

Problem 2 – Distillation of air (20%)

A feed with two components (79 mole% N₂ and 21% O₂) is to be separated by continuous distillation.

(a) Compute equilibrium data (y,x) at 1 atm for N₂-O₂ at x=0, 0.2, 0.4, 0.6, 0.8 and 1, given the relative volatility $\alpha = 3.5$.

(b) Find the minimum number of theoretical stages to get products with 99.99% N₂ (distillate, top) and 99.5% O₂ (bottom)? (You may find this graphically, but it is simpler to use the Fenske formula $N = \ln S / \ln \alpha$)

(c) What is the minimum reflux (L_{\min}/F) and corresponding minimum boilup (V_{\min}/F) when the feed F is saturated liquid and saturated vapor, respectively? (You may find this graphically)

Given: Relative volatility, $\alpha = (y_L/x_L) / (y_H/x_H) = y(1-x) / x(1-y)$, where y and x are the mole fractions of light component. The normal boiling point is 77.4 K for N₂ and 90.2 K for O₂.

Solution

(a) Note from the boiling points that Nitrogen is the light component (x,y).

$$\alpha = (y/x) / ((1-y)/(1-x)) = 3.5$$

Solve with respect to y:

$$y = \alpha x / (1 + (\alpha-1)x)$$

Get with $\alpha=3.5$:

x	0	0.2	0.4	0.6	0.8	1
y	0	0.47	0.7	0.84	0.93	1

(b) Minimum stages (N_{\min}) is obtained with infinite reflux, so $L/V=1$ and the operating line is on diagonal in the xy-diagram. Note that the feed composition and feed condition (liquid, vapor) does not matter when obtaining N_{\min} .

Separation factor,

$$S = (x_L/x_H)_D / (x_L/x_H)_B = (0.9999/0.0001) / (0.005/0.995) = 1989801$$

$$\ln S = 14.51$$

$$\ln \alpha = 1.25$$

$$\text{Fenske: } N_{\min} = \ln S / \ln \alpha = 11.59$$

(You can also do this graphically on the xy-diagram by making stair case between the diagonal and equilibrium curve, but it is very difficult to get accurate values in the corners).

(c) Want to find minimum reflux (L_{\min}), corresponding to infinite stages. This will correspond to a pinch at the feed.

Overall mass balance for the column

$$F = D + B$$

$$z_F F = x_D D + x_B B$$

Assume $F=1$, so all flows are relative to F. Introduce $B=1-D$. Get

$$z_F = x_D D + x_B (1-D)$$

or

$$D = (z_F - x_B) / (x_D - x_B) = (0.79 - 0.005) / (0.9999 - 0.005) = 0.789$$

Minimum flows in top section (see figure)

$$(L/V)_{\min} = (x_D - y') / (x_D - x')$$

where $x_D = 0.9999$ and (x', y') is at crossing between feed line and equilibrium curve (see Figure).

Note here that: $R = L/D = L/(V-L) = L/V / (1 - L/V)$

And then with known R_{\min} and D we can find L_{\min} and V_{\min} (see Figure of column below).

Feed liquid (see figure): $x' = z_F = 0.79$ and $y' = \alpha x' / (1 + (\alpha-1)x') = 0.9294$

$$(L/V)_{\min} = (x_D - y') / (x_D - x') = (0.9999 - 0.9294) / (0.9999 - 0.79) = 0.3359$$

$$R_{\min} = (L/D)_{\min} = 0.3359 / (1-0.3359) = 0.5058$$

$$L_{\min} = (L/D)_{\min} * D = 0.5058 * 0.789 = \mathbf{0.399} \text{ (in top of column)}$$

$$V_{\min} = D+L = 0.789 + 0.399 = \mathbf{1.188} \text{ (in top and btm of column)}$$

Feed vapor: $y' = zF = 0.79$ and $x'=0.518$ (in equilibrium with y')

$$(LT/VT)_{\min} = (xD-y') / (xD-x') = (0.9999 - 0.79)/(0.9999-0.0.518) = 0.4356$$

$$R_{\min} = (L/D)_{\min} = 0.4356 / (1-0.4356) = 0.7718$$

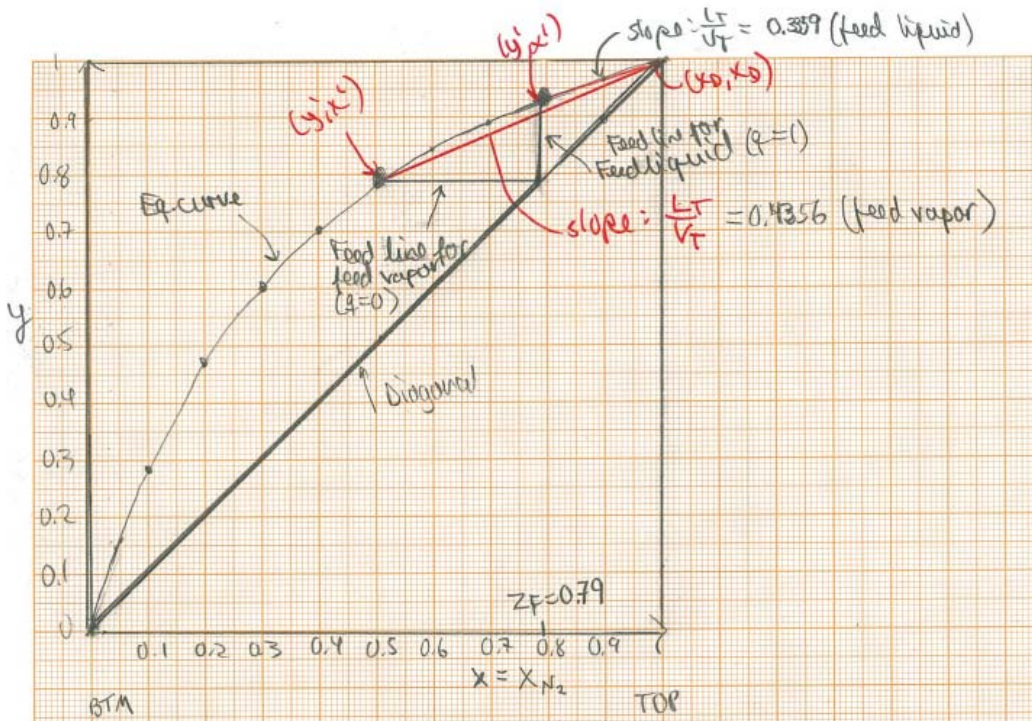
$$L_{\min} = (L/D)_{\min} * D/F = 0.7718 * 0.789 = \mathbf{0.609} \text{ (in top of column)}$$

$$VT_{\min} = D + L = 0.789 + 0.609 = 1.398 \text{ (in top of column, see figure)}$$

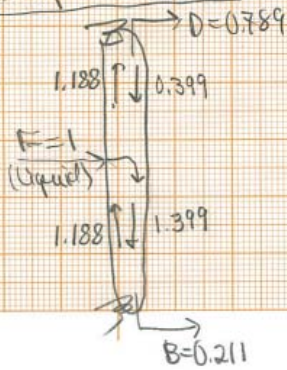
$$VB_{\min} = VT_{\min} - F = \mathbf{0.398} \text{ (in btm of column, see figure)}$$

The resulting minimum flows are summarized in the figure.

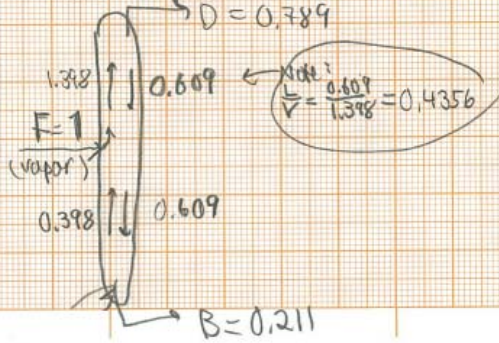
Comment: For feed liquid, Kings's formula from the lectures gives (assuming pure products)
 Feed liquid: $L_{\min}/F = 1/(\alpha-1) = 1/2.5 = 0.4$ (which agrees with the above)
 Feed vapor: $L_{\min}/F = 1/(\alpha-1) + B/F = 0.611$ (which agrees) or equivalently
 $V_{\min}/F = 1/(\alpha-1) = 1/2.5 = 0.4$ (which also agrees)



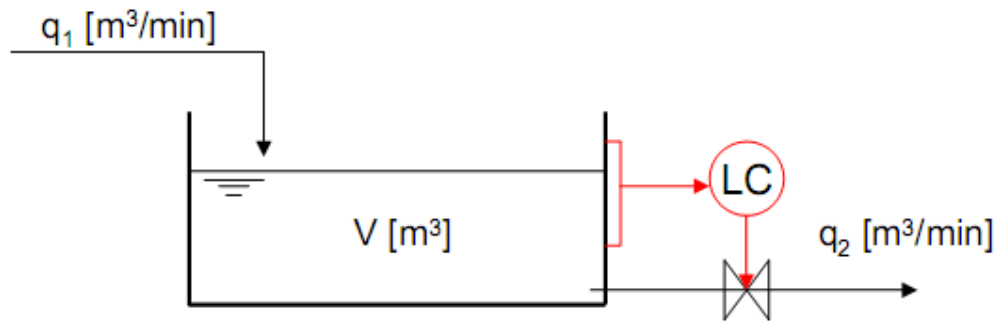
Feed liquid Min flows



Feed vapor Min flows



Problem 3 –Process control (15%)



(a) PI-controller:

$$q_2(t) = q_{20} + K_c (V(t) - V_0) + (K_c / \tau_I) \int (V(t) - V_0) dt$$

Advantage with I-action: No steady-state offset in V (but this may not be an important control objective as the main reason for having the tank is to have smooth changes in q_2).

Disadvantage: More tuning parameters and may get cycling. Also, may actually want V to change (increase) when there is a larger flow (because then we have more capacity to get a smooth decrease in q_2 when q_1 drops).

(b) P-control: $q_2 = q_{20} + K_c (V - V_0)$

(i) $0.2 = 0.1 + 0.5 (V - 1) \rightarrow V = 1.2 \text{ m}^3$

$0.3 = 0.1 + 0.5 (V - 1) \rightarrow V = 1.4 \text{ m}^3$

Comment: With a PI-controller the steady-state value of V is 1 m^3 in all cases.

(ii) Mass balance around tank

$$dm/dt = w_{in} - w_{out} \text{ [kg/s]}$$

where $m = \rho V$, $w_{in} = \rho_1 q_1$, $w_{out} = \rho_2 q_2$

Assume: constant (equal) density ρ [kg/m³]. Then:

$$dV/dt = q_1 - q_2$$

(iii) From the P-controller equation

$$dq_2/dt = K_c dV/dt$$

Inserted into the material balance

$$(1/K_c) dq_2/dt = q_1 - q_2$$

Separate variables

$$dq_2/(q_1 - q_2) = K_c dt$$

Note that $q_1 = 0.3$ (step in q_1). Integrate from $t=0$ where $q_2 = 0.2$:

$$\ln(0.3 - q_2)/(0.3 - 0.2) = -K_c t$$

or

$$q_2 = 0.3 - 0.1 \exp(-K_c t)$$

so the time constant is

$$\tau = 1/K_c = 1/0.5 = 2 \text{ min}$$

Get

t [min]	0	1	2	5	∞
q_2 [m³/min]	0.2	0.239	0.263	0.292	0.3

see the Figure on the next page

c)

$$i) \underset{0.2}{q_2} = \overset{0.1}{q_1} + \overset{0.5}{k} (V - \overset{1 \text{ m}^3}{V_0}) \Rightarrow \underline{V = 1.2 \text{ m}^3}$$

ii) Materialbalansen
Beholdning: $\rho \cdot V$, In: $q_1 \cdot \rho_1$, Ut: $q_2 \cdot \rho_2$
 kg/m^3

Anta: $\rho = \rho_1$ (Blandetank), $\rho_1 = \rho_2$

För då $\frac{d}{dt}(\rho V) = \rho_1 q_1 - \rho_2 q_2 \Rightarrow \underline{\frac{dV}{dt} = q_1 - q_2}$

iii) Fra ligningen for P-regulatoren fås

$$\frac{dq_2}{dt} = k \frac{dV}{dt}$$

Indsatt i materialbalansen

$$\frac{1}{k} \frac{dq_2}{dt} = q_1 - q_2 \Rightarrow \underline{\frac{dq_2}{q_1 - q_2} = k dt}$$

Integrerer fra startpunkt ved $t=0$ der $q_2 = 0.2$. $q_1 = 0.3$ (konst.)

$$\int_{0.2}^{q_2} \frac{dq_2}{0.3 - q_2} = \int_0^t k dt \Rightarrow -\ln(0.3 - q_2) \Big|_{0.2}^{q_2} = kt$$

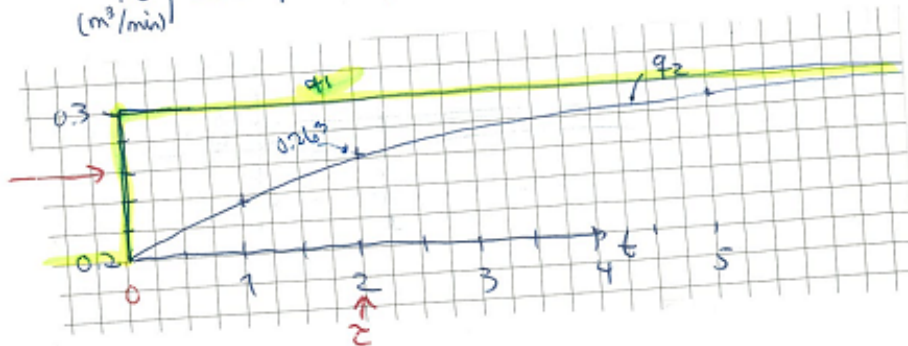
$$\Rightarrow \ln \frac{0.3 - q_2}{0.1} = -kt \Rightarrow \underline{q_2 = 0.3 - 0.1 e^{-kt}}$$

ders tidskonstanten

$$\underline{C = 1/k = 1/0.5 = 2 \text{ min}}$$

t (min)	0	1	2	5	∞
q_2 (m ³ /min)	0.2	0.239	0.263	0.292	0.3

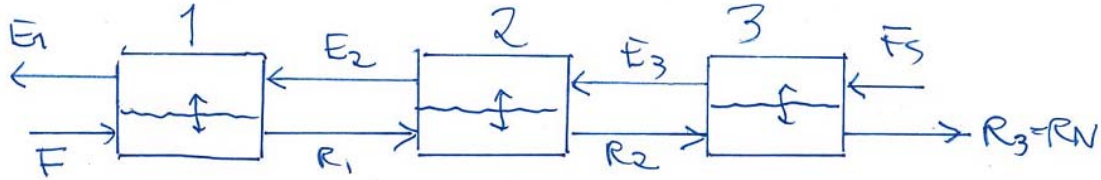
63% af
endelig
ændring efter
 $t = \tau$.



Problem 4 – Extraction (15%)

We have a given feed F (250 kg/min) with components A and C, and we want to reduce the amount of A using countercurrent extraction with pure component S. The desired products are R_N (main product) og E_1 (side product), see the figure.

(a) Make a flowsheet of the process for the case with three stages ($N=3$).



(b) From the liquid-liquid equilibrium diagram we read the compositions in red in the table.

Mass balances then give the flows.

$$\begin{aligned} \text{Total:} \quad & F + F_S = R_N + E_1 \\ \text{A:} \quad & 0.26 \cdot 250 = 0.025 R_N + 0.37 E_1 \\ \text{C:} \quad & 0.74 \cdot 250 = 0.90 R_N + 0.07 E_1 \end{aligned}$$

From the last two balances: $E_1 = 162.6 \text{ kg}$, $R_N = 192.9 \text{ kg}$

From total mass balance: $F_S = 105.5 \text{ kg}$

Component/Stream	F (feed)	F_S (feed)	R_N (product)	E_1 (product)
Total (kg/min)	250	105.5 kg	192.9 kg	162.6 kg
Weight fraction A	0.26	0	0.025	0.37
Weight fraction C	0.74	0	0.90	0.07
Weight fraction S	0	1	0.075	0.56

Note: Can alternatively use point M and level arm rule (which is the graphical mass balance). This is probably more accurate in this case since we use the diagram more directly.

Lever arm rule for F_S -M-R:

$$F_S / F = F-M / F_S-M = 3.05 \text{ cm} / 7.35 \text{ cm} = 0.415 \rightarrow F_S = 0.415 \cdot 250 = 103.7 \text{ kg}$$

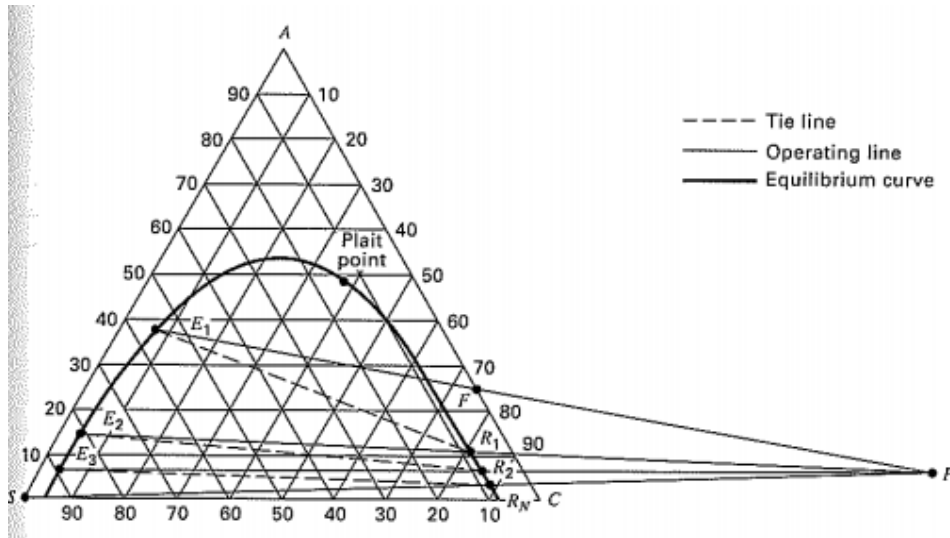
Lever arm rule for E_1 -M- R_N :

$$E_1 / R_N = R_N-M / E_1-M = 3.75 \text{ cm} / 4.55 \text{ cm} = 0.824$$

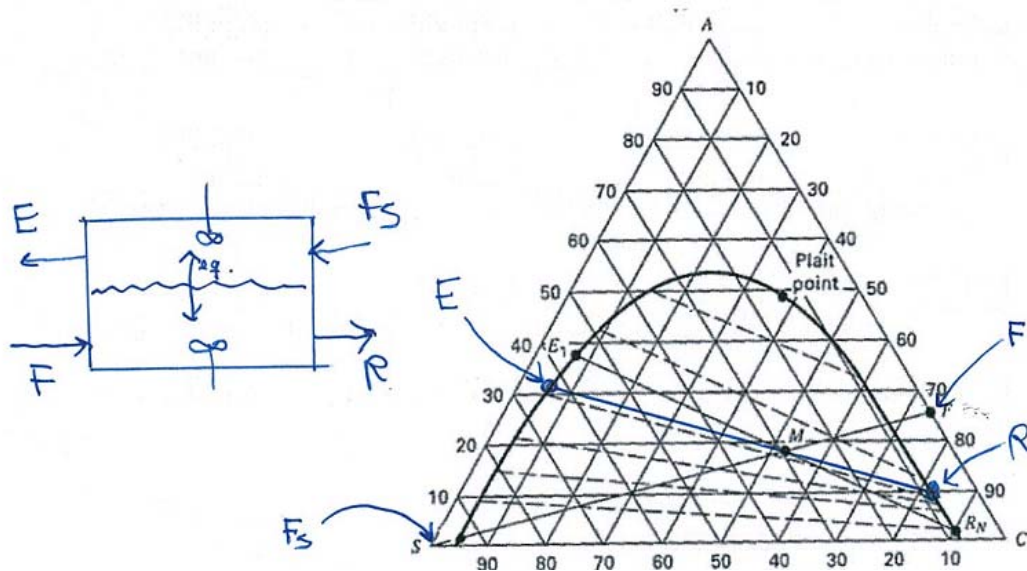
And since $E_1 + R_N = M = 353.7 \text{ kg}$, we get, $E_1 = 159.8 \text{ kg}$ and $R_N = 193.9 \text{ kg}$.

(c) Determine graphically the number of stages (N) required to get the desired products R_N og E_1 .

The operating point (P in the figure below) is found as the crossing between the E1-F and FS-RN lines. The number of stages is then found by stepping between the equilibrium (on stages) and operating (between stages) points. We start from F and move to RN. Need about 2.8 stages (see below)



(d) One-stage extraction (N=1)



From the liquid-liquid diagram we read the compositions of the products E and R (given in red in the table below).

Mass balances then give:

$$\text{Total: } 250 + 105.5 = R + E$$

$$\text{A: } 0.26 \cdot 250 = 0.1 R + 0.32 E$$

Get from these two: $E = 133.9 \text{ kg}$, $R = 221.6 \text{ kg}$

Component/Stream	F (feed)	F _S (feed)	R (product)	E (product)
Total (kg/min)	250	105.5 kg	221.6 kg	133.9 kg
Weight fraction A	0.26	0	0.1	0.32
Weight fraction C	0.74	0	0.82	0.05
Weight fraction S	0	1	0.08	0.63

Check using C-balance: In = 0.74*250 = 185 kg

Out = 0.82*221.6 + 0.05*133.9 = 188.4 kg (hm...some error)

Note: Could alternatively use point M and level arm rule.

Lever arm rule for E-M-R:

$E/R = R-M/E-M = 3.05\text{ cm}/4.85\text{ cm} = 0.629$

And since $E+R = M = 353.7\text{ kg}$, we get, $E = 136.6\text{ kg}$ and $R = 217.1\text{ kg}$.