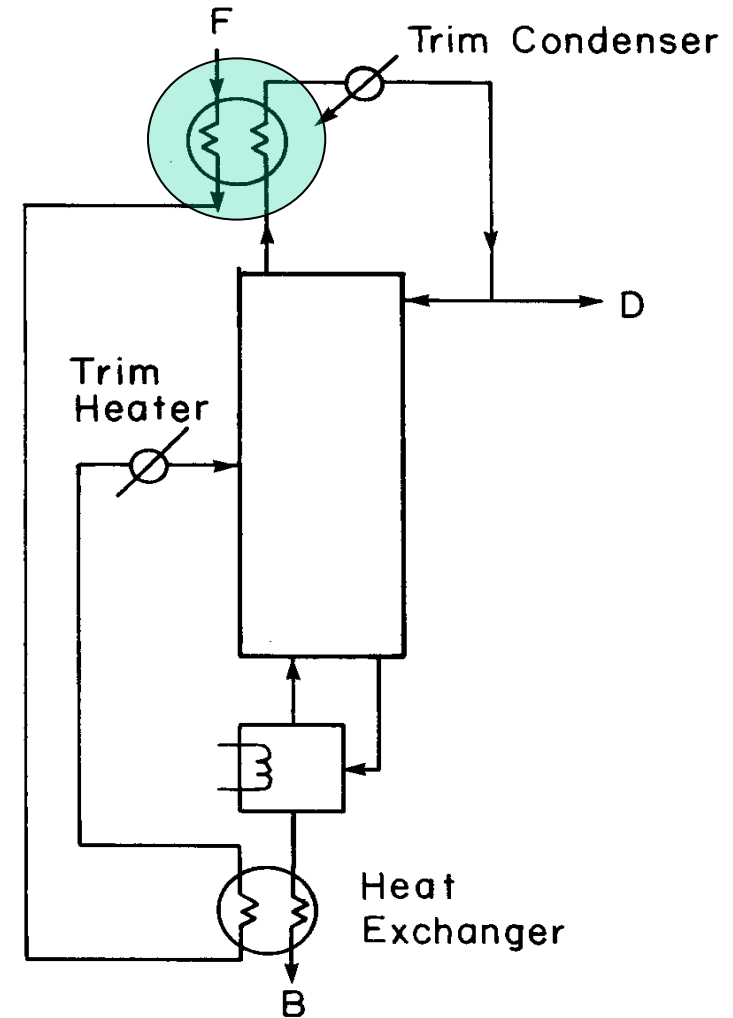


Energetic integration

Hot streams are used to warm cold streams

Examples:

- Feed is pre-heated in the condenser where the exiting vapor condenses in contact with the cold feed, that in turn will heat

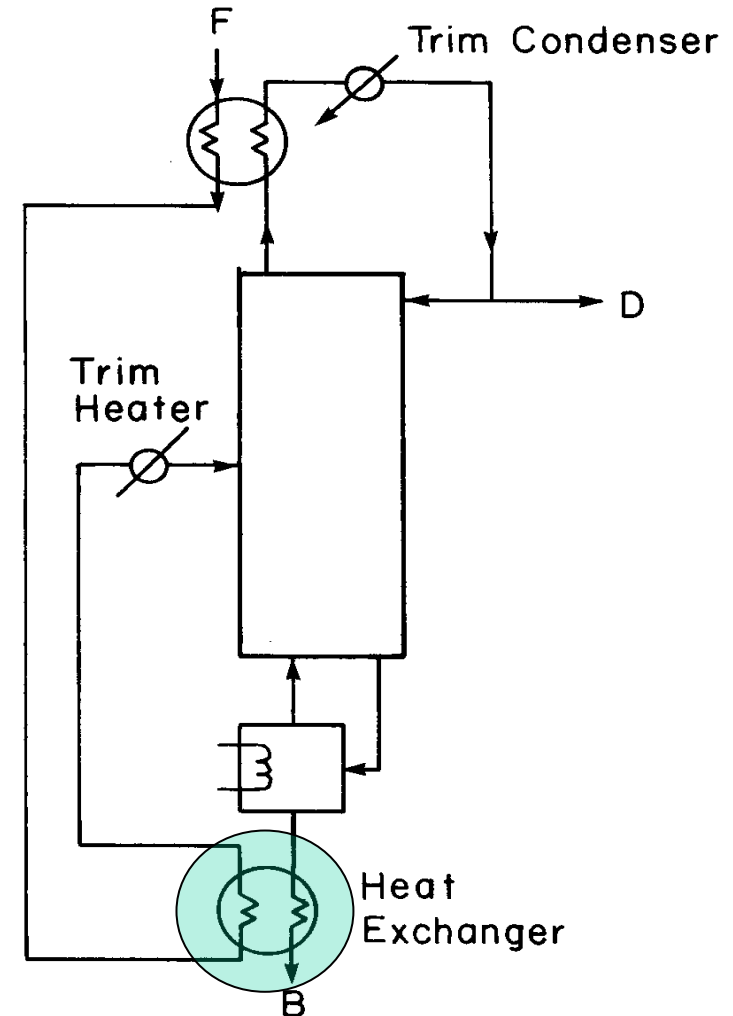


Energetic integration

Hot streams are used to warm cold streams

Examples:

- Feed is pre-heated in the condenser where the exiting vapor condenses in contact with the cold feed, that in turn will heat
- Feed is pre-heated by the bottom current, that acts as a heating fluid



Integrated several distillation columns

The vapour of column 1 is condensed in the boiler of column 2

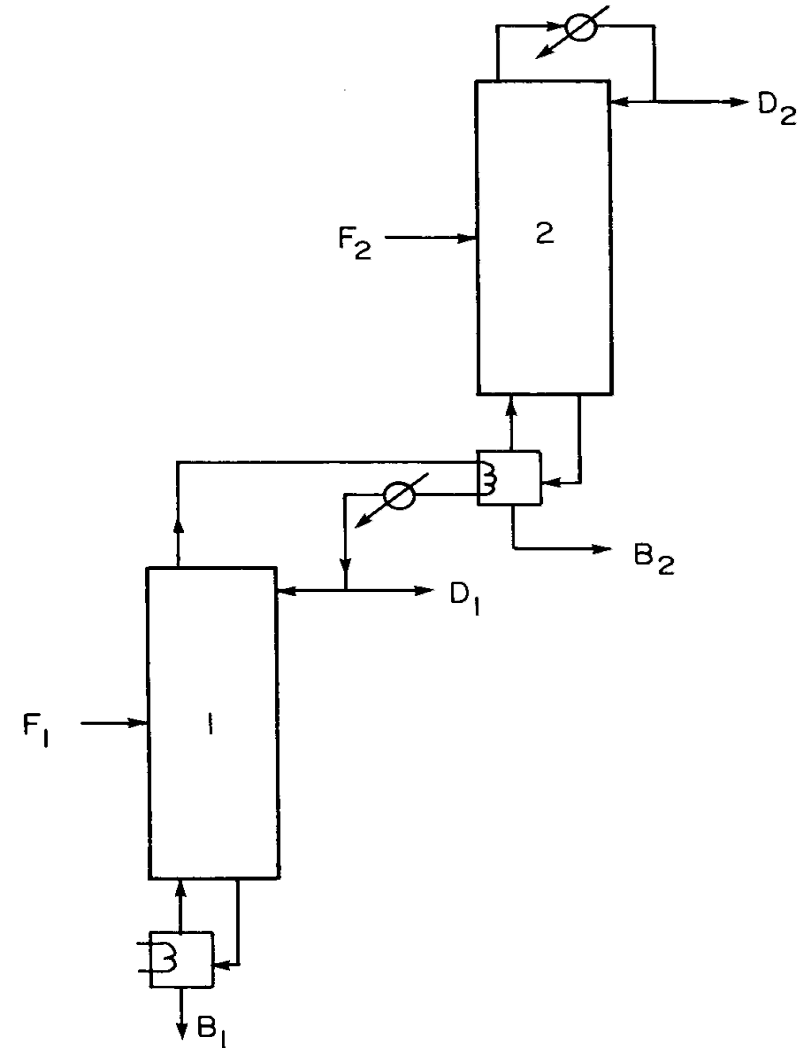




Table 7.1 Representative Commercial Binary Distillation Operations

Binary Mixture	Average Relative Volatility	Number of Trays	Typical Operating Pressure, psia	Reflux-to-Minimum-Reflux Ratio
1,3-Butadiene/vinyl acetylene	1.16	130	75	1.70
Vinyl acetate/ethyl acetate	1.16	90	15	1.15
<i>o</i> -Xylene/ <i>m</i> -xylene	1.17	130	15	1.12
Isopentane/ <i>n</i> -pentane	1.30	120	30	1.20
Isobutane/ <i>n</i> -butane	1.35	100	100	1.15
Ethylbenzene/styrene	1.38	34	1	1.71
Propylene/propane	1.40	138	280	1.06
Methanol/ethanol	1.44	75	15	1.20
Water/acetic acid	1.83	40	15	1.35
Ethylene/ethane	1.87	73	230	1.07
Acetic acid/acetic anhydride	2.02	50	15	1.13
Toluene/ethylbenzene	2.15	28	15	1.20
Propane/1,3-butadiene	2.18	40	120	1.13
Ethanol azeotrope/water	2.21	60	15	1.35
Isopropanol/water	2.23	12	15	1.28
Benzene/toluene	3.09	34	15	1.15
Methanol/water	3.27	60	45	1.31
Cumene/phenol	3.76	38	1	1.21
Benzene/ethylbenzene	6.79	20	15	1.14
HCN/water	11.20	15	50	1.36
Ethylene oxide/water	12.68	50	50	1.19
Formaldehyde/methanol	16.70	23	50	1.17
Water/ethylene glycol	81.20	16	4	1.20

Mix, T.W., J.S. Dweck, M. Weinberg, and R.C. Armstrong, Chem. Eng. Prog., 74(4), 49–55 (1978).