

Distillation.

Optimal operation using simple control structures

Sigurd Skogestad, NTNU, Trondheim

EFCE Working Group on Separations, Göteborg, Sweden, June 2019

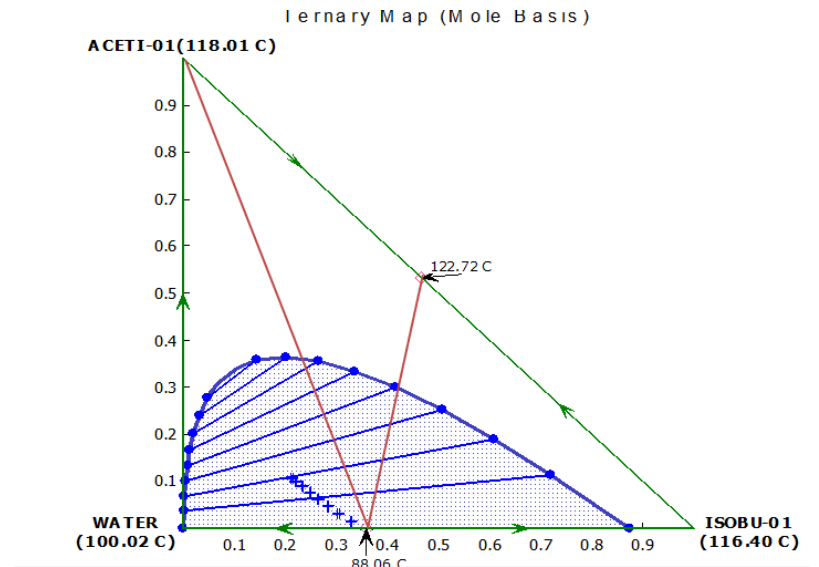
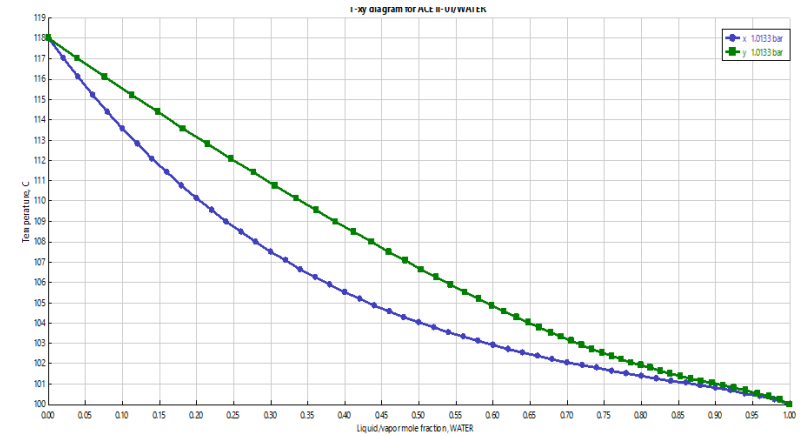
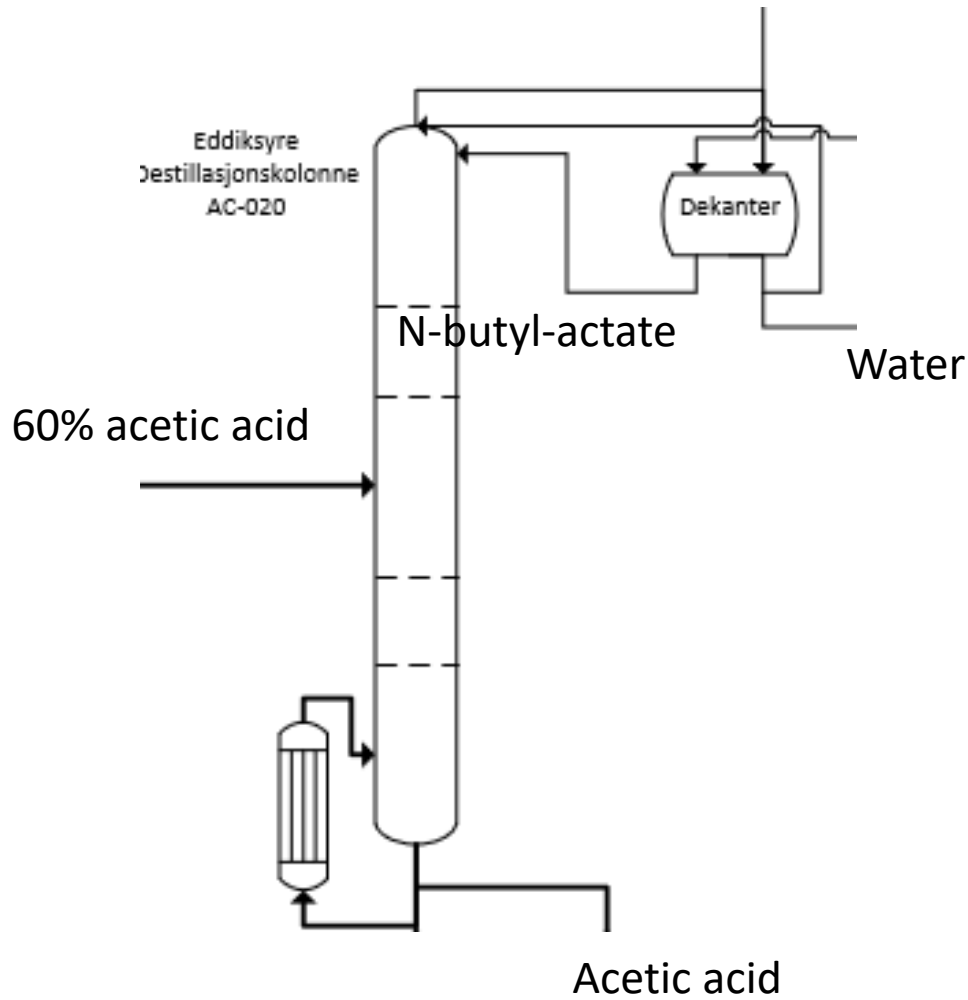
Distillation is part of the future

1. It's a myth that distillation is bad in terms of energy
2. Better operation and control can save energy
3. Integrated schemes can save energy and capital
 - Divided-wall / Petlyuk columns

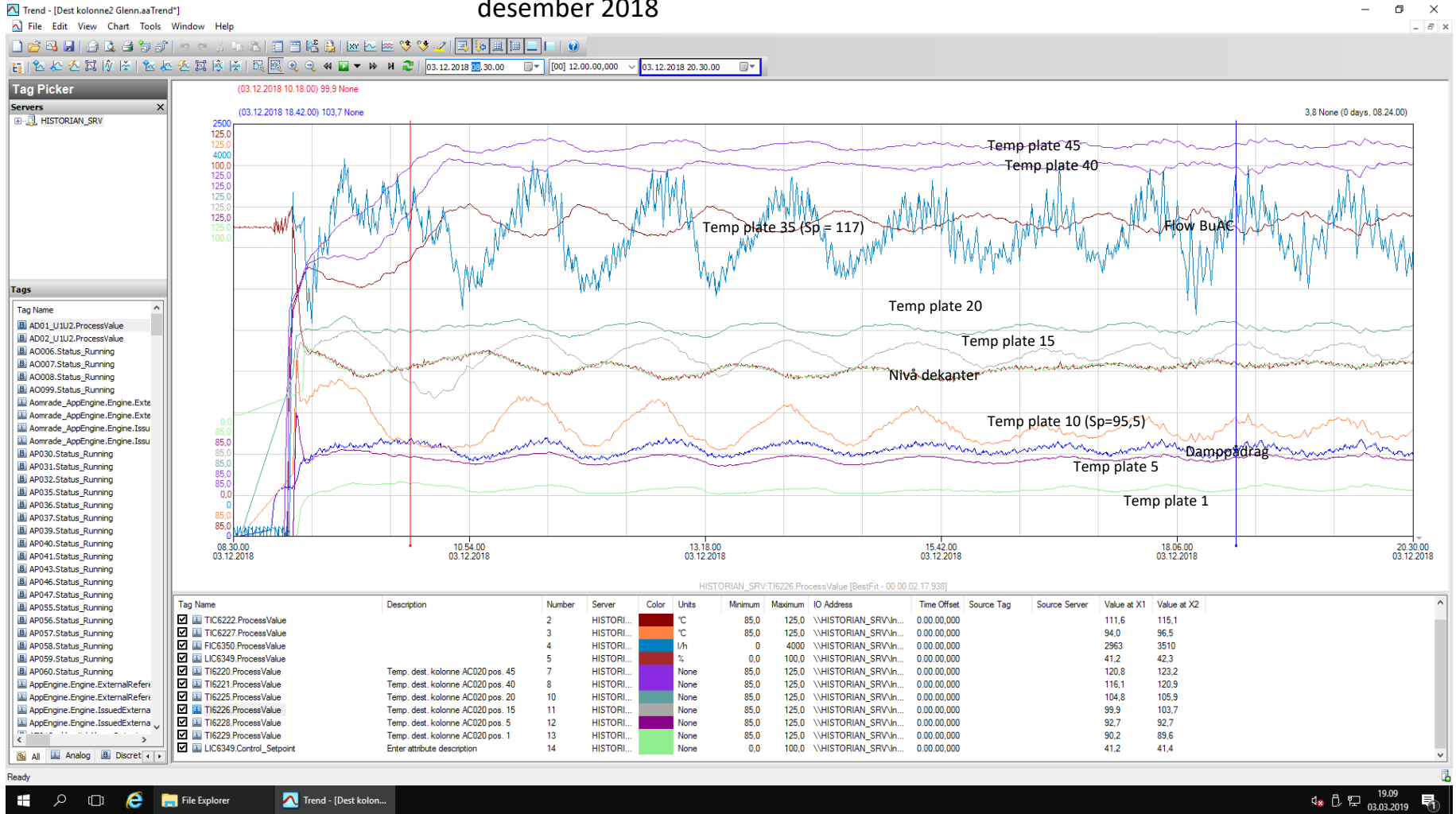
OUTLINE

- Many columns operate poorly because of poor control
- Myths about distillatons
 - Ineffecient (large energy usage)
 - Slow response
- Petlyuk distillation
 - Vmin-diagram for insight and initialization of detailed simulation

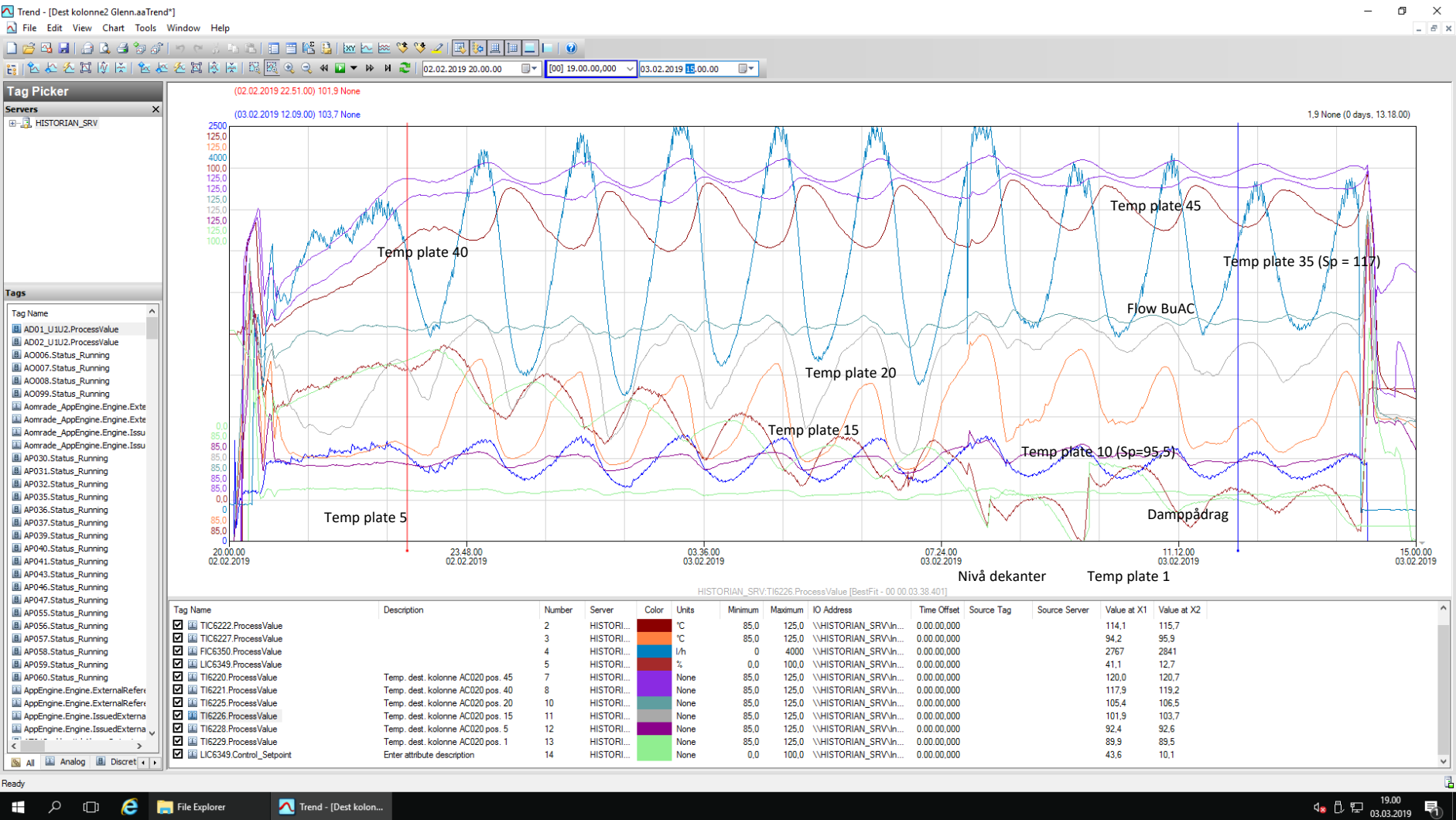
Solvent recovery. Explosives plant, Norway



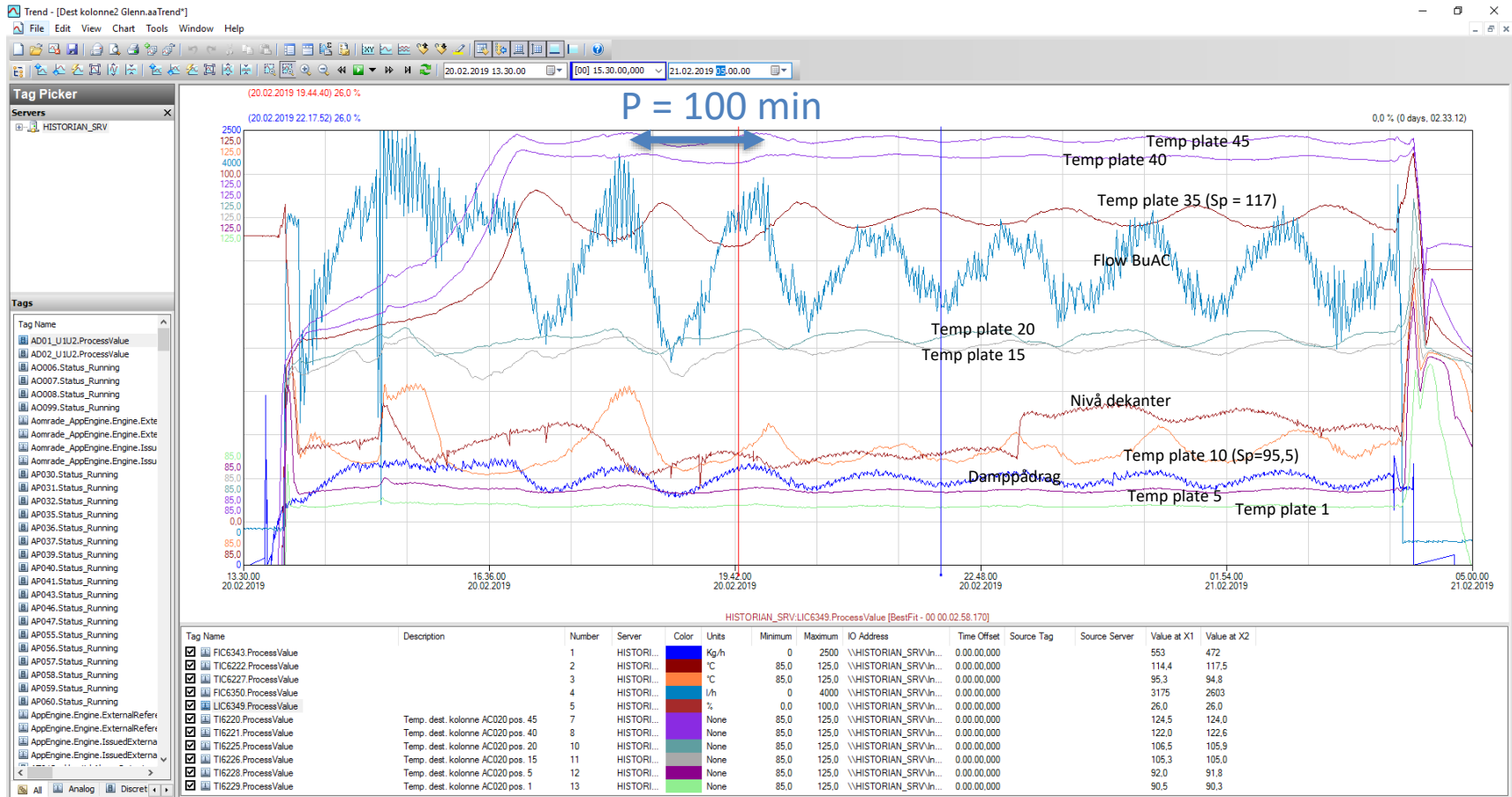
desember 2018



02 feb. 2019.



20 feb. 2019. After replacing some column internals (in the hope of fixing the problem)



Tray 10 temperature controlled using butyl-acetate reflux: Integral time (taui) = 10 minutes. TOO MUCH INTEGRAL ACTION!

Sigurd's formula*: Increase $K_c \cdot \tau_{i0}$ by factor $f = 0.1 \cdot (P/\tau_{i0})^2 = 0.1 \cdot (100/10)^2 = 10$. Problem solved by increasing integral time to 50 minutes.

*Sigurd Skogestad. "Simple analytic rules for model reduction and PID controller tuning"

«Distillation is an inefficient process which uses a lot of energy»

- This is a myth!
- By itself, distillation is an efficient process.
- It's the heat integration that may be inefficient.
- Yes, it can use a lot of energy, but it provides the same energy at a lower temperature
 - Difficult separations (close-boiling): use a lot of energy -- but well suited for heat pumps
 - Easy separations: Use little energy

Typical distillation Case

Example 8.20 from Skogestad (2008)

By defining “useful energy in” as “heat supply” Q_H , the thermodynamic efficiency (8.12) for distillation of this feed mixture is then

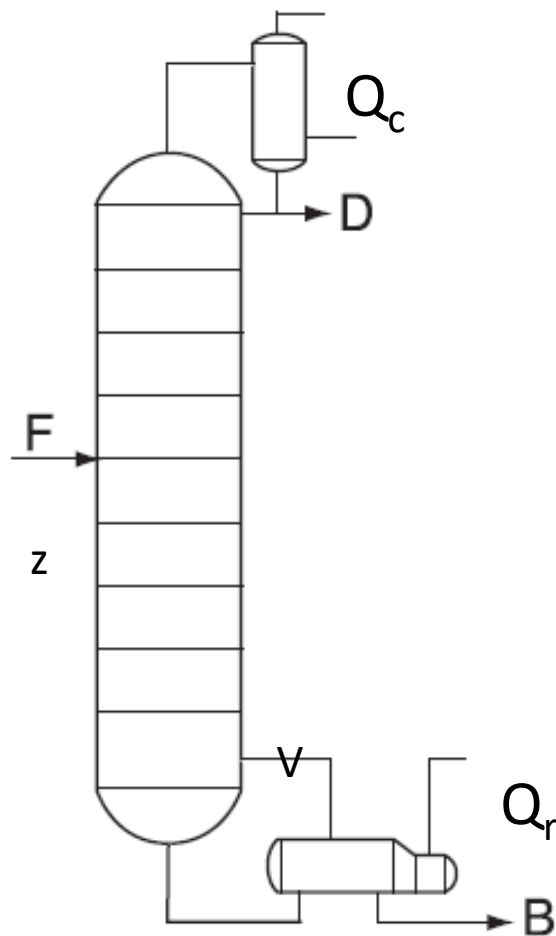
$$\eta = \frac{\text{ideal (minimum) heat supply}}{\text{actual heat supply distillation}} = \frac{Q_H^{\text{min}}}{Q_H} = \frac{20 \text{ kJ/mol}}{31.8 \text{ kJ/mol}} = 0.63$$

Thus, distillation as a separating process has a good thermodynamic efficiency (63% in this case). On the other hand, the “energy efficiency” (8.13) for the distillation process obtained by defining “useful energy out” as “separation work” is only 5.2%:

$$\eta_{\text{energy}} = \frac{\text{separation work}}{\text{heat supply distillation}} = \frac{W_s^{\text{id}}}{Q_H} = \frac{1.66 \text{ kJ/mol}}{31.8 \text{ kJ/mol}} = 0.052$$

Thermodynamic
(exergy) efficiency
is 63%

Energy efficiency is
only 5% (with no
heat Integration)



King's formula:
 (binary, feed liquid, constant α ,
 Infinite* no. Stages, pure products)

$$Q_r = \lambda V_{\min} = \left(z + \frac{1}{\alpha - 1} \right) \lambda F$$

$Q_r = \text{reboiler duty [W]}$

$\lambda = \text{heat of vaporization}$

$\alpha = \text{relative volatility}$

$z = \text{mole fraction light component in feed}$

*Actual energy only 5-10% higher

Ideal separation work

- Minimum supplied work (for any process)

$$W_{s,id} = \Delta H - T_0 \Delta S$$

- Assume $\Delta H=0$ for the separation. Minimum separation work

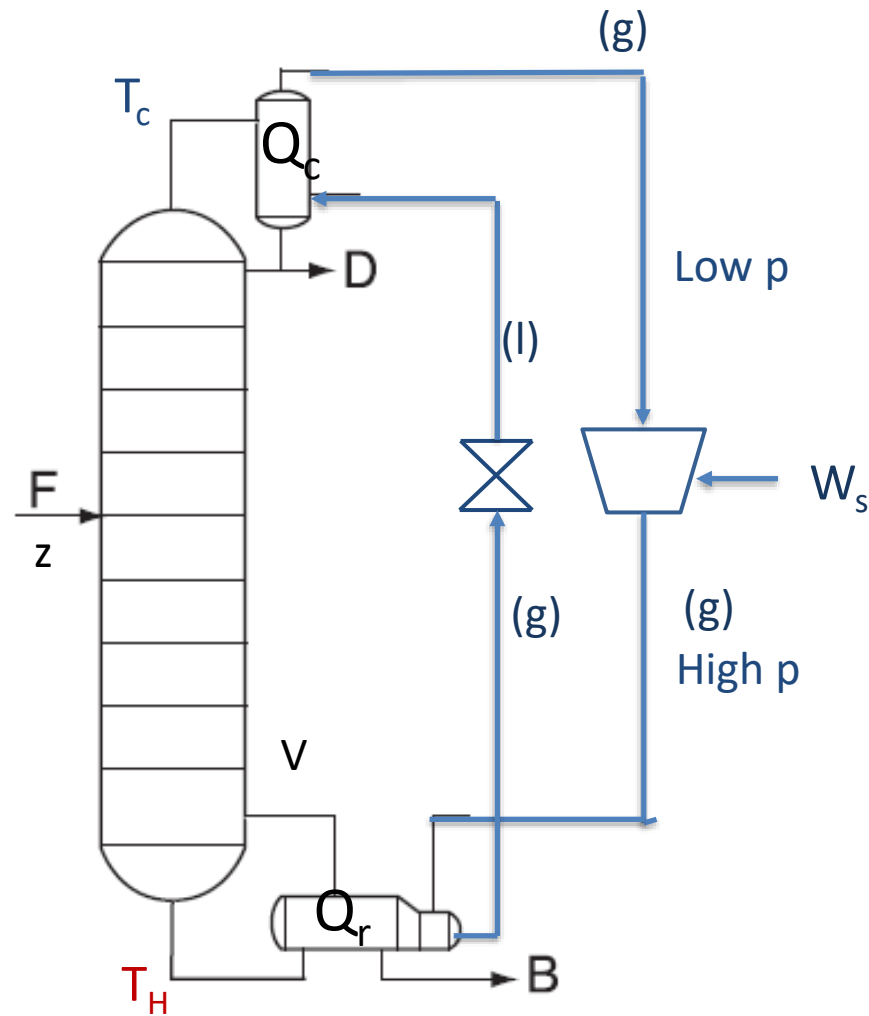
$$W_{s,id} = - T_0 \Delta S$$

- Separation of feed into pure products

$$\Delta S = F R \sum_{i=1}^N z_i \ln z_i$$

- This is a negative number so the minimum separation work $W_{s,id}$ is positive!

Distillation with heat pump



Minimum work (Carnot)

Assume $Q_c \approx -Q_r$

$$W_{s,carnot} = Q_r T_0 \left(\frac{1}{T_C} - \frac{1}{T_H} \right)$$

Thermodynamic efficiency (exergy) for conventional distillation

- Thermodynamic Efficiency =

Ideal work for the separation/Actual work:

$$\eta = \frac{W_s^{id}}{W_{s,carnot}} = \frac{-FRT_0 \sum_1^N z_i \ln z_i}{Q_r T_0 \left(\frac{1}{T_C} - \frac{1}{T_H} \right)}$$

Note that T_0 drops out

Thermodynamic efficiency

Special case: Binary, constant α

- King's formula

$$Q_r = \left(z + \frac{1}{\alpha - 1}\right) \lambda F$$

- Ideal binary mixture (Claperyon equation) + no pressure drop. King shows:

$$\frac{1}{T_C} - \frac{1}{T_H} = \frac{R}{\lambda} \ln \alpha$$

- So

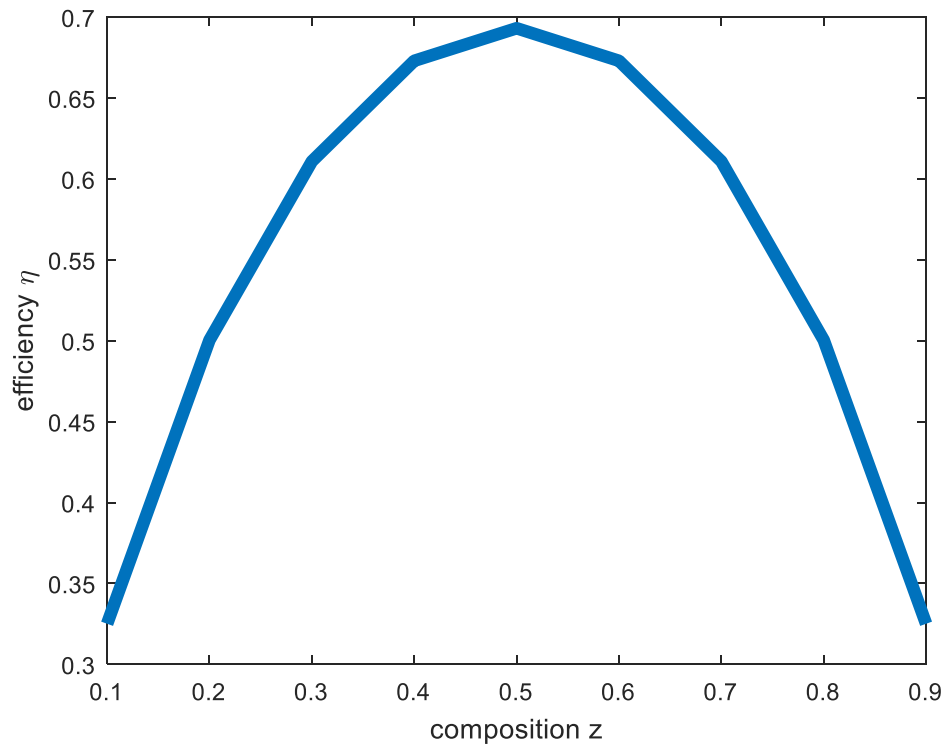
$$\eta = \frac{W_s^{id}}{W_{s,carnot}} = \frac{-F \sum_1^N z_i \ln z_i}{Q_r \left(\frac{1}{T_C} - \frac{1}{T_H}\right)} \stackrel{\text{Binary}}{\downarrow} = \frac{-(z \ln z + (1 - z) \ln(1 - z))}{\left(z + \frac{1}{\alpha - 1}\right) \ln \alpha}$$

Note that λ drops out

$$\text{Use: } \lim_{\alpha \rightarrow 1} \frac{\ln \alpha}{\alpha - 1} = 1 \quad \lim_{\alpha \rightarrow 1} \eta = -(z \ln z + (1 - z) \ln(1 - z))$$

Thermodynamic efficiency of binary close-boiling mixtures ($\alpha \rightarrow 1$)

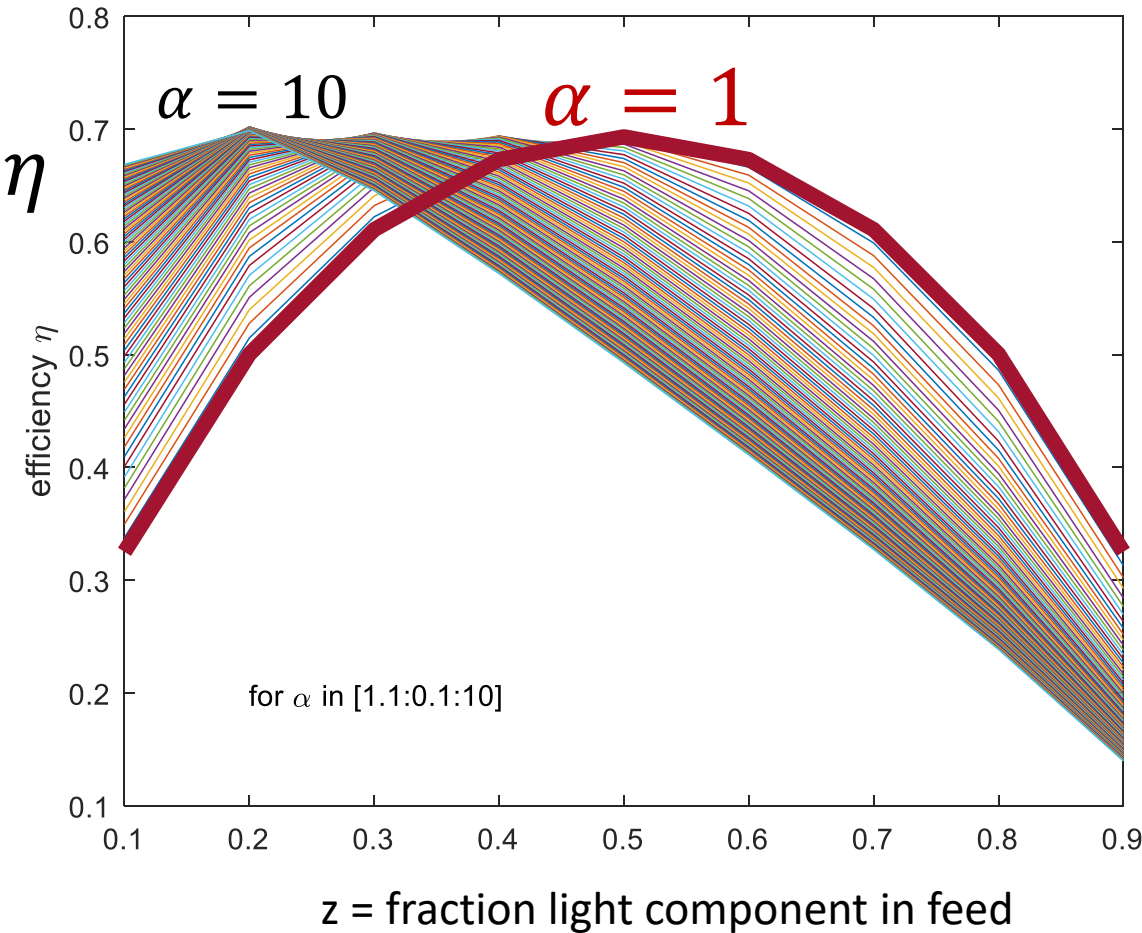
$$\lim_{\alpha \rightarrow 1} \eta = -(z \ln z + (1 - z) \ln(1 - z))$$



Comment: Above 50% for z from 0.2 to 0.8

Peak efficiency is $-\ln 0.5 = 0.693$ at $z=0.5$

Thermodynamic efficiency of binary distillation



$$\eta = \frac{W_s^{id}}{W_{s,tot}} = \frac{-(z \ln z + (1-z) \ln(1-z))}{\left(z + \frac{1}{\alpha-1}\right) \ln \alpha}$$

- High efficiency at small z for easy separations with large α
- Reason: Must evaporate light component to get it over top

$$Q_r = \lambda V_{\min} = \left(z + \frac{1}{\alpha-1}\right) \lambda F$$

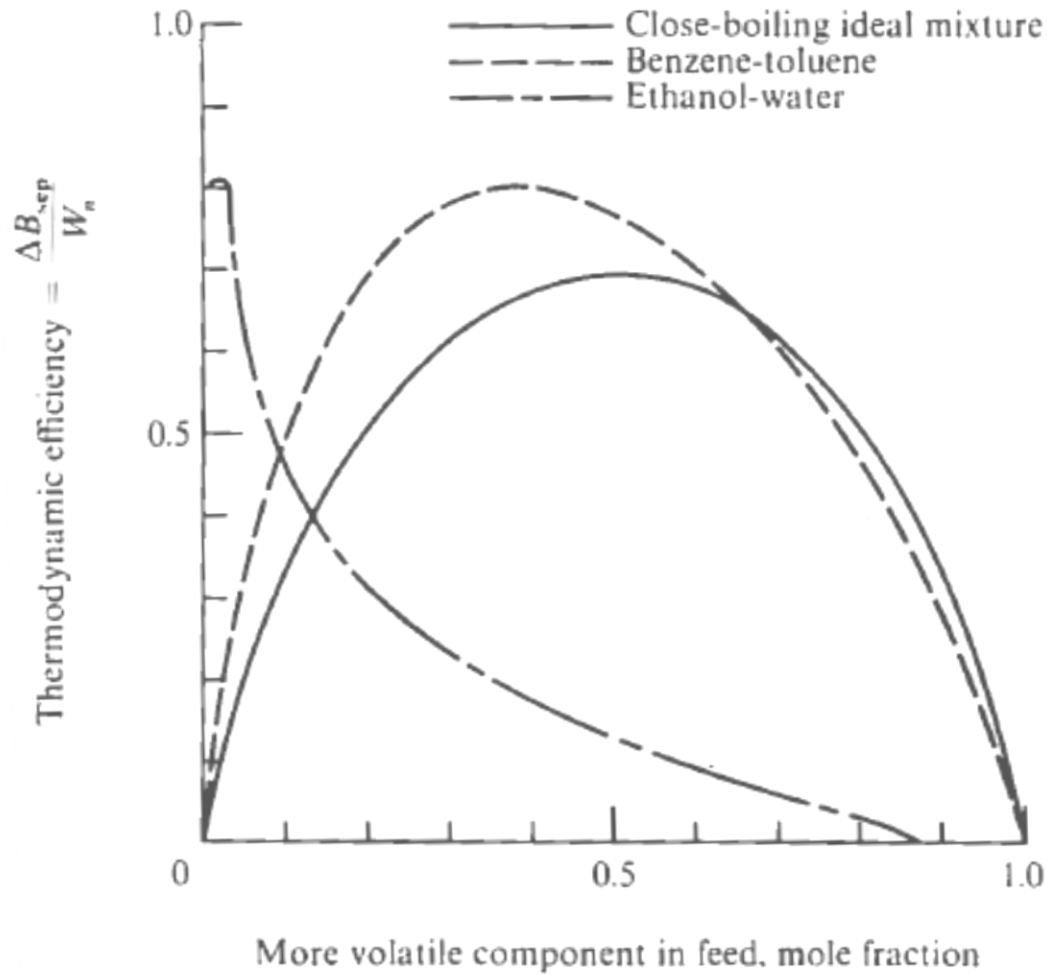
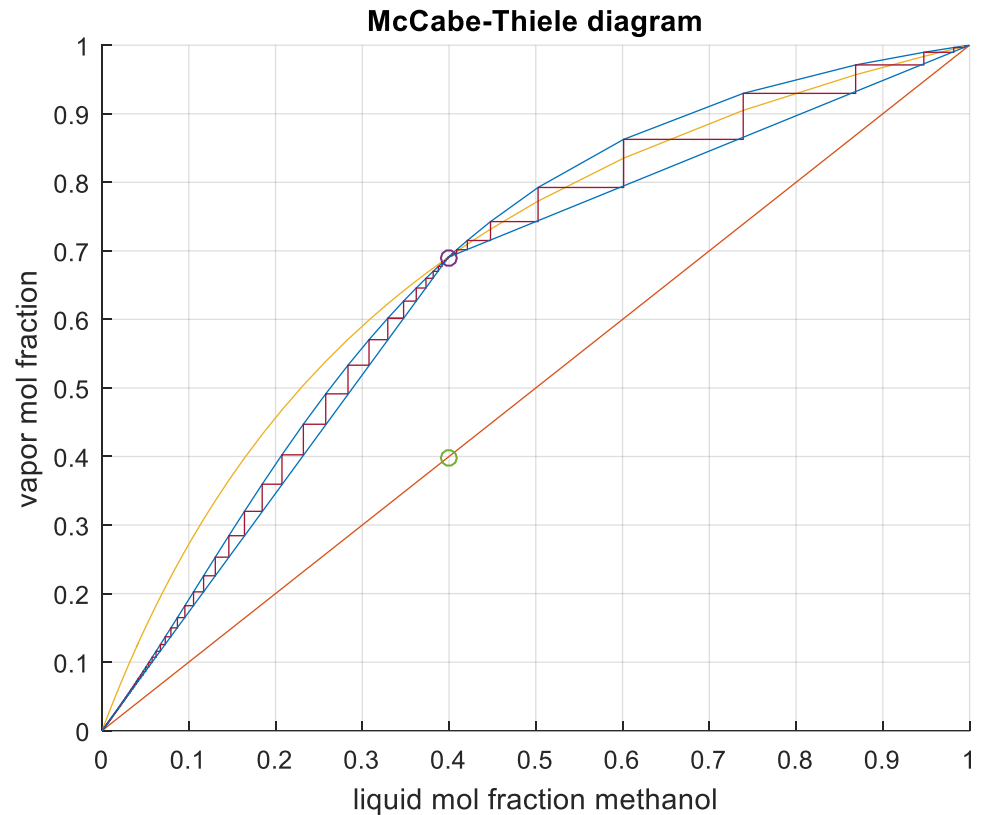


Figure 13-10 Thermodynamic efficiency of distillation of various mixtures at minimum reflux. (Data from Robinson and Gilliland, 1950.)

Note: Non-ideality does not necessarily imply lower thermodynamic efficiency

Why is it not perfect – where are the losses?

- Irreversible mixing loss at every stage.
- Largest losses in the middle of each section – where the bulk separation takes place
- Small losses at the high-purity column ends



Reversible binary distillation

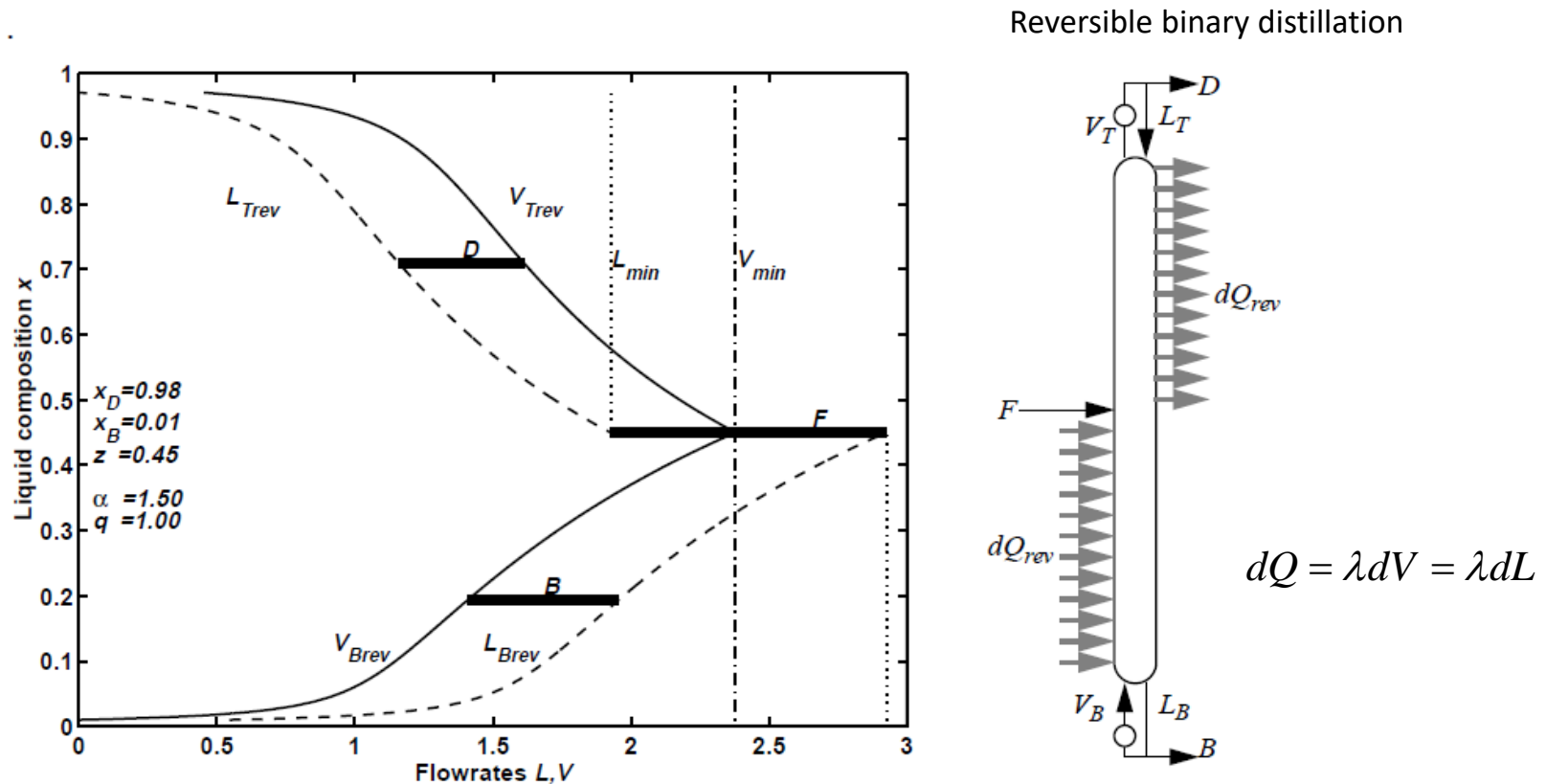
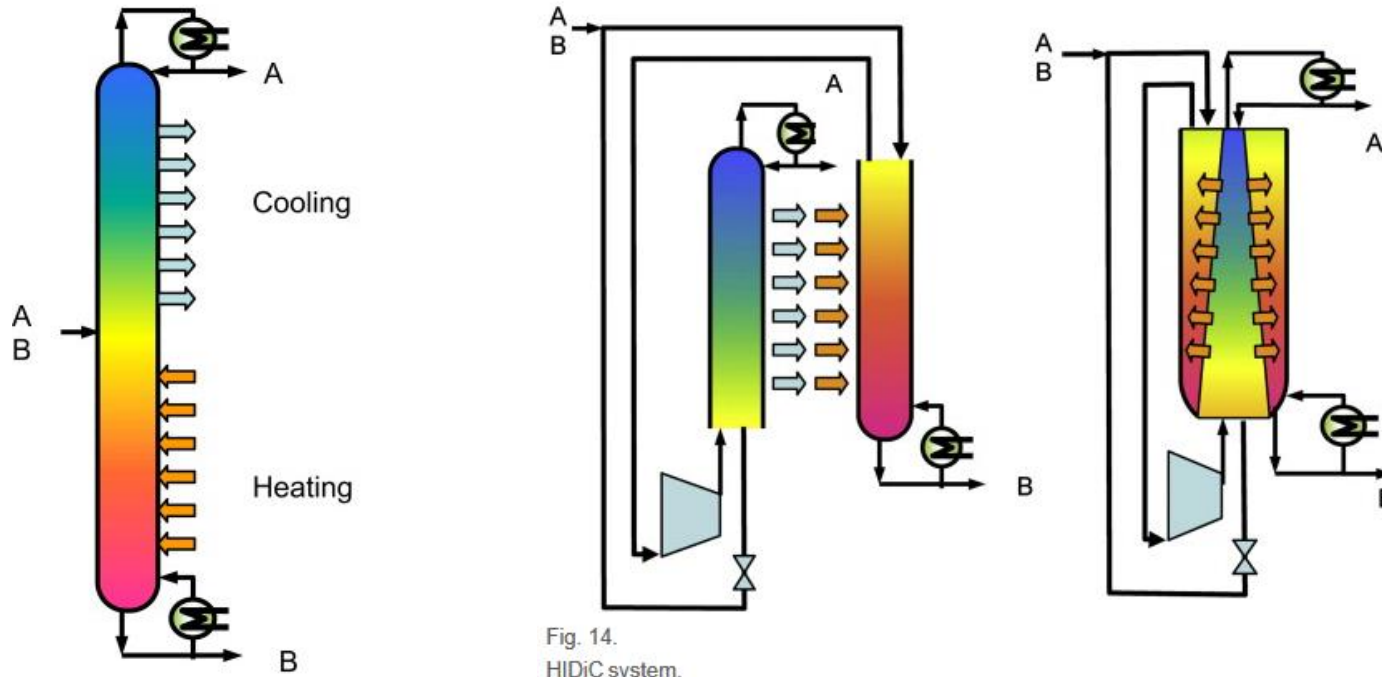


Figure 6.9: Reversible binary distillation: Internal flow rates (L_{rev}, V_{rev}) as function of composition. Minimum flow rates in an adiabatic column are also indicated (L_{min}, V_{min}). Note that $V_T L_T = D$ and $L_B - V_B = B$

Reversible binary distillation

HIDiC (Heat Integrated Distillation Column)



I have written papers on HIDiC, but don't believe in it.....
Too complicated, too much investment, not enough savings

Distillation is unbeatable for high-purity separations

- Operation: Energy usage essentially independent of product purity
- Capital: No. of stages increases with $\log(\text{impurity})$

$$\text{Fenske: } N_{\min} = \ln S / \ln \alpha$$

$$\text{Actual: } N \approx 2.5 N_{\min}$$

$$\text{Separation factor: } S \approx \frac{1}{x_{L,B} x_{H,D}}$$

OPERATION

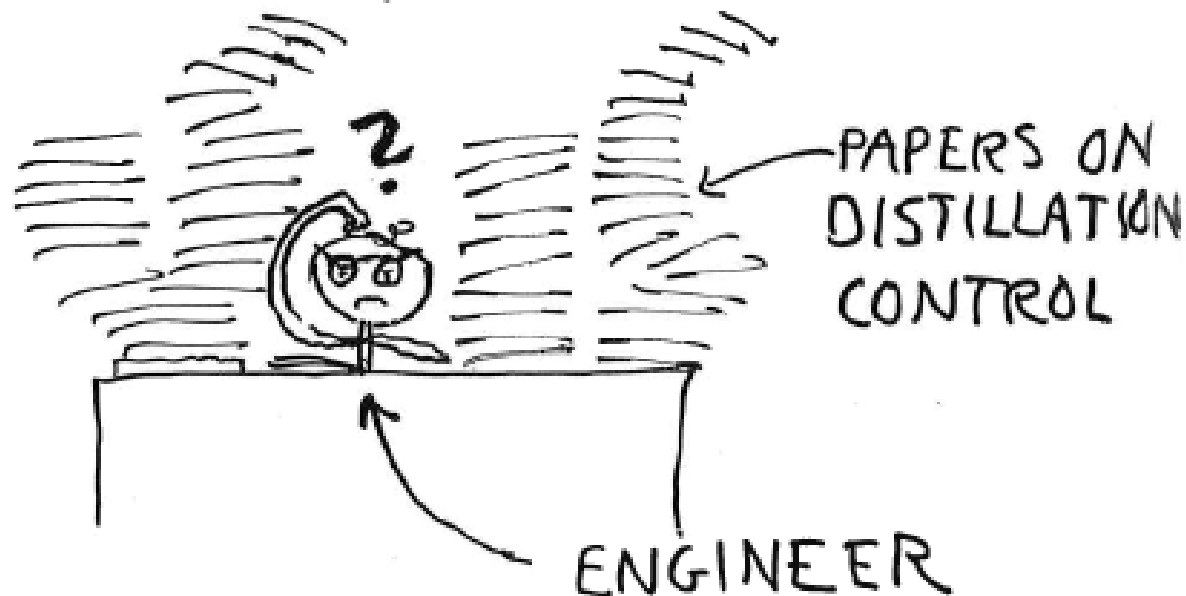
Economics and sustainability for operation of distillation columns

Is there a trade-off?

- No, not as long as the column is operated in a region of constant (optimal) stage efficiency
- Yes, if we operate at too high or too load so that the stage efficiency drops

DISTILLATION CONTROL

- Studied in hundreds of research and industrial papers over the last 40 years



Problem industrial papers: Ad-hoc

Problem academic papers: Not distillation, but show that "my control theory is best"

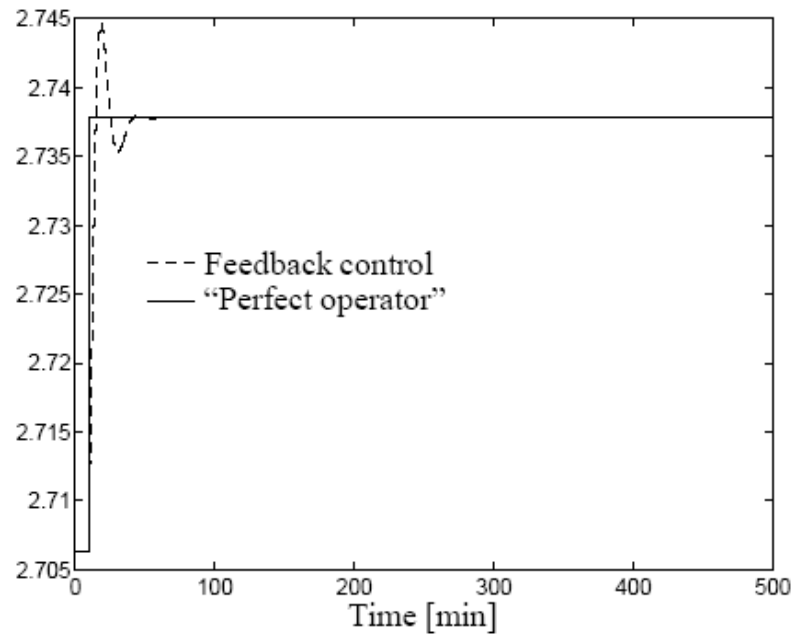
Myth of slow control

- Use extra energy because control is poor
- **Let us get rid of it!!!**

- Compare manual (“perfect operator”) and automatic control for typical column:
 - 40 stages,
 - Binary mixture with 99% purity both ends,
 - relative volatility = 1.5
 - First “one-point” control: Control of top composition only
 - Then “two-point” control: Control of both compositions

Myth about slow control

One-point control



(a) Reflux L

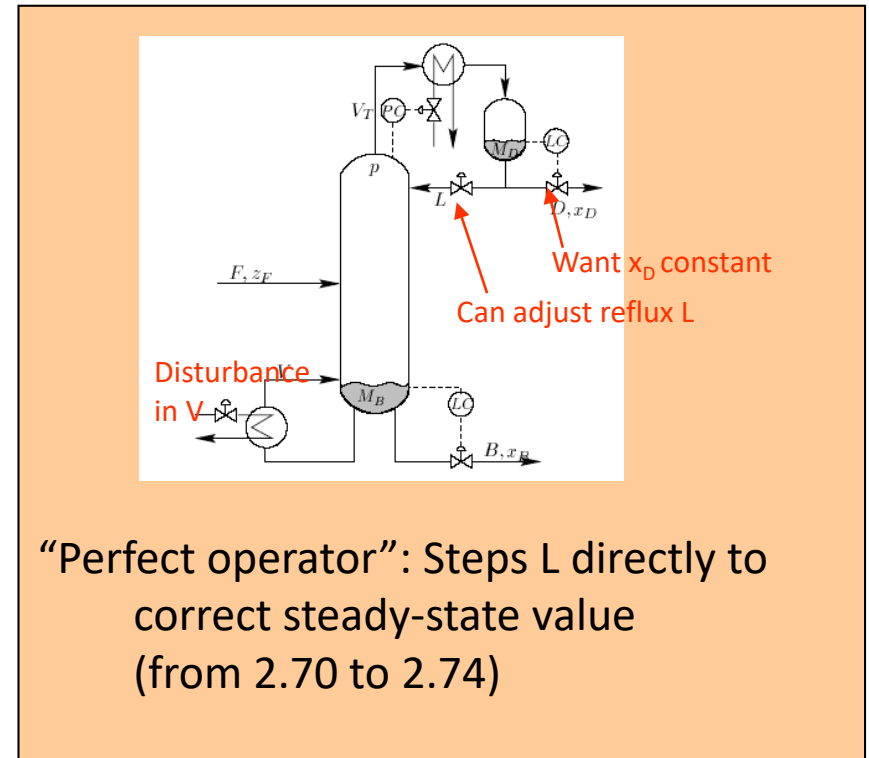
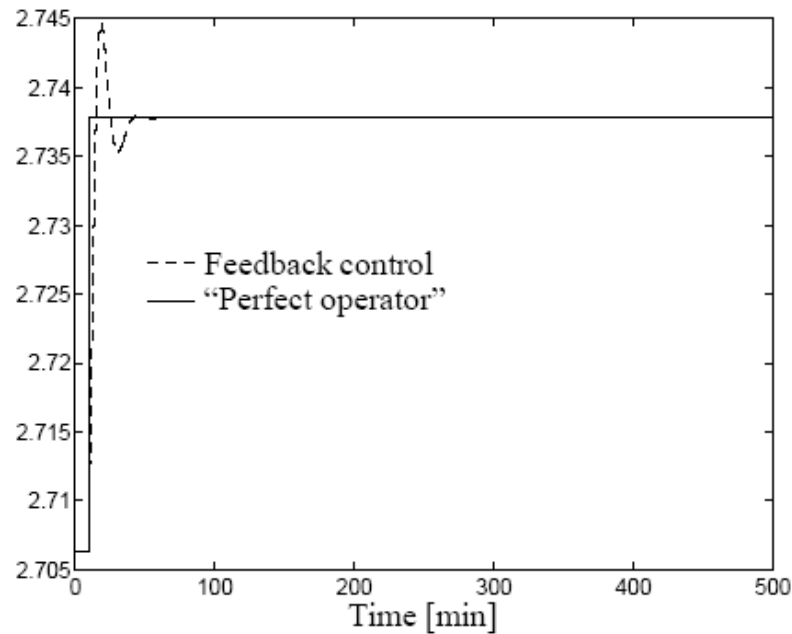


Figure 12: One-point control of x_D : Response to a 1% step increase (disturbance) in V . Solid line: Simultaneous step increase in L (“perfect operator”). Dashed line: Feedback where L is used to control x_D (PI-settings: $k = 60$, $\tau_I = 3.6$ min)

Myth about slow control

One-point control



(a) Reflux L

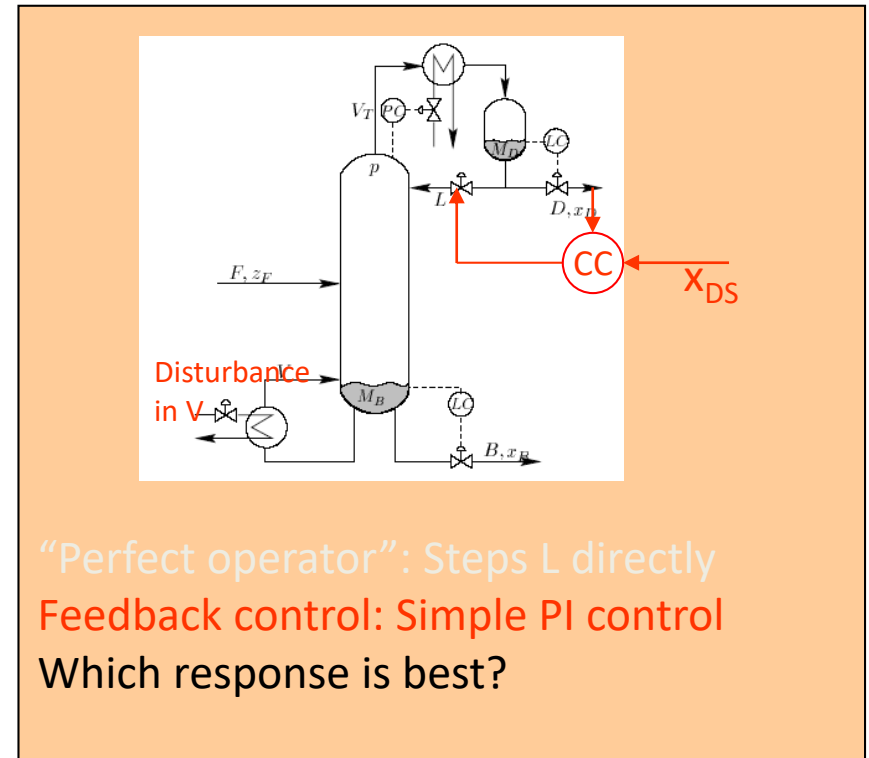
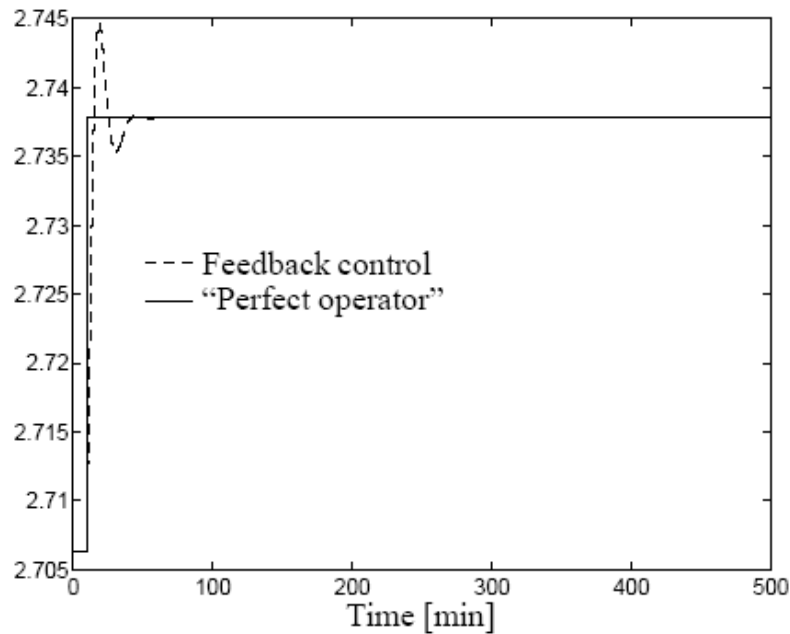


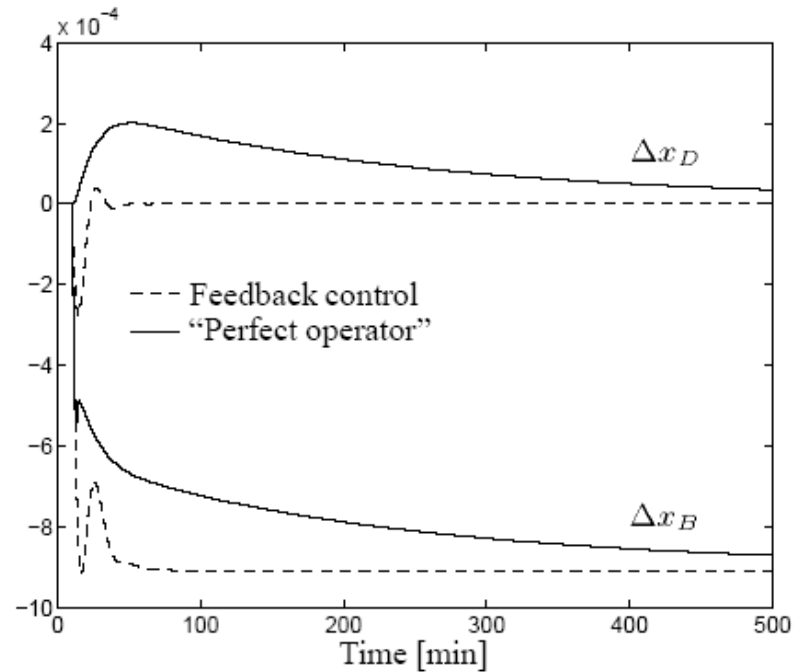
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Myth about slow control

One-point control



(a) Reflux L



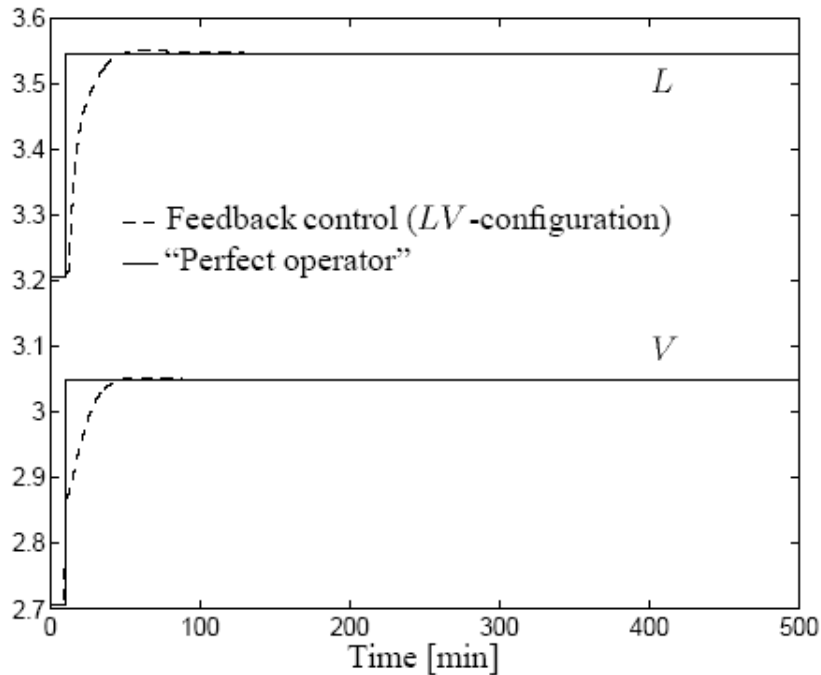
(b) Compositions, $\Delta x_D(t)$ and $\Delta x_B(t)$

Figure 12: One-point control of x_D : Response to a 1% step increase (disturbance) in V . Solid line: Simultaneous step increase in L (“perfect operator”). Dashed line: Feedback where L is used to control x_D (PI-settings: $k = 60$, $\tau_I = 3.6$ min)

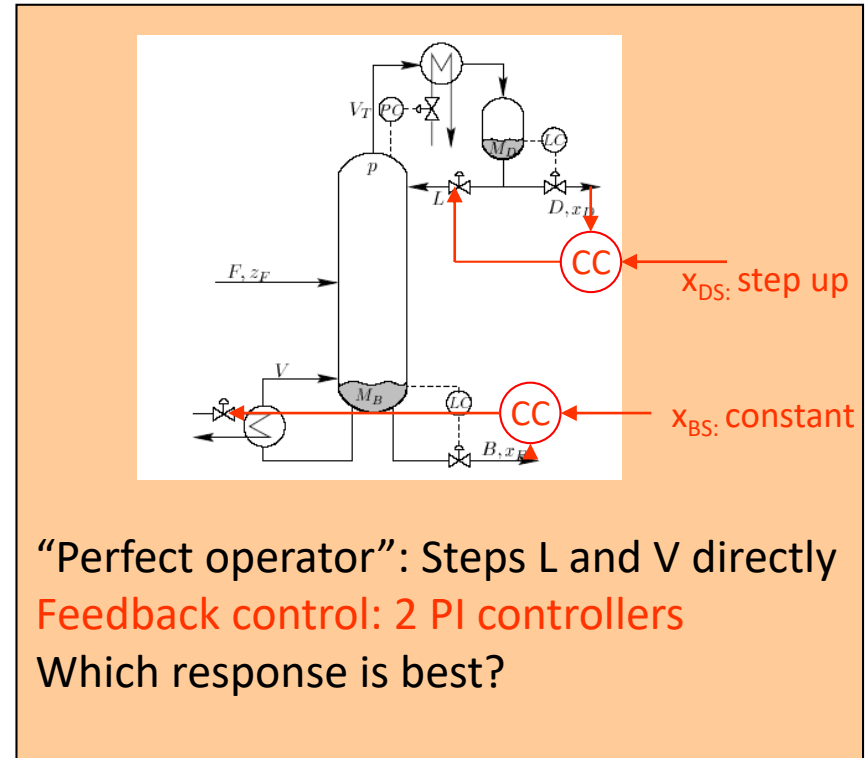
SO SIMILAR (inputs)
... and yet SO DIFFERENT (outputs)

Myth about slow control

Two-point control



(a) Reflux L and boilup V

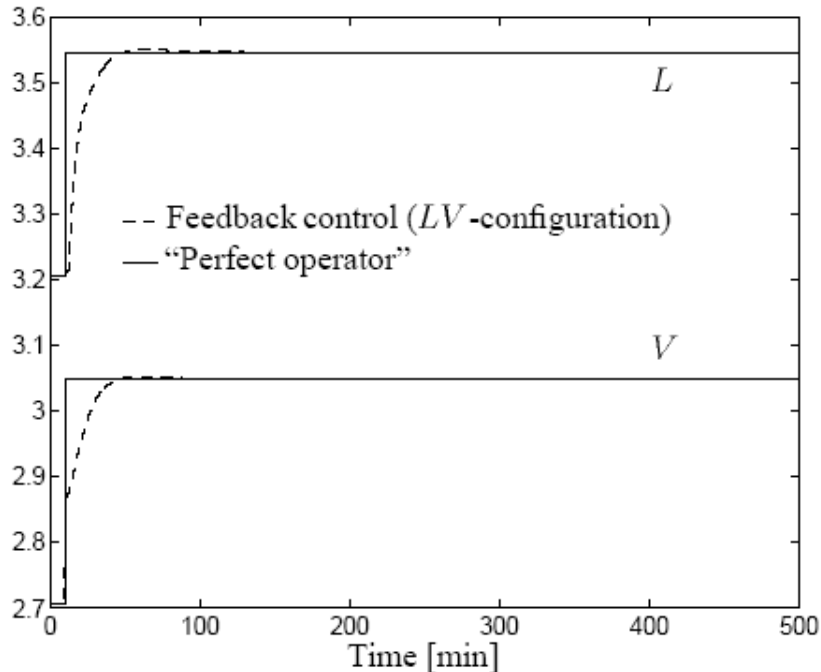


“Perfect operator”: Steps L and V directly
 Feedback control: 2 PI controllers
 Which response is best?

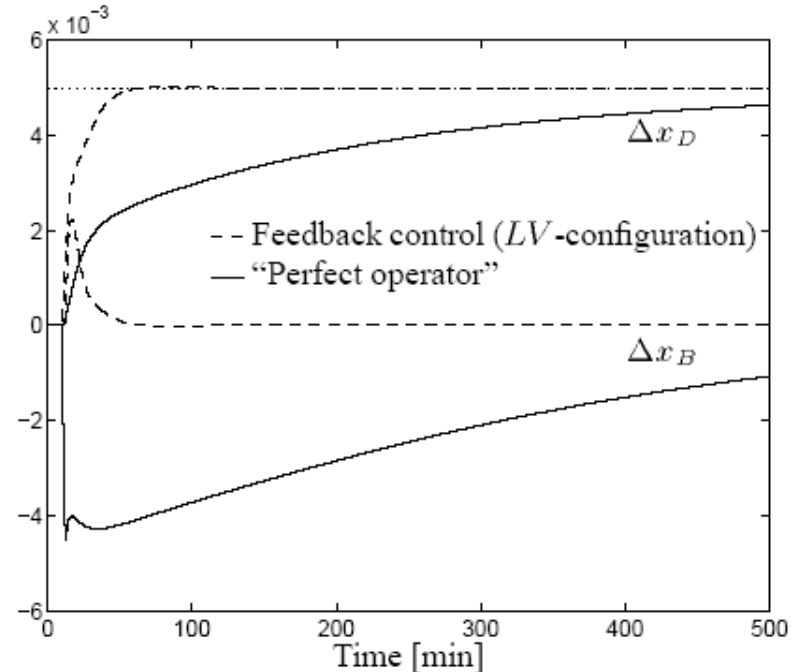
Figure 13: Two-point control: Setpoint change in x_D from 0.99 to 0.995 with x_B constant. Solid line: Simultaneous step increase in L and V to their new steady-state values (“perfect operator”). Dashed line: Feedback control using the LV -configuration with PI-settings in (82).

Myth about slow control

Two-point control



(a) Reflux L and boilup V



(b) Compositions, $\Delta x_D(t)$ and $\Delta x_B(t)$

Figure 13: Two-point control: Setpoint change in x_D from 0.99 to 0.995 with x_B constant. Solid line: Simultaneous step increase in L and V to their new steady-state values (“perfect operator”). Dashed line: Feedback control using the LV -configuration with PI-settings in (82).

SO SIMILAR (inputs)
... and yet SO DIFFERENT (outputs)

Myth about slow control

Conclusion:

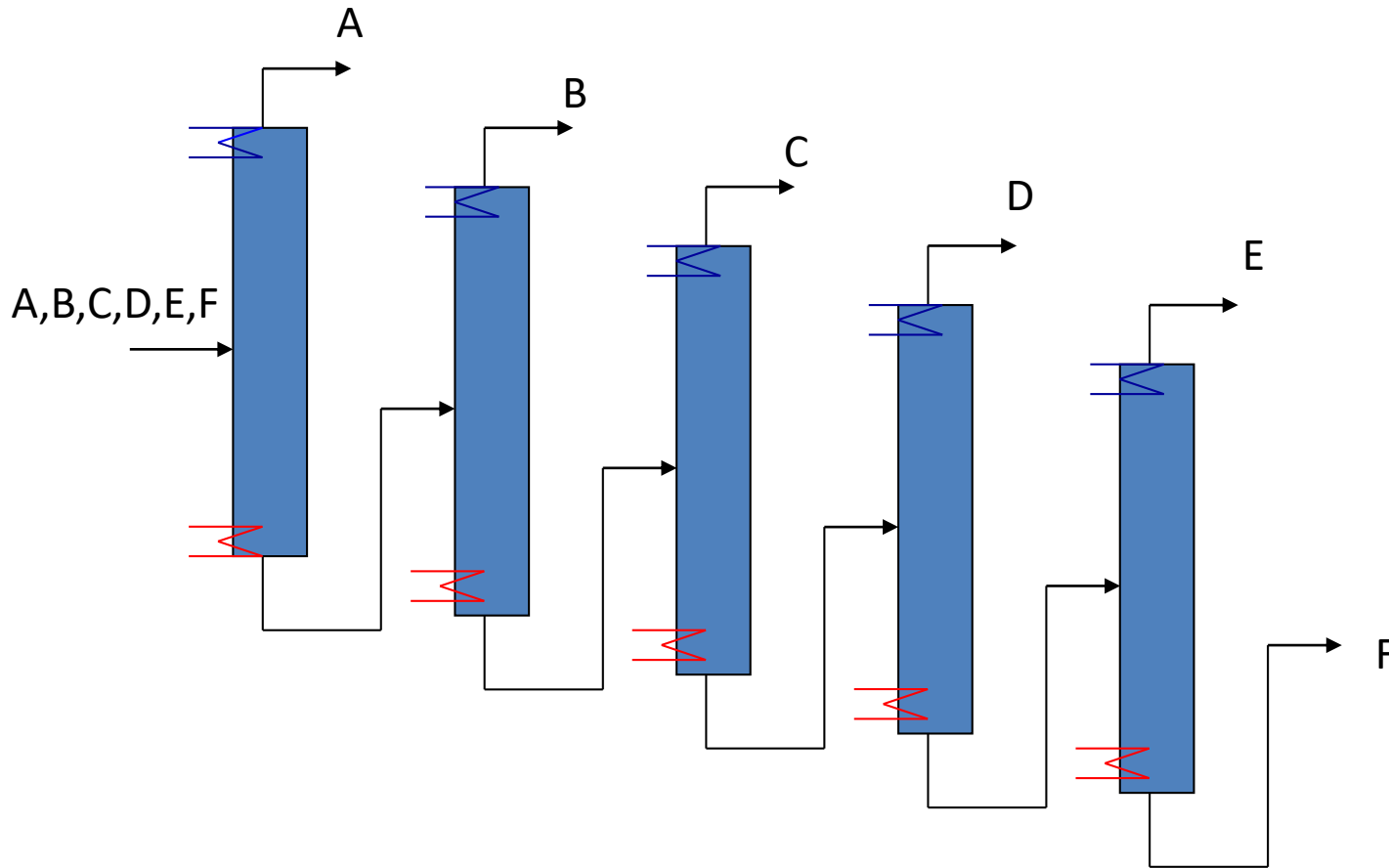
- Experience operator: Fast control impossible
 - “takes hours or days before the columns settles”
- BUT, with feedback control the response can be fast!
 - Feedback changes the dynamics (eigenvalues)
 - Requires continuous “active” control
- Most columns have a single slow mode (without control)
 - Sufficient to close a single loop (typical on temperature) to change the dynamics for the entire column

Complex columns

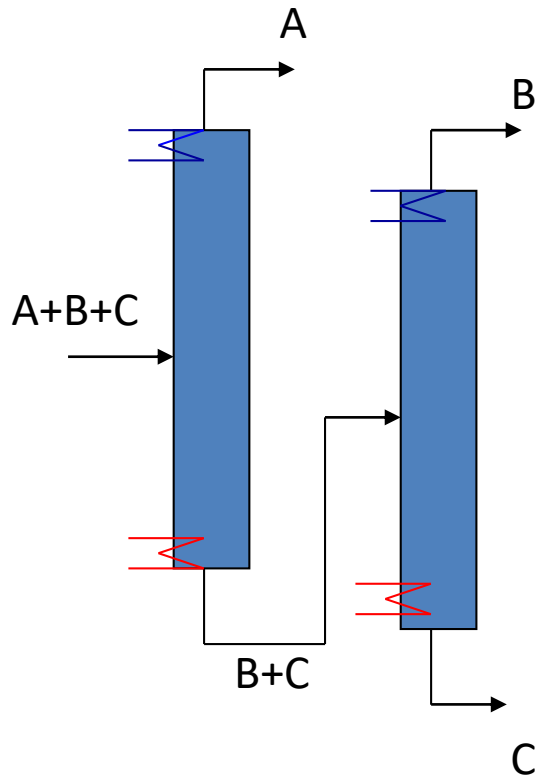
- Sequence of columns for multicomponent separation
- Heat integration
- Pressure levels
- Integrated solutions
- Non-ideal mixtures (azeotropes)

- Here: Will consider “Petlyuk” columns

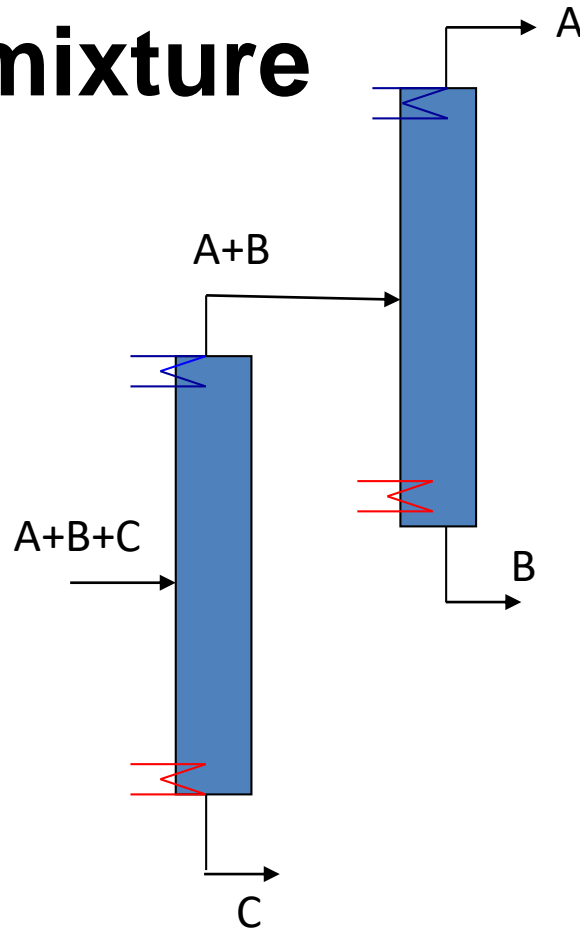
Typical sequence: “Direct split”



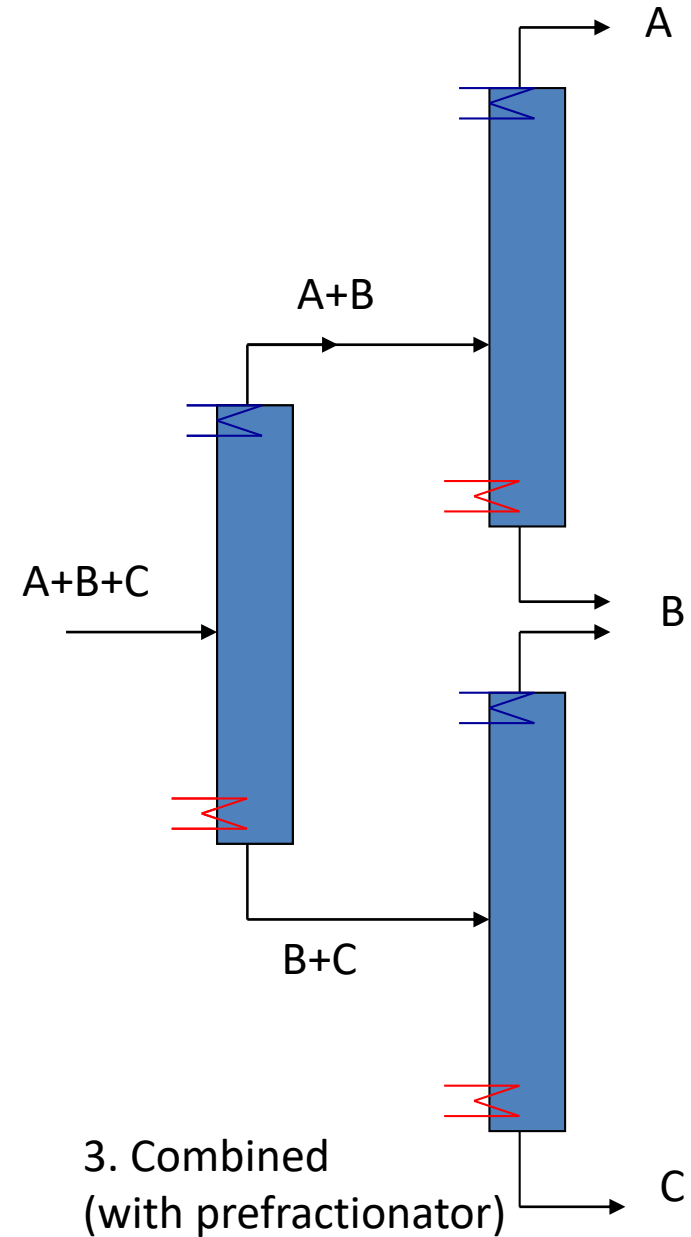
3-product mixture



1. Direct split

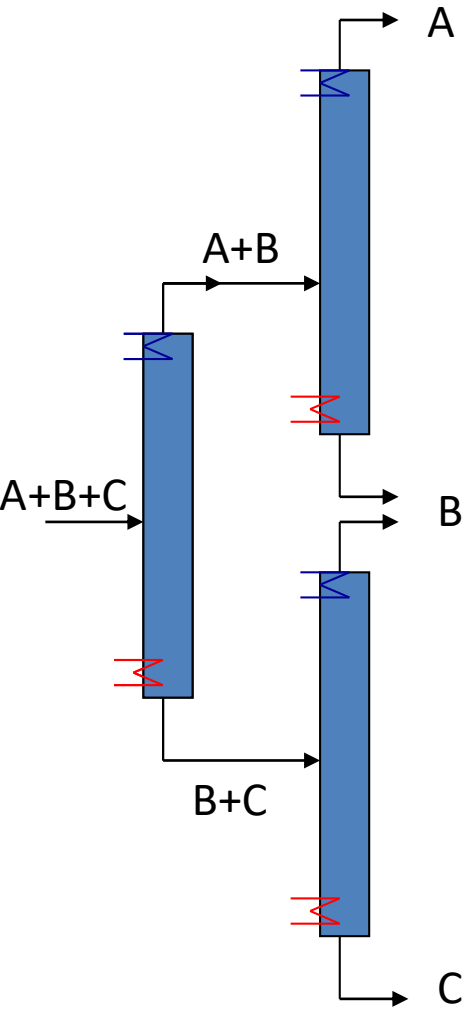


2. Indirect split

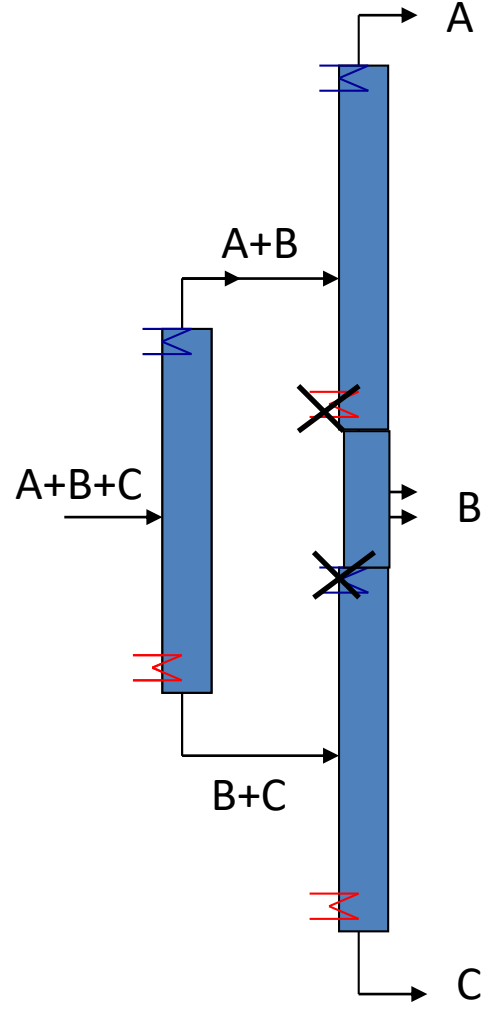


3. Combined (with prefractionator)

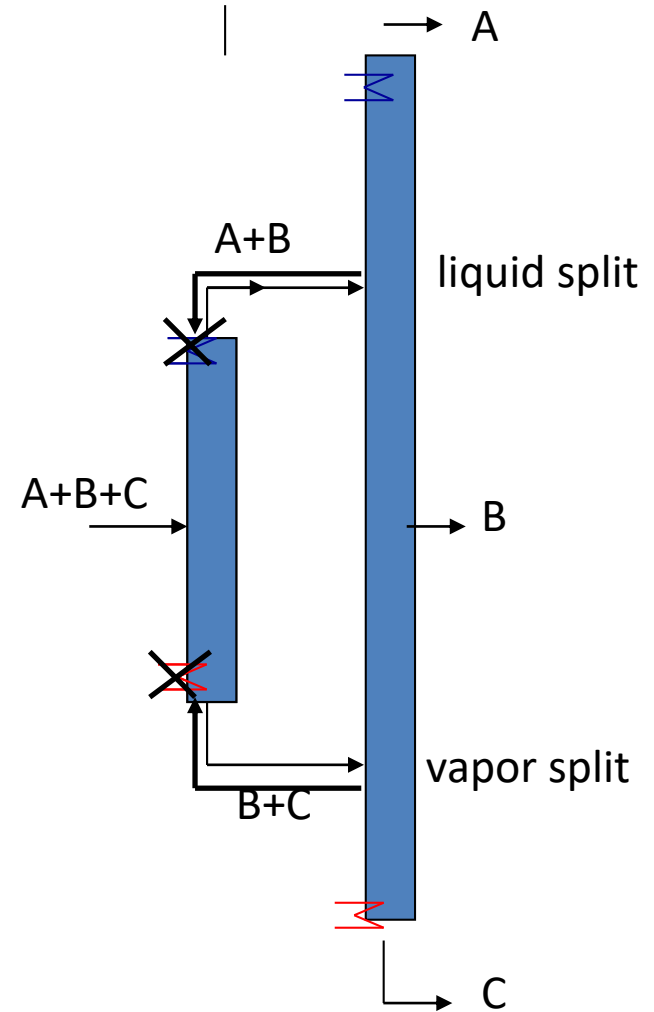
Towards the Petlyuk column



3.



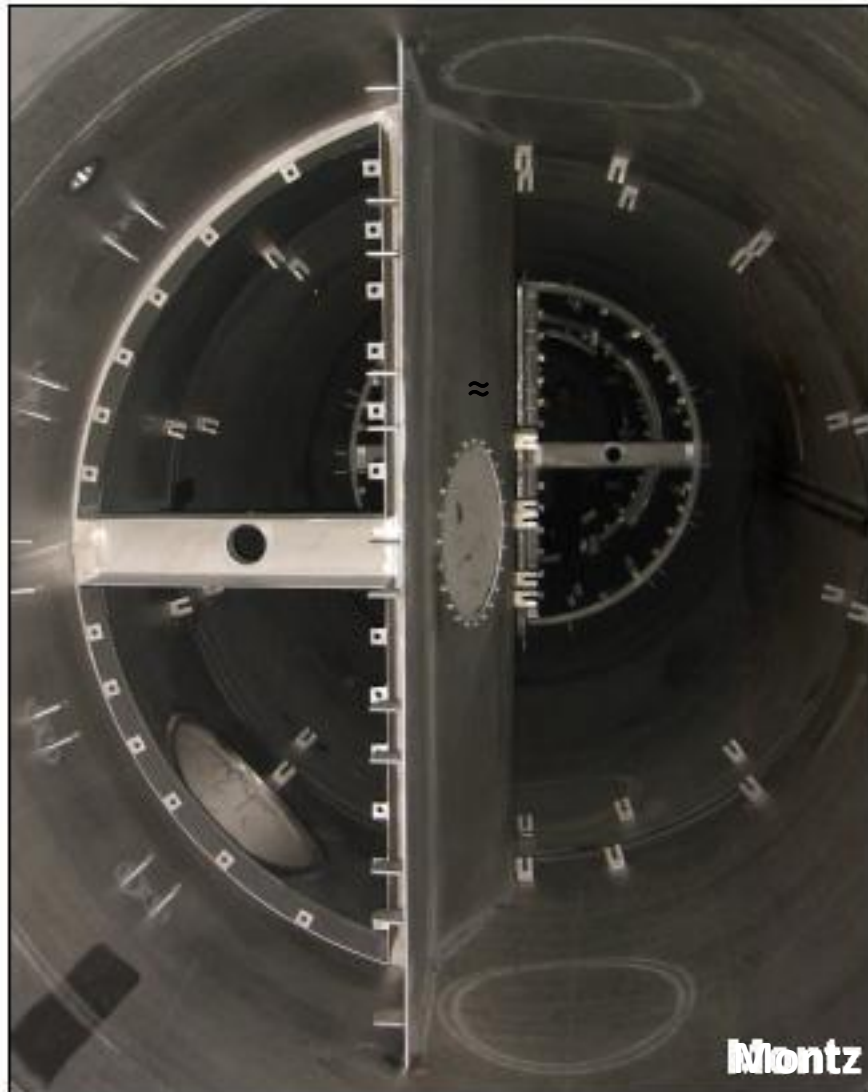
4. Prefractionator
+ sidestream column



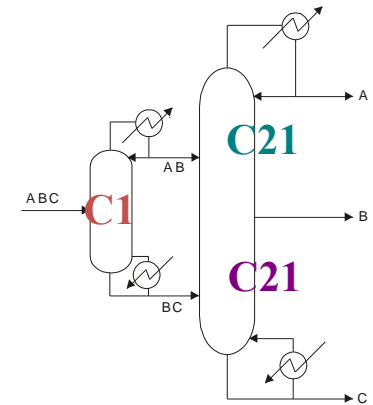
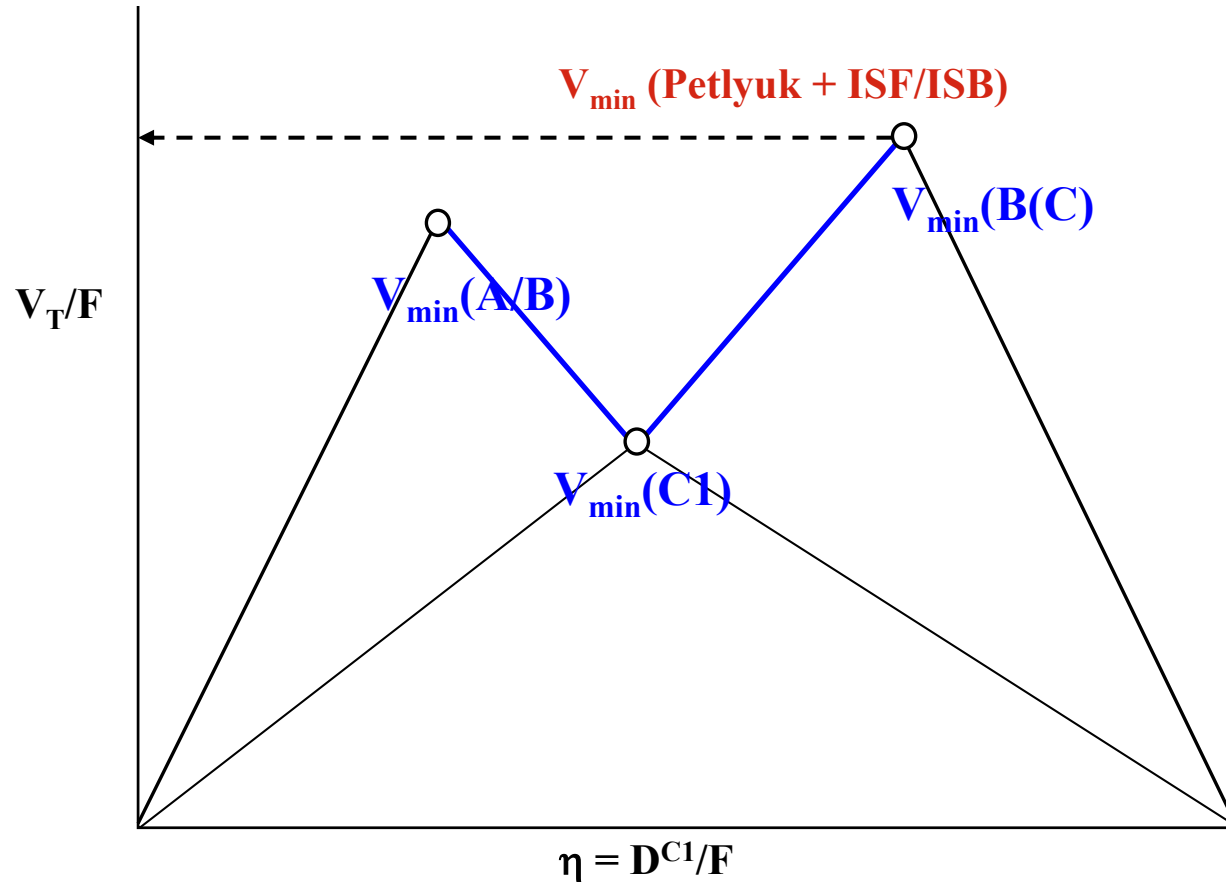
5. Petlyuk
30-40% less energy

Dividing Wall Columns

Off-center Position of the Dividing Wall



V_{\min} -diagram (Halvorsen)

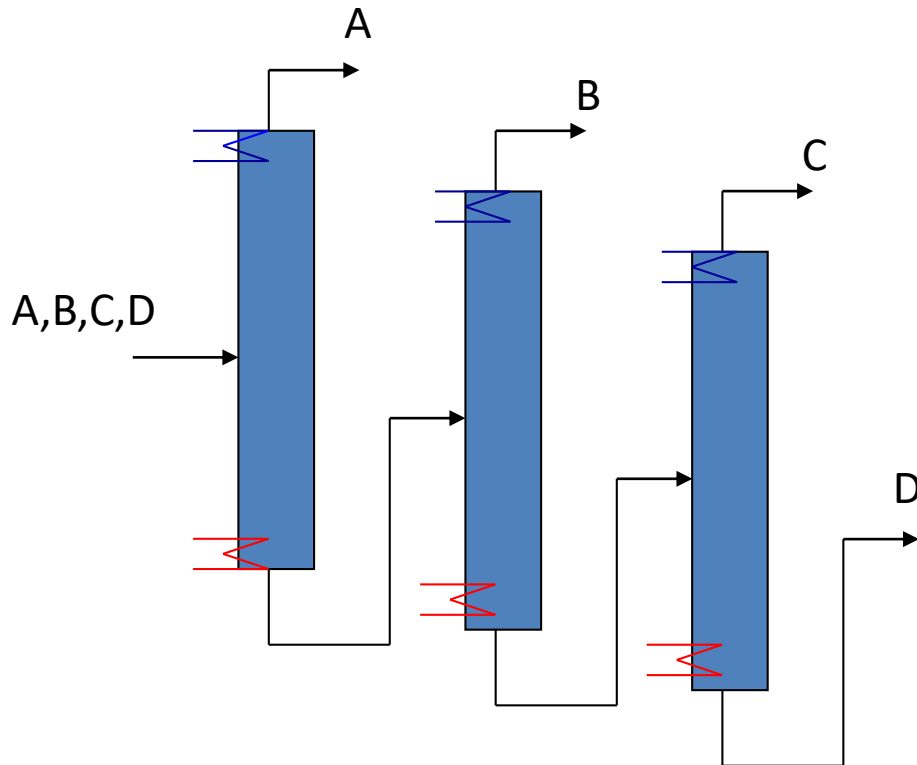


Petlyuk saves 30-40% energy but may be less efficient in terms of exergy
How fix?

Add side cooler or side reboiler : Can see from V_{\min} diagram!

4-product mixture

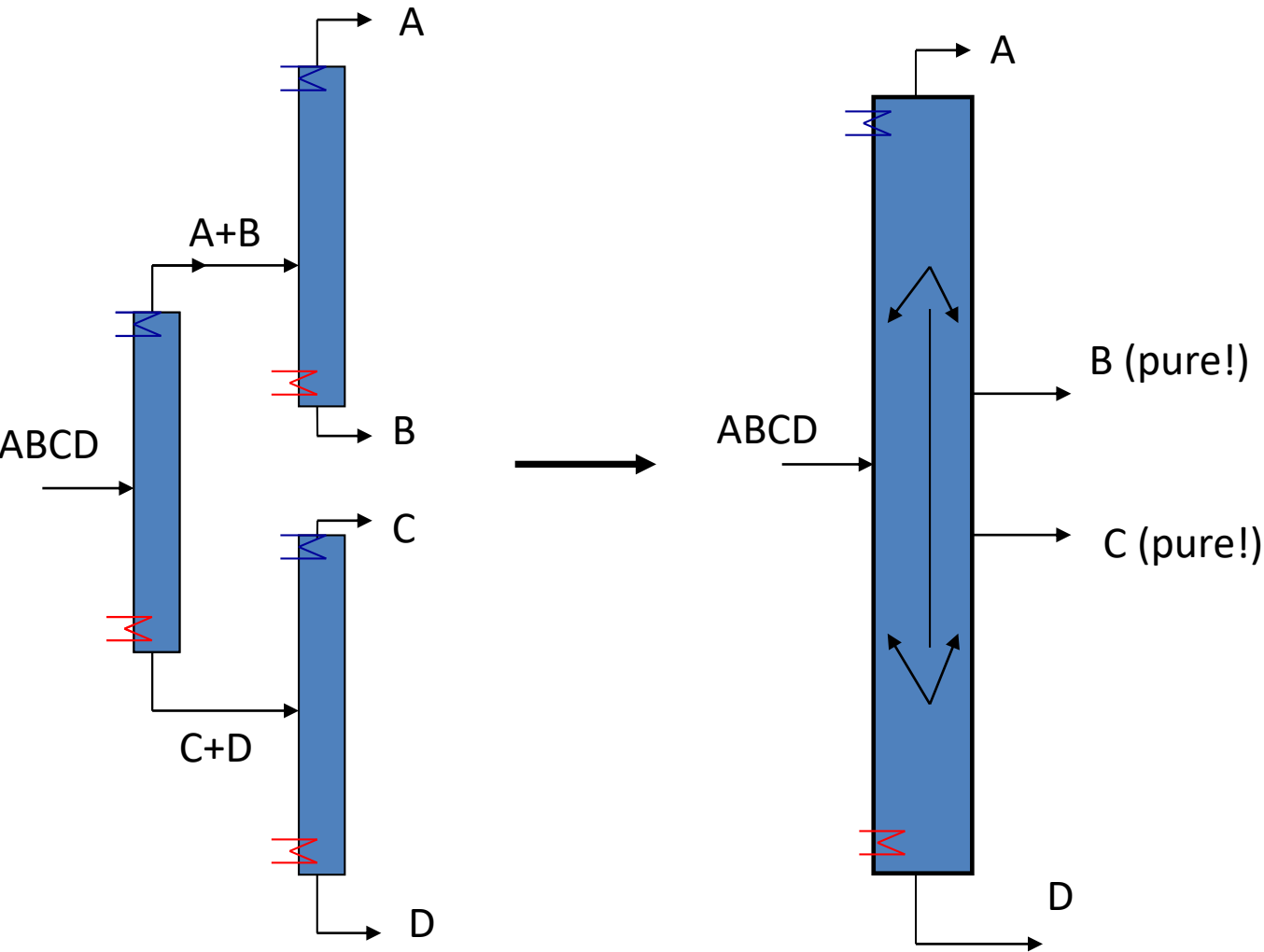
Conventional sequence with 3 columns



A – methanol
B – ethanol
C – propanol
D – butanol

Direct optimal extension of Petlyuk ideas requires two divided walls.
Will look for something simpler

4-product mixture: Kaibel column



- A – methanol
- B – ethanol
- C – propanol
- D – butanol

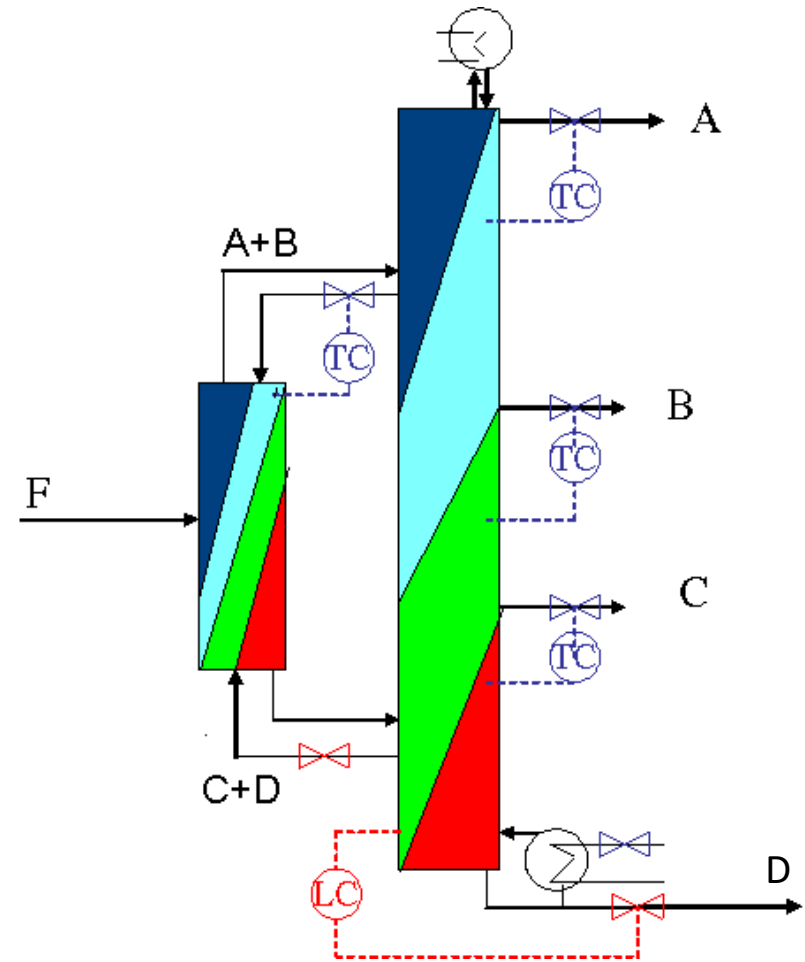
Alternative 3-column
sequence

Kaibel: 1 column!! More than 50% capital savings
Also saves energy (but maybe not exergy)

Control of Kaibel column

Close a “stabilizing” temperature (profile) loop for each split

- Prefractionator:
 - Close 1 “stabilizing” temperature loop
- Main column
 - Close 3 “stabilizing” temperature loops



H=6m
D=5cm

F →

D →

S1 →

S2 →

B →



Conclusion

- Distillation is important
- Distillation is unbeatable (in some cases)
- Distillation is fun
- Distillation is complex yet simple... and vice versa

Romania February 2019



Optimal operation distillation column

- Distillation at steady state with given p and F : $N=2$ DOFs, e.g. L and V (\mathbf{u})
- Cost to be minimized (economics)

$$J = -P \text{ where } P = \underbrace{p_D D + p_B B}_{\text{value products}} - p_F F - p_V V$$

↙ cost energy (heating+ cooling)
↖ cost feed

- Constraints

Purity D: For example $x_{D, \text{impurity}} < \max$

Purity B: For example, $x_{B, \text{impurity}} < \max$

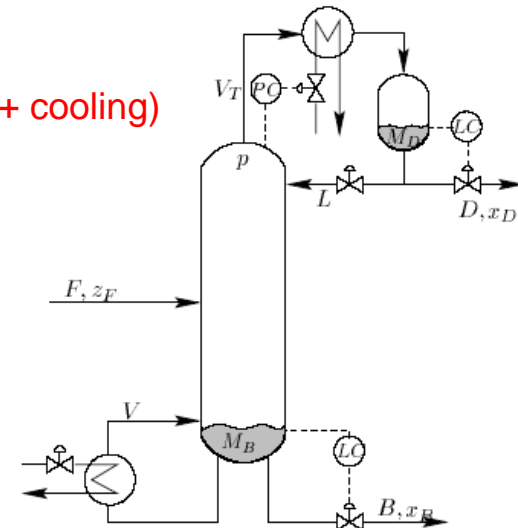
Flow constraints: $\min < D, B, L \text{ etc.} < \max$

Column capacity (flooding): $V < V_{\max}$, etc.

Pressure: p has given setpoint (can be given up, but need $p_{\min} < p < p_{\max}$)

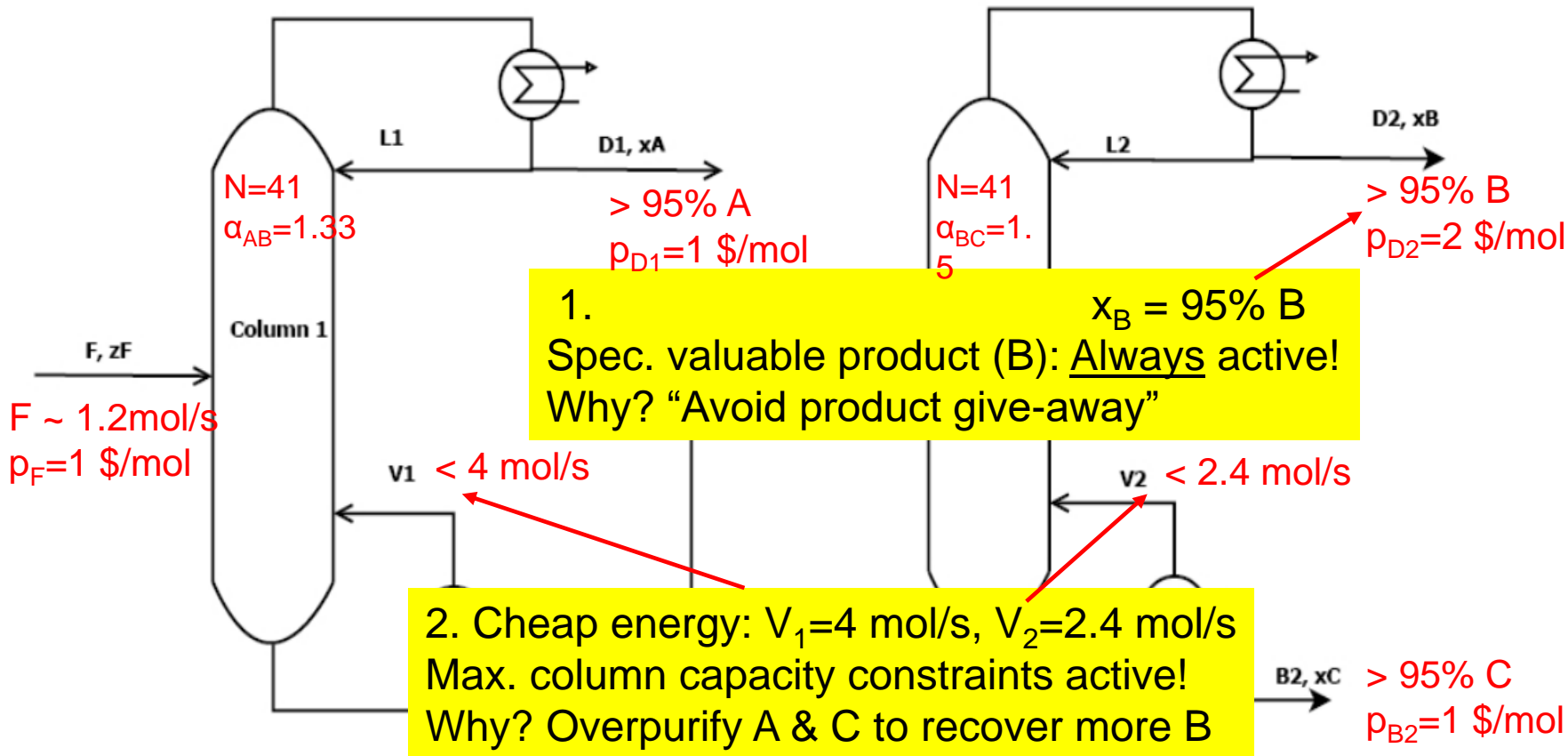
Feed: F has given setpoint (can be given up)

- Optimal operation: Minimize J with respect to steady-state DOFs (\mathbf{u})



Operation of Distillation columns in series

With given F (disturbance): 4 steady-state DOFs (e.g., L and V in each column)



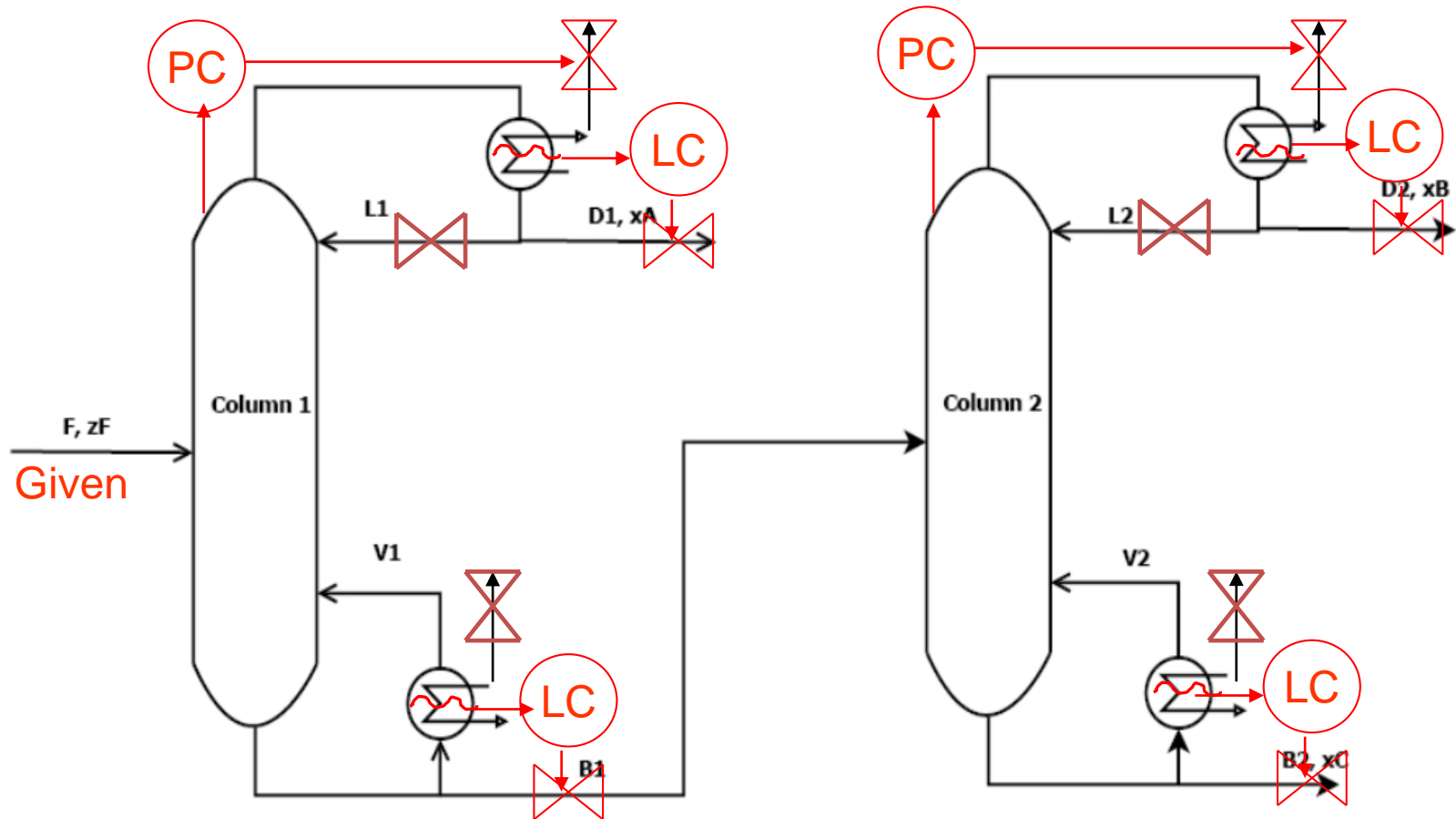
1. $x_B = 95\% \text{ B}$
 Spec. valuable product (B): Always active!
 Why? "Avoid product give-away"

2. Cheap energy: $V_1=4 \text{ mol/s}$, $V_2=2.4 \text{ mol/s}$
 Max. column capacity constraints active!
 Why? Overpurify A & C to recover more B

Cost (J) = - Profit = $p_F F + p_V(V_1+V_2) - p_{D1}D_1 - p_{D2}D_2 - p_{B2}B_2$
 Energy price: $p_V=0-0.2 \text{ $/mol}$ (varies)

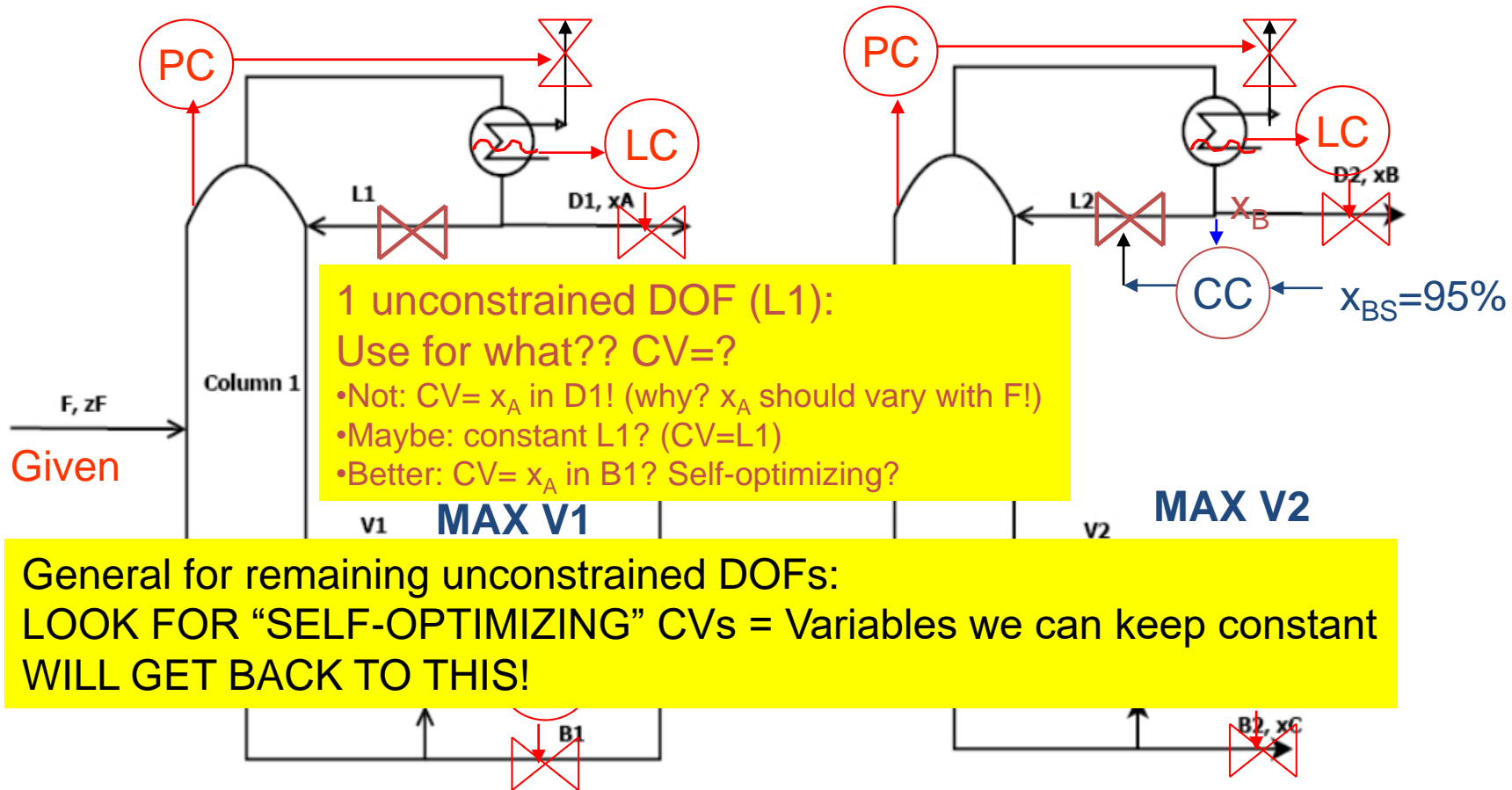
QUIZ 1: What are the expected active constraints?
 1. Always. 2. For low energy prices.

Control of Distillation columns in series



QUIZ 2. Assume low energy prices ($p_V=0.01$ \$/mol)
How should we control the columns?
HINT: CONTROL ACTIVE CONSTRAINTS

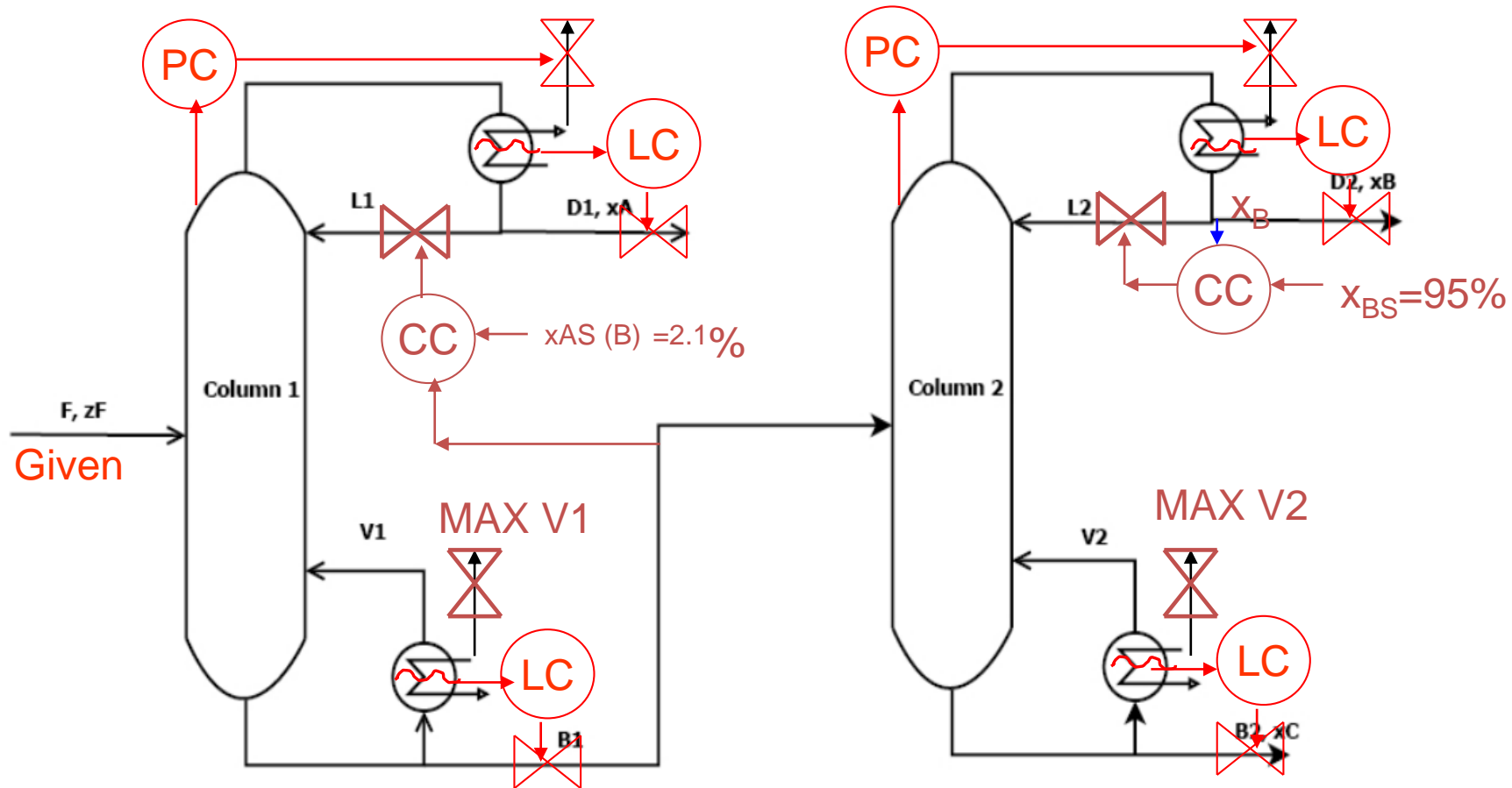
Control of Distillation columns in series



QUIZ 2. Assume low energy prices ($p_V=0.01$ \$/mol)
 How should we control the columns?
 HINT: CONTROL ACTIVE CONSTRAINTS

Solution.

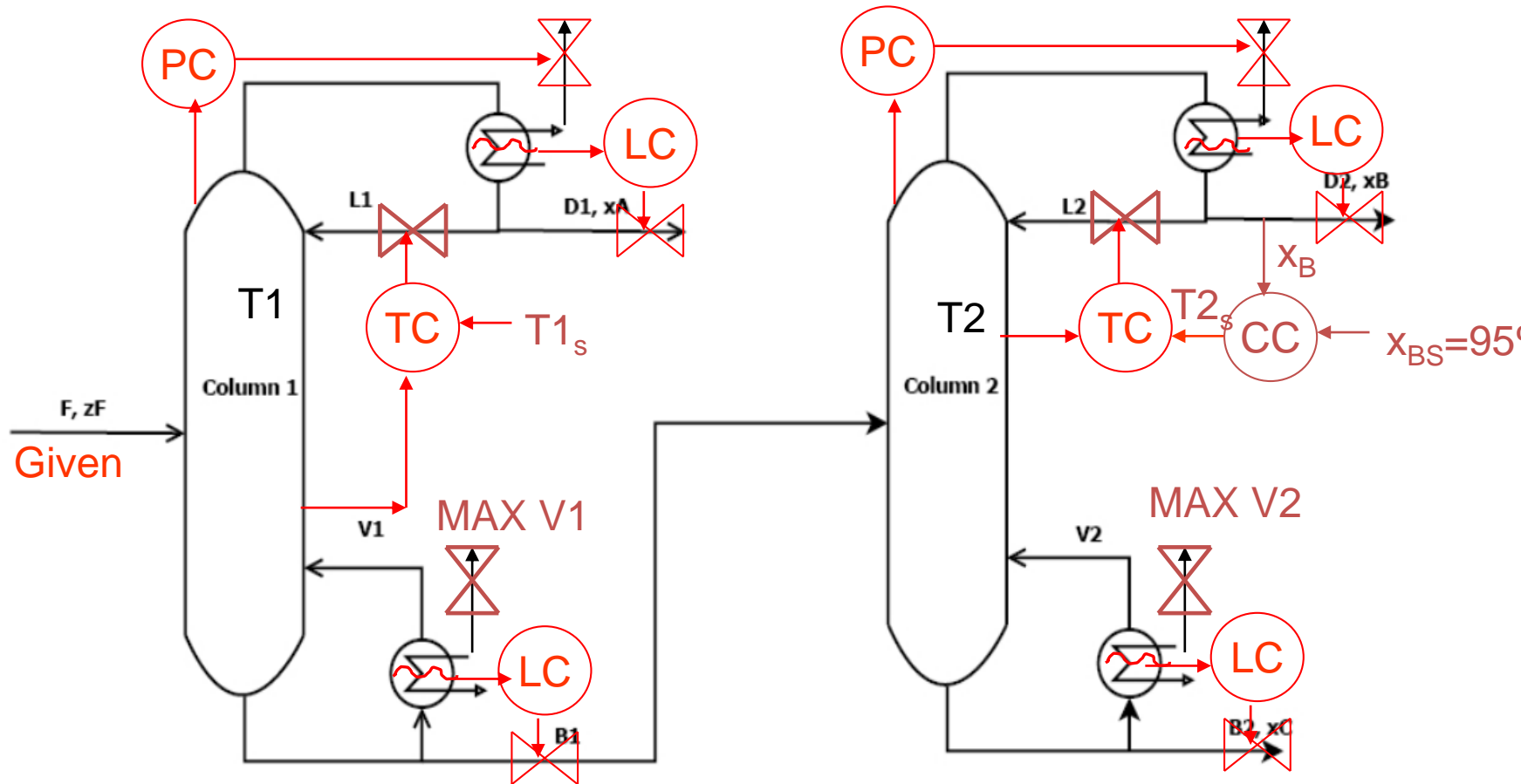
Control of Distillation columns. Cheap energy



Comment: Distillation column control in practice

1. Add stabilizing temperature loops
 - In this case: use reflux (L) as MV because boilup (V) may saturate
 - T_{1s} and T_{2s} then replace L_1 and L_2 as DOFs.
 2. Replace $V1=\max$ and $V2=\max$ by DPmax-controllers (assuming max. load is limited by flooding)
- See next slide

Control of Distillation columns in series



More on: Optimal operation

minimize $J = \text{cost feed} + \text{cost energy} - \text{value products}$

Two main cases (modes) depending on marked conditions:

Mode 1. Given feedrate

Mode 2. Maximum production

Comment: Depending on prices, Mode 1 may include many subcases (active constraints regions)

Acknowledgement

- Ivar Halvorsen, NTNU (20%) and SINTEF (80%)

$$\alpha \approx \exp \left[\frac{\Delta_{\text{vap}} H}{RT_b} \cdot \frac{T_{bH} - T_{bL}}{T_b} \right]$$

Thermodynamic efficiency for conventional distillation

- Use heat pumps for reboiler and condenser. Ideal work with surroundings at T_0 (Carnot):

$$W_r = Q_r \left(1 - \frac{T_0}{T_H}\right) \quad W_c = Q_c \left(1 - \frac{T_0}{T_C}\right)$$

- Assume feed liquid and constant molar flows so $Q_c \approx -Q_r$

$$W_{s,tot} = W_r + W_c = Q_r T_0 \left(\frac{1}{T_C} - \frac{1}{T_H}\right)$$

- Thermodynamic Efficiency = **Ideal** work/Actual work:

$$\eta = \frac{W_s^{id}}{W_{s,tot}} = \frac{-FRT_0 \sum_{i=1}^N z_i \ln z_i}{Q_r T_0 \left(\frac{1}{T_C} - \frac{1}{T_H}\right)}$$