

Introduction to Column Distillation

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Agenda

- ◆ Flash drums in series
- ◆ Developing a distillation cascade
- ◆ Specifications
- ◆ External column balances

Introduction to Column Distillation

◆ Distillation is **important**

- 90-95% of separations in chemical industry
- Approximately 40.000 distillation column operate around the world
- Consuming 40% of the energy used in US process industry
- Equivalent to 1.2 million barrels of crude oil per day

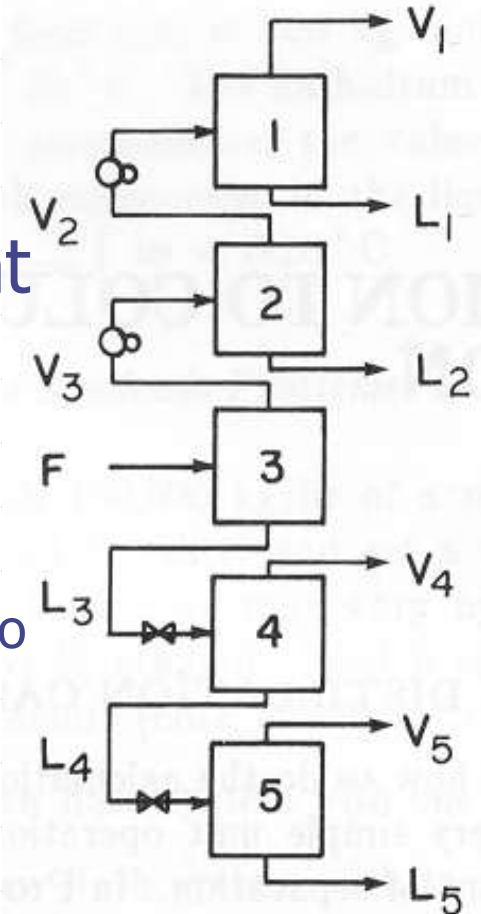
◆ **Flash** distillation provides a method of separation, but the **amount of separation obtained is limited.**

◆ What if we need to have a **greater separation** to obtain essentially pure components?

◆ We could place **flash drums in series** or as a cascade...

Flash Drums in Cascade

- ◆ One can obtain a high level of separation using **cascading flash drums**.
- ◆ The problem with this arrangement is that we generate a **large number of intermediate** liquid and vapor streams, which would need to be separated.
 - One could feed these intermediate streams to another flash drum cascade, but even more intermediate streams are formed, and so on and so on.
- ◆ Let's look at what we can do with the intermediate streams...

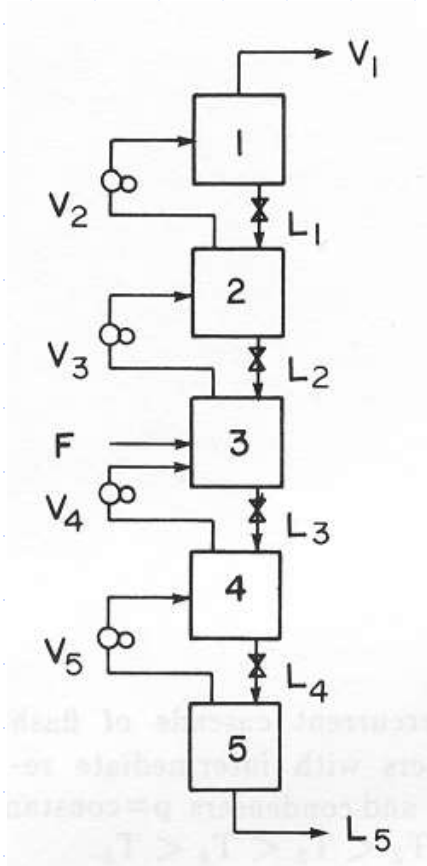


Cascade of flash chambers:
 $p_1 > p_2 > p_3 > p_4 > p_5$

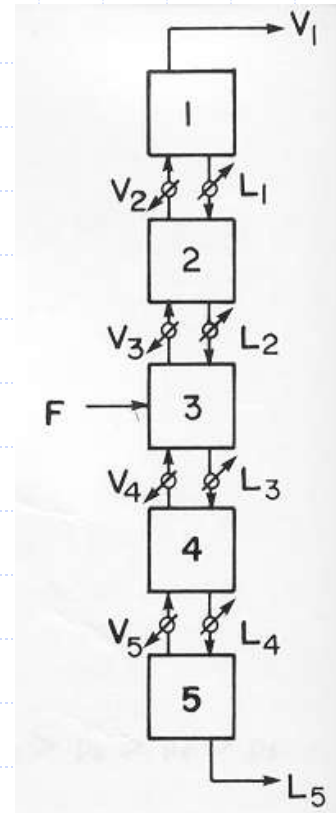
Flash Drums in Counter-Current

◆ Use of intermediate steams

◆ Isobaric operation



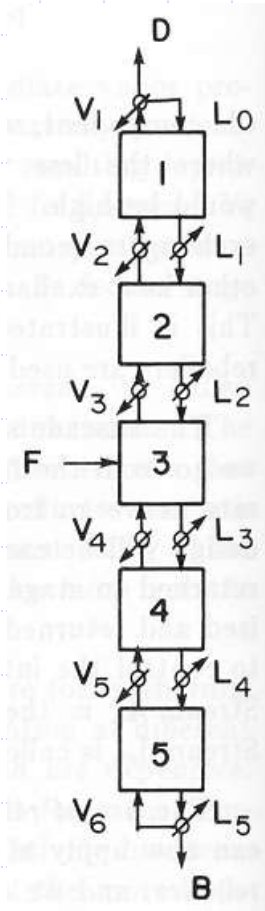
Counter current cascade of flash chambers: $p_1 > p_2 > p_3 > p_4 > p_5$



Counter current cascade of flash chambers with intermediate reboilers and condensers. $P = \text{constant}$: $T_1 < T_2 < T_3 < T_4 < T_5$

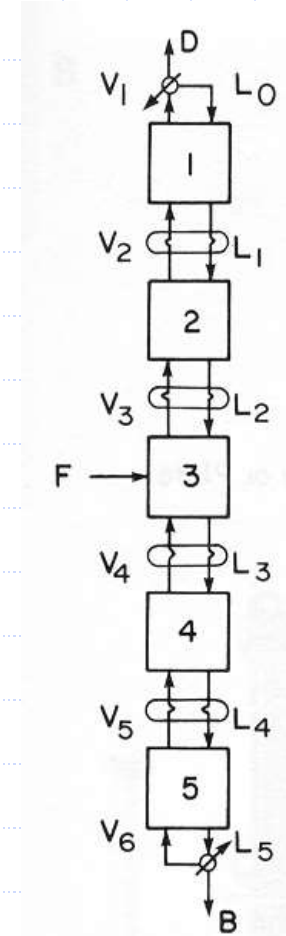
Flash Drums in Counter-Current

◆ Reflux and boilup



Counter current cascade of flash chambers with reflux and boilup. $P = \text{constant}$: $T_1 < T_2 < T_3 < T_4 < T_5$

◆ Intermediate heat exchange

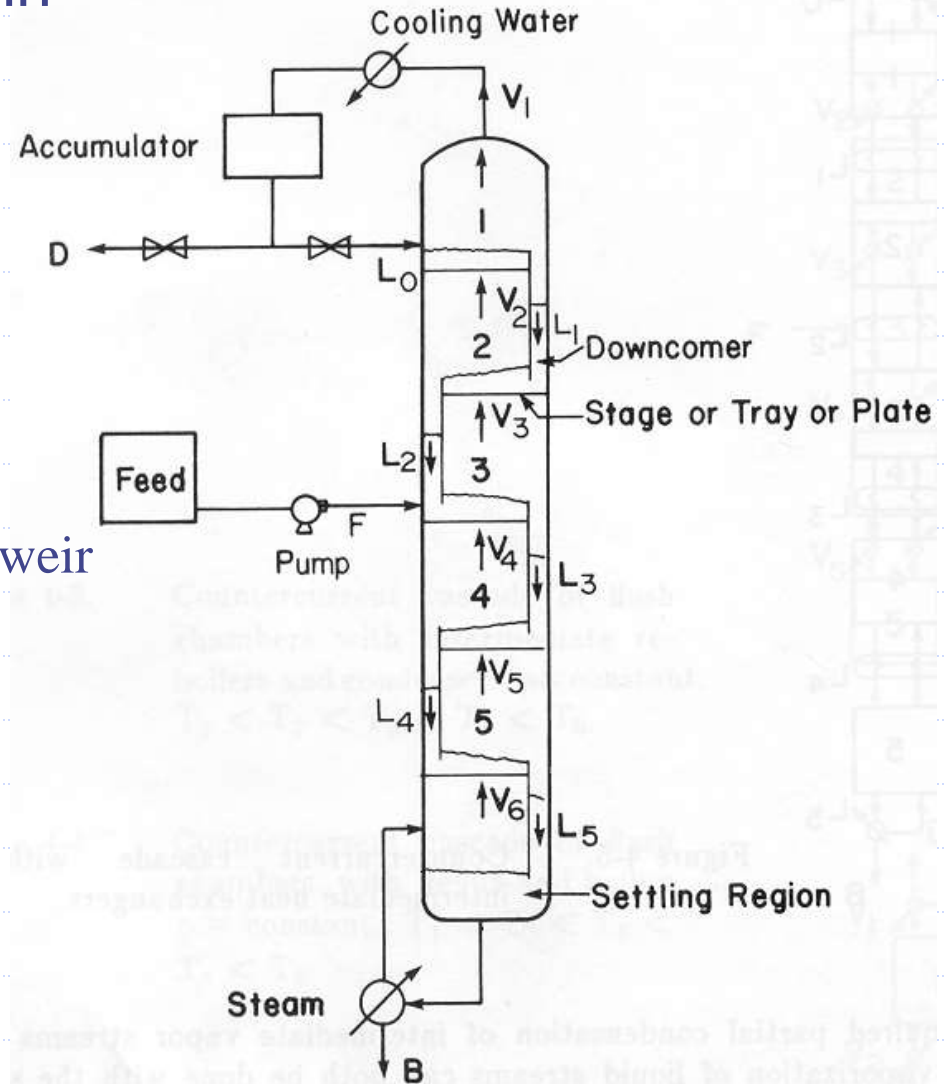
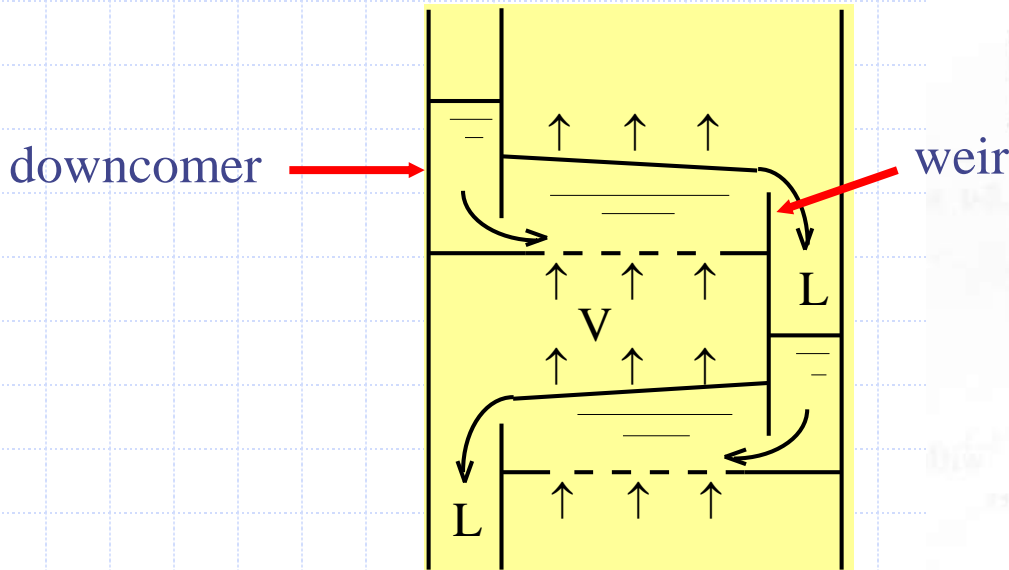


Counter current cascade of flash chambers with intermediate heat exchangers.

Distillation Column

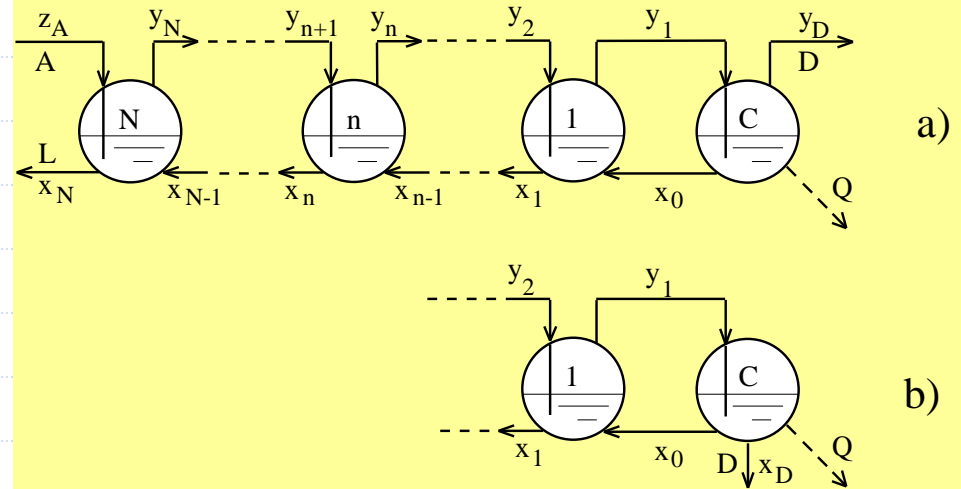
◆ Schematic of 5 stage column

- $T_1 < T_2 < T_3 < T_4 < T_5 < T_6$

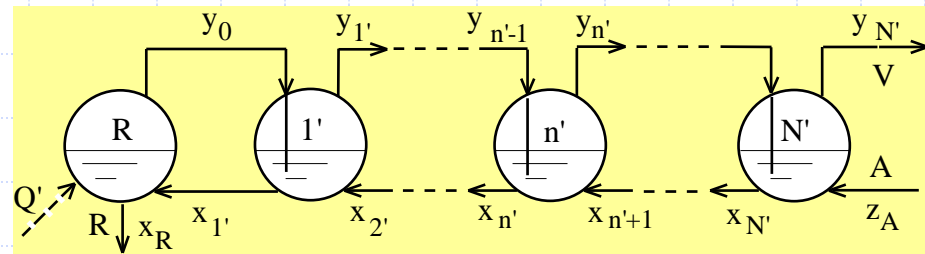


The idea of multistage distillation

rectifying section

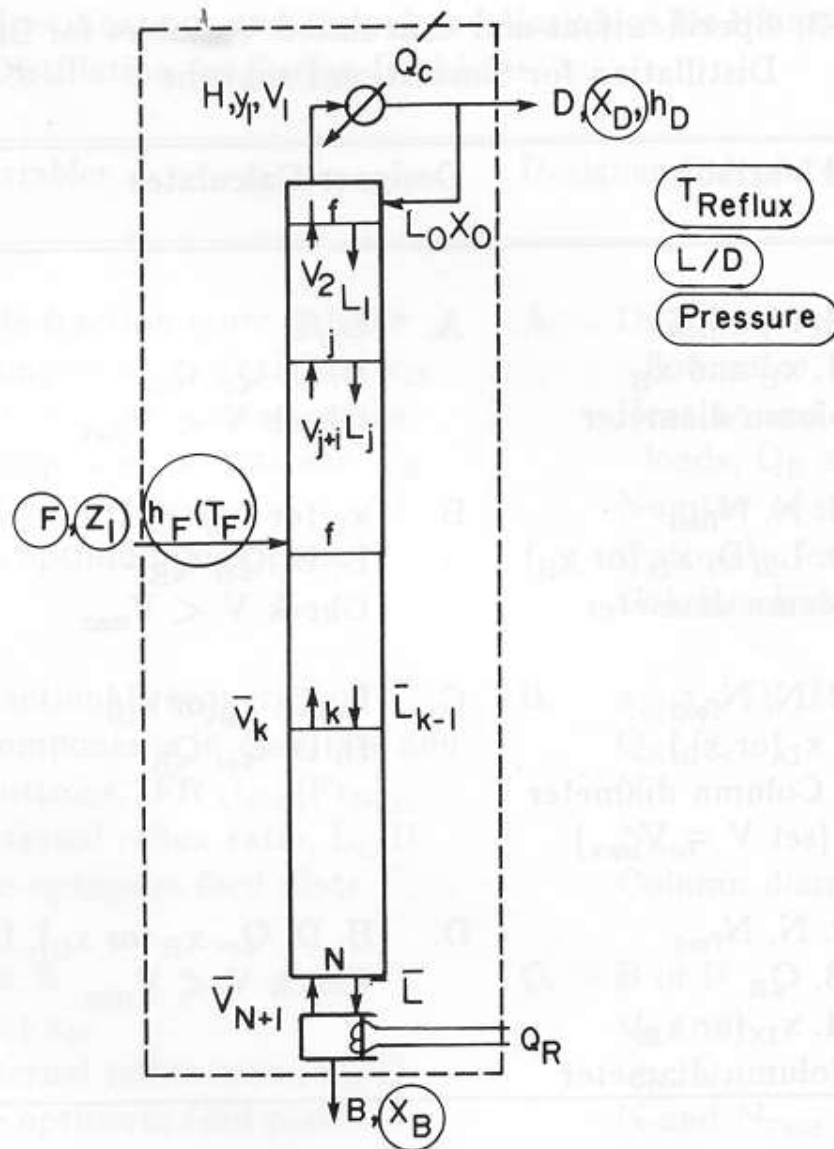


stripping section



- Heat is supplied at the bottom only and withdrawn at the top only
- All other condensations/vaporizations are by direct internal contacts

Distillation Column



Specifications

Table 3-1. Usual specified variables for binary distillation

1. Column pressure
2. Feed flow rate
3. Feed composition
4. Feed temperature or enthalpy or quality
5. Reflux temperature or enthalpy (usually saturated liquid)

Specifications and calculated variables for binary distillation for simulation problems

| <i>Specified Variables</i> | <i>Designer Calculates</i> |
|--|--|
| A. 1,2. N, N_{feed} 3,4. x_D and x_B Column diameter | A. L_v/D B, D, Q_c, Q_R Check $V < V_{max}$ |
| B. 1,2. N, N_{feed} 3,4. $L_v/D, x_D$ (or x_B) Column diameter | B. x_B (or x_D) B, D, Q_c, Q_R Check $V < V_{max}$ |
| C. 1,2. N, N_{feed} 3. x_D (or x_B) 4. Column diameter (set $V = \text{fraction} \times V_{max}$) | C. $L_v/D, x_B$ (or x_D) B, D, Q_c, Q_R D. B, D, Q_c, x_B (or x_D), L_v/D Check $V < V_{max}$ |
| D. 1,2. N, N_{feed} 3. Q_R 4. x_D (or x_B) Column diameter | |

Table 3-2. Specifications and calculated variables for binary distillation for design problems

Specified Variables

- A. 1. Mole fraction more volatile component in distillate, x_D
 2. Mole fraction more volatile component in bottoms, x_B
 3. External reflux ratio, L_v/D
 4. Use optimum feed plate
- B. 1,2. Fractional recoveries of components in distillate and bottoms, $(FR_A)_{dist}$, $(FR_B)_{bot}$
 3. External reflux ratio, L_v/D
 4. Use optimum feed plate
- C. 1. D or B
 2. x_D or x_B
 3. External reflux ratio, L_v/D
 4. Use optimum feed plate
- D. 1,2. x_D and x_B
 3. Boilup ratio, V/B
 4. Use optimum feed plate

Designer Calculates

- A. Distillate and bottoms flow rates, D and B
 Heating and cooling loads, Q_R and Q_c
 Number of stages, N
 Optimum feed plate
 Column diameter
- B. x_B , x_D , D, B
 Q_R , Q_c
 N
 N_{feed}
 Column diameter
- C. B or D
 x_B or x_D
 Q_R , Q_c
 N and N_{feed}
 Column diameter
- D. D and B, Q_R and Q_c
 N, N_{feed}
 Column diameter

Distillation Column:

◆ Typical Specified Variables

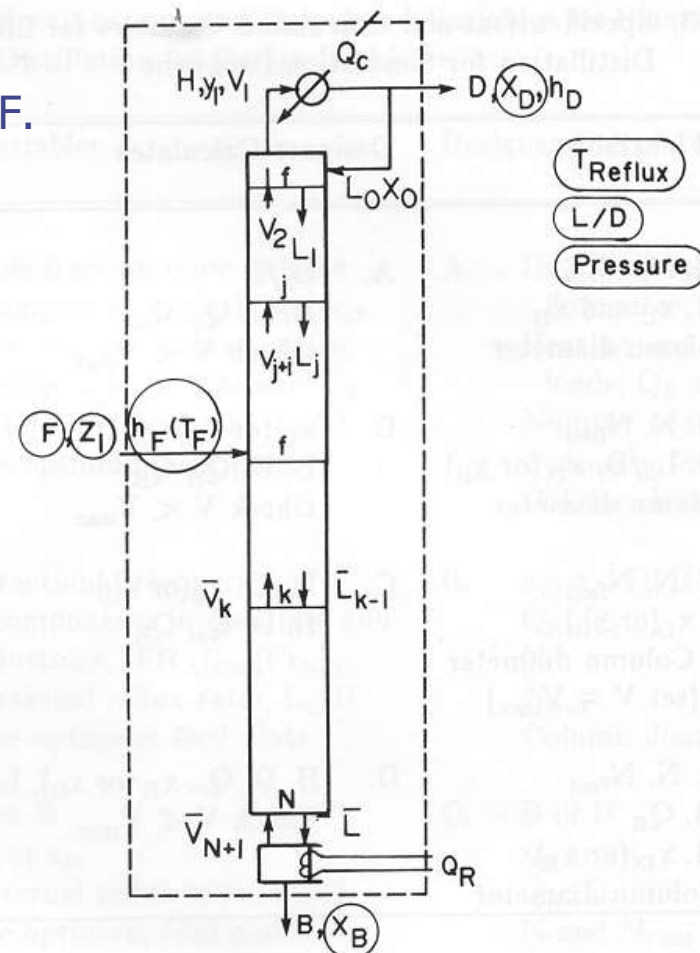
- Column pressure, P_C .
- Feed flow rate, F .
- Feed composition, z .
- Feed temp. T_F ; enthalpy, h_F ; or quality, $q = L/F$.
- Reflux temperature, T_R ; or enthalpy, h_D .
- Reflux ratio, L/D ; or distillate composition, x_D .
- Bottoms composition, x_B .

◆ Tools for Solution

- Equilibrium relationships
- Mass balances
- Energy balances

◆ Methods of solution

- External column balances
 - ◆ Overall
 - ◆ Condenser
 - ◆ Reboiler
- Internal column balances
 - ◆ Stage-by-stage calculations



External Column Balances

- ◆ Overall mass balance

$$F = B + D$$

- ◆ More volatile component

$$Fz_F = Bx_B + Dx_D$$

- ◆ Distillate Stream D

$$D = \left(\frac{z_F - x_B}{x_D - x_B} \right) F$$

- ◆ Bottoms Stream B

$$B = F - D = \left(\frac{x_D - z}{x_D - x_B} \right) F$$

External Column Balances

◆ Energy balance

$$Fh_F + Q_C + Q_R = Dh_D + Bh_B$$

◆ With

- $h_F(z, T_F, p)$, $h_D(x_D, T_{\text{reflux}}, p)$, $h_B(x_B, \text{saturated liquid}, p)$
- Can be calculated from enthalpy composition diagrams (or correlations)

◆ Energy balance equation

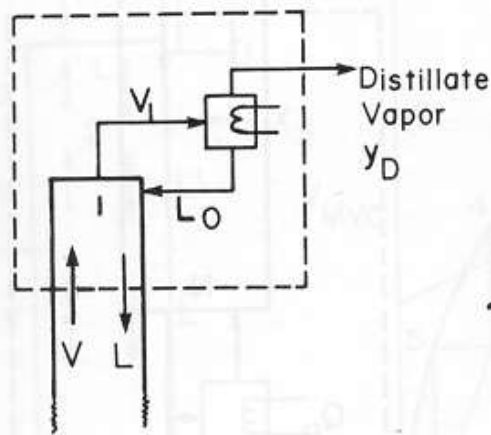
- 2 unknowns (Q_R and Q_C)
- 1 more equation is needed → that comes from the condenser condition

Condenser Conditions

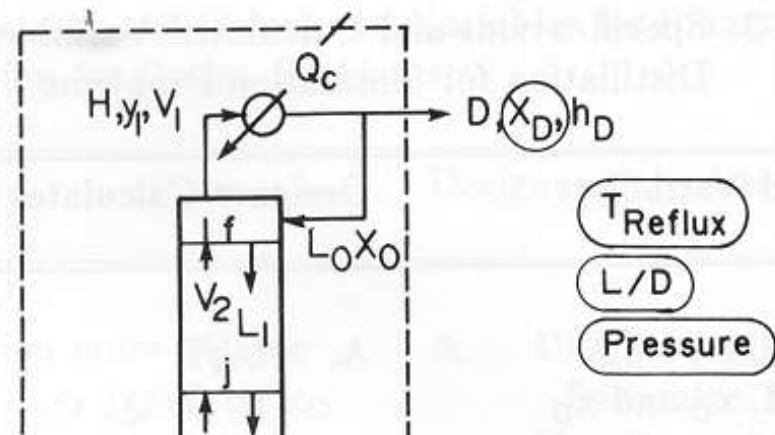
- ◆ **Partial** – only part of the incoming vapor stream, V_1 , is condensed.
 - We have both vapor and liquid streams at saturated conditions.
 - D is taken as a saturated vapor and the liquid reflux, L_o , is returned to the column as a saturated liquid.
 - Both must be at saturated conditions since we have equilibrium – in fact the partial condenser is an additional equilibrium stage.
 - We will look at partial condensers later.
- ◆ **Total** – all of the incoming vapor stream, V_1 , is condensed to liquid.
 - We then split the resulting liquid outlet into the distillate stream, D , and the reflux L_o , which is returned to the column.
 - We will consider only total condensers for now.

Condenser conditions

Partial



Total



Important note for total condenser

- ◆ the mole fraction, y_1 , of the vapor stream from the top of the column is equal to both the mole fractions, x_D and x_o , of the liquid streams!

$$y_1 = x_D = x_o$$

- ◆ The condition of the outlet of the condenser has to be specified as either a saturated liquid or a subcooled liquid in order to use the energy balance.
- ◆ The state of the liquid determines the heat duty of the condenser.

Total-Condenser Mass Balance

- ◆ Total mass balance

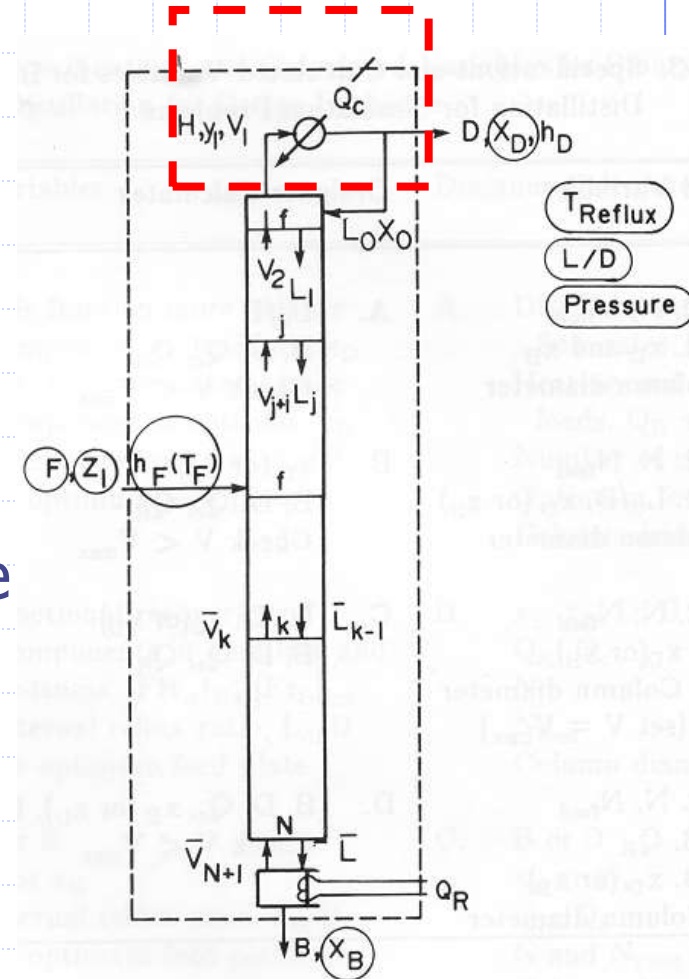
$$V_1 = L_0 + D$$

- ◆ Component mass balance

$$V_1 y_1 = L_0 x_0 + D x_D$$

- ◆ Since external reflux ratio L_0/D is specified we can substitute its value

$$V_1 = \left(\frac{L_0}{D} \right) D + D = \left(1 + \frac{L_0}{D} \right) D$$



Total-Condenser Energy Balance

$$V_1 H_1 + Q_C = D h_D + L_0 h_0$$

$$Q_C = \left(1 + \frac{L_0}{D}\right) D (h_D - H_1)$$

- ◆ Recall the D from the total material balance.

$$Q_C = \left(1 + \frac{L_0}{D}\right) \left(\frac{z_F - x_B}{x_D - x_B}\right) F (h_D - H_1)$$

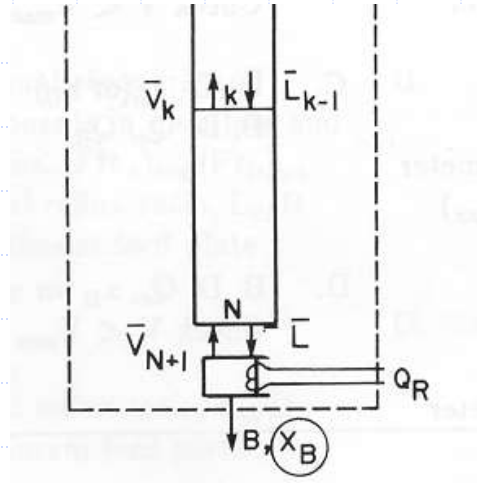
- ◆ Note that $Q_C < 0$ because the liquid enthalpy is less than that of vapor

Reboiler Conditions

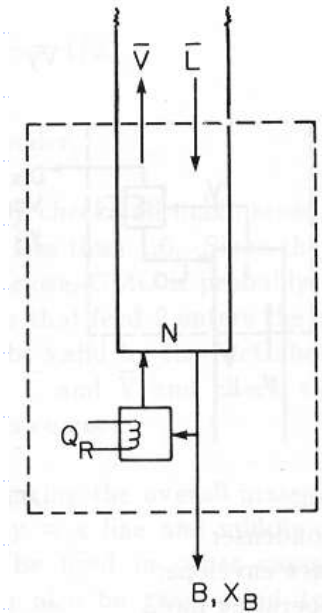
- ◆ **Partial** – only part of the incoming liquid stream, L , is vaporized.
 - We have both vapor and liquid streams exiting the reboiler at saturated conditions.
 - B is taken as a saturated liquid and the boilup, V_{n+1} , is returned to the column as a saturated vapor.
 - Both streams must be at saturated conditions since we have equilibrium in the reboiler – in fact the partial reboiler is an additional equilibrium stage.
- ◆ **Total** – the incoming liquid stream, L , is split first to obtain our bottoms stream, B , as a saturated liquid.
 - It has to be a saturated liquid since it leaves the equilibrium stage at the bottom of the column.
 - We then reboil all of the remaining liquid and return it to the column as a vapor stream, V .

Reboiler Conditions

Partial



Total



Important note for total reboiler

- ◆ the mole fraction, x_N , of the liquid stream from the bottom of the column is equal to the mole fraction, x_B , of the bottom liquid stream and that fed to the partial reboiler!

$$x_B = x_N$$

- ◆ The condition of the outlet of the reboiler has to be specified as either a saturated vapor or a superheated vapor in order to use the energy balance.
- ◆ The state of the vapor determines the heat duty of the reboiler.

Partial-Reboiler Energy Balance

$$Q_R = Dh_D + Bh_B - Fh_F - Q_C$$

$$Q_R = Dh_D + Bh_B - Fh_F + \left(1 + \frac{L_o}{D}\right) D(H_1 - h_D)$$

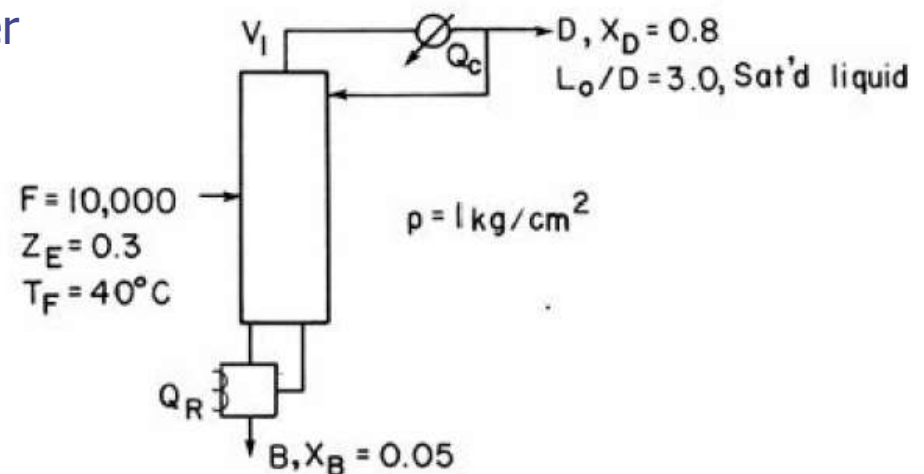
$$Q_R = \left(\frac{z_F - x_B}{x_D - x_B}\right) Fh_D + \left(\frac{x_D - z_F}{x_D - x_B}\right) Fh_B - Fh_F + \left(1 + \frac{L_o}{D}\right) D(H_1 - h_D)$$

- ◆ Note that $Q_R > 0$ because the liquid enthalpy is less than that of vapor

Example: external balances for binary distillation

- ◆ A steady-state, countercurrent, staged distillation column is to be used to separate ethanol from water.
 - The feed is a 30 wt % ethanol, 70 wt % water mixture at 40°C. Flow rate of feed is 10,000 kg/h.
 - The column operates at a pressure of 1 kg/cm².
 - The reflux is returned as a saturated liquid. Reflux ratio $L_0/D=3.0$ is used.
 - We desire a bottoms composition of $x_B = 0.05$ (weight fraction ethanol) and a distillate composition of $x_D = 0.80$ (weight fraction ethanol).
 - The system has a total condenser and a partial reboiler.

◆ Find D , B , Q_C , and Q_R .



Example: external balances for binary distillation

◆ From mass balance eq. $D = F \left(\frac{z - x_B}{x_D - x_B} \right) = 10,000 \left[\frac{0.3 - 0.05}{0.8 - 0.05} \right] = 3333 \text{ kg/h}$

■ And $B = F - D = 10,000 - 3333 = 6667 \text{ kg/h}$

◆ From Ponchon – Savarit graph, enthalpies are

■ $h_D(x_D = 0.8, \text{ saturated liquid}) = 60 \text{ kcal/kg}$

■ $h_B(x_B = 0.05, \text{ saturated liquid}) = 90 \text{ kcal/kg}$

■ $h_f(z = 0.3, 40^\circ \text{ C}) = 30 \text{ kcal/kg}$

■ $H_1(y_1 = x_D = 0.8, \text{ saturated vapor}) = 330 \text{ kcal/kg}$

◆ From energy balance around the condenser

$$Q_c = \left(1 + \frac{L_0}{D}\right)D(h_D - H_1) = (1 + 3)(3333)(60 - 330) = -3,599,640 \text{ kcal/h}$$

◆ From the column external energy balance:

■ $Q_R = D_{hD} + B_{hB} - F_{hF} - Q_C$

■ $Q_R = (3333)(60) + (6667)(90) - (10,000)(30) - (-3,599,640) = 4,099,650 \text{ kcal/h}$