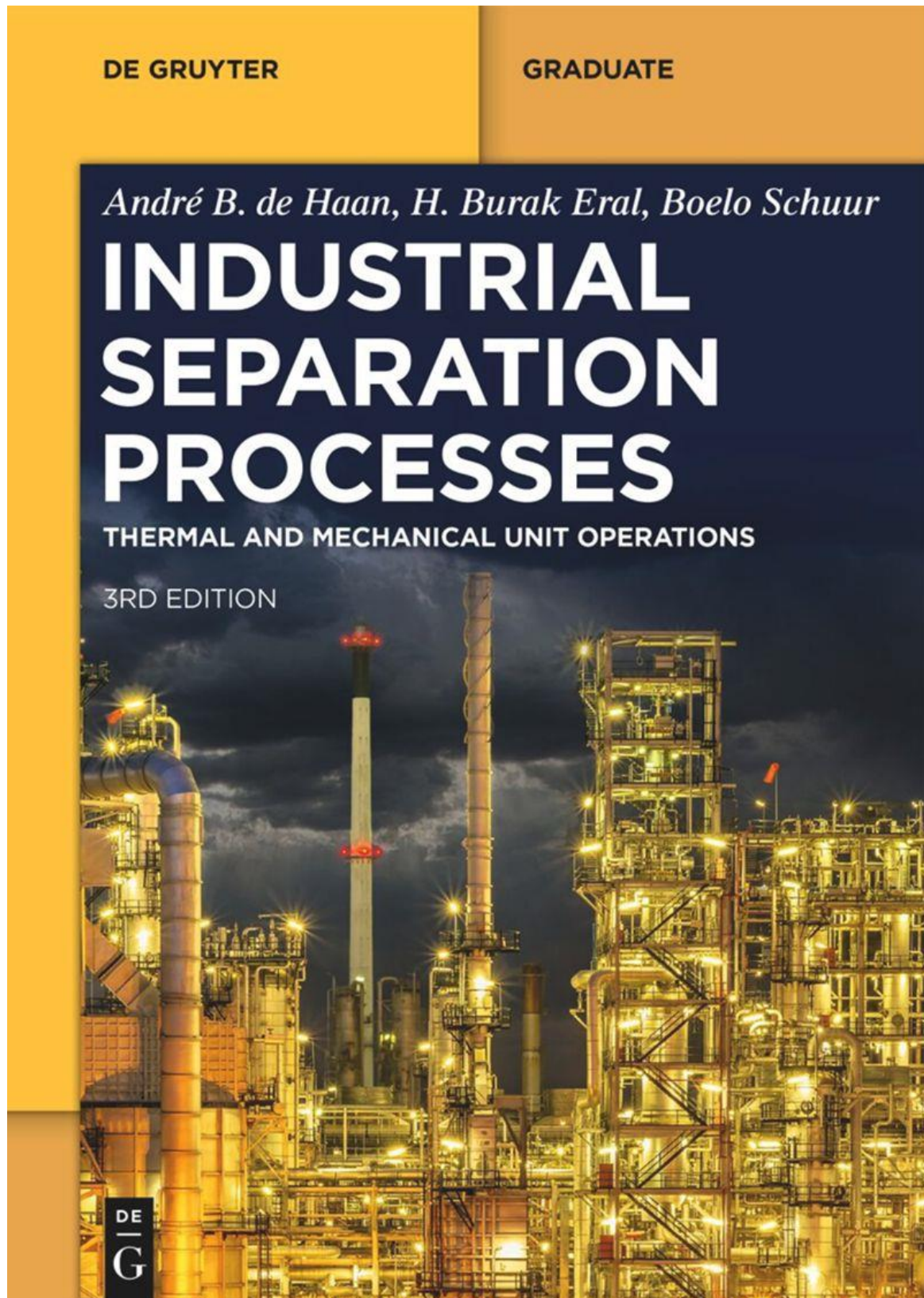


SOLUTIONS TO EXERCISES

Belonging to:



Chapter 1: INTRODUCTION

Exercise 1.1

Compare and discuss the advantages and disadvantages of making separations using an energy-separating agent (ESA) versus using a mass-separating agent (MSA).

Exercise 1.2

The system benzene-toluene adheres closely to Raoult's law. The vapor pressures of benzene and toluene at 121°C are 300 and 133 kPa.

Calculate the relative volatility.

Exercise 1.3

As a part of the life system support for spacecrafts it is necessary to provide a means of continuously removing carbon dioxide from air. If it is not possible to rely upon gravity in any way to devise a CO₂-air separation process.

Suggest at least two separation schemes, which could be suitable for continuous CO₂ removal from air under zero gravity conditions.

Exercise 1.4

Gold is present in seawater to a concentration level between 10⁻¹² and 10⁻⁸ weight fraction, depending upon the location.

Briefly evaluate the potential for recovering gold economically from seawater.

Exercise 1.5

Assuming that the membrane characteristics are not changed as the upstream pressure increases, will the product-water purity in a reverse-osmosis seawater desalination process increase, remain constant or decrease?

Exercise 1.6

Propylene and propane are among the light hydrocarbons produced by thermal and catalytic cracking of heavy petroleum fractions. Although propylene and propane have close boiling points, they are traditionally separated by distillation. Because distillation requires a large numbers of stages and considerable reflux and boilup flow rates compared to the feed flow, considerable attention has been given to the possible replacement of distillation with a more economical and less energy-intensive option. Based on the properties of both species, propose some alternative properties that can be exploited to enhance the selectivity of propylene and propane separation. What separation processes are based on these alternative properties?

<i>Property</i>	<i>Propylene</i>	<i>Propane</i>
Molecular weight (kg/mol)	0,04208	0,04410
VdWaals volume (m ³ /mol)	0,0341	0,0376
VdWaals area (m ² /mol)	5,06	5,59
Acentric factor	0,142	0,152
Dipole moment (Debye)	0,4	0,0
Radius of gyration (m·10 ¹⁰)	2,25	2,43
Melting point (K)	87,9	85,5
Boiling point (K)	225,4	231,1
Critical temperature (K)	364,8	369,8
Critical pressure (MPa)	4,61	4,25

SOLUTIONS

Exercise 1.1

Compare and discuss the advantages and disadvantages of making separations using an energy-separating agent (ESA) versus using a mass-separating agent (MSA).

Answer

	<i>Advantages</i>	<i>Disadvantages</i>
<i>ESA</i>	Known technology Phase separation Relatively simple	Energy consumption may be expensive Heat integration required
<i>MSA</i>	High selectivities possible	Recovery of agent requires additional separation

Exercise 1.2

Calculate the relative volatility of benzene-toluene.

Answer

For an ideal system such as benzene-toluene, by definition (Eqs. 1.3 and 1.5)

$$SF \equiv \alpha \equiv \frac{y_B / x_B}{(1 - y_B) / (1 - x_B)} = \frac{P_B^o}{P_T^o} = \frac{300}{133} = 2.26$$

Exercise 1.3

Suggest at least two separation schemes that could be suitable for continuous CO₂ removal from air under zero gravity conditions.

Answer

- Adsorption of CO₂ on activated carbon. Regeneration by exposing to vacuum outside the spacecraft.
- Absorption of CO₂ in a suitable solvent at the low temperature outside the vessel. Regeneration of the loaded solvent at room temperature, releasing the overpressure via a valve to the outer space.

Exercise 1.4

Briefly evaluate the potential for recovering gold economically from seawater.

Answer

The lower the concentration of the desired substance, the more expensive the required technology. At the given extremely low concentrations, huge amounts of seawater have to be treated, either by evaporation or by (membrane)filtration. Either method is not feasible due to the high-energy costs or pumping costs.

The thermodynamical basis is given by the change in chemical potential upon mixing, which is proportional to $\ln(\text{activity})$ a negative value, indicating a spontaneous process. Separation of the mixture, on the other hand, requires at least that amount of work, which is extremely large at very low values of the activity (or concentration).

Exercise 1.5

Assuming that the membrane characteristics are not changed, as the upstream pressure increases, will the product-water purity in a reverse-osmosis seawater desalination process increase, remain constant or decrease?

Answer

The water flux is proportional to $\Delta P_{\text{ext}} - \Delta \Pi_{\text{osm}}$ and will increase with increasing ΔP_{ext} . The salt flux is proportional to $c_{\text{feed}} - c_{\text{permeate}}$ and will not be effected by a change in ΔP_{ext} . Hence, the product purity will increase with increasing ΔP_{ext} .

Exercise 1.6

Based on the properties of propylene and propane, propose some alternative properties that can be exploited to enhance the selectivity of propylene and propane separation. What processes are based on these alternative properties?

<i>Property</i>	<i>Propylene</i>	<i>Propane</i>
Molecular weight (kg/mol)	0,04208	0,04410
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Critical pressure (Mpa)	4,61	4,25

Answer

Difference in molecular weight

Ultracentrifuge

Difference in VdWaals volume

Kinetic separation by preferential adsorption of the component with the smallest volume in a molecular sieve adsorbent

Difference in dipole moment

Absorption, adsorption, extraction or extractive distillation

Chapter 2: EVAPORATION & DISTILLATION

Exercise 2.1

In *Vapor-Liquid Equilibrium Data Collection* the following form of Antoine-equation is used

$$\log P_i^0 = A' - \frac{B'}{T + C'}$$

with P_i^0 saturation pressure in mmHg and temperature T in $^{\circ}\text{C}$ (760 mmHg = 1 atm).

For benzene in benzene-toluene mixtures the following values are reported:

$$A' = 6.87987, B' = 1196.760 \text{ and } C' = 219.161.$$

Calculate the constants A , B and C in the Antoine-equation as defined in Eq. 2.9.

$$\ln P_i^0 = A - \frac{B}{T + C} \quad \text{with pressure units in atm and temperature in } ^{\circ}\text{C}.$$

Exercise 2.2

With temperature in degrees Fahrenheit and pressure in pounds per sq. inch, the following Antoine constants apply for benzene

$$A' = 5.1606, B' = 2154.2, C' = 362.49 \text{ (psi, } ^{\circ}\text{F, } ^{10}\log P \text{ expression)}.$$

Noting that 1 atm = 14.696 psi = 1.01325 bar and $T(^{\circ}\text{F}) = T(^{\circ}\text{C}) \cdot 9/5 + 32$, calculate the saturation pressure of benzene in bar at 80.1°C .

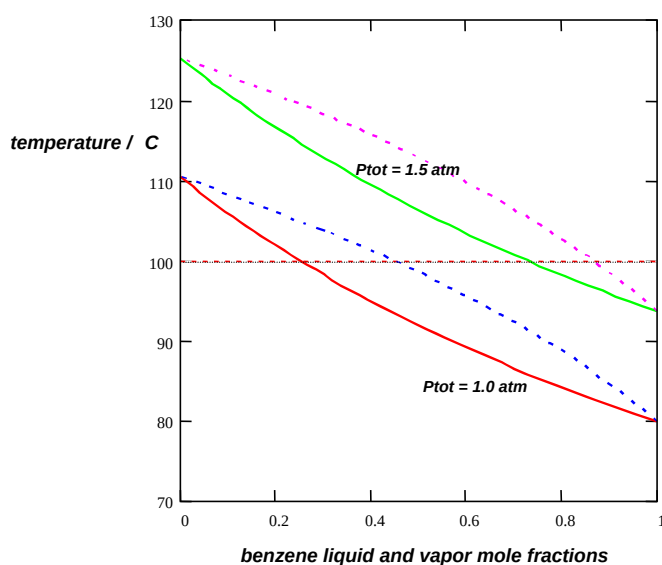
Exercise 2.3

Vapor-liquid equilibrium data for benzene-toluene are given at 1.0 atm and at 1.5 atm in the T-x diagram below. Expressing the saturated vapor pressure in atm as a function of temperature in K, the Antoine constants for benzene and toluene are, respectively

$$A = 9.2082, B = 2755.64, C = -54.00 \text{ and}$$

$$A = 9.3716, B = 3090.78, C = -53.97 \text{ (atm, K, Eq. 2.9)}.$$

Check the phase compositions at 100°C and total pressures of 1.0 and 1.5 atm.



Exercise 2.4

A liquid benzene-toluene mixture with $z = 0.40$ should produce a liquid with $x = 0.35$ in a flash drum at 1.0 atm.

- Calculate the required feed temperature
- Calculate the equilibrium vapor-liquid ratio in the flash drum.

Exercise 2.5

We wish to flash distill isothermally a mixture containing 45 mole% of benzene and 55 mole% of toluene. Feed rate to the still is 700 moles/h. Equilibrium data for the benzene-toluene system can be approximated with a constant relative volatility of 2.5, where benzene is the more volatile component. Operation of the still is at 1 atm.

- Plot the y - x diagram for benzene-toluene.
- If 60% of the feed is evaporated, find the liquid and vapor compositions
- If we desire a vapor composition of 60 mole%, what is the corresponding liquid composition and what are the liquid and vapor flow rates?
- Find the compositions and flow rates of all unknown streams for a two stage flash cascade where 40% of the feed is flashed in the first stage and the liquid product is sent to a second flash chamber where 30% is flashed.

Exercise 2.6

Distillation is used to separate pentane from hexane. The feed amounts 100 mol/s and has a mole ratio pentane/hexane = 0.5. The bottom and top products have the compositions $x_B = 0.05$ and $x_D = 0.98$. The reflux ratio is 2.25. The column pressure is 1 bar. The feed, at the bubbling point, enters the column exactly on the feed tray. The tray temperature is equal to the feed temperature. The pentane vapor pressure is given by:

$$P_5^o = e^{11 \cdot (1 - \frac{310}{T(K)})} \text{ (bar)}$$

The vapor pressure of hexane is 1/3 of the pentane vapor pressure over the whole temperature range. The average density of liquid pentane and hexane amounts to 8170 and 7280 mol/m³, resp. The heat of vaporization amounts to 30 kJ/mol. The distance between the trays amounts to 0.50 m.

- Calculate the feed temperature
- Calculate the vapor stream from the reboiler
- Calculate the required energy in the reboiler
- Construct the y - x diagram
- Construct the operating lines and locate the feed line
- Determine the number of equilibrium stages
- Determine the height of the column

Exercise 2.7

Methanol (M) is to be separated from water (W) by atmospheric distillation. The feed contains 14.46 kg/h methanol and 10.44 kg/hr water. The distillate is 99 mol% pure, the bottom product contains 5 mol% of methanol. The feed is subcooled such that $q = 1.12$.

- Determine the minimum number of stages and minimum reflux
- Determine the feed stage location and number of theoretical stages required for a reflux ratio of 1.

Vapor-liquid equilibrium data (1.0 atm, mole fraction methanol)

x	0.0321	0.0523	0.075	0.154	0.225	0.349	0.813	0.918
y	0.1900	0.2940	0.352	0.516	0.593	0.703	0.918	0.963

Exercise 2.8

A feed to a distillation unit consists of 50 mol% benzene in toluene. It is introduced to the column at its bubble point to the optimal plate. The column is to produce a distillate containing 95 mol% benzene and a bottoms of 95 mol% toluene. For an operating pressure of 1 atm, calculate:

- the minimum reflux ratio
- the minimum number of equilibrium stages to carry out the desired separation
- the number of actual stages needed, using a reflux ratio (L'/D) of 50% more than the minimum,
- the product and residue stream in kilograms per hour if the feed is 907.3 kg/h
- the saturated steam required in kilograms per hour for heat to the reboiler using enthalpy data below

Steam: $\Delta H_{VAP} = 2000$ kJ/kg

Benzene: $\Delta H_{VAP} = 380$ kJ/kg

Toluene: $\Delta H_{VAP} = 400$ kJ/kg

Vapor-liquid equilibrium data (1 atm, mole fraction benzene)

x	0.10	0.20	0.30	0.40	0.50	0.60	0.70	0.80	0.90
y	0.21	0.37	0.51	0.64	0.72	0.79	0.86	0.91	0.96

SOLUTIONS

Exercise 2.1

Given Antoine-equation in the form: $\log P_i^0 = A' - \frac{B'}{T + C'}$ with P_i^0 saturation pressure in mmHg and T temperature in °C. (760 mmHg = 1 atm)
For benzene $A' = 6.87987$ [-], $B' = 1196.760$ [-] and $C' = 219.161$ [°C].

Find The constants A , B and C in the Antoine-equation as defined in Eq. 2.9.

Thoughts Compare both forms of the Antoine-equation side by side:

$$\log P_{benzene}^0 = A' - \frac{B'}{T + C'} \quad (I) \quad \text{and} \quad \ln P_{benzene}^0 = A - \frac{B}{T + C} \quad (II),$$

convert the pressure in (I) into form (II) and compare the three resulting constants with A , B and C , respectively

$$\text{Solution} \quad \ln P_B^0 (\text{atm}) = \ln \frac{10^{\frac{A' - \frac{B'}{T + C'}}{760 (\text{mmHg/atm})}} = A' \ln(10) - \ln(760) - \frac{B' \ln(10)}{T + C'}$$

Hence

$$A = A' \ln(10) - \ln(760) = \ln(10) [A' - \log(760)] \quad A = 9.2082 \quad [-]$$

$$B = B' \ln(10) \quad B = 2755.6 \quad [-]$$

$$C = C' \quad C = 219.16 \quad [^\circ\text{C}]$$

Given Antoine constants benzene ($^{10}\log P$ expression; units in psi and °F)

$$A' = 5.1606 \quad [-], \quad B' = 2154.2 \quad [-], \quad C' = 362.49 \quad [^\circ\text{F}]$$

Find Saturation pressure of benzene in bar at 80.1°C.

Thoughts Two different approaches:

- convert temperature to °F, calculate saturation pressure at this temperature in psi and convert result to bar, or
- recalculate Antoine constants in the required units as shown in Exercise 2.1.

Solution Method a:

$$\text{Temperature conversion:} \quad 80.1^\circ\text{C} = 176.18^\circ\text{F} \quad \left(^\circ\text{C} \cdot \frac{9}{5} + 32\right)$$

$$\text{Saturation pressure} \quad P_{benzene}^0(176.18) = 10^{\frac{A' - \frac{B'}{176.18 + C'}}{1}} = 14.696 \text{ psi}$$

$$\text{Pressure conversion} \quad 14.696 \text{ psi} = \frac{14.696}{14.696} \text{ atm} = 1.01325 \text{ bar}$$

Method b:

$$P_{benzene}^0 [\text{bar}] = 1.01325 [\text{bar/atm}] \cdot \frac{10^{\frac{A' - \frac{B'}{T(^{\circ}\text{F}) + C'}}{1}} [\text{psi}]}{14.696 [\text{psi/atm}]} = \exp\left(A - \frac{B}{T(^{\circ}\text{C}) + C}\right) \quad \text{with}$$

$$T(^{\circ}\text{F}) = T(^{\circ}\text{C}) \cdot \frac{5}{9} + 32$$

$$\text{hence } A = A' \cdot \ln(10) + \ln \frac{1.01325}{14.696} \quad A = 9.2083 [-]$$

$$B = \frac{5}{9} \cdot B' \cdot \ln(10) \quad B = 2755.64 [-]$$

$$C = \frac{5}{9} \cdot (C' + 32) \quad C = 219.16 [^{\circ}\text{C}]$$

$$\text{Saturation pressure of benzene at } 80.1^{\circ}\text{C} = \exp\left(A - \frac{B}{80.1 + C}\right) = 1.01325 \text{ bar}^1$$

Exercise 2.3

Given Antoine constants benzene (B) 9.2082, 2755.64, -54.0
 Antoine constants toluene (T) 9.3716, 3090.78, -53.97 (atm., K, Eq. 2.9)

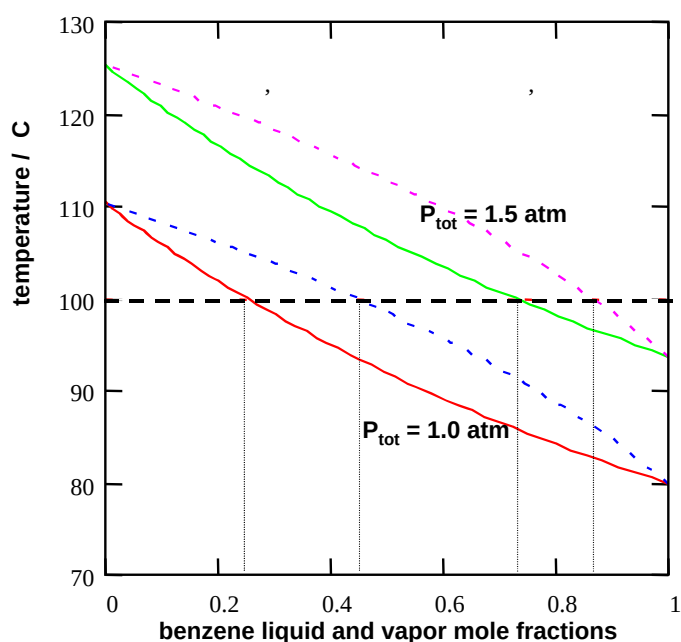
Find The phase compositions at 100°C and total pressure of 1.0 and 1.5 atm

Thoughts The mole fraction in a binary mixture at a given temperature and total pressure can be calculated from the distribution coefficients of both components, see Eq. 2.18. The distribution coefficient of a component follows from its saturation pressure and the total pressure, Eq. 1.5. The saturation pressure at a certain temperature is given through the Antoine equation, Eq. 2.9.

Alternatively, for a given value of P_{tot} and calculated saturation pressures, solve both Eqs. 2.6 for x and y :

$$P_{\text{tot}} \cdot y = P_B^0 \cdot x \text{ and } P_{\text{tot}} \cdot (1 - y) = P_T^0 \cdot (1 - x)$$

Solution



¹ Check boiling point of benzene in T - x diagram in exercise 2.3

First approach

$$P_B^0(T) = \exp\left(A_B - \frac{B_B}{T + C_B}\right) \quad P_B^0(100 + 273.16) = 1.776 \text{ atm}$$

$$P_T^0(T) = \exp\left(A_T - \frac{B_T}{T + C_T}\right) \quad P_T^0(100 + 273.16) = 0.732 \text{ atm}$$

$$P_{tot} = 1.0 \text{ atm}$$

$$K_B(T) = \frac{1.776}{1.0} = 1.776 \quad K_T(T) = \frac{0.732}{1.0} = 0.732 \quad \alpha = \frac{K_B(T)}{K_T(T)} = 2.425$$

$$x = \frac{1 - K_T(T)}{K_B(T) - K_T(T)} = 0.257 \quad y = \frac{\alpha x}{1 + (\alpha - 1)x} = 0.456$$

$$P_{tot} = 1.5 \text{ atm}$$

$$K_B(T) = \frac{1.776}{1.5} = 1.184 \quad K_T(T) = \frac{0.732}{1.5} = 0.488$$

$$x = \frac{1 - K_T(T)}{K_B(T) - K_T(T)} = 0.736 \quad y = \frac{\alpha x}{1 + (\alpha - 1)x} = 0.871$$

Second approach

$$P_{tot} = 1.0 \text{ atm} \quad P_{tot} \cdot y = P_{ben}^0 \cdot x \quad \text{and} \quad P_{tot} \cdot (1 - y) = P_{tol}^0 \cdot (1 - x)$$

$$x = 0.257 \quad \text{and} \quad y = 0.456$$

$$P_{tot} = 1.5 \text{ atm} \quad P_{tot} \cdot y = P_{ben}^0 \cdot x \quad \text{and} \quad P_{tot} \cdot (1 - y) = P_{tol}^0 \cdot (1 - x)$$

$$x = 0.736 \quad \text{and} \quad y = 0.871$$

Exercise 2.4

Given mole fraction benzene in feed $z = 0.40$, mole fraction in liquid product $x_{liq} = 0.35$

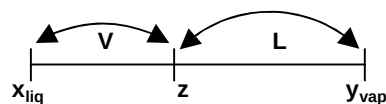
Find a. boiling point of benzene – toluene feed mixture at 1.0 atm
b. vapor – liquid ratio in the flash drum at 1.0 atm

Thoughts At a given P_{tot} and x_{liq} , all other parameters, such as temperature and y_{vap} , are fixed. Summation of the partial pressures of benzene and toluene (Eq. 2.6) gives the total pressure:

$$P_{tot} = x \cdot P_{ben}^0(T) + (1 - x) \cdot P_{tol}^0(T)$$

Solve this equation for T , calculating the saturation pressures as shown in the previous exercise. Use the Antoine constants given in Exercise 2.3.

The vapor – liquid ratio follows either from applying the lever rule (see diagram) to the values of x_{liq} , y_{vap} and z , or via Eq. 2.18 with $q = L/F = 1/(1 + V/L)$.



MathCad work sheet

$$P_{\text{tot}} := 1.0 \text{ atm} \quad x_L := 0.35 \quad z := 0.4$$

$$\text{Ant_Ben} := \begin{bmatrix} 9.2082 \\ 2755.64 \\ -54.00 \end{bmatrix} \quad \text{Ant_Tol} := \begin{bmatrix} 9.3716 \\ 3090.78 \\ -53.97 \end{bmatrix}$$

+

a) boiling point at 1 atm

$$\text{given } P_{\text{tot}} = x_L \cdot \exp \left\{ \text{Ant_Ben}_0 - \frac{\text{Ant_Ben}_1}{T + \text{Ant_Ben}_2} \right\} + (1 - x_L) \cdot \exp \left\{ \text{Ant_Tol}_0 - \frac{\text{Ant_Tol}_1}{T + \text{Ant_Tol}_2} \right\}$$

$$T_b := \text{find}(T) \quad T_b = 369.9 \text{ K} \quad T_b - 273.16 = 96.8 \text{ } ^\circ\text{C}$$

b) vapor/liquid ratio at boiling point

$$K_{\text{Ben}}(T) := \frac{\exp \left\{ \text{Ant_Ben}_0 - \frac{\text{Ant_Ben}_1}{T + \text{Ant_Ben}_2} \right\}}{P_{\text{tot}}} \quad K_{\text{Ben}}(T_b) = 1.625 \quad y_V := K_{\text{Ben}}(T_b) \cdot x_L$$

$$y_V = 0.569$$

$$\frac{V}{L} = \frac{z - x_L}{y_V - z} = 0.296$$

Alternatively, using Eq. 2.18a and

$$K_{\text{Tol}}(T) := \frac{\exp \left\{ \text{Ant_Tol}_0 - \frac{\text{Ant_Tol}_1}{T + \text{Ant_Tol}_2} \right\}}{P_{\text{tot}}} \quad K_{\text{Tol}}(T_b) = 0.663$$

$$q(T) := -z \cdot \frac{K_{\text{Tol}}(T)}{1 - K_{\text{Tol}}(T)} - (1 - z) \cdot \frac{K_{\text{Ben}}(T)}{1 - K_{\text{Ben}}(T)} \quad q(T_b) = 0.772 \quad \frac{V}{L} = \frac{1}{q(T_b)} - 1 = 0.296$$

Exercise 2.5*Given* $F = 700 \text{ mol/s}$, $x_F = 0.45^{(2)}$, isothermal flash

- Find*
- the y - x diagram for benzene - toluene
 - liquid and vapor composition at 60% evaporation
 - liquid and vapor flow rates at a vapor composition of 60%
 - compositions and flow rates for a two-stage flash cascade where 40% is flashed in the first stage and 30% of the remaining liquid in the second stage

Thoughts

- Eq. 2.7 relates equilibrium vapor composition y and liquid composition x for any α
- $V/F = 0.6$, hence $q = 0.4$ (Eq. 2.13). The operating line (Eq. 2.14) relates y and x between two subsequent stages for any q , while the equilibrium line still holds. This defines a set of

two equations with two unknowns, y and x . Note that

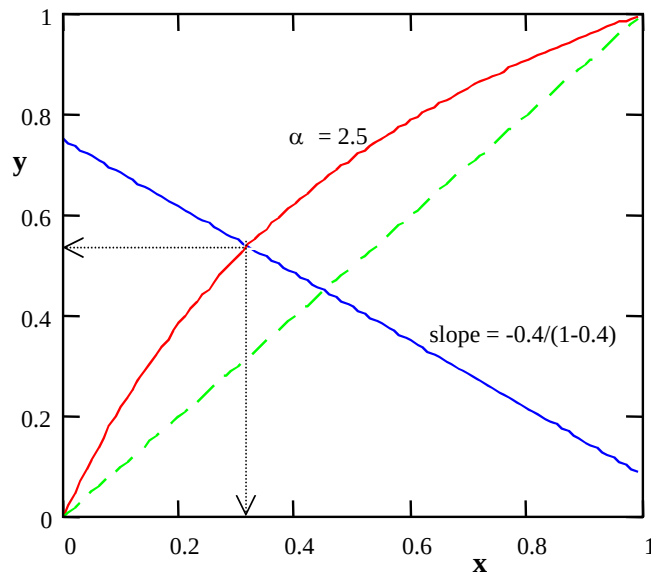
$$y = \frac{\text{mol A in gas}}{\text{mol gas}} = \frac{\text{total mol A} - \text{mol A in liquid}}{\text{mol gas}} = \frac{x_F F - q x F}{(1 - q) F} = \frac{x_F - q x}{1 - q}$$

- the same idea as before, here with $y = 0.6$
- draw a process scheme and apply mass balances and equilibrium equations

Solution

- Apply Eq. 2.7b for $\alpha = 2.5$, see diagram on next page.

⁽²⁾ Note that either z or x_F is used to represent the mole fraction of the more volatile component in the feed

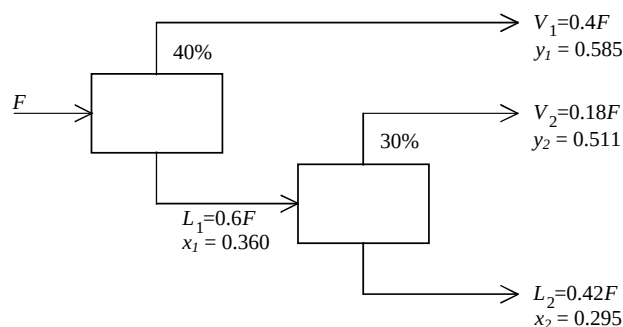


- b. The compositions are found at the intersection of the operating and equilibrium line. This point can be found either graphically (draw in the x-y diagram the operating line with slope $-q/(1-q)$ and read the required values, see diagram) or solve the equation: $(y =) \frac{x_F - q \cdot x}{1-q} = \frac{\alpha x}{1 + (\alpha - 1) \cdot x}$ for x with $q = 0.4$.

In either case follows $x = 0.318$ and $y = 0.538$

- c. Again, two equations determine the system: the operating line and the equilibrium line. Solve for the two unknowns, x and q, or eliminate, e.g., x and solve the remaining equation for q: $(x =) \frac{x_F - (1-q) \cdot y}{q} = \frac{y}{\alpha - y \cdot (\alpha - 1)}$.

With $y = 0.6$ follows $q = 0.667$, $L = q \cdot F = 466.7$ mol/h, $V = F - L = 233.3$ mol/h and, via either equation, $x = 0.375$.



- d. The following scheme applies, including the over-all mass balances:
Repeat the procedure in (b) (given the feed composition and fraction flashed) for the first stage. With $q_1 = 0.6$ and $z = 0.45$ it follows $x_1 = 0.360$ and $y_1 = 0.585$.
The second stage has also a given feed composition $x_1 = 0.360$ and a known fraction flashed ($q_2 = 0.700$).
Following the same procedure, it follows that $x_2 = 0.295$ and $y_2 = 0.511$.
Check: $x_F = 0.295 \cdot (1 - 0.4) \cdot (1 - 0.3) + 0.511 \cdot (1 - 0.4) \cdot 0.3 + 0.585 \cdot 0.4 = 0.45$

Exercise 2.6

Given	F	$=$	100 mol/s	x_D	$=$	0.98
	F_P/F_H	$=$	0.5	x_B	$=$	0.05
	ρ_P	$=$	8170 mol/m ³	P_{tot}	$=$	1 bar
	ρ_H	$=$	7280 mol/m ³	R	$=$	2.25
	ΔH_{vap}	$=$	30 kJ/mol	H	$=$	0.50 m

The feed enters exactly on the feed tray at its boiling point.

The pentane vapor pressure is given by: $P_P^o = e^{11 \left(1 - \frac{310}{T(K)} \right)}$ (bar)

The vapor pressure of hexane is 1/3 of the pentane vapor pressure.

- Find**
- the feed temperature
 - the vapor stream from the reboiler
 - the required energy in the reboiler
 - the y - x diagram
 - the operating lines and locate the feed line
 - number of equilibrium stages
 - height of column

Thoughts Raoult's law (Eq. 2.5) relates the partial pressures of both components in the vapor phase to the concentrations in the liquid phase. The feed temperature can now be calculated by the given equation for the pentane vapor pressure. Mass balances define the vapor stream from the reboiler. The last four problems can be solved graphically. Note that $q = 1$.

Solution a. Combination of Raoult's law (Eq. 2.5) and Dalton's law (Eq. 2.4) yields

$$P_{tot} = x_F P_P^o + (1 - x_F) P_H^o \quad \text{with} \quad P_H^o = \frac{1}{3} P_P^o \quad \text{and} \quad x_F = \frac{F_P}{F} = \frac{F_P}{F_P + F_H} = \frac{0.5}{0.5 + 1} = \frac{1}{3}$$

hence

$$P_{tot} = x_F P_P^o + (1 - x_F) P_P^o / 3 \Rightarrow P_{tot} = \frac{1}{3} P_P^o + \left(1 - \frac{1}{3}\right) \frac{P_P^o}{3} \Rightarrow P_{tot} = \frac{5}{9} P_P^o \quad \text{or}$$

$$P_P^o = \frac{9}{5} P_{tot}$$

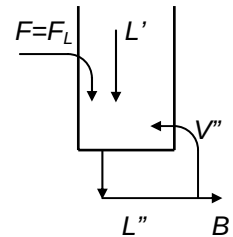
$$\text{Now the temperature is calculated from } \frac{9}{5} P_{tot} = e^{11 \left(1 - \frac{310}{T} \right)} \Rightarrow T = 327.5 \text{ K}$$

b. The mass balances over the reboiler and the feed stage give

$$V'' = L'' - B = q \cdot F + L' - B \quad \text{with} \quad q = 1$$

while, by definition, $L' = R \cdot D$, hence

$$V'' = F + L' - B = F + R \cdot D - B$$



D and B are calculated from the mass balance over the entire column (Eq. 2.19) and the component material for pentane (Eq. 2.20):

$$F = D + B \quad \text{and} \quad x_F F = x_D D + x_B B, \quad \text{respectively}$$

Solving these two equations for the unknowns D and B yields

$$D = 31.2 \text{ mole/s and } B = 68.8 \text{ mole/s, hence } V'' = 101.4 \text{ mole/s.}$$

- c. The total energy to vaporize a total mole flow V'' required is:

$$Q_{reb} = \Delta H_{vap} \cdot V'' = 3042 \text{ kJ/s}$$

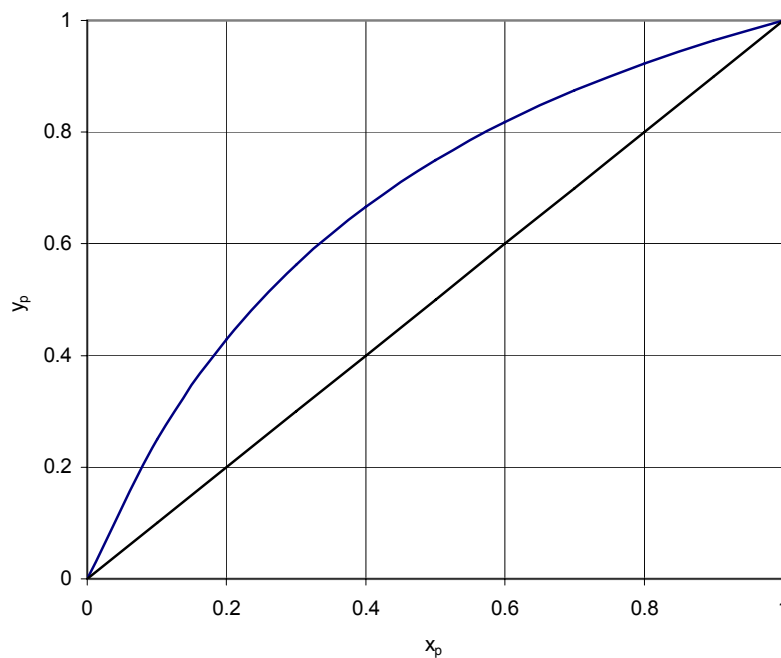
- d. For an ideal binary mixture, with constant relative volatility α , the equilibrium curve is given by Eq. 2.7b:

$$y_P = \frac{\alpha_{PH} x_P}{1 + (\alpha_{PH} - 1)x_P}$$

The relative volatility for this (assumed) ideal mixture equals to (Eq.1.5):

$$\alpha_{PH} = \frac{P_P^o}{P_H^o} = \frac{P_P^o}{\frac{1}{3}P_P^o} = 3$$

The calculated values are plotted in the x-y diagram below:



- e. The operating line for the rectification section is written as (Eq. 2.22):

$$y = \frac{L'}{V'} \cdot x + \frac{D}{V'} \cdot x_D = \frac{L'}{V'} \cdot x + \frac{V' - L'}{V'} \cdot x_D \quad \text{or} \quad y_P - x_D = \frac{L'}{V'}(x - x_D)$$

which represents a straight line through $y = x_D$ and $x = x_D$ with slope L'/V' . Herein the slope L'/V' can be calculated from Eq. 2.23:

$$\frac{L'}{V'} = \frac{R}{R+1} = \frac{2.25}{2.25+1} = 0.692$$

Similarly, the operating line for the stripping section is (Eq. 2.26):

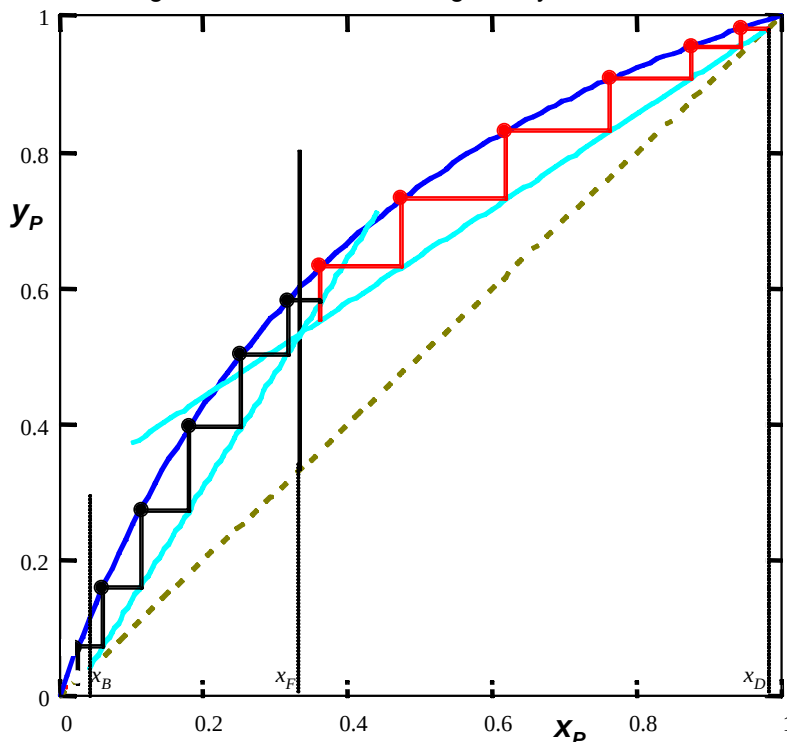
$$y = \frac{L''}{V''} \cdot x_P - \frac{B}{V''} \cdot x_B = \frac{L''}{V''} \cdot x_P - \frac{L'' - V''}{V''} \cdot x_B \quad \text{or} \quad y - x_B = \frac{L''}{V''}(x - x_B)$$

representing a straight line through $y = x_B$ and $x = x_B$ with slope V''/L'' .

$$L'' = V'' + B = 170.2 \text{ mole/s and } V''/L'' = 1.678.$$

Both operating lines are plotted in the figure below. The feed line is fixed by two points: the intersection of the two operating lines and the intersection of the vertical through $x_p = 1/3$ and the diagonal, see page 2-17.

The feed enters the column exactly at bubbling point (no vapor), so $q = 1$. The resulting feed line is vertical and given by $x = 1/3$.



Operating and feed lines in y - x diagram for pentane/hexane.

- f. Start from (x_D, y_D) , the point on the operating line at the upper exit of the column. Find the composition of the liquid leaving the upper tray, which is in equilibrium with V' leaving the column, on the equilibrium line by drawing a horizontal line. The composition of the vapor from the 2nd tray is found on the operating line by drawing a vertical line between the first equilibrium point and the operating line. Continue carefully this staircase construction. Read in the diagram above that 6.7 equilibrium trays are required in the rectifying section. A similar construction in the stripping section, starting from the bottom composition, gives 4.8 equilibrium stages. In total 11.5 equilibrium stages, including the reboiler and the feed tray.

- g. The number of equilibrium stages (minus the condenser) estimates the *minimum* column height $= H_{spacing} \cdot (N_{ts} - 1) = 5.3$ m.

The real height requires estimation of the number of real stages $N_s = N_{ts} / E_o$ (E_o = over-all efficiency, see Chapter 4).

Exercise 2.7

Given $F_M = 14.46 \text{ kg/h}$ $x_D = 0.98$ $q = 1.12$
 $F_W = 10.44 \text{ kg/h}$ $x_B = 0.05$ $P = 1 \text{ bar}$

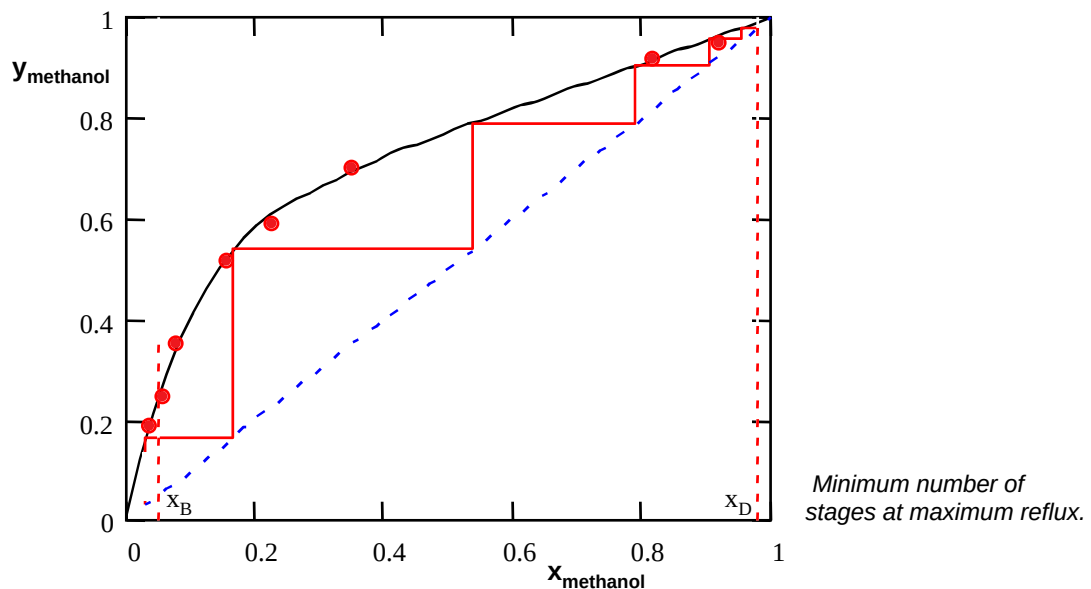
x	0.0321	0.0523	0.075	0.154	0.225	0.349	0.813	0.918
y	0.1900	0.2490	0.352	0.516	0.593	0.703	0.918	0.953

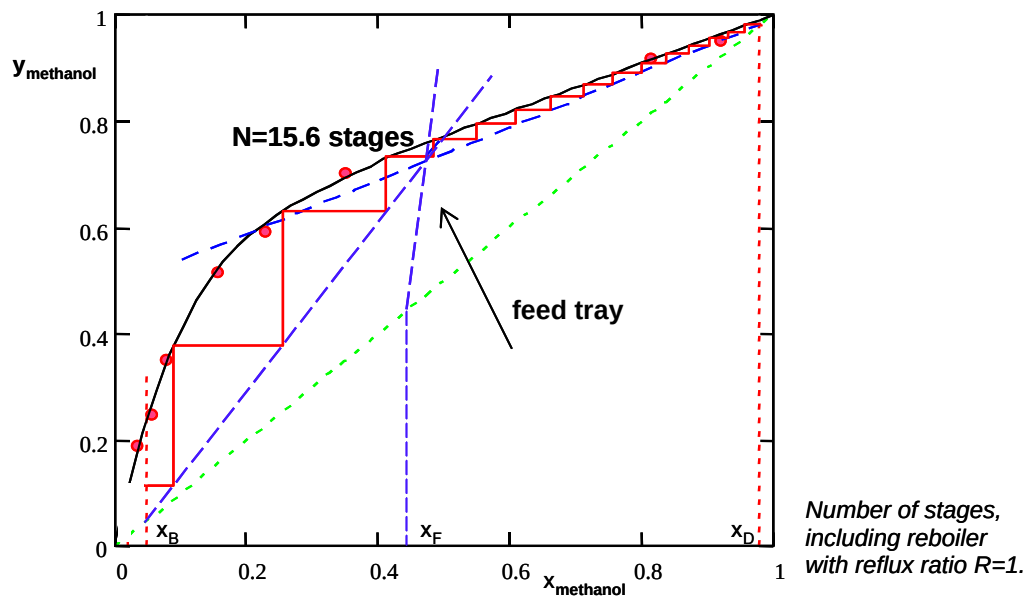
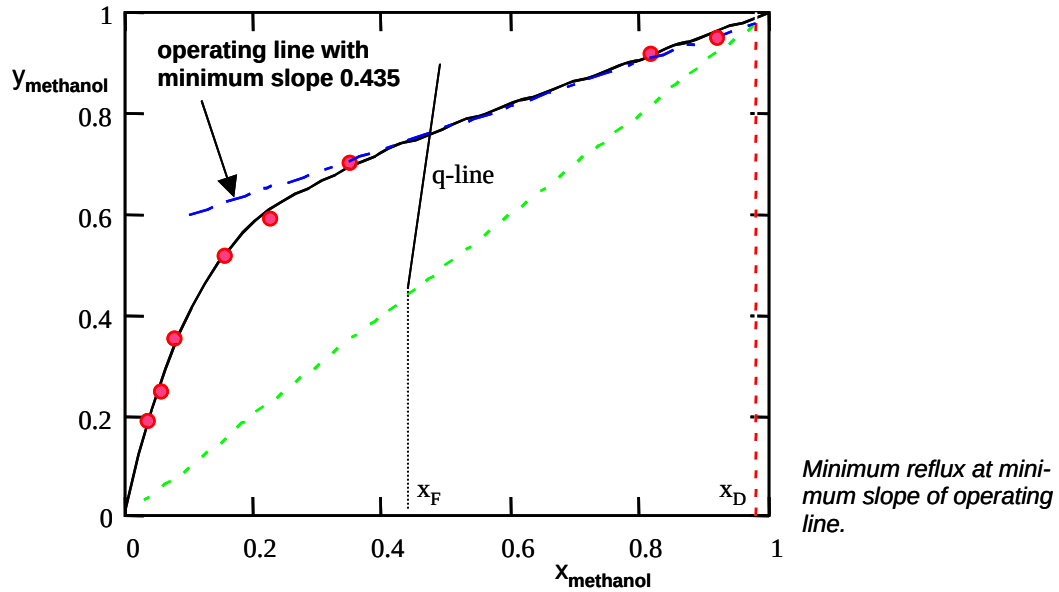
Find a. the minimum number of stages and minimum reflux.
 b. the feed stage location and number of theoretical stages for a reflux ratio $R = 1.0$.

Thoughts The minimum number of stages is obtained at maximum reflux, i.e. $D = 0$ and $B = 0$. Hence the slope of both operating lines equals unity.
 The minimum reflux is obtained at infinite number of stages, i.e. both operating lines intersect the feed line on the equilibrium line.
 Draw the operating line for the rectifying section for $R = 1.0$ according to Eq. 2.23. Calculate V'' and L'' as explained in Exercise 2.6b and draw the operating line for the stripping section.

Solution a. The minimum number of stages is obtained at maximum reflux, slopes of operating lines amount to unity. The stair case is constructed between the diagonal of the y - x diagram and the equilibrium curve. As shown in the first diagram below, the minimum number of stages amounts to 5.8. With $\alpha_{av} = 3.58$ (Eq. 2.48), the Fenske expression (Eq. 2.47) would give $N_{min} = 5.4$. Note that in this case the reboiler does not contribute to the separation because the liquid entering the reboiler is entirely vaporized: its composition equals that of the vapor leaving the reboiler (total reboiler).
 The reflux R is related to the slope of the operating line (L'/V') through Eq. 2.23:

$$R = \frac{L'/V'}{1-L'/V'}$$
 Any operating line goes through (x_D, x_D) , the line with the smallest slope goes also through the intersection of the feed line with the equilibrium line. From the next diagram one reads $(L'/V')_{min} = 0.435$. Hence, according to Eq. 2.38, $R_{min} = 0.77$. The Underwood expression (Eq. 2.54) would give $R_{min} = 0.82$ (however, q should equal unity for Eq. 2.54 to be applicable).





- b. The slope of the operating line in the rectifying sections follows from Eq. 2.23:

$$L'/V' = \frac{R}{R+1} = 0.5$$

The operating line in the stripping section goes through (x_B, x_B) and the intersection of the upper operating line and the feed line, $(0.469, 0.724)$.

Draw the operating line through both points.

Start the staircase construction at (x_D, x_D) between the equilibrium line and the upper operating line downwards.

The equilibrium stage closest to the intersection of the feed line and the equilibrium line constitutes the optimal feed stage. From that point, the staircase lies between the equilibrium and the lower operating line, see diagram.

Alternatively, the compositions of all equilibrium stages in the rectifying section can be calculated from that operating line and the equilibrium line (Eq. 2.22)

$$y_{n+1} = x_D + \frac{L'}{V'} \cdot [x_n(y_n) - x_D]$$

Starting at the top with $y_1 = x_D$, calculate $x_1 = x_1(y_1)$, the composition of the liquid from tray 1 in equilibrium with the vapor leaving that tray (or read x_1 from the equilibrium line in the x-y-diagram if no equilibrium function such as that in Eq. 2.7 is given). Determine the composition of the liquid leaving tray 2, in equilibrium with y_2 and calculate the composition of the vapor coming from tray 3, y_3 , from Eq. 2.22.

Repeat this procedure until the equilibrium composition passes the intersection of the feed and the equilibrium line. After that point, repeat the same procedure using Eq. 2.29, the operating line in the stripping section. This numerical procedure gives accurate results then a drawing.

In either procedure, the number of equilibrium stages found includes the reboiler. Note that in case of total reflux, the *minimum number* of stages does not include the reboiler because all liquid is evaporated entirely (total reboiler), see note in part a.

Exercise 2.8

<i>Given</i>	$F = 907.3 \text{ kg/h}$	$x_D = 0.95$	$\Delta H_{\text{vap, H}_2\text{O}} = 2000 \text{ kJ/kg}$
	$P = 1 \text{ bar}$	$x_B = 0.05$	$\Delta H_{\text{vap, C}_6\text{H}_6} = 380 \text{ kJ/kg}$
	$q = 1.0$	$z = x_F = 0.50$	$\Delta H_{\text{vap, C}_6\text{H}_5\text{CH}_3} = 400 \text{ kJ/kg}$

x	0.10	0.20	0.30	0.40	0.50	0.60	0.70	0.80	0.90
y	0.21	0.37	0.51	0.64	0.72	0.79	0.86	0.96	0.98

- Find*
- the minimum reflux ratio.
 - the minimum number of equilibrium stages.
 - the number of equilibrium stages required at a reflux ratio 1.5 times the minimum.
 - the top and bottom product in kg/h.
 - the energy demand in the reboiler in kg steam/h

Thoughts The minimum reflux is obtained at infinite number of stages, i.e. both operating lines intersect the feed line on the equilibrium line. Apply the Underwood expression (Eq. 2.54) or find graphically.

The minimum number of stages is obtained at maximum reflux, i.e. $D = 0$ and $B = 0$. Hence the slope of both operating lines equals unity. Apply the Fenske equation, Eq. 2.48, or solve in the x-y-diagram.

The compositions are given in mole fractions, so to calculate the product streams in kg/time the appropriate mole balances have to be converted to mass balances. The conversion factor of a particular stream is its molecular weight, which depends on the composition of that stream.

Solution a. Applying the Underwood expression, Eq. 2.54 ($q = 1$)

$$R_{\min} = \frac{\frac{x_D}{x_F} - \alpha_{av} \frac{1-x_D}{1-x_F}}{\alpha_{av} - 1}$$

with an average value of the relative volatility according to Eq. 2.48:

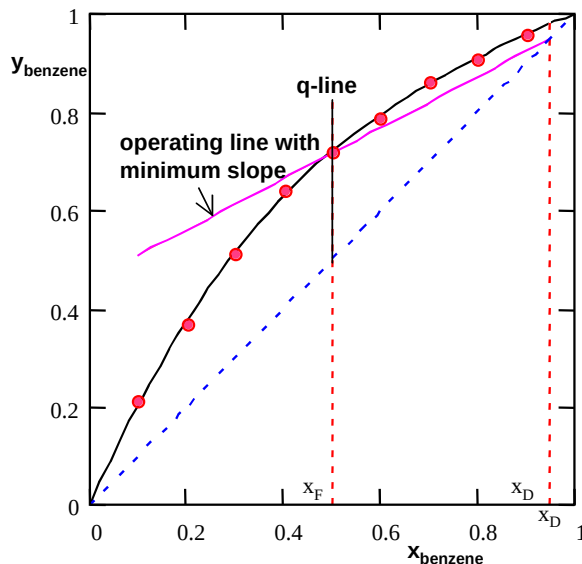
$$\alpha_{av} = \sqrt{\alpha_B \cdot \alpha_D} \approx \sqrt{\frac{y_1}{1-y_1} \cdot \frac{1-x_1}{x_1} \cdot \frac{y_9}{1-y_9} \cdot \frac{1-x_9}{x_9}} = 2.53 \text{ gives } R_{\min} = 1.080.$$

Alternatively, draw the equilibrium line in a x-y-diagram. Eq. 2.7 may be applied,

$$y_{eq}(x) = \frac{\alpha_{av} \cdot x}{1 + (\alpha_{av} - 1) \cdot x}$$

The operating line in the rectifying section goes through (x_D, x_D) and the intersection of the vertical feed line ($q = 1$) and the equilibrium line:

$$y = \frac{\alpha_{av} \cdot x_F}{1 + (\alpha_{av} - 1) \cdot x_F} = 0.716, \quad x = x_F.$$



Hence, the minimum slope of the operating line equals $\frac{x_D - 0.716}{x_D - x_F} = 0.519$.

Follows with Eq. 2.38 $R_{\min} = \frac{0.519}{1 - 0.519} = 1.079$,

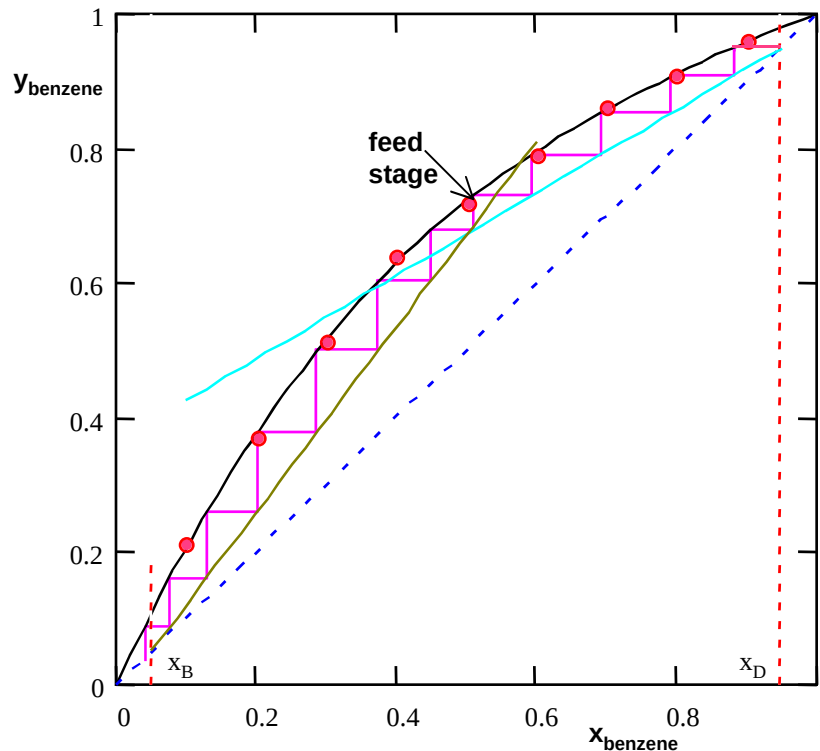
for this rather ideal system, in excellent agreement with the Underwood approach.

b. Applying the Fenske expression, Eq. 2.48:
$$N_{\min} = \frac{\ln\left[\frac{x_D}{1-x_D} \cdot \frac{1-x_B}{x_B}\right]}{\ln \alpha_{av}} = 6.4$$

Like in Exercise 2.7b, an alternative method would be to calculate each subsequent equilibrium y-value y_{n+1} from the operating line, Eq. 2.21, with $L'/V' = 1$: $y_{n+1} = x_n$

with x_n , the equilibrium on the previous stage n , calculated from the reciprocal of the equilibrium expression: $x_n = \frac{y_n}{\alpha_{av} - (\alpha_{av} - 1) \cdot y_n}$ from which $N_{\min} \approx 6.8$, in

good agreement with the result from the Fenske expression, see diagram below.



- c. The slope of the operating line in the rectifying sections follows from Eq. 2.23:

$$L'/V' = \frac{1.5 \cdot R_{\min}}{1.5 \cdot R_{\min} + 1} = 0.618$$

The operating line in the stripping section goes through (x_B, x_B) and the intersection of the upper operating line and the feed line, $x = x_F$. Substitution of $x = x_F$ into the upper operating line gives $y = 0.672$.

Draw the operating line through (x_B, x_B) and $(x_F, 0.672)$. Start the staircase construction at (x_D, x_D) between the equilibrium line and the upper operating line downwards. Beyond the feed stage, apply the stripping operating line, see x-y-diagram.

Similarly to the outline given in exercise 2.7b, the y-compositions of all equilibrium stages in the rectifying section can be calculated by elimination of x_n from the operating line using $\frac{y_n}{\alpha_{av} - (\alpha_{av} - 1) \cdot y_n} = x_n$, the reciprocal form of

Eq. 2.7:

$$y_{n+1} = x_D + \frac{L'}{V'} \cdot \left[\frac{y_n}{\alpha_{av} - (\alpha_{av} - 1) \cdot y_n} - x_D \right]$$

The slope of the operating line in the stripping section is calculated as

$$\frac{L''}{V''} = \frac{0.672 - x_B}{x_F - x_B} = 1.382$$

- d. The following overall mole balances apply (Eqs. 2.19 and 2.20)

$$F = B + D$$

$$x_F \cdot F = x_B \cdot B + x_D \cdot D$$

With all x mole fractions and F , B and D in mol/h. However, the feed Φ_F is given in kg/h. The conversion is reads

$$\Phi_F = M_F \cdot F = [x_F \cdot M_{ben} + (1-x_F) \cdot M_{tol}] \cdot F = 907.3 \text{ kg/h}$$

With $x_F = 0.5$ it follows that $F = 10675 \text{ mol/h}$. Solving Eqs. 2.19 and 2.20 gives

$$B = D = 5337 \text{ mol/h}$$

and

$$\Phi_D = M_{dis} \cdot D = [x_D \cdot M_{ben} + (1-x_D) \cdot M_{tol}] \cdot D = 420.0 \text{ kg/h}$$

$$\Phi_B = \Phi_F - \Phi_D = 487.3 \text{ kg/h}$$

or

$$\Phi_B = M_{bot} \cdot B = [x_B \cdot M_{ben} + (1-x_B) \cdot M_{tol}] \cdot B = 487.3 \text{ kg/h}$$

$$\Phi_D = \Phi_F - \Phi_B = 420.0 \text{ kg/h}$$

- e. The heat required to evaporate liquid bottom product is delivered through condensation of steam.

The amount to be evaporated equals $V'' = 13981 \text{ mol/h}$ (following the procedure explained in exercise 2.6b). The steam consumption required at the given reflux:

$$\Phi_{\text{steam}} = \frac{x_B \cdot V'' \cdot M_{ben} \cdot \Delta H_{\text{vap,ben}} + (1-x_B) \cdot V'' \cdot M_{tol} \cdot \Delta H_{\text{vap,tol}}}{\Delta H_{\text{vap,H}_2\text{O}}} = 254.7 \text{ kg/h}$$

Chapter 2: EVAPORATION & DISTILLATION (continued)

Exercise 2.9

During the synthesis of nitro-toluene ortho-nitrotoluene as well as para-nitrotoluene are formed. The customer requires pure para-nitrotoluene, the most volatile of the two isomers. The product requires a minimal purity of 95%. Another customer is interested in ortho-nitrotoluene, also with a minimal purity of 95%.

The feed, of which 40% is in the vapor phase, amounts 100 kmol/uur and consists of 55% ortho-nitrotoluene. At the chosen operation pressure and temperature of the column the relative volatility of ortho-nitrotoluene over para-nitrotoluene is 2.0.

- a) calculate the size of the top- and bottom streams
- b) determine the minimum number of stages for this separation
- c) determine the minimum reflux ratio
- d) the applied reflux ratio is 3.76, determine the boilup ratio ($=V''/B$) in the reboiler.

Exercise 2.10

550 kmol/h of a binary mixture water/acetic acid (70 mol% water) is separated by distillation in a bottom fraction with 98 mol% acetic acid and a distillate containing 5 mol% acetic acid. The relative volatility of water over acetic acid is 1.85

- a) Calculate the amount of distillate and bottom product this distillation yields?
- b) Determine graphically the minimum number of stages

The external reflux ratio $R (= L'/D)$ is set at 3. The boilup ratio in the stripping section ($= V''/B$), is set to 10.

- c) Determine graphically the required number of equilibrium stages under these conditions.
- d) Determine graphically the optimal location for the feed stage.
- e) Determine graphically the slope of the feed line and from that the vapor fraction in the feed.

Exercise 2.11

Isopropyl alcohol (IPA, boiling point 82.3 °C) needs to be separated from a 30 mol% IPA solution in water. The total IPA production in the plant amounts 50 kmol/hr and the maximum residual concentration in the water should be only 1 mole%. The feed consists for 90% of liquid and the efficiency of the trays is 90%.

- What is the maximum purity of the IPA that can be obtained by distillation under the current circumstances?
- Calculate the size of the bottom and distillate stream to obtain an IPA purity of 70 mol%.
- Draw the y - x -diagram including the pseudo-equilibrium line
- Determine the minimum reflux ratio.
- The real reflux ratio is 2.5, determine the boilup ratio ($=V''/B$) in the reboiler.
- Determine graphically the number of real trays that is required for this distillation.

Vapor-liquid equilibrium data (1 atm, mole fraction IPA)

x	0.10	0.20	0.35	0.50	0.65	0.75	0.80	0.85	0.95
y	0.33	0.43	0.53	0.61	0.70	0.77	0.81	0.85	0.94

Exercise 2.9

<i>Given</i>	F=100 kmol/hr	$x_D=0.95$	$\alpha=2.0$
	P=1 bar	$x_B=0.05$	R=3.76
	q=0.6	$x_F=0.55$	

- Find*
- size of the top- and bottom streams
 - minimum number of stages for this separation
 - minimum reflux ratio
 - boilup ratio ($=V''/B$) in the reboiler

Solution

a) $F = D + B$

$$X_F F = x_D D + x_B B \quad 0.45 \cdot 100 = 0.95D + 0.05B$$

Solve for D or B

$$D = F - B \text{ for other stream}$$

$$B = 55 \text{ kmol/hr, } D = 45 \text{ kmol/hr}$$

- b) Construct y-x diagram for $\alpha=2.0$

Draw stages from x_B to x_D

$$N_{\text{MIN}}=8$$

- c) q-line starts in (0.45;0.45), slope $-q/(1-q)=-0.6/(1-0.6)$

minimal reflux at "pinch", draw correct rectifying operating line

$$\text{slope } \Delta y/\Delta x = (0.95-0.28)/(0.95-0) = 0.71$$

$$R/(R+1) = L'/V' = \text{helling} \rightarrow R = 2.45$$

- d) $L'/V' = 3.76/4.76 = 0.79$,

Draw rectifying operating line

Operating line stripping section through (0.05;0.05) and Intersection q-line with operating line rectifying section

Draw stripping section operating line

$$\text{Slope} = \Delta y/\Delta x = (0.8-0.05)/(0.55-0.05) = 1.5$$

$$L'' = V'' + B$$

$$L''/V'' = 1.5, \rightarrow B = 1.5 - 1 = 0.5, V''/B = 2$$

Exercise 2.10

Given $F=550\text{kmol/hr}$ $x_D=0.95$ $\alpha=1.85$
 $P=1\text{ bar}$ $x_B=0.02$ $R=3$
 $V''/B=10$ $x_F=0.70$

Find a) amount of distillate and bottom product this distillation yields?
 b) minimum number of stages
 c) required number of equilibrium stages
 d) optimal location for the feed stage
 e) slope of the feed line and the vapor fraction in the feed.

Solution a) $F = B + D = 550$

$$F x_F = B x_B + D x_D \rightarrow 550 * 0.7 = B * 0.02 + D * 0.95$$

$$550 * 0.7 = (550-D) * 0.02 + D * 0.95$$

$$D = 402 \text{ kmol/h} \quad B = 148 \text{ kmol/h}$$

b) Construct y-x diagram for $\alpha=1.85$

 Draw stages from $x_B=0.02$ to $x_D=0.95$

$N_{\min}=12$

c) Slope rectifying section operating line $(L'/V'=3/(3+1)=0.75$

$$L''=V''+B, \text{ given is } B=V''/10$$

 Slope stripping section operating line $L''/V'' = 1.1$

 Intersection operating lines at $y=0.70$

 Drawing stages from $x_B=0.02$ to $x_D=0.95 \rightarrow N=17$

d) Tray 10

e) Intersection operating lines at $y=0.7$, feed $x_F=0.7$

 Slope of feed line $-q/(1-q)=0$

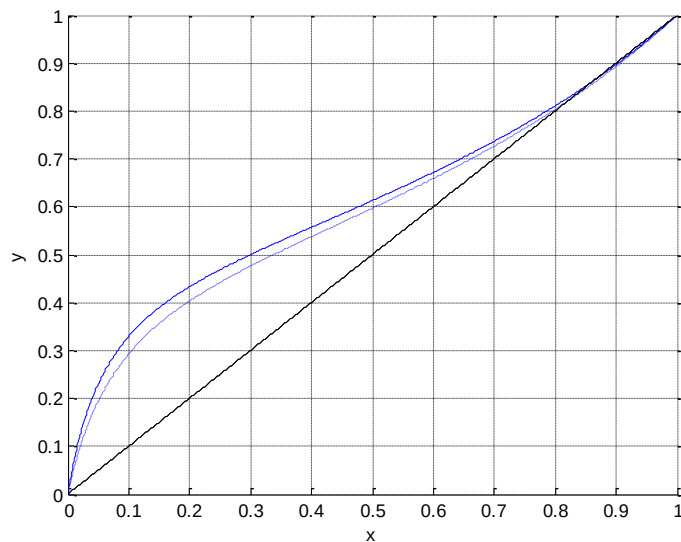
$q=0 \rightarrow$ feed completely saturated vapor

Exercise 2.11

<i>Given</i>	$F=50\text{ kmol/hr}$	$x_D=0.70$	$R=2.5$
	$P=1\text{ bar}$	$x_F=0.30$	$\eta_{MV}=0.90$
	$q=0.1$	$x_B=0.01$	

- Find*
- Maximum purity of the IPA that can be obtained
 - Size of the bottom and distillate stream for IPA purity of 70 mol%.
 - yx-diagram including the pseudo-equilibrium line
 - Miniumal reflux ratio.
 - Boilup ratio ($=V''/B$)
 - number of real trays required for this distillation

Solution a) Draw y-x diagram



$x_{B,\max} = 0.85$ due to azeotrope

b) $F = D + B$

$$x_F F = x_D D + x_B B \quad 0.30F = 0.70 \cdot 50 + 0.01B$$

Solve for D or B

$$D = F - B \text{ for other stream}$$

$$F = 119 \text{ kmol/hr}, B = 69 \text{ kmol/hr}$$

c) Dotted line in diagram under a)

d) q-line starts in (0.3;0.3), slope $-0.9/(1 - 0.9)$

minimal reflux at "pinch", draw correct rectifying operating line

determine slope $\Delta y/\Delta x$

$$R/(R+1) = L'/V' = \text{slope} \rightarrow R_{\min} = 1.33$$

- e) slope rectifying operating line $L'/V' = 2.5/3.5 = 0.714$

Stripping section operating line through (0.01;0.01) and intersection q-line with operating line rectifying section

$$\text{Slope} = \Delta y/\Delta x = (0.416-0.01)/(0.3-0.05) = 1.4$$

$$L'' = V'' + B$$

$$L''/V'' = 1.4, \rightarrow B/V'' = 1.4 - 1 = 0.4$$

$$V''/B = 2.5$$

- f) Use the pseudo equilibrium line

Draw stages from x_B to x_D

Chapter 3: ABSORPTION & STRIPPING

Exercise 3.1

A plate tower providing six equilibrium stages is employed for stripping ammonia from a wastewater stream by means of countercurrent air at atmospheric pressure and 25°C. Calculate the concentration of ammonia in the exit water if the inlet liquid concentration is 0.1 mole% ammonia in water, the inlet air is free of ammonia and 2000 standard cubic meter (1 atm, 25°C) of air are fed to the tower per m³ of waste water. The absorption equilibrium at 25°C is given by the relation $y_{\text{NH}_3} = 1.414 x_{\text{NH}_3}$.

$M_{\text{water}} = 0.018 \text{ kg mol}^{-1}$; $\rho_{\text{water}} = 1000 \text{ kg m}^{-3}$; $R = 8.314 \text{ J mol}^{-1}\text{K}^{-1}$; 1 atm = 1.01325 bar.

Exercise 3.2

When molasses is fermented to produce a liquor containing ethanol, a CO₂-rich vapor containing a small amount of ethanol is evolved. The alcohol can be recovered by absorption with water in a sieve tray tower. For the following conditions, determine the number of equilibrium stages required for countercurrent flow of liquid and gas, assuming isothermal, isobaric conditions in the tower and neglecting mass transfer of all components except ethanol.

Entering gas:	180 kmol/h, 98% CO ₂ , 2% ethyl alcohol, 30°C, 1.1 bar
Entering absorbing liquid:	100% water, 30°C, 1.1 bar
Required ethanol recovery:	95%

The vapor pressure of ethanol amounts 0.10 bar at 30°C, and its liquid phase activity coefficient at infinite dilution in water can be taken as 7.5.

Exercise 3.3

A gas stream consists of 90 mole% N₂ and 10 mole% CO₂. We wish to absorb the CO₂ into water. The inlet water is pure and is at 5°C. Because of cooling coils the operation can be assumed to be isothermal. Operation is at 10 bar. If the liquid flow rate is 1.5 times the minimum liquid flow rate, how many equilibrium stages are required to absorb 92% of the CO₂. Choose a basis of 1 mole/hr of entering gas. The Henry coefficient of CO₂ in water at 5°C is 875 bar.

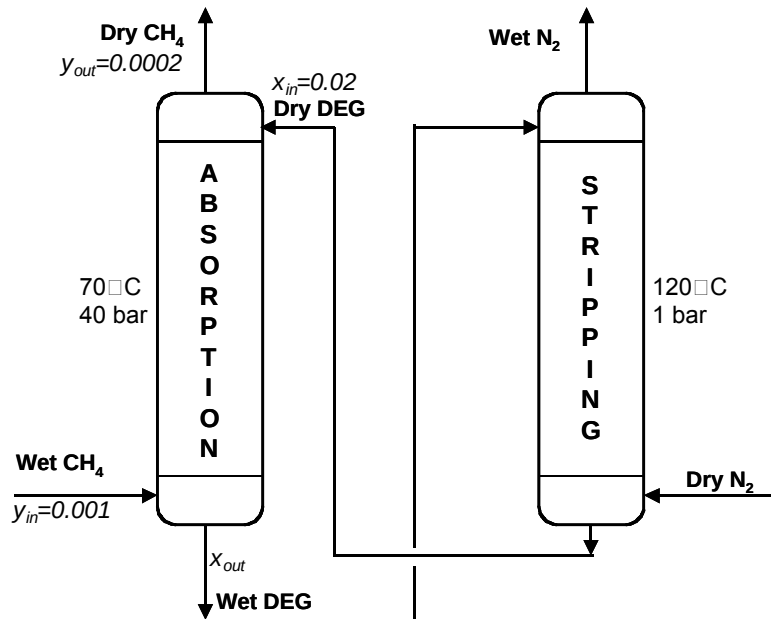
Exercise 3.4

A vent gas stream in your chemical plant contains 15 wt% of a pollutant, the rest is air. The local authorities want to reduce the pollutant concentration to less than 1 wt%. You have decided to build an absorption tower using water as the absorbent. The inlet water is pure and at 30°C. The operation is essentially isothermal. At 30°C your laboratory has found that at low concentrations the equilibrium data can be approximated by $y = 0.5 \cdot x$ (where y and x are weight fractions of the pollutant in vapor and liquid). Assume that air is not soluble in water and that water is nonvolatile.

- Find the minimum ratio of water to air $(L'/G')_{\min}$ on a solute-free basis
- With an $L'/G' = 1.22 (L'/G')_{\min}$ find the total number of equilibrium stages and the outlet liquid concentration

Exercise 3.5

A gas treatment plant often has both absorption and stripping columns as shown in the figure. In this operation the solvent is continually recycled. The heat exchanger heats the saturated solvent, changing the equilibrium characteristics of the system so that the solvent can be stripped. A very common type of gas treatment plant is used for the drying of natural gas by physical absorption of water in a hygroscopic solvent such as diethylene glycol (DEG). In this case dry nitrogen is used as the stripping gas.



Schematic of natural gas absorptive drying operation

- At a temperature of 70°C and a pressure of 40 bar the saturated vapor pressure of water is equal to 0,2 bar. It is known that water and DEG form a nearly ideal solution. Calculate the vapor-liquid equilibrium coefficient and draw the equilibrium line.
- Construct the operating line for $x_0=0.02$, $y_1=0.0002$ and $L/G = 0.01$. Determine the number of theoretical stages required to reduce the water mole fraction from $y_{N+1} = 0.001$ to $y_1 = 0.0002$.
- How many stages are required for $L/G = 0.005$. What happens for $L/G = 0.004$. Determine the minimal L/G -ratio to obtain the desired separation.
- Desorption takes place at 120°C and 1 bar. The saturated vapor pressure of water is equal to 2 bars. Construct the equilibrium and operating lines for desorption with $y_{N+1} = 0$ and $L/G = (L/G)_{\max}/1.5$. x_N and x_0 are to be taken from the absorber operating at $L/G = 0.01$. Determine the number of theoretical stages in the stripping section graphically.
- Calculate analytically the number of theoretical stages in both sections.
- Comment on the chosen value of the liquid mole fraction at the outlet of the absorber.

SOLUTIONS

Exercise 3.1

Given Waste water with ammonia, clean air as strip gas $K = 1.414$
 $x_{\text{ammonia, feed}} = 0.001$ $T = 298.16 \text{ K}$
 $P_{\text{tot}} = 1 \text{ atm}$ $N_{\text{ts}} = 6 \text{ theoretical stages}$
 $G/L = 2000 \text{ standard cubic meter air per cubic meter waste water}$
 $M_{\text{water}} = 0.018 \text{ kg mol}^{-1}$; $\rho_{\text{water}} = 1000 \text{ kg m}^{-3}$; $R = 8.314 \text{ J mol}^{-1}\text{K}^{-1}$; $1 \text{ atm} = 1.01325 \text{ bar}$.

Find The ammonia concentration in the exit water

Thoughts Because the concentrations are low, mole fractions can be used. The equilibrium line is a straight line and the Kremser equation (Eqn. 3.33) is applicable. This equation links the number of theoretical stages and the aqueous concentrations at inlet and exit ($y_{\text{in}} = 0$). However, Eq. 3.34, giving the fraction stripped, is equally applicable and more convenient in this case. The flow rates have to be converted to molar units.

Note that the actual number of stages is larger than the number of theoretical stages because an actual plate does not operate at 100% efficiency. This will be discussed in the next chapter, see Eq. 4.9.

Solution $1 \text{ m}^3 \text{ water / unit time equals } \rho_{\text{water}}/M_{\text{water}} = 55556 \text{ mol / unit time}$
 $2000 \text{ m}^3 \text{ NTP / unit time equals } P \cdot V / R \cdot T = \frac{1.01325 \cdot 10^5 \cdot 2000}{8.314 \cdot 298.16} = 81750 \text{ mol / unit time}$

The L/G -ratio in mole units: $\frac{L}{G} = \frac{55556}{81750} = 0.680$

The stripping factor: $S = \frac{KG}{L} = \frac{1.414}{0.680} = 2.081$

From Eqn. 3.34: $\frac{x_{\text{in}} - x_{\text{out}}}{x_{\text{in}} - \frac{y_{\text{in}}}{K}} = \frac{S^{N_{\text{ts}}+1} - S}{S^{N_{\text{ts}}+1} - 1} = 0.99356$

$y_{\text{in}} = 0 \rightarrow x_{\text{out}} = x_{\text{in}} \cdot (1 - 0.99356) = 6.44 \cdot 10^{-6}$

Alternatively, with known N_{ts} , S , K , x_{in} and $y_{\text{in}} = 0$, solve the Kremser Eq. 3.33, for x_{out} .

Exercise 3.2

Given gas flow rate = 180 kmol/hr , 98% CO_2 , 2% $\text{C}_2\text{H}_5\text{OH}$ $T = 30^\circ\text{C}$
 $P = 1.1 \text{ bar}$ $P_{\text{vap, EtOH}} = 0.1 \text{ bar}$
 Absorbent: pure water ($x_{\text{in}} = 0$) $\gamma_{\infty, \text{EtOH in water}} = 7.5$

Find The required number of stages to recover 95% of the ethanol.

Thoughts Mole fractions can be used because the concentrations are sufficiently small. Eqs. 3.2 and 3.3 are used to calculate the distribution coefficient K_A . The volume change due to absorption is neglected. The liquid-to-gas-ratio is not given in this exercise, so a reasonable value has to be assumed: take $L/G = 1.5 \cdot (L/G)_{\text{min}}$. The required number of stages is then found from a McCabe Thiele diagram or from the Kremser equation.

Solution Calculation of the distribution coefficient: $K_A = \frac{\gamma_A \cdot p_A^0}{p_{\text{tot}}} = \frac{7.5 \cdot 0.1}{1.1} = 0.682$

Calculation of the minimum slope of the operating line, $(L/G)_{\text{min}}$, with Eq. 3.7:

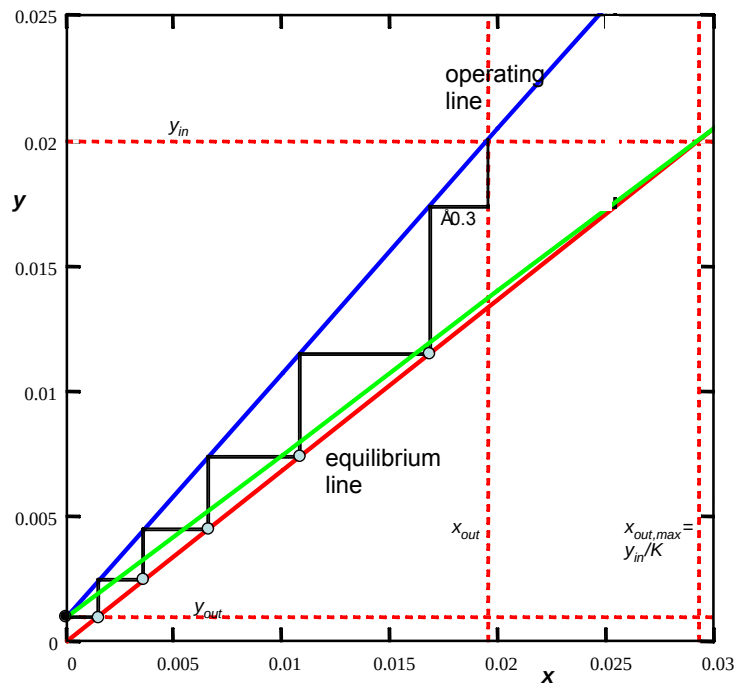
$$\left. \frac{L}{G} \right|_{\text{min}} = \frac{y_{\text{in}} - y_{\text{out}}}{x_{\text{out, max}} - x_{\text{in}}} = \frac{0.02 - 0.02 \cdot (1 - 0.95)}{0.02 / 0.682 - 0} = 0.648, \quad (y_{\text{out}} = 10^{-3})$$

hence $\frac{L}{G} = 1.5 \cdot \frac{L}{G} \Big|_{min} = 0.972$. With $A = \frac{L}{G \cdot K} = \frac{0.972}{0.682} = 1.425$ Eq. 3.28 gives:

$$N_{ts} = \frac{\ln\left(\frac{1}{A} + \left(1 - \frac{1}{A}\right) \cdot \frac{y_{in}}{y_{out}}\right)}{\ln A} = 5.36 \text{ theoretical stages}$$

For a graphical McCabe-Thiele solution, draw the operating line through (x_{in}, y_{out}) with slope 0.972 and the equilibrium line with slope $K = 0.682$. Start the step case construction at $(0, 0.001)$.

The number of equilibrium stages, counted on the equilibrium line, is appr. 5.3.



Exercise 3.3

Given	$N_{2,in} = 90 \text{ mol\%}$	$P_{tot} = 10 \text{ bar}$
	$CO_{2,in} = 10 \text{ mol\%}$	$T = 5^\circ\text{C}$
	Isothermal operation	$H_{CO_2, 5^\circ\text{C}} = 875 \text{ bar}$
	Absorbent = pure water	$L/G = 1.5 (L/G)_{min}$

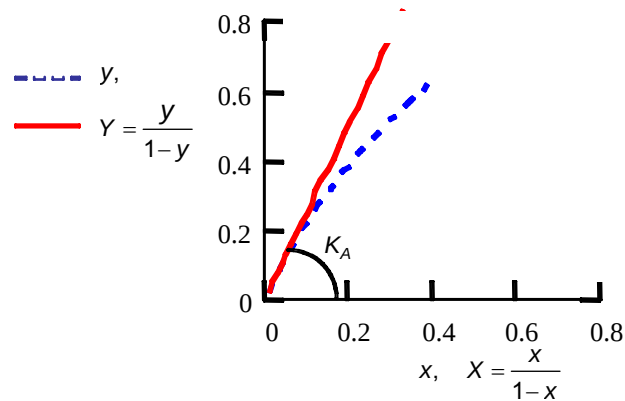
Find N_{ts} , number of equilibrium stages for a CO_2 recovery of 92%

Thoughts Mole ratios have to be used because of the rather large concentrations. The distribution coefficient K_A is calculated from the Henry coefficient according to Eqs. 3.3 and 3.4.

While the linear expression $y = K_A \cdot x$ (Eq. 3.3) applies to low concentrations only, conversion to mole ratios extends the applicability: $Y = K_A \cdot X$ still holds at higher concentrations. Both expressions have the same initial slope, K_A , see YX diagram on next page.

The number of stages can be determined graphically from a McCabe Thiele diagram or by applying a Kremser equation (Eq. 3.28). The minimum absorbent flow rate can be found either graphically or from a mass balance assuming equilibrium between the exiting absorbent and the entering gas, i.e.

$$X_{out,max} = Y_{in}/K_A = Y_{in}/K_{CO_2}$$



Solution $y_{N+1} = y_{in} = 0.1$

$$Y_{in} = 0.111$$

$$Y_{out} = (1 - 0.92) \cdot 0.111 = 8.9 \cdot 10^{-3}$$

$$x_{in} = X_{in} = 0$$

Dividing Eq. 3.3 by Eq. 3.4 gives the distribution coefficient $K_{CO_2} = H_{CO_2} / P_{tot} = 875/10 = 87.5$, hence, $Y_{CO_2} = 87.5 \cdot X_{CO_2}$.

All operating lines intersect at $(X_{in}, Y_{out}) = (0, 8.9 \cdot 10^{-3})$.

Minimum absorbent flow rate from mass balance Eq. 3.7, see also Fig. 3.6.

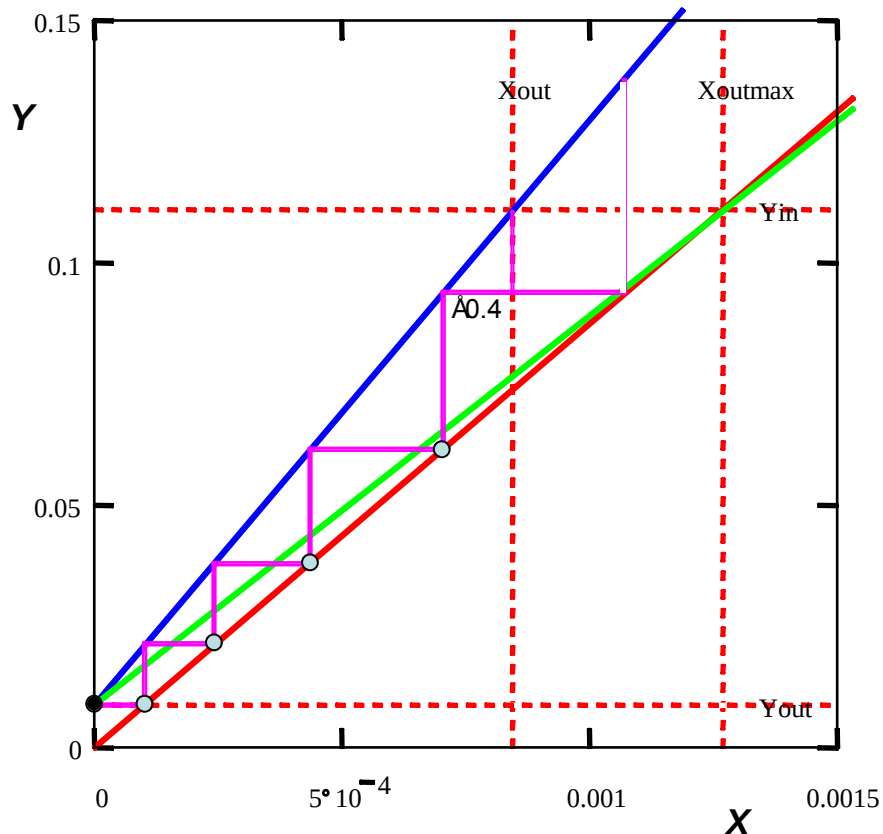
After conversion to mole ratios it follows:

$$\left(\frac{L}{G}\right)_{min} = \frac{Y_{in} - Y_{out}}{X_{out,max} - X_{in}} = \frac{0.111 - 8.9 \cdot 10^{-3}}{\frac{0.111}{87.5} - 0} = 80.5$$

The actual absorbent flow rate: $L/G = 1.5 \cdot 80.5 = 120.8$

In the McCabe-Thiele diagram below one can count the number of equilibrium stages on the equilibrium line. Check in the graph below that $N_{ts} \approx 4.4$.

Alternatively, Eq. 3.28 can be applied with $A = 120.8 / 87.5 = 1.38$, resulting in $N_{ts} \approx 4.2$.



Exercise 3.4

Given $y_{\text{pollutant}} = 15 \text{ wt\%}$ in gas stream $T = 30^\circ\text{C}$, isothermal operation
 Desired exit concentration: $y_{\text{pollutant}} = 1 \text{ wt\%}$ Absorbent: pure water
 Equilibrium: $y = 0.5 \cdot x$ (30°C , weight fractions, low concentration)

Find The minimum water-to-air-ratio $(L'/G')_{\min}$ and the number of stages and outlet liquid concentration at $L'/G' = 1.22 \cdot (L'/G')_{\min}$

Thoughts Weight ratios have to be used because of the high concentrations to allow working on a *solute-free* basis. This conversion extends the linear equilibrium line with $K = 0.5$, see previous exercise. The minimum L'/G' ratio can be found from the mass balance assuming equilibrium between the entering gas and the exiting liquid because that gives the highest possible value for x_{out} , see Fig. 3.6.

Solution $Y_{\text{in}} = \frac{y_{\text{in}}}{1 - y_{\text{in}}} = 0.176$, $Y_{\text{out}} \approx y_{\text{out}}$

Equilibrium line: $Y = 0.5 \cdot X$

All operating lines through the point: $X_{\text{in}} = x_{\text{in}} = 0$, $Y_{\text{out}} = y_{\text{out}} = 0.01$

The highest value of x_{out} is obtained at the lowest possible liquid flow rate.

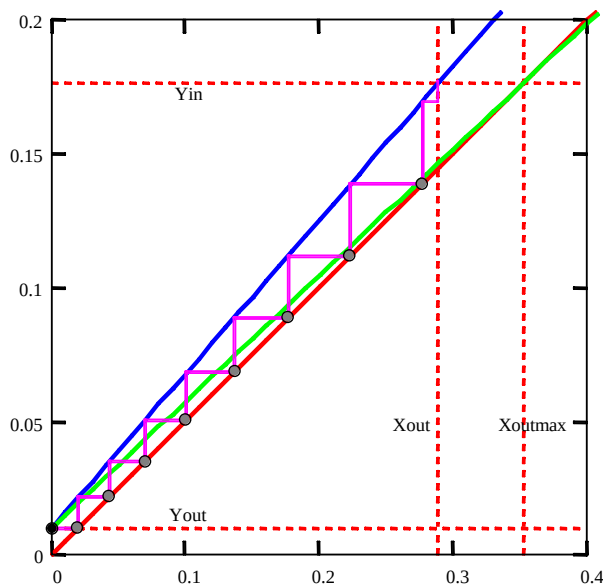
The slope of the corresponding operating line is calculated from:

$$\left(\frac{L'}{G'}\right)_{\min} = \frac{Y_{\text{in}} - Y_{\text{out}}}{X_{\text{out,max}} - X_{\text{in}}} = \frac{0.176 - 0.01}{\frac{0.176}{0.5} - 0} = 0.471$$

The L'/G' ratio to be applied is $L'/G' = 1.22 \cdot 0.471 = 0.575$ and the expression for the operating line becomes

$$Y = 0.575 \cdot X + Y_{\text{out}}$$

The McCabe Thiele diagram looks as follows



The Kremser equation (Eq. 3.28) gives with $A = 0.575/0.5 = 1.15$: $N_{\text{ts}} = 8.2$.

X_{out} follows from the overall balance, Eq. 3.26: $X_{\text{out}} = \frac{0.176 - 0.01}{0.575} = 0.289$ or $x_{\text{out}} = 0.224$.

Note: Some textbooks use $A^N = \frac{Y_{\text{in}} - K \cdot X_{\text{out}}}{Y_{\text{out}}}$ which is essentially Eq. 3.25 for $x_{\text{in}} = 0$.

Exercise 3.5**Given** Absorption:

$T = 70\text{ }^{\circ}\text{C}$

$P_W^0 = 0.2\text{ bar}$

$y_{N+1} = y_{in} = 0.001$

$P = 40\text{ bar}$

$x_0 = x_{in} = 0.02$

$y_1 = y_{out} = 0.0002$

Desorption:

$T = 120\text{ }^{\circ}\text{C}$

$P_{\text{sat, H}_2\text{O}} = 2\text{ bar}$

$L/G = (L/G)_{\text{max}}/1.5$

$P = 1\text{ bar}$

$y_{N+1} = y_{in} = 0$

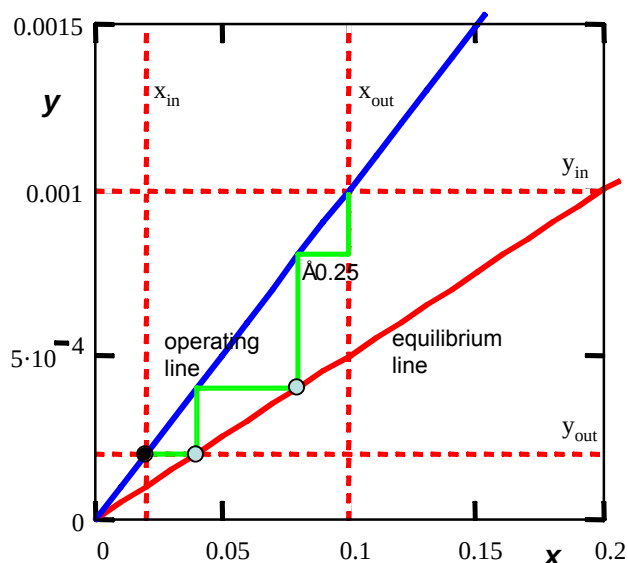
Find

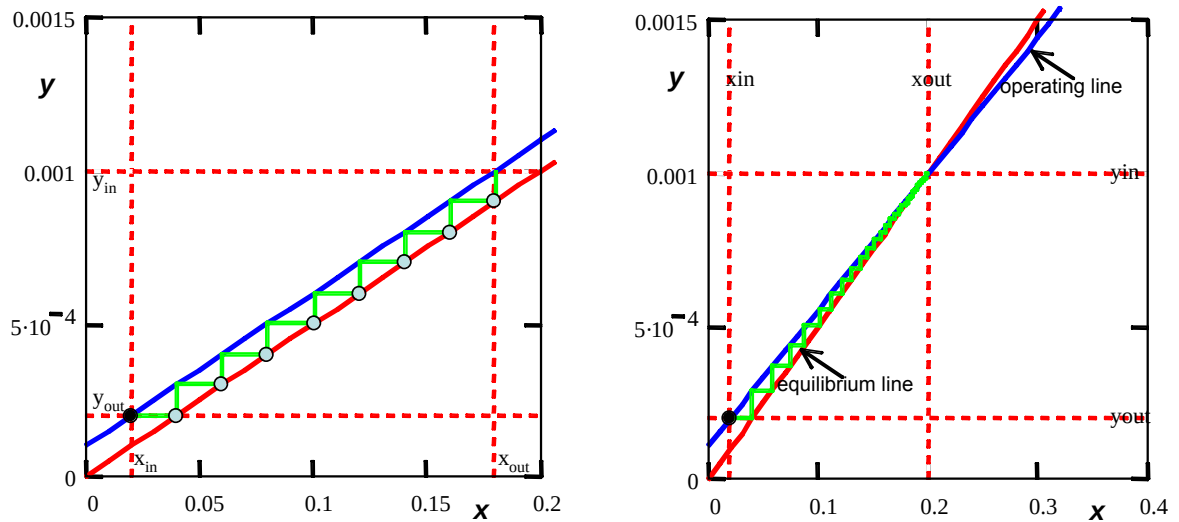
- the distribution coefficient and the equilibrium curve
- the operating line and number of theoretical stages for $L/G = 0.01$
- idem for $L/G = 0.005$ and $L/G = 0.004$; the minimum L/G both from the McCabe-Thiele diagram and from a mass balance
- the number of theoretical stages for desorption at $L/G = (L/G)_{\text{max}}/1.5$
- the number of theoretical stages in both absorption and desorption using the Kremser-equations.
- the concentration in liquid phase at inlet of stripper

Thoughts

- K_w can be calculated from Eqs. 3.1 and 3.3. The concentrations are sufficiently small to assume the equilibrium curve to be a straight line.
- All operating lines intersect at $(x_{in}, y_{out}) = (0.02, 0.0002)$ and have as slope (L/G) .
- Idem; the lower (L/G) , the more difficult the separation will be. At the minimum value of (L/G) , an infinite number of stages is required.
- The K -value for stripping can be calculated from Eq. 3.1. The operating line will now be located below the equilibrium, because the direction of mass transfer is reversed.
- The Kremser equation is given in Eq. 3.28 (absorption) and in Eq. 3.33 (desorption/stripping). Conversion of mole fractions to mole ratios is not necessary. It can be expected that the results of the Kremser equations with mole fractions agree quite well with the graphical results.
- Investigate the limiting values at absorber inlet and stripper outlet.

- Solution** (a) From Eqs. 3.1 and 3.3: $K_w = K_{w, \text{abs}} = \frac{P_W^0}{P} = \frac{0.2}{40} = 5 \cdot 10^{-3}$. The line is shown in the figure below.
- (b) Operating line through $(x_{in}, y_{out}) = (0.02, 0.0002)$ with slope $L/G = 0.01$
 In this case the intercept $y_1 - x_0 \cdot L/G = 0 \rightarrow y = 0.01 \cdot x$.
 Reduction of the water mole fraction in the gas from 0.001 to 0.0002: $N_{ts} = 2.25$.





- (c) For $L/G = 0.005$, the operating line and the equilibrium line are parallel and $N = 8$.
 For $L/G = 0.004$, the separation cannot be achieved: the operating line and equilibrium line intersect at a y -value lower than 0.001.

The minimum L/G -ratio can be found by drawing an operating line which intersects the equilibrium line at the specified concentrations (0.02, 0.001), meaning equilibrium between entering gas and exiting 'wet' DEG:

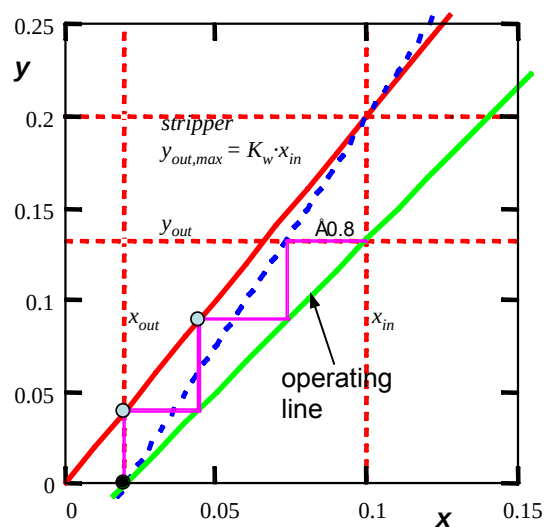
$$\left. \frac{L}{G} \right|_{min} = \frac{y_{in} - y_{out}}{x_{out,max} - x_{in}} = \frac{y_{N+1} - y_1}{y_{N+1}/K_w - x_0} = \frac{0.001 - 0.0002}{0.2 - 0.02} = 4.44 \cdot 10^{-3}$$

(d) $K_w = K_{w, str} = \frac{P_w^0}{P} = \frac{2}{1} = 2$

The water concentration in the exiting DEG stream can be found from the operating line: $x_N = 0.1$. After desorption, the water concentration has to be reduced to $x = 0.02$.
 As strip gas, dry nitrogen is used: $y = 0$.

The maximum L/G ratio follows from the mass balance:

$$\left(\frac{L}{G} \right)_{max} = \frac{K_w x_{in} - y_{in}}{x_{in} - x_{out}} = \frac{2 \cdot 0.1 - 0}{0.1 - 0.02} = 2.5 \Rightarrow \frac{L}{G} = \frac{2.5}{1.5} = 1.667$$



- (e) Kremser equation for absorption, Eq. 3.28, with $A = 0.01 / 5 \cdot 10^{-3} = 2$, $y_{in} = 0.001$, $x_{in} = 0.02$, and $y_{out} = 2 \cdot 10^{-4}$. Follows $N_{ts} = 2.32$.
Kremser equation for stripping, Eq. 3.33, with $S = 2 / 1.667 = 1.20$, $x_{S_{in}} = 0.1$, $y_{S_{in}} = 0$, and $x_{S_{out}} = 0.02$. Follows $N_{ts} = 2.80$.

- (f) At a given L/G ratio x_{out} at the absorber outlet follows from the overall mass balance (Eq. 3.6 and $L/G = 0.01$)

$$x_{out} = \frac{G}{L}(y_{in} - y_{out}) + x_{in} = 0.10$$

The upper and lower limits of L/G are determined by the maximum concentration leaving the absorber and the minimum concentration at the inlet.

The highest possible liquid concentration at the outlet of the absorber amounts to $y_{in} / K_{w,abs} = 0.2$.

The lowest possible liquid concentration at the inlet of the absorber equals the lowest outlet concentration of the stripped liquid and amounts to zero.

Any value in between would work and would fix L/G in both columns.

Chapter 4: GENERAL DESIGN OF G/L CONTACTORS

Exercise 4.1

An aqueous droplet with radius R is surrounded by a stagnant, inert vapor phase containing a low concentration of ammonia.

Show that $y|_r - y|_{r=R}$, the difference in ammonia concentration in the vapor phase and at the LV-interface, is proportional to $1/R - 1/r$, where r is the distance to the centre of the droplet. Assume that the resistance against mass transfer from the vapor to the liquid is mainly situated in the vapor phase.

Exercise 4.2

Derive the rate expression in Eq. 4.6, starting from the stationary state balance given in Eq. 4.3.

Exercise 4.3

A tray column is to be designed to reduce the water content in natural gas ($M_{\text{methane}} = 0.016 \text{ kg mol}^{-1}$) from 0.10 to 0.02 mole% by absorption of water at 70°C and 40 bar in diethylene glycol ($M_{\text{DEG}} = 0.106 \text{ kg mol}^{-1}$, $\rho_{\text{DEG}} = 1100 \text{ kg m}^{-3}$) containing 2.0 mol% H_2O . The equilibrium constant $K = 5.0 \cdot 10^{-3}$ at this temperature. Preliminary calculations (see exercise 3.5) resulted in a slope of the operating line $L/V = 0.010$ and $N_{\text{ts}} = 2.25$.

The column should have the capacity to treat $Q_V = 3 \text{ m}^3$ gas per unit time at this condition (40 bar, 70°C). Lab scale experiments showed that the overall mass transfer coefficient $k_{OV} = 3 \cdot 10^{-3} \text{ m s}^{-1}$ at this gas load.

The gas phase is assumed to obey the ideal gas law. $H_{\text{spacing}} = 0.5 \text{ m}$, $g = 9.81 \text{ m s}^{-2}$.

Calculate:

- the minimum column diameter
- the plate efficiency
- the height of the column

Exercise 4.4

An aqueous waste stream of $0.015 \text{ m}^3 \text{ s}^{-1}$, saturated with benzene, should be purified by reducing the benzene content by at least 99.9%. This is possible by stripping with (pure) air. To this purpose an existing tray column with 5 trays is available. The effective surface area of a single tray amounts to 1.77 m^2 . Laboratory experiments at 1 bar and 294K show that the overall mass transfer coefficient based on the gas phase, $k_{OV} = 0.0080 \text{ m s}^{-1}$.

The stripping process is carried isothermically out at the same conditions with an airflow¹ of $0.932 \text{ m}^3 \text{ STPs}^{-1}$. At this temperature the distribution coefficient $K_{\text{benzene}} = 152$ and the saturation pressure of benzene is 0.104 bar. At this airflow the interfacial surface area is 50 m^2 per m^2 tray area.

$M_{\text{air}} = 0.029 \text{ kg mol}^{-1}$, $M_{\text{water}} = 0.018 \text{ kg mol}^{-1}$, $M_{\text{benzene}} = 0.078 \text{ kg mol}^{-1}$, $g = 9.8 \text{ ms}^{-2}$,

$R = 8.31 \text{ J mol}^{-1} \text{ K}^{-1}$, $\rho_{\text{water}} = 1000 \text{ kg m}^{-3}$, 1 atm = 1.01325 bar.

- How many theoretical stages are required to reduce the benzene content by 99.9%?
- Calculate the plate efficiency E_{MV} .
- Show by calculation that this column can produce the required reduction at the given flow rates.
- Calculate the mole fraction of benzene in the effluent.
- Comment on the chosen value of the airflow.
- How should the exiting gas flow be treated to avoid dumping of benzene?

¹ 1 $\text{m}^3 \text{ STP}$ means 1 m^3 at standard temperature (0°C) and standard pressure (1atm)

Exercise 4.5

An air-ammonia mixture, containing 5 mol% NH_3 at a total flow rate of 5 mol s^{-1} , is scrubbed in a packed column by a countercurrent flow of $0.5 \text{ kg water s}^{-1}$. At 20°C and 1 bar 90% of the ammonia is absorbed.

$M_{\text{air}} = 0.029 \text{ kg mol}^{-1}$, $M_{\text{ammonia}} = 0.017 \text{ kg mol}^{-1}$, $M_{\text{water}} = 0.018 \text{ kg mol}^{-1}$, the density of water may be taken as 1000 kg m^{-3} .

Calculate the flooding velocity for two different packing materials:

- 25 mm ceramic Raschig rings with a packing factor of 540 m^{-1} , and
- 25 mm metal Hiflow rings with a packing factor of 125 m^{-1} .

Exercise 4.6

To study the rate of absorption of SO_2 in water, a laboratory scale column packed with plastic *Hiflow* rings is used. The diameter of the rings is 15 mm. The bed has a porosity $\epsilon_{\text{bed}} = 0.90$ and a surface area per unit bed volume $a = 200 \text{ m}^2 \text{ m}^{-3}$. The height of the packing is 2.0 m, the internal diameter of the column 0.33m.

Air containing 2.0 mol% SO_2 is fed to the column at a rate of $V = 2.25 \text{ mol s}^{-1}$, clean water is fed to the top of the column. The absorption of SO_2 in water is studied in counter-current operation at 1 bar and 285 K. At this temperature the distribution coefficient $K_{\text{SO}_2} = 32$.

At a liquid flow rate $L = 65 \text{ mol s}^{-1}$ the SO_2 -content in the effluent is reduced to 0.50 mol%.

$M_{\text{lucht}} = 0.029 \text{ kg mol}^{-1}$, $M_{\text{water}} = 0.018 \text{ kg mol}^{-1}$,

$g = 9.81 \text{ m s}^{-2}$, $R = 8.31 \text{ J mol}^{-1} \text{ K}^{-1}$, $\rho_{\text{water}} = 1000 \text{ kg m}^{-3}$.

- How much larger is the chosen value of L compared to the theoretical minimum value, L_{min} ?
- How many transfer units N_{OV} characterize this absorption process at the given process conditions?
- Calculate the overall mass transfer coefficient based on the gas phase, k_{OV} .
- At what (superficial) velocity of the gas feed flooding will start?
- Calculate the concentration of SO_2 in the gaseous effluent in case the air feed is increased to 75% of the flooding velocity. Temperature, pressure and liquid flow rate remain unchanged

SOLUTIONS

Exercise 4.1

Given A water droplet with radius R absorbing ammonia from a stagnant gas phase.

Find The concentration of ammonia as a function of the distance r from the droplet centre.

Thoughts The concentration of ammonia is small, thus the droplet may absorb ammonia at a constant rate for quite a while. This rate is given by Eq. 4.1a, with $\phi_{air} = 0$ and y_{NH_3} being very small.

Solution The second term in the RHS of Eq. 4.1a, $y_{NH_3} \cdot \phi_{NH_3}$, is small compared to the first term and, replacing z by r and with $A_r = 4\pi r^2$, this equation transforms into

$$\phi_{NH_3} = -D_{NH_3 \cdot air} \cdot 4\pi r^2 \cdot \rho_V \cdot \frac{d y_{NH_3}}{d r} = \text{constant, as long as } y_{NH_3} \text{ is sufficiently small.}$$

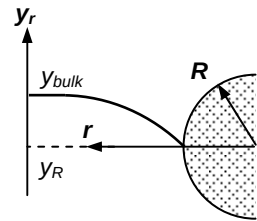
$$\text{Separation of the variables gives } d y_{NH_3} = \frac{\phi_{NH_3}}{4\pi \cdot \rho_V \cdot D_{NH_3 \cdot air}} d r^{-1}$$

Integration from $y_{NH_3} = y_R (= y_{Ai})$ at the L/V interface ($r = R$) to y_r at distance r from the centre of the droplet results in

$$y_r - y_R = -\frac{\phi_{NH_3}}{4\pi \cdot \rho_V \cdot D_{NH_3 \cdot air}} \cdot \left[\frac{1}{R} - \frac{1}{r} \right]$$

As $r \rightarrow \infty$, $y_{NH_3} \rightarrow y_{bulk}$. Hence the rate of absorption is given by

$$\phi_{NH_3} = -4\pi R \cdot D_{NH_3 \cdot air} \cdot \rho_V [y_{NH_3, bulk} - y_R], \text{ in mol/s.}$$



Note that $\phi_{NH_3} < 0$ meaning transport in the direction of decreasing r (towards the L/V -interface).

Exercise 4.2

Derive Eq. 4.6 from Eq. 4.3.

Thoughts Try to eliminate the composition at the interface. Note that $y_{Ai} = y_{Ai}^*$

Solution Extract the differences in compositions from the two balance equations, Eq. 4.3, replacing x by the equilibrium relation y^*/K ,

$$\begin{aligned} y_A - y_{Ai}^* &= \frac{\phi_A}{k_V A_{LV} \rho_V} \\ y_{Ai}^* - y_A &= \frac{K \phi_A}{k_L A_{LV} \rho_L} \end{aligned}$$

$$y_A - y_A^* = \frac{\phi_A}{A_{LV} \rho_L} \left[\frac{1}{k_V} + \frac{1}{k_L} \frac{K \rho_V}{\rho_L} \right]$$

and, after rearrangement,

$$\phi_A = \frac{A_{LV} \rho_L}{\left[\frac{1}{k_V} + \frac{1}{k_L} \cdot \frac{K \rho_V}{\rho_L} \right]} (y_A - y_A^*) = k_{OV} A_{LV} \rho_L (y_A - y_A^*)$$

with

$$\frac{1}{k_{OV}} = \frac{1}{k_V} + \frac{1}{k_L} \cdot \frac{K \rho_V}{\rho_L}$$

Exercise 4.3**Given** Drying of natural gas by absorption of water in DEG

$x_{\text{water,in}}$	$= 0.001$	$x_{\text{water,out}}$	$= 0.0002$	$x_{\text{water,DEG}}$	$= 0.02$
T	$= 343 \text{ K}$	P_{tot}	$= 40 \cdot 10^5 \text{ Pa}$	Q_V	$= 3 \text{ m}^3 \text{ s}^{-1}$
M_{DEG}	$= 0.106 \text{ kg mol}^{-1}$	$\rho_{\text{DEG}} = \rho_L$	$= 1100 \text{ kg m}^{-3}$	M_{CH_4}	$= 0.016 \text{ kg mol}^{-1}$
L/V	$= 0.010$	K_{eq}	$= 5.0 \cdot 10^{-3}$	k_{OV}	$= 3 \cdot 10^{-3} \text{ m s}^{-1}$
N_{ts}	$= 2.25$	H_{spacing}	$= 0.5 \text{ m}$	g	$= 9.81 \text{ m s}^{-2}$

- Find**
- the minimum column diameter
 - the plate efficiency and the overall efficiency
 - the height of the column

Thoughts Because the concentrations are very small, the flow rate is constant throughout the column. Conversion of mole fractions to mole ratios is not necessary in this case.

- The flooding velocity is the key parameter, which can be read from Fig. 4.10 once the flow parameter is known.
Note that L and V (parameters in slope of operating line) are in mole s^{-1} and Q_V and Q_L (parameters in flow parameter) in $\text{m}^3 \text{s}^{-1}$.
- Eqs. 4.12 and 4.13 are applicable for trays. However, the interface area A_{LV} in Eq. 4.12 is not known. On the other hand, its value is known to range from 30 to 100 m^2 per unit surface tray area. Assume the lowest value to be on the safe side.
- Calculate the number of real trays with Eq. 4.9 ($N_{\text{ts}} = 2.25$) and the column height with Eq. 4.8.

Solution a) $L = (L/V) \cdot V$ and $V = Q_V \cdot \rho_y$ with $\rho_y = P/RT = 1403 \text{ mol m}^{-3}$.
 $V = 4209 \text{ mol s}^{-1}$, $L = 42.1 \text{ mol s}^{-1}$ and $Q_L = L \cdot M_{\text{DEG}} / \rho_{\text{DEG}} = 4.06 \cdot 10^{-3} \text{ m}^3 \text{s}^{-1}$.

$$\text{Flow parameter} = \frac{Q_L}{Q_V} \sqrt{\frac{\rho_L}{\rho_y \cdot M_{\text{CH}_4}}} = 0.0095.$$

In Fig. 4.10 the corresponding value on the vertical axis is the limiting value, 0.046.
 (This value can be calculated with Eq. 4.25, taking $\gamma = 0.03 \text{ N m}^{-1}$ and $\phi = 0.1$)

$$u_{\text{flood}} = 0.046 \cdot \sqrt{\frac{g \cdot H_{\text{spacing}} \cdot \rho_{\text{DEG}}}{\rho_y \cdot M_{\text{CH}_4}}} = 0.713 \text{ m s}^{-1}.$$

$$A_{\text{min}} = \frac{Q_V}{0.85 \cdot u_{\text{flood}}} = 4.95 \text{ m}^2 \quad \text{and} \quad D_{\text{min}} = \sqrt{\frac{4A_{\text{min}}}{\pi \cdot 0.85}} = 2.7 \text{ m (Eq. 4.27)}$$

- b) The interfacial area $A_{LV} = 30 \cdot 4.95 = 148.5 \text{ m}^2$, the number of transfer units

$$N_{\text{OV}} = \frac{k_{\text{OV}} \cdot A_{LV}}{Q_V} = 0.148 \text{ (Eq. 4.12) and the tray efficiency}$$

$$E_{\text{MV}} = 1 - \exp(-N_{\text{OV}}) = 0.138 \text{ (Eq. 4.13).}$$

- c) The overall efficiency E_O is given by Eq. 4.19:

$$E_O = \frac{\ln[1 + E_{\text{MV}}(S - 1)]}{\ln(S)}$$

In this case of absorption, $S = A_{\text{abs}} = L/KV = 2$.

Hence, $E_O = 0.186$ and $N_s = N_{\text{th}}^{2}) / E_O = 2.25 / 0.186 = 12.1$ (Eq. 4.9)

$$H_{\text{column}} = H_{\text{spacing}} \cdot N_s = 0.5 \cdot 12.1 = 6.0 \text{ m (Eq. 4.8).}$$

How would a change in the value of L/V , P_{tot} or Q_V influence the column dimensions?
 (see Exercises 4.4e and 4.6e for a discussion)

²⁾ $N_{\text{th}} = 2.25$, see exercise 3.5.

Exercise 4.4

<i>Given</i>	$Q_L = 0.015 \text{ m}^3 \text{ s}^{-1}$	$\rho_{\text{water}} = 1000 \text{ kg m}^{-3}$	fraction f stripped = 0.999
	$Q_V = 0.932 \text{ m}^3 \text{ STPs}^{-1}$	$P_{\text{tot}} = 1 \text{ bar}$	$N = 5 \text{ trays}$
	$A_{\text{tray}} = 1.77 \text{ m}^2$	$A = 50 \text{ m}^2 \text{ per m}^2 \text{ tray area}$	$k_{OV} = 0.0080 \text{ m s}^{-1}$
	$T = 294 \text{ K}$	$K_B = 152$	$P_B^0 = 0.104 \text{ bar}$
	$M_{\text{air}} = 0.029 \text{ kg mol}^{-1}$	$M_{\text{water}} = 0.018 \text{ kg mol}^{-1}$	$M_B = 0.078 \text{ kg mol}^{-1}$
	$R = 8.31 \text{ J mol}^{-1} \text{ K}^{-1}$	$g = 9.8 \text{ ms}^{-2}$	1 atm = 1.01325 bar

- Find*
- the number of theoretical stages, N_{ts}
 - the plate efficiency, E_{MV}
 - out whether the fraction benzene stripped is at least 99.9% under the given conditions
 - x_{out}
 - out how a change in airflow will effect the operation
 - a way to dispose of the benzene in the effluent safely.

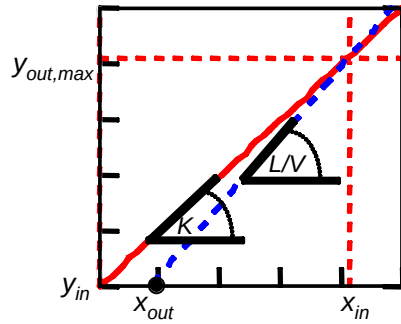
- Thoughts*
- The Kremser equation in the form of the amount stripped (Eq. 3.34) is applicable
 - Use the number of transfer units N_{OV} from Eq. 4.12 to calculate E_{MV} in Eq. 4.13.
 - The real number of stages should be not larger than $N = 5$. Hence the overall efficiency $E_o = N_{ts}/N_s$ has to be calculated from Eq. 4.19.
 - x_{out} follows from x_{in} (saturated solution) and the actual stripping factor.
 - Check the minimum and maximum allowable gas flow. Take $H_{spacing} = 0.5 \text{ m}$.

- Solution*
- In order to apply Eq. 3.34, the stripping factor $S = K \cdot V/L$ has to be calculated, where V and L are in mol/s.
 $V = Q_V \cdot \rho_g = Q_V \cdot P_{\text{tot}} / RT = 0.932 \cdot 10^5 \cdot 1.01325 / (8.31 \cdot 273) = 41.6 \text{ mol/s}$,
 $L = Q_L \cdot \rho_{\text{water}} / M_{\text{water}} = 833.3 \text{ mol/s}$ and follows $S = K \cdot V/L = 7.59$.

$$f = \frac{S^{N_{ts}+1} - S}{S^{N_{ts}+1} - 1} = 0.999$$
, hence $N_{ts} = 3.34$ theoretical stages³.
 - The total LV-bubble interphase area $A_{LV} = A \cdot A_{\text{tray}} = 88.5 \text{ m}^2$.
The volumetric flow rate at 1 bar and 294 $Q = 0.932 \cdot 294 / 273 = 1.00 \text{ m}^3 \text{ s}^{-1}$.
With Eq. 4.12 follows $N_{OV} = 0.0080 \cdot 88.5 / 1.00 = 0.706$ transfer units.
From Eq. 4.13 the resulting plate efficiency $E_{MV} = 1 - \exp(-0.706) = 0.506$.
 - The real number of stages, N_s , is given by Eq. 4.9: $N_s = N_{ts} / E_o$.
Substitution of the values of S and E_{MV} gives $E_o = 0.724$ and $N_s = 4.61$.
 $N_s < 5$, hence the stripping factor can be even (slightly) higher than the required value of 0.999 or, with $f = 0.999$, the capacity can be somewhat higher.
 - The mole fraction benzene y_B in the gas phase = $P_B^0 / P_{\text{tot}} = 0.104$ (Eq. 2.3).
In equilibrium (saturated solution), $x_{in} = y_B / K_B = 6.84 \cdot 10^{-4}$ (Eq. 1.5). The actual number of theoretical stages $N_{ts} = E_o \cdot N = 0.724 \cdot 5 = 3.62$ and the actual value of

$$f = \frac{S^{N_{ts}+1} - S}{S^{N_{ts}+1} - 1} = 0.99943$$
.
 $x_{out} = (1 - f) \cdot x_{in} = 0.388 \cdot 10^{-6}$ (0.4 ppm).
 - The *minimum* airflow is determined by the maximum slope of the operating line which, because $f \approx 1$, coincidences with the equilibrium line:

³) = N_{th} in Eq. 4.19



$$\left. \frac{L}{V} \right|_{\max} = \frac{y_{out,\max} - y_{in}}{x_{in} - x_{out}} = \frac{y_{out,\max} - 0}{x_{in} - (1-f)x_{in}} \approx \frac{y_{out,\max}}{x_{in}} = K$$

For clarity, the value of x_{out} in the figure above corresponds to $f = 0.80$ (with $f = 0.9996$ this point would almost coincide with the origin).

$Q_{min} = V_{min}/\rho_y$, $V_{min} = L/K = 5.48 \text{ mol s}^{-1}$, $\rho_V = P_{tot}/RT = 41.473 \text{ mol m}^{-3}$, and $Q_{min} = 0.132 \text{ m}^3 \text{ s}^{-1}$, which is appr. 6 times smaller than the actual flow.

The flooding velocity determines the maximum airflow.

The flow parameter = $\frac{Q_L}{Q} \sqrt{\frac{\rho_{water}}{\rho_V}}$ with $\rho_V = \rho_y \cdot M_{air} = 1.203 \text{ kg m}^{-3}$ and

$$Q = Q_V \cdot 294/273 = 1.00 \text{ m}^3 \text{ s}^{-1}.$$

$$\frac{Q_L}{Q} \sqrt{\frac{\rho_{water}}{\rho_V}} = \frac{0.015}{1.00} \sqrt{\frac{1000}{1.203}} = 0.431.$$

From Fig. 4.10

$$u_{flood} = \frac{0.013}{\sqrt{\frac{1}{H_{spacing} \cdot g} \cdot \frac{\rho_V}{\rho_{water}}}} = 0.830 \text{ m s}^{-1}. \quad u_{max} = 0.85 \cdot 0.830 = 0.705 \text{ m s}^{-1}.$$

$Q_{max} = u_{max} \cdot A_{tray} = 1.25 \text{ m}^3 \text{ s}^{-1}$, which is 25% higher than the actual flow.

$$\frac{Q_{min}}{Q} \approx 0.16, \quad \frac{Q_{max}}{Q} = 1.25$$

It should be noted that in the comparison above, the efficiency is assumed to be constant, a reasonable assumption because generally k_{OV} and Q_V are proportional in a wide range of flow rates. Working at a higher capacity by increasing both L and V does not change the stripping factor either.

Not taken into account is the influence of flow rate on the interphase area, higher flow rates may decrease bubble diameter and increase A_{LV} .

- f. Benzene may be recovered by cooling, however, at low concentrations it is standard practice to burn the traces.

Exercise 4.5

Given	$V = 5 \text{ mol s}^{-1}$	$M_{\text{air}} = 0.029 \text{ kg mol}^{-1}$	$T = 20^\circ\text{C}$, isothermal operation
	$M_{\text{NH}_3} = 0.017 \text{ kg mol}^{-1}$	$x_{\text{NH}_3} = 0.05$	fraction ammonia absorbed 90%
	$\Phi_L = 0.5 \text{ kg water s}^{-1}$	$\rho_L = 1000 \text{ kg m}^{-3}$	$M_{\text{air}} = 0.029 \text{ kg mol}^{-1}$
	$F_{25\text{raschig}} = 540 \text{ m}^{-1}$	$F_{25\text{hiflow}} = 125 \text{ m}^{-1}$	$P_{\text{tot}} = 1 \text{ bar}$

Find the flooding velocity for both types of packings

Thoughts Fig. 4.18 is to be used. To calculate u_{flood} at a given value of the packing factor, F_p , the volume flow rate and the density of both phases have to be calculated.
The largest vapor stream is found at the bottom of the column and the flooding velocity should be calculated at these conditions.

Solution $Q_L = 0.5 / 1000 = 5 \cdot 10^{-4} \text{ m}^3 \text{ s}^{-1}$, $Q_V = 5 \cdot 8.31 \cdot 293 / 10^5 = 0.122 \text{ m}^3 \text{ s}^{-1}$.

$$M_V = 0.95 \cdot 0.029 + 0.05 \cdot 0.017 = 0.028 \text{ kg mol}^{-1}.$$

$$\rho_V = \frac{10^5 \cdot M_V}{R \cdot T} = 1.166 \text{ kg m}^{-3}, \text{ hence the flow parameter} = \frac{Q_L}{Q_V} \sqrt{\frac{\rho_L}{\rho_V}} = 0.120.$$

$$\text{Read the ordinate in Fig. 4.18: } u_{\text{flood}} \sqrt{\frac{F_p}{g} \cdot \frac{\rho_V}{\rho_L}} = 0.30$$

$$u_{\text{flood}}(F_p = 540) = 1.18 \text{ m s}^{-1}, u_{\text{flood}}(F_p = 125) = 2.46 \text{ m s}^{-1}.$$

Exercise 4.6

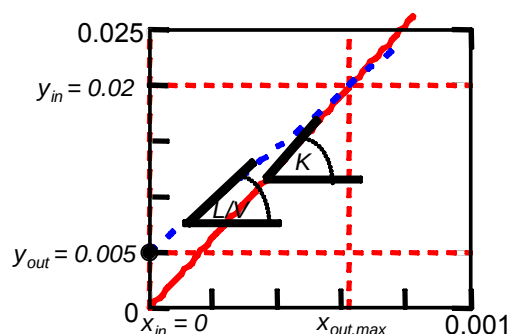
Given	$V = 2.25 \text{ mol s}^{-1}$	$M_{\text{air}} = 0.029 \text{ kg mol}^{-1}$	$M_{\text{water}} = 0.018 \text{ kg mol}^{-1}$
	$y_{\text{in}} = 0.02$	$y_{\text{out}} = 0.005$	$x_{\text{in}} = 0$
	$L = 65 \text{ mol water s}^{-1}$	$\rho_L = 1000 \text{ kg m}^{-3}$	$K_{\text{SO}_2} = 32$
	$R = 8.31 \text{ J mol}^{-1} \text{ K}^{-1}$	$P_{\text{tot}} = 1 \text{ bar}$	$T = 285 \text{ K}$, isothermal operation
	$H = 2.0 \text{ m}$	$D = 0.33 \text{ m}$	$\epsilon_{\text{bed}} = 0.90$
	$a = 200 \text{ m}^2 \text{ m}^{-3}$	$g = 9.81 \text{ m s}^{-2}$	

Find a. L_{min} b. N_{OV} c. k_{OV}
d. minimum flooding velocity
e. y_{SO_2} in effluent at 75% of maximum airflow rate

Thoughts a. translate figure in solution to exercise 4.4 (page 4.6) to *absorption* (position operating line!)
b. low concentration, constant flow, Equation 4.36 applicable, calculate absorption factor A
c. Eq. 4.32 relates H_{column} , k_{OV} (via H_{tu}) and N_{OV}
d. calculate the flow parameter and u_{flood} from the corresponding value at the ordinate in Fig. 4.18.
e. Just changing the air volume flow rate changes N_{OV} as well as the absorption factor.

Solution a. $L_{\text{min}} = \frac{V \cdot (y_{\text{in}} - y_{\text{out}})}{x_{\text{out,max}} - x_{\text{in}}} = \frac{V \cdot (y_{\text{in}} - y_{\text{out}})}{\frac{y_{\text{in}}}{K_{\text{SO}_2}} - x_{\text{in}}} = \frac{2.25 \cdot (0.02 - 0.005)}{\frac{0.02}{32} - 0} = 54.0 \text{ mol s}^{-1},$

the actual liquid flow rate is 20% higher.



- b. Absorption factor $S_{abs} = A = L/KV = 0.903$. Linear operating and equilibrium lines, hence Eq. 4.36 is applicable:

$$N_{OV} = \frac{0.903}{0.903-1} \cdot \ln \left[\frac{1}{0.903} + \frac{0.020-0}{0.005-0} \cdot \left(1 - \frac{1}{0.903}\right) \right] = 3.62$$

- c. From Eq. 4.32 $H_{tu} = H_{column}/N_{OV} = 2.0/3.62 = 0.552$ m.

By definition (Eq. 4.33): $k_{OV} = \frac{Q_V}{H_{tu} \cdot A_h}$ with

$$Q_V = \frac{V}{\rho_y} \frac{\text{mol/s}}{\text{mol/m}^3} = V \cdot \frac{RT}{P_{tot}} = 2.25 \cdot \frac{8.31 \cdot 285}{10^5} = \frac{2.25}{42.2} = 0.0533 \text{ m}^3 \text{ s}^{-1} \text{ and}$$

$$A_h = a \cdot \frac{\pi}{4} D^2 = 200 \cdot 0.0850 = 17.0 \text{ m}^2/\text{m}$$

$$k_{OV} = \frac{Q_V}{H_{tu} \cdot A_h} = \frac{0.0533}{0.552 \cdot 17.0} = 5.68 \cdot 10^{-3} \text{ m s}^{-1}.$$

- d. Estimation of the packing factor (page 4.24 – 4.25) $F_p = a/\varepsilon^3 = 274.3 \text{ m}^{-1}$.

$$Q_L = L \cdot M_{water}/\rho_L = 1.17 \cdot 10^{-3} \text{ m}^3 \text{ s}^{-1}.$$

$$\text{Flow parameter} = \frac{Q_L}{Q_V} \cdot \sqrt{\frac{\rho_L}{\rho_y \cdot M_{air}}} = 0.617 \rightarrow \text{ordinate in Fig. 4.18} = 0.172.$$

$$u_{flood} = \frac{0.172}{\sqrt{\frac{F_p}{g} \cdot \frac{\rho_y}{\rho_L}}} = 0.916 \text{ m s}^{-1}.$$

$$(u_{max} = 0.85 \cdot u_{flood} = 0.779 \text{ m s}^{-1}, \text{ corresponding to } 0.0662 \text{ m}^3 \text{ s}^{-1})$$

- e. Increasing the gas flow decreases $N_{OV} = H_{column}/H_{tu} = H_{column} \cdot k_{OV} \cdot A_h/Q_y$ and decreases the absorption factor $S_{abs} = A = L/KV$.

k_{OV} does not change, assuming A_h is constant.

$$Q_y = (\pi D^2/4) \cdot u_{flood} \cdot 0.75 = 0.0584 \text{ m}^3 \text{ s}^{-1}$$

$$H_{tu} = 0.605 \text{ m}, N_{OV} = 2/0.605 = 3.31$$

$$V = Q_y \cdot \rho_y = 0.0584 \cdot 42.2 = 2.466 \text{ mol s}^{-1}$$

$$S_{abs} = A = L/KV = 0.842$$

Now solve Eq. 4.36 for y_{out} : $y_{out} = 5.93 \cdot 10^{-3}$, which is almost 20% higher than the value at the lower gas flow.

Chapter 4: GENERAL DESIGN OF G/L CONTACTORS (continued)**Exercise 4.7**

Ground water with a flow of $0.009 \text{ m}^3\text{s}^{-1}$, consisting of water with a small amount (15 mg/l) dissolved MTBE, needs to be purified by reducing the MTBE content by 99.9%. This will be executed by stripping with (clean) air in an available existing column with 30 trays. The effective area of one tray is 1.10 m^2 . The air flow amounts $0.900 \text{ m}^3\text{s}^{-1}$. Laboratory experiments have shown that $k_{OV} = 0.012 \text{ m s}^{-1}$. Stripping conditions are 1 bar and 293 K and the process can be considered isothermal. At this temperature the equilibrium constant $K = 20$. For an air flow of $0.900 \text{ m}^3\text{s}^{-1}$ the area of the bubbles is 50 m^2 per m^2 tray area.

Other data: $M_{\text{air}} = 0.029 \text{ kg mol}^{-1}$, $M_{\text{water}} = 0.018 \text{ kg mol}^{-1}$, $M_{\text{MTBE}} = 0.088 \text{ kg mol}^{-1}$

$g = 9.81 \text{ m s}^{-2}$, $R = 8.31 \text{ J mol}^{-1} \text{ K}^{-1}$, $\rho_{\text{water}} = 1000 \text{ kg m}^{-3}$.

- How many theoretical stages are required for the requested reduction of 99.9%?
- Calculate the tray efficiency E_{MV}
- Show that for the given flows the column can achieve the requested reduction.
- Calculate the residual amount (ppm) of MTBE in the water flow leaving the column
- Show that the air flow is sufficiently low to avoid flooding (use figure 4.10)

Exercise 4.8

The gaseous product stream of an ethylene oxide reactor contains 2 mol% ethylene oxide in ethylene, which can be considered as a gas. Water is used to absorb the ethylene oxide at a temperature of 30°C and a pressure of 20 bar. The gaseous feed stream amounts 2500 mol/s and the water stream 3500 mol/s . At these operating conditions the distribution coefficient K of ethylene oxide equals 0.85 (molal base).

The absorption takes place in a packed column with a packing height of 20 m and a diameter of 3 m that contains pall rings with a specific area a of $115 \text{ m}^2/\text{m}^3$ and a packing factor F_p of 88 m^{-1} . $k_{OV} = 0.0025 \text{ m s}^{-1}$.

Other data:

$M_{\text{ethylene}} = 0.028 \text{ kg mol}^{-1}$, $M_{\text{water}} = 0.018 \text{ kg mol}^{-1}$, $M_{\text{ethylene oxide}} = 0.044 \text{ kg mol}^{-1}$

$g = 9.81 \text{ m s}^{-2}$, $R = 8.31 \text{ J mol}^{-1} \text{ K}^{-1}$, $\rho_{\text{water}} = 1000 \text{ kg m}^{-3}$.

- How many stages are required to absorb 99% of the ethylene oxide?
- Calculate the average gas flow (m^3/s) and average superficial gas velocity (m/s) in the column.

- c. Calculate the interface area per unit column height A_h (m²/m) for this column assuming that the whole packing surface is wetted.
- d. Show that for the given flows the column can achieve the requested 99% ethylene oxide absorption.
- e. How many equilibrium stages does the column contain and which fraction of the ethylene oxide can finally be absorbed?
- f. At which % of the flooding capacity is the available column operated? (use figure 4.18).

Exercise 4.9

A CO₂ rich gas stream from fermentation contains small amounts of ethanol, which can be recovered by absorption in water using a packed column (metal cascade mini ring packing) with a height of 5 meter and an effective diameter of van 2 meter.

Absorption conditions:

- constant temperature of 30°C
 - Gas feed flow (3800 m³/uur)
 - Gas composition: 98% CO₂ (molar mass 44 g/mol), 2% ethanol at 120 kPa
 - $K = 0.57$
 - $L/V = 2$
 - $a = 300$ m² per unit bed volume
- a. Determine the total number of gas transfer units (Nov)
 - b. Determine the overall mass transport coefficient (k_{ov})?
 - c. Calculate the residual amount of ethanol left in the gas stream after absorption?
 - d. Calculate the amount of water (m³/uur) required to remove 95% of the ethanol?
 - e. Flooding occurred as a gas velocity of 1 m/s. Determine the bed porosity of this type of packing. (Used figure 4.18).

Exercise 4.7

Given $Q_V = 0.900 \text{ m}^3 \text{ s}^{-1}$ $A_{\text{tray}} = 1.10 \text{ m}^2$ $P = 1 \text{ bar}$ $A = 50 \text{ m}^2 \text{ m}^{-2}$
 $Q_L = 0.009 \text{ m}^3 \text{ s}^{-1}$ $k_{OV} = 0.012 \text{ m s}^{-1}$ $T = 293 \text{ K}$
 $x_{in} = 15 \text{ ppm}$ $N_s = 30$ $K = 20$

$M_{\text{air}} = 0.029 \text{ kg mol}^{-1}$ $M_{\text{water}} = 0.018 \text{ kg mol}^{-1}$
 $M_{\text{MTBE}} = 0.088 \text{ kg mol}^{-1}$ $g = 9.81 \text{ m s}^{-2}$
 $R = 8.31 \text{ J mol}^{-1} \text{ K}^{-1}$ $\rho_{\text{water}} = 1000 \text{ kg m}^{-3}$.

Find a) Theoretical stages required for 99.9% reduction
 b) Tray efficiency EMV
 c) Show the column can achieve the requested reduction
 d) Residual MTBE amount (ppm) in the water flow leaving the column
 e) Show that air flow is sufficiently low to avoid flooding (Fig 4.10)

Solution a) In order to apply Eq. 3.34, the stripping factor $S = K \cdot V/L$ has to be calculated, where V and L are in mol/s.

$$V = Q_V \cdot \rho_y = Q_V \cdot P_{\text{tot}}/RT = 0.900 \cdot 101325/(8.31 \cdot 293) = 37.5 \text{ mol/s},$$

$$L = Q_L \cdot \rho_{\text{water}}/M_{\text{water}} = 500 \text{ mol/s}$$

and follows $S = K \cdot V/L = 1.5$.

$$f = \frac{S^{N_{ts}+1} - S}{S^{N_{ts}+1} - 1} = 0.999, \text{ hence } N_{ts} = 14.34 \text{ theoretical stages}^1.$$

b) The total LV-bubble interphase area $A_{LV} = A \cdot A_{\text{tray}} = 55 \text{ m}^2$

With Eq. 4.16 follows $N_{OV} = 0.0120 \cdot 55/0.90 = 0.733$ transfer units.

From Eq. 4.15 the resulting plate efficiency
 $E_{MV} = 1 - \exp(-0.733) = 0.520$

c) The real number of stages, N_s , is given by Eq. 4.11: $N_s = N_{ts}/E_o$.

Substitution of the values of S and E_{MV} gives $E_o = 0.570$ and $N_s = 25.1$.

$N_s < 30$, dus OK

d) Actual number of theoretical stages $N_{ts} = E_o \cdot N = 0.570 \cdot 30 = 17.1$

and the actual value of $f = \frac{S^{N_{ts}+1} - S}{S^{N_{ts}+1} - 1} = 0.99967$.

$$x_{out} = (1 - f) \cdot x_{in} = 0.0049 \text{ ppm} = 4.9 \text{ ppb}$$

¹⁾ = N_{th} in Eq. 4.21

- e) The flooding velocity determines the maximum airflow.

$$\text{The flow parameter} = \frac{Q_L}{Q} \sqrt{\frac{\rho_{\text{water}}}{\rho_V}} \quad \text{with } \rho_V = \rho_y \cdot M_{\text{air}} = 1.207 \text{ kg m}^{-3}$$

$$\text{and } \frac{Q_L}{Q} \sqrt{\frac{\rho_{\text{water}}}{\rho_V}} = \frac{0.009}{0.90} \sqrt{\frac{1000}{1.207}} = 0.2878 .$$

(2)

From Fig. 4.10 gasfactor = 0.015

Take $H_{\text{spacing}} = 0.5 \text{ m}$

$$u_{\text{flood}} = \frac{0.015}{\sqrt{\frac{1}{H_{\text{spacing}} \cdot g} \cdot \frac{\rho_V}{\rho_{\text{water}}}}} = 1.037 \text{ ms}^{-1} . \quad u_{\text{max}} = 0.85 \cdot 1.037 =$$

$$0.881 \text{ m s}^{-1} .$$

$$Q_{\text{max}} = u_{\text{max}} \cdot A_{\text{tray}} = 0.969 \text{ m}^3 \text{ s}^{-1},$$

which is 8% higher than the actual flow.

Exercise 4.8

<i>Given</i>	$Q_V = 2500 \text{ mol s}^{-1}$	$D = 3 \text{ m}$	$P = 20 \text{ bar}$	$a = 115 \text{ m}^2 \text{ m}^{-2}$
	$Q_L = 3500 \text{ mol s}^{-1}$	$k_{OV} = 0.0025 \text{ m s}^{-1}$	$T = 303 \text{ K}$	
	$x_{\text{in}} = 2 \text{ mol\%}$	$H = 20 \text{ m}$	$K = 0.85$	

$$\text{Methylene} = 0.028 \text{ kg mol}^{-1}$$

$$M_{\text{water}} = 0.018 \text{ kg mol}^{-1},$$

$$\text{Methylene oxide} = 0.044 \text{ kg mol}^{-1}$$

$$g = 9.81 \text{ m s}^{-2}$$

$$R = 8.31 \text{ J mol}^{-1} \text{ K}^{-1}$$

$$\rho_{\text{water}} = 1000 \text{ kg m}^{-3}.$$

- Find*
- Number of stages required to absorb 99% of the ethylene oxide
 - Average gas flow (m³/s) and average superficial gas velocity (m/s) in the column
 - Interface area per unit column height A_h (m²/m)
 - Show that the column can achieve 99% ethylene oxide absorption
 - Amount of equilibrium stages the column contains and fraction of the ethylene oxide that can finally be absorbed
 - % of the flooding capacity the column is operated? (Fig. 4.18)

Solution a) Absorption factor $S_{\text{abs}} = A = L/KV = (3500/2500/0.85) = 1.647$

Linear operating and equilibrium lines, hence Eq. 4.38 is applicable:

$$N_{OV} = \frac{1.647}{1.647 - 1} \cdot \ln \left[\frac{1}{1.647} + \frac{0.020 - 0}{0.0002 - 0} \cdot \left(1 - \frac{1}{1.647} \right) \right] = 9.38$$

$$b) \quad Q_v = V/p_y = V/(P_{tot}/RT) = 2500/(20 \times 10^5/(8.31 \cdot 303)) = 3.147 \text{ m}^3/\text{s}$$

$$\text{column cross sectional area} = \pi/4 D^2 = 7.069 \text{ m}^2$$

$$\text{superficial gas velocity } u_v = Q_v/A_k = 3.147/7.069 = 0.445 \text{ m/s}$$

$$c) \quad A_h = A_k \cdot a = 7.069 \cdot 115 = 813 \text{ m}^2/\text{m}$$

$$d) \quad H_{tu} = \frac{Q_v}{k_{ov} A_h} = \frac{3.147}{0.0025 \cdot 813} = 1.548(m)$$

$$\text{Column contains } 20/1.548 = 12.92 \text{ transfer units}$$

This is more than the required 9.38, so 99% can be achieved

$$e) \quad N_{ts} = N_{ov} \frac{A-1}{A \cdot \ln A} = 12.92 \frac{1.647-1}{1.647 \cdot \ln 1.647} = 10.2$$

$$f_{abs} = \frac{A^{N_{ts}+1} - A}{A^{N_{ts}+1} - 1} = 0.9976$$

$$f) \quad \text{The flow parameter } \frac{Q_L}{Q_v} \sqrt{\frac{\rho_{water}}{\rho_v}} = \frac{0.063}{3.147} \sqrt{\frac{1000}{22.24}} = 0.1342$$

$$\rho_v = p_y \cdot M_{air} = (P_{tot}/RT) \cdot y \cdot M_{ethene} = (20 \times 10^5/(8.31 \cdot 303)) \cdot 0.028 = 22.24 \text{ kg m}^{-3}$$

$$Q_L = L \cdot M_{water} / \rho_{water} = 3500 \cdot 0.018 / 1000 = 0.063 \text{ m}^3/\text{s}$$

From Fig. 4.18 gas factor ≈ 0.3

$$u_{v, flood} = \frac{0.30}{\sqrt{\frac{F_p}{g} \cdot \frac{\rho_v}{\rho_L}}} = \frac{0.30}{\sqrt{\frac{88}{9.81} \cdot \frac{22.24}{1000}}} = 0.672 \text{ ms}^{-1}$$

$$\% \text{Flood} = u_v / u_{v, flood} \cdot 100\% = 0.445 / 0.672 \cdot 100\% = 66\%$$

Exercise 4.9

<i>Given</i>	$Q_v=3800 \text{ m}^3 \text{ s}^{-1}$	$D=2 \text{ m}$	$P=20 \text{ bar}$
	$L/V=2$	$a=300 \text{ m}^2 \text{ m}^{-2}$	$T=303 \text{ K}$
	$x_{in}=2 \text{ mol\% EtOH}$	$H=5 \text{ m}$	$K=0.57$

- Find*
- Total number of gas transfer units (Nov)
 - Overall mass transport coefficient (kov)
 - Residual amount of ethanol left in the gas stream after absorption
 - Amount of water (m³/uur) required to remove 95% of the ethanol
 - Bed porosity of this type of packing. (Fig. 4.18)

- Solution*
- Ideal gas law $3800 \text{ m}^3/\text{uur} \rightarrow 181 \text{ kmol}/\text{uur} \rightarrow 7964 \text{ kilo}/\text{uur} \rightarrow 0.7 \text{ kg}/(\text{m}^2 \text{ s})$

HETP from figuur 4.15 $\rightarrow 0.32 \text{ m}$

$$H_{\text{column}} = N_{ts} H_{\text{etP}} \quad N_{ts} = 15.6$$

$$A=L/(KV)=3.5 \quad N_{ov} = N_{ts} \frac{A \ln A}{A-1} \rightarrow N_{ov} = 27.4$$

$$\text{b) } H_{\text{column}} = H_{tu} N_{ov} \rightarrow H_{tu} = 0.18 \text{ m}$$

$$H_{tu} = \frac{Q_v}{k_{ov} A_h}$$

$$A_h = 300 \text{ m}^2 \text{ per unit volume} \rightarrow 300 \times 3.14 = 942$$

$$k_{ov} = 6.2 \cdot 10^{-3} \text{ m/s}$$

$$\text{c) } f_s = \frac{A^{N_{ts}+1} - A}{A^{N_{ts}+1} - 1} = 1$$

$$\text{d) } f_s = \frac{A^{N_{ts}+1} - A}{A^{N_{ts}+1} - 1}$$

$$A=1.02$$

$$A=L/(KV)=1 \text{ so } L/V=0.57$$

$$Q_l=1896 \text{ kg/hr}$$

$$\text{e) } \frac{Q_l}{Q_v} \sqrt{\frac{\rho l}{\rho v}} = 1.89/3800 \times (1000/2.1)^{0.5} = 0.011$$

$$u_{v, \text{flood}} \sqrt{\frac{F_p \rho v}{g \rho l}} = 0.5$$

$$F_p=46.7=a/e^3=1168$$

$$e=0.64$$

Chapter 5: LIQUID-LIQUID EXTRACTION

Exercise 5.1

A water solution containing 0.005 mole fraction of benzoic acid is to be extracted by pure toluene as the solvent. If the feed rate is 100 moles/h and the solvent rate is 100 moles/h, find the number of equilibrium stages required to reduce the water concentration to 0.0001 mole fraction benzoic acid. Operation is isothermal and countercurrent.

The equilibrium represented by: mole fraction benzoic acid in water = $0.446 \times$ mole fraction benzoic acid in toluene.

Compare the McCabe-Thiele method with the analytical Kremser method.

Exercise 5.2

The system shown in Fig. 5.2.1 is extracting acetic acid from water using toluene as the solvent. A temperature shift is used to regenerate the solvent and return the acid to the water phase. The distribution coefficient of acetic acid between the toluene and water phase ($K_D = y_A/x_A$) amounts 0.033 at 25°C and 0.102 at 40°C.

The indicated number of stages refers to equilibrium stages.

- Determine y_1 and y_{N+1} for the extraction column
- Determine R' and x_N' for the regeneration column
- Is this a practical way to concentrate the acid?

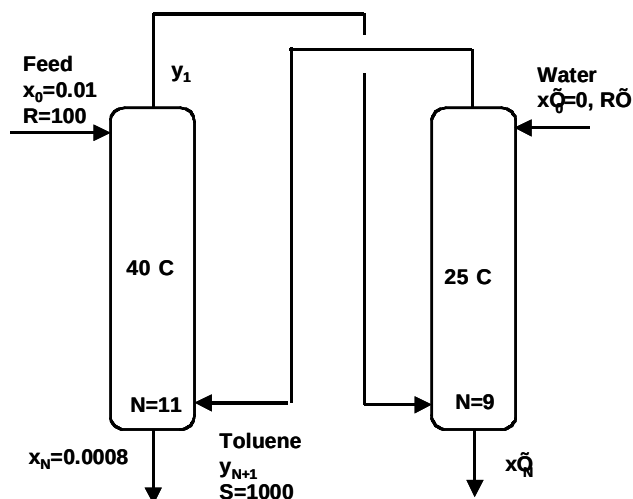


Figure 5.2.1. Extraction of acetic acid

Exercise 5.4

An 11.5 wt% mixture of acetic acid in water is to be extracted with 1-butanol at atmospheric pressure and 25°C in countercurrent operation. We desire outlet concentrations of 0.5 wt% in the water and 9.6 wt% in the butanol. Inlet butanol is pure. Find the number of equilibrium stages required and the ratio of water to 1-butanol by using the McCabe-Thiele diagram design method. The acetic acid equilibrium distribution is given by $K = 1.613$ (wtfr. in butanol/wtfr. in water). Required steps:

- Convert the streams and equilibrium data to mass ratio units
- Plot the equilibrium curve
- Plot the operating curve to determine the water to 1-butanol ratio
- Step off the equilibrium stages

Exercise 5.5

1000 kg per hour of a 45 wt% acetone in water solution is to be extracted at 25 °C in a continuous countercurrent system with pure ethyl acetate to obtain a raffinate containing 10 wt% acetone. Using the following equilibrium data, determine with the aid of a ternary diagram:

- The minimum flow rate of solvent
- number of stages required for a solvent rate equal to 1.5 times the minimum
- flow rate and composition of each stream leaving each stage

	Acetone wt%	Water wt%	Ethyl acetate wt%
<i>Extract</i>	60	13	27
	50	4	46
	40	3	57
	30	2	68
	20	1.5	78.5
	10	1	89
<i>Raffinate</i>	55	35	10
	50	43	7
	40	57	3
	30	68	2
	20	79	1
	10	89.5	0.5
	<i>Raffinate</i>	<i>Extract</i>	
	wt% acetone	w% acetone	
<i>Tie-line data</i>	44	56	
	29	40	
	12	18	

Exercise 5.7

Caprolactam is industrially recovered and purified by extraction with benzene. The flow diagram of this extraction section is shown in Fig. 5.7.1 on next page.

In the first column caprolactam is extracted from the aqueous phase. The initial concentration amounts 65 wt% caprolactam. The objective of this extraction is to obtain an aqueous raffinate stream with only minimal amounts of residual caprolactam.

- a. Determine the minimal required solvent flow for the first column.
- b. Determine the caprolactam concentration (wt%) in and the amount (ton/hr) of the extract stream when 1.5 time the minimal required benzene stream is used.
- c. When 15 ton/hr water is used for the extraction in the 2nd column, determine the final concentration of caprolactam in the water after both extractions.

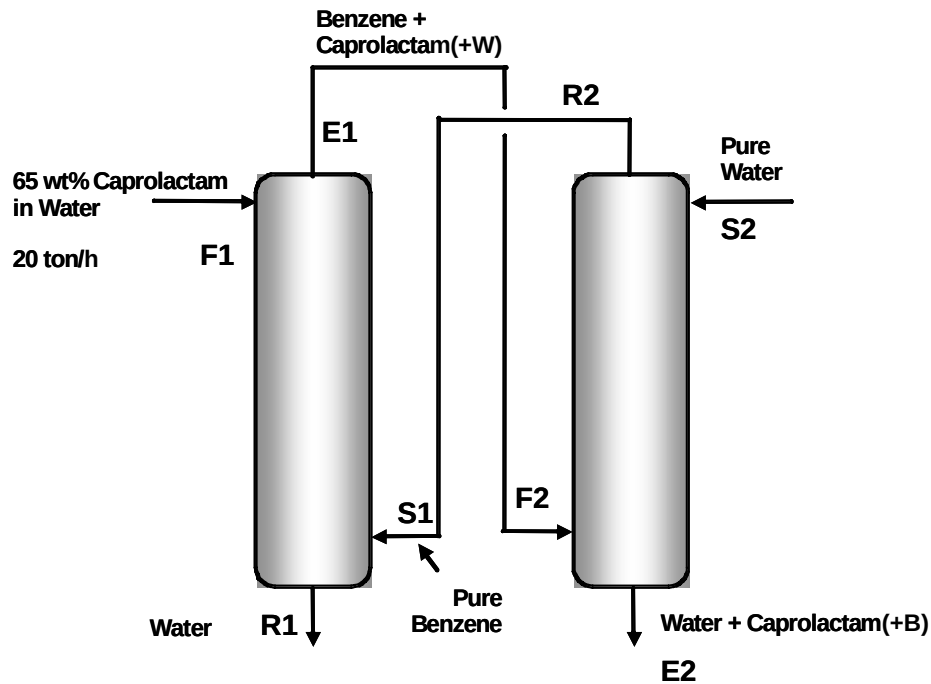


Figure 5.7.1. Extraction of caprolactam

Equilibrium data at 20°C are as follows:

Extract			Raffinate		
Caprolactam wt%	Water wt%	Benzene wt%	Caprolactam wt%	Water wt%	Benzene Wt%
0	0.0	100.0	0	99.9	0.1
1.5	0.1	98.4	9.5	90.3	0.2
4.0	0.2	95.8	18.6	81.0	0.4
8.2	0.4	91.3	28.2	71.1	0.7
10.9	0.7	88.4	37.0	61.6	1.3
16.2	1.2	82.6	45.9	51.0	3.0
23.6	2.4	74.0	53.4	39.5	7.1

Exercise 5.8

Suggest the extraction equipment that should be considered for:

- Extraction of large volumes of copper ore leachate with only 2 to 3 stages required
- Recovery of caprolactam from aqueous carrier stream in chemical plant
- Recovery of penicillin from an fermentation broth

Exercise 5.9

An aqueous stream F of 0.11 kg s^{-1} comprises of pyridine and water in equal weights. This stream should be purified in a counter current extraction process (see Fig. 5.9.1). The concentration of pyridine in the raffinate R_N should be reduced to 5 wt% (or less). Pure benzene is to be used as solvent S . The ternary equilibrium diagram of water-pyridine-benzene is given in Fig. 5.9.2.

- Calculate the minimum solvent stream, S_{min} .
- The solvent stream S is chosen to be 0.11 kg s^{-1} . Determine the value of flow E_1 .
- Determine the number of equilibrium separation stages, N .
- Determine the size and composition of the extract leaving the second stage, E_2 .

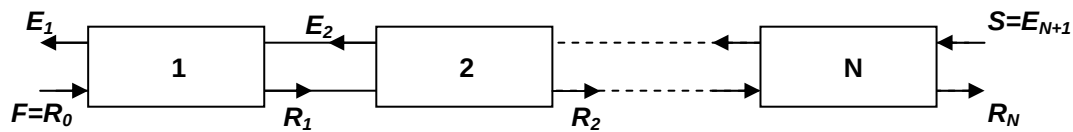


Figure 5.9.1. N -stages countercurrent extraction

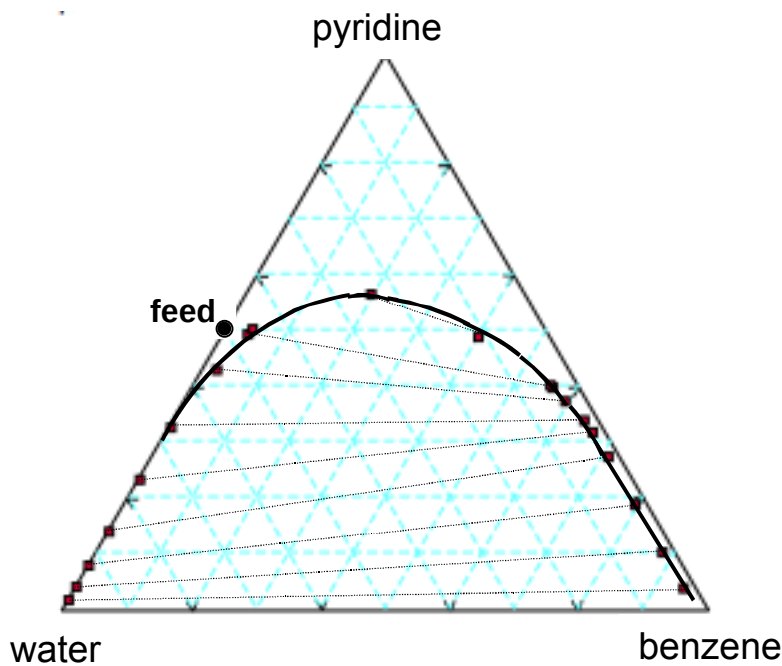


Figure 5.9.2. Equilibrium diagram water-benzene-pyridine, compositions in wt%.

SOLUTIONS

Exercise 5.1

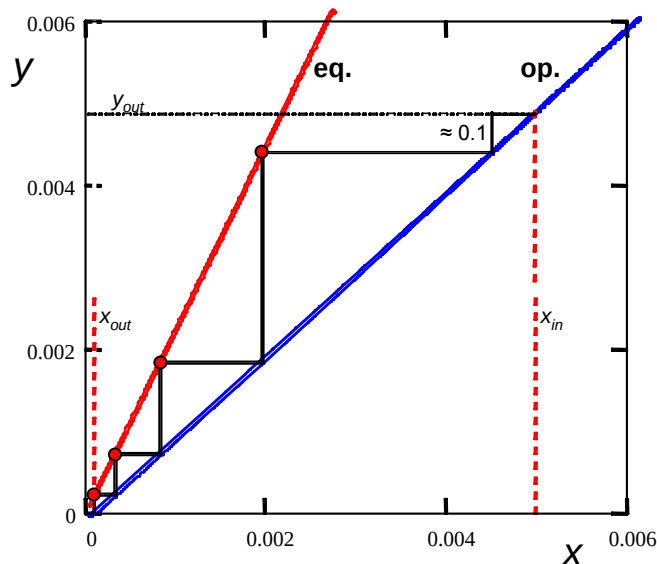
Given $x_0 = 0.005$ $y_{N+1} = 0$
 $x_N = 0.0001$ $F/S = 100/100 = 1$
 $x = 0.446 \cdot y$ ($K = 2.242$) isothermal operation

Find N , number of equilibrium stages, by the McCabe-Thiele method and the Kremser equation

Thoughts The equilibrium relationship is given, the operating line intersects (x_N, y_{N+1}) and has slope = 1. Both lines are straight, Kremser Eq. 5.33 is applicable with mole fractions. $E = K \cdot F/S = 2.242$.

Solution Number of stages from the Kremser equation: $N = 4.13$

$$N = \frac{\ln \left[\frac{x_{in} - y_{in}/K}{x_{out} - y_{in}/K} \left(1 - \frac{1}{E} \right) + \frac{1}{E} \right]}{\ln E} = \frac{\ln \left[\frac{0.005 - 0}{0.0001 - 0} \cdot \left(1 - \frac{1}{2.242} \right) + \frac{1}{2.242} \right]}{\ln 2.242} = 4.13$$



In the McCabe-Thiele diagram above, 4 equilibrium stages are plotted on the equilibrium line (the upper one), the remainder can be estimated as 0.1 equilibrium stage. The two solutions agree well.

Exercise 5.2

Given	<u>Extraction:</u>	<u>Regeneration:</u>
	$x_0 = 0.01$ $x_N = 0.0008$	$x'_0 = 0$
	$K_D = y_A/x_A = 0.102$ $T = 40^\circ\text{C}$	$K_D = y_A/x_A = 0.033$ $T = 25^\circ\text{C}$
	$N = 11$	$N = 9$
	$S = 1000$ (a.u.) $R = 100$ (a.u.)	

Find

- y_1 and y_{N+1} for the extraction column
- x'_N and R' for the regeneration column
- is this a practical way to concentrate the acid?

Thoughts The Kremser equation (Eq. 5.33) can be used because the solute concentrations are sufficiently low.

- The only unknown in the Kremser equation is $y_{in} = y_{N+1}$. Apply overall mass balance to extraction column with $y_{out} = y_1$ and $E = K \cdot S/R$.
- All 'y' (toluene) concentrations are known from the previous part, so the Kremser equation can be used once more. As the direction of mass transfer is changed, the Kremser equation should be adapted (compare Eq. 3.28 for absorption and Eq. 3.33 for stripping): $A = R'/K \cdot S$.
Note that $y_{out}(\text{extraction}) = y_{in}(\text{regeneration})$ and vice versa.
Alternatively, the phases x and y should be renamed and the equilibrium constant recalculated.

Solution a. Calculation of y_{in} and y_{out} in a MathCad work sheet:

$$E := K \cdot \frac{S}{R} \quad E = 1.02$$

$$\text{given} \quad N = \frac{\ln \left[\frac{1}{E} + \frac{x_{in} - \frac{y_{in}}{K}}{x_{out} - \frac{y_{in}}{K}} \left\{ 1 - \frac{1}{E} \right\} \right]}{\ln(E)} \quad y_{in} := \text{find}(y_{in}) \quad y_{in} = 5.996 \cdot 10^{-6}$$

$$\text{given} \quad R \cdot x_{in} + S \cdot y_{in} = R \cdot x_{out} + S \cdot y_{out} \quad y_{out} := \text{find}(y_{out}) \quad y_{out} = 9.26 \cdot 10^{-4}$$

- Adapted Kremser equation for the regeneration column with $x_{in} = 0$:

$$N = 9 = \frac{\ln \left[\left(1 - \frac{1}{A} \right) \frac{y_{in}}{y_{out}} + \frac{1}{A} \right]}{\ln A} \quad \text{with } A = \frac{R'}{KS} \text{ and } y = K \cdot x$$

Solving for A gives $A = 1.565 \rightarrow R' = K \cdot S \cdot A = 51.6$ (a.u.)

x'_N is calculated from the overall mass balance of the regeneration section:

$$S \cdot y'_{N+1} + R' \cdot x'_0 = S \cdot y'_1 + R' \cdot x'_N \rightarrow x'_N = 0.0178$$

- No. This operation requires two columns, a large amount of recycle toluene and a temperature shift. The resulting aqueous stream is still quite large and the concentration is hardly twice the feed concentration (0.01 resp. 0.018).

Exercise 5.4

<i>Given</i>	x_0	= 11.5 wt%	y_1	= 0 wt%
	x_N	= 0.5 wt%	y_{N+1}	= 9.6 wt%
	T	= 298 K	p	= 1 bar
	y/x	= 1.613 (weight fractions)		

Find The number of stages and the water-to-butanol ratio

Thoughts The steps that have to be taken are described in the assignment. Assume that conversion to weight ratio's assures a constant K -value in the given concentration range (see also Exercise 5.3). The solvent-to-feed ratio is the slope of the operating line.

Solution is given on page 5.9 as a MathCad worksheet. The straight line through the origin is the equilibrium line, almost 4 equilibrium stages are required for an acetic acid reduction from 11.5% to 0.5 wt%.
Check that the Kremser equation is also applicable, because both operating and equilibrium line are straight.

$$K := 1.613$$

$$x_{in} := 0.115$$

$$x_{out} := 0.005$$

$$y_{in} := 0$$

$$y_{out} := 0.095$$

$$X_{in} := \frac{x_{in}}{1 - x_{in}}$$

$$X_{out} := \frac{x_{out}}{1 - x_{out}}$$

$$Y_{in} := \frac{y_{in}}{1 - y_{in}}$$

$$Y_{out} := \frac{y_{out}}{1 - y_{out}}$$

$$X_{in} = 0.13$$

$$X_{out} = 5.025 \cdot 10^{-3}$$

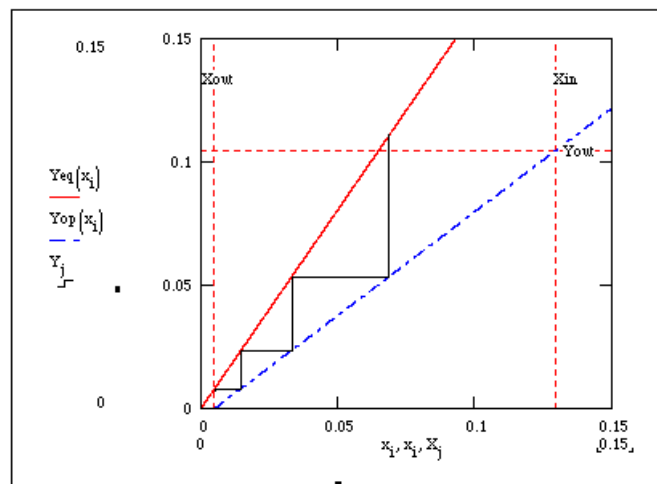
$$Y_{out} = 0.105$$

Operating line (solute free) through (X_{out}, Y_{in}) and (X_{in}, Y_{out})

$$\text{slope operating line } \text{slop} := \frac{Y_{out} - Y_{in}}{X_{in} - X_{out}} \quad \text{slop} = 0.84$$

$$Y_{op}(x) := \text{slop} \cdot (x - X_{out}) + Y_{in} \quad Y_{eq}(x) := K \cdot x \quad X_0 := X_{out}$$

$$X_{j+1} := \frac{K \cdot X_j - Y_{in}}{\text{slop}} + X_{out} \quad Y_j := K \cdot X_j$$



Exercise 5.5

Given Ternary phase diagram for acetone (AC)-water(H₂O)-ethyl acetate (EA), with tie lines
 Feed F : (AC-H₂O-EA) = (0.45; 0.55; 0)
 Desired raffinate: R_N = (AC-H₂O-EA) = (0.10; 0.895; 0.005)
 Solvent: pure EA = (AC-H₂O-EA) = (0; 0; 1)

Find

- minimum flow rate of solvent S_{min}
- number of stages N for $S = 1.5 \cdot S_{min}$
- flow rate and composition of each stream leaving each stage

Thoughts All possible indifference points P are located on the line through S and R_N .

- The minimum solvent flow rate S_{min} is found if an infinite number of equilibrium stages is used. In that case, the operating line through the feed point F and the first extract E_1^* is parallel to the nearest tie line; the point P_{min} can be found by extending the line through E_1^* and F . The corresponding mixing point M_{min} is located at the intersection of $F-S$ and $R_N-E_1^*$. The composition of E_1^* can be read from the chart. To calculate the compositions and flow rates, the overall material balance and component balances for two of the components have to be evaluated.
- The new mixing point M can be calculated from the mass balances. The first extract composition E_1 is found by extending the line through R_N and the new mixing point. The indifference point P is found by extending the line between F and E_1 . Now, R_1 is connected to E_1 by a tie line (equilibrium composition); E_2 is found from extending the operating line through P and R_1 , etcetera.

- c. The compositions can be read from the charts; the flow rates are calculated from the material balances.

Solution a. The composition of E_1^* can be read from the chart: (AC-H₂O-EA) = (0.57, 0.10, 0.33)
Material balances:

overall: $M = F + S_{\min} = E_1^* + R_N$

per component: $x_{AC,M} \cdot M = x_{AC,F} \cdot F + x_{AC,S} \cdot S = x_{AC,E} \cdot E + x_{AC,R} \cdot R \rightarrow$

$$0.45 \cdot 1000 + 0 = 0.57 \cdot E_1 + 0.10 \cdot R$$

$$x_{H_2O,M} \cdot M = x_{H_2O,F} \cdot F + x_{H_2O,S} \cdot S = x_{H_2O,E} \cdot E + x_{H_2O,R} \cdot R \rightarrow$$

$$0.55 \cdot 1000 + 0 = 0.10 \cdot E + 0.895 \cdot R$$

The flow rates of E and R can be solved from both component balances:

$$E = 695.3 \text{ kg/h and } R = 536.8 \text{ kg/h; } M = E + R = 1232.1 \text{ kg/h} \rightarrow$$

$$S_{\min} = 232.1 \text{ kg/h}$$

Location of point $M_{\min}$: $x_{AC,M} = 0.45 \cdot 1000/M = 0.365$ and $x_{H_2O,M} = 0.55 \cdot 1000/M = 0.446$

These values agree with the graphical result, see ternary diagram below.

- b. $S = 1.5 \cdot S_{\min} = 348 \text{ kg/h}$ and $M = 1000 + 348 = 1348 \text{ kg/h}$

$$x_{AC,M} = 0.45 \cdot 1000/M = 0.333 \text{ and } x_{H_2O,M} = 0.55 \cdot 1000/M = 0.408$$

Solve E and R from both material balances:

$$x_{AC,M} \cdot M = x_{AC,E} \cdot E + x_{AC,R} \cdot R \rightarrow 450 = 0.51 \cdot E + 0.10 \cdot R$$

$$x_{H_2O,M} \cdot M = x_{H_2O,E} \cdot E + x_{H_2O,R} \cdot R \rightarrow 550 = 0.04 \cdot E + 0.895 \cdot R$$

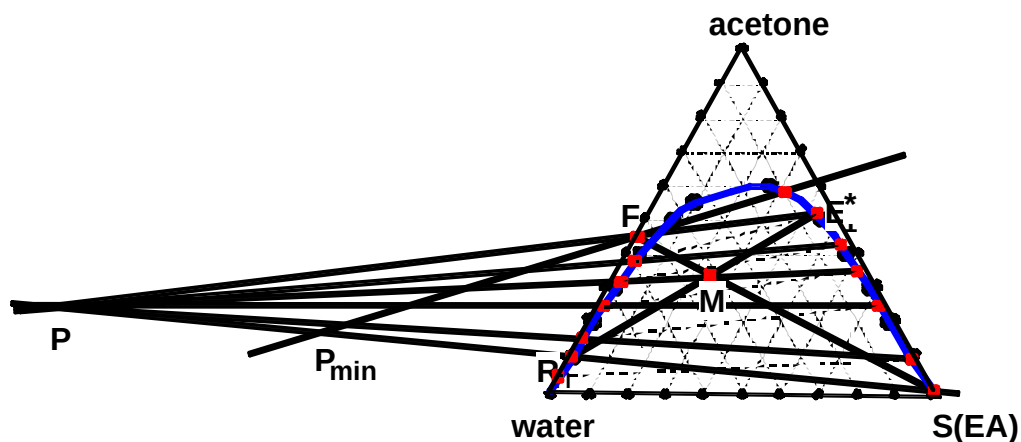
Results: $E = 768.6 \text{ kg/h}$ and $R = 580.2 \text{ kg/h}$ and the total $M = E + R = 1348.8 \text{ kg/h}$.

Continuing the graphical solution defined above (see diagram) or solve the material balances for each subsequent stage (see c), it follows that $N \approx 4.5$

- c. Use the material balance: $x_{R,N-1} \cdot R_{N-1} + x_{E,N+1} \cdot E_{N+1} = x_{R,N} \cdot R_N + x_{E,N} \cdot E_N$

The approximate results are given in the table below. A small error is made when reading the compositions from the chart, so the mass balances do not close completely.

Extr.	Ac	H ₂ O	EA	kg/hr	Raff.	Ac	H ₂ O	EA	kg/hr
E ₁	0.51	0.04	0.45	769	R ₁	0.38	0.59	0.03	914
E ₂	0.43	0.03	0.54	673	R ₂	0.32	0.66	0.02	809
E ₃	0.34	0.025	0.635	590	R ₃	0.24	0.74	0.02	714
E ₄	0.24	0.02	0.74	472	R ₄	0.15	0.84	0.01	622
E ₅	0.10	0.01	0.89	≈350	R ₅	0.05	0.945	0.005	≈500



Exercise 5.7

Given A caprolactam extraction process is given in Fig. 5.7.1 together with relevant equilibrium data.
 $F_1 = 20$ ton (C)aprolactam / h with $x_{F,C} = 0.60$; $x_{F,W} = 0$;
 $R_2 = S_1$; $F_2 = E_1$; $S_2 = 15$ ton W(ater) / h;

Find a. minimal solvent flow for first column
 b. concentration C(aprolactam) in E_1 for $S = 1.5 \cdot S_{min}$
 c. final concentration of C in E_2 .

Thoughts The solution can be obtained from a ternary diagram with equilibrium line, defining the two liquid phases region, and the node lines, representing particular raffinate – extract equilibria. Construct this diagram and mark the composition of the feed on the CW-axes with F .

Solution a. Draw a line through F parallel to the closest node line. Intersection of this line with the equilibrium line at the extract (benzene-rich) side gives the extract phase, E , with the highest possible concentration of C. This corresponds to the smallest amount of solvent B(enzene). The figure on the next page shows how the mixing point M is constructed. The solvent-to-feed ratio follows from the ratio of the segments FM and MB :

$$(S/F)_{min} = \frac{\text{feed} - M}{\text{benzene} - M} = 1.36, \text{ hence } S_{min} = 27.3 \text{ ton/h.}$$

b. $S = 1.5 \cdot S_{min} = 40.8$ ton B/h; $W = 0.4 \cdot F = 0.4 \cdot 20 = 8.0$ ton/h; $C = 0.6 \cdot F = 12.0$ ton/h, giving a total of 60.8 ton/h.

The mixing point M in the first column now has the following overall composition:

$x_{M,B} = 40.8 / 60.8 = 0.67$; $x_{M,W} = 8 / 60.8 = 0.13$ and $x_{M,C} = 0.20$. Plot point M , extend the line from W through M to the extract side of the equilibrium curve and read the composition of E_1 : $x_B = 0.76$; $x_W = 0.02$ and $x_C = 0.22$.

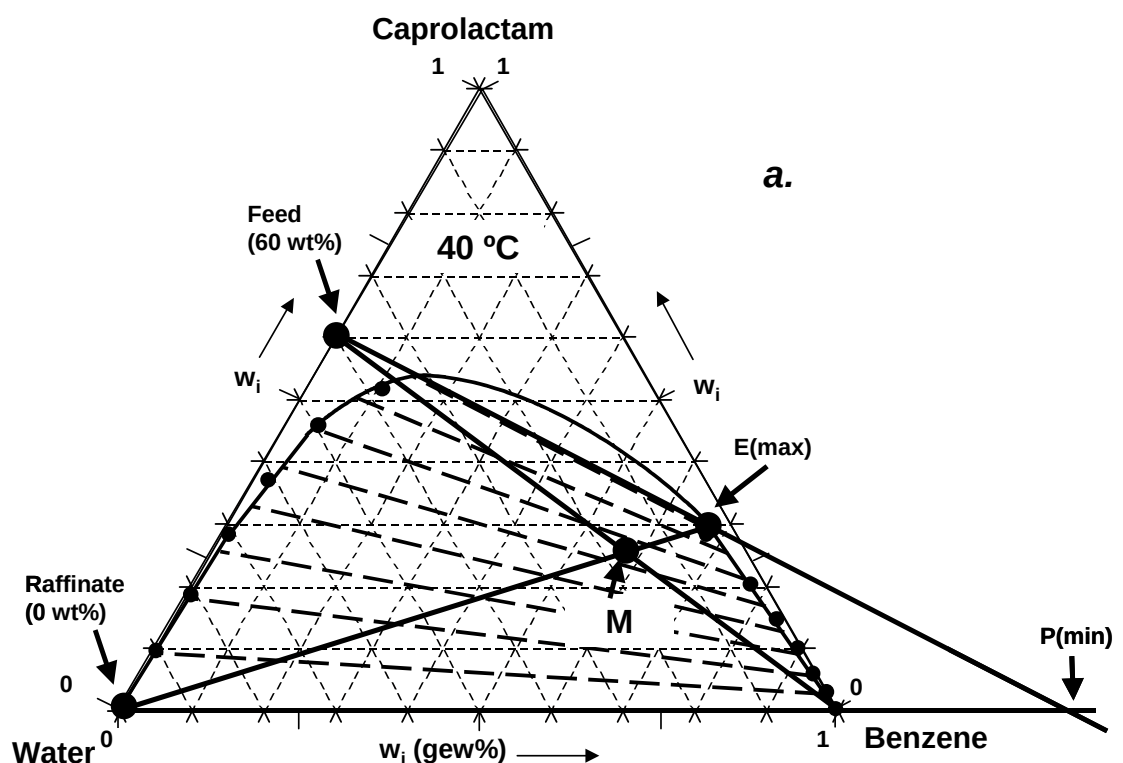
This corresponds to 40.8 (B) + $0.02 \cdot 40.8 / 0.76$ (W) + $0.22 \cdot 40.8 / 0.76$ (C) = 53.7 ton E_1 /h.

c. $B = 40.8$ ton/h; $W = 1.1 + 15.0 = 16.1$ ton/h; $C = 11.8$ ton/h, giving a total of 68.7 ton/h.

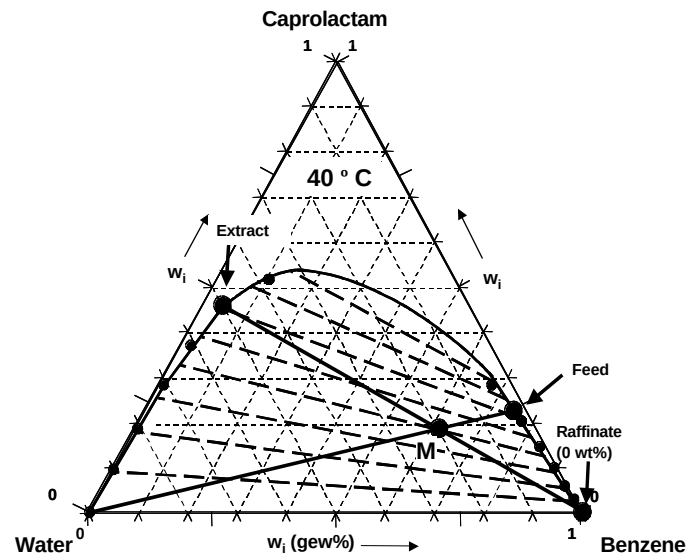
The mixing point M in the 2nd column now has the following overall composition:

$x_{M2,B} = 40.8 / 68.7 = 0.594$; $x_{M2,W} = 16.1 / 68.7 = 0.197$ and $x_{M2,C} = 11.8 / 68.7 = 0.172$.

Extend the line BM and read the composition of E_2 : $x_B = 0.04$; $x_W = 0.50$; $x_C = 0.46$



5.7c.



Exercise 5.8

Given Three extraction cases:

- extraction of large volumes of copper ore leachate with only 2 to 3 stages required,
- recovery of caprolactam from aqueous carrier stream,
- recovery of penicillin from a fermentation broth

Find A suitable piece of extraction equipment for each case

Thoughts Apply the scheme in Fig. 5.18 on page 5-24 of Fund. Ind. Sepns.

- Solution**
- Extraction of large volumes of copper ore leachate with only 2 to 3 stages required*
Large volume; no extreme risk of emulsification; small number of stages; depending on the available space in the facility, either a series of mixer-settlers (quite common for the large volumes in metallurgy) or a column separator. A centrifugal extractor is probably too expensive for a relatively simple, large-volume separation.
 - Recovery of caprolactam from aqueous carrier stream.*
Large volume; potential emulsion formation ($\rightarrow$ centrifugal extractors or certain columns); probably more difficult separation ($N > 5$); small floor area (common in chemical plants); a column extractor is most useful. More data are required for the selection of the most appropriate column.
 - Recovery of penicillin from a fermentation broth*
Volume will be relatively low $\rightarrow$ separators or centrifugal extractors. The material to be extracted is relatively unstable and vulnerable; therefore the short residence time in a centrifugal extractor is very advantageous.

Exercise 5.9

Given Countercurrent extraction of pyridine (50 wt%) from an aqueous feed F (0.11 kg s^{-1}) with benzene.

- Find**
- Minimum solvent flow S_{min} for the process to work,
 - Extract flow E_1 leaving the first stage for $S = F$
 - The number of equilibrium extraction stages, N .
 - Size and composition of E_2 .

- Thoughts**
- Firstly, find E_{min} leaving the 1st stage by drawing a straight line through F parallel to the nearest node line. Then, calculate S_{min} from the overall mass balance: $F + S_{min} = R_N + E_{min}$
 - Apply the overall mass balance: $F + S = R_N + E_1$ and an appropriate component balance
 - A graphical solution: Mark the intersection of the extensions of $F-E_1$ and R_N-S . This is the operating point P . The difference in flows between any two subsequent stages ($F - E_1$, $R_1 - E_2$, $R_N - E_{N+1} = R_N - S$) all point to this operating point. R_1 follows from E_1 via the appropriate node line (equilibrium). $R_1 - E_2$ points to P , hence E_2 is found as the intersection of R_1-P and the phase equilibrium line.
 - Repeat b for the stage number 1.

- Solution**
- Three unknowns, S_{min} , E_{min} , R_N , hence three equations are required:

$$\text{overall} \quad F + S_{min} = R_N + E_{min}$$

$$\text{pyridine} \quad xF_{pyr} \cdot F = xE_{pyr} \cdot E_{min} + xR_{pyr} \cdot R_N$$

$$\text{benzene} \quad S_{min} = xE_{ben} \cdot E_{min}$$

$xF_{pyr} = 0.50$ (given), $xR_{pyr} = 0.05$ (required), $xE_{pyr} = 0.40$ and $xE_{ben} = 0.56$ (read from the ternary diagram). Solve for S_{min} , E_{min} and R_N .

$$S_{min} = 0.073 \text{ kg s}^{-1} \quad (E_{min} = 0.131 \text{ and } R_N = 0.052 \text{ kg s}^{-1})$$

Alternatively, a graphical solution leads to approximately the same result. Draw $F-S_{min}$ and R_N-E_{min} . The intersection is the mixing point M (see Fig. 5.9.2A).

$$\text{Lever rule: } S_{min}/F = FM/MS_{min} = 0.644. \quad S_{min} = 0.071 \text{ kg s}^{-1}.$$

- $S = F$, hence the new mixing point M is exactly halfway F and S_{min} (see Fig. 5.9.2B). Draw a straight line from R_N to M and extend until its intersection with the phase line. This gives point E_1 . Lever rule: $E_1/(E_1 + R_N) = MR_N/E_1R_N = E_1/(F + S)$, $E_1 = 0.173 \text{ kg s}^{-1}$.

Alternatively, the unknowns S_{min} and R_N can be calculated from the overall balance and the pyridine balance:

$$\text{overall} \quad F + S = R_N + E_1$$

$$\text{pyridine} \quad xF_{pyr} \cdot F = xE_{pyr} \cdot E_1 + xR_{pyr} \cdot R_N$$

$$xE_{pyr} \approx 0.31 \text{ (read from diagram), solving for the two unknowns gives } E_1 = 0.17 \text{ kg s}^{-1}.$$

$$xE_{ben} \approx 0.66 \text{ (from diagram) or } xE_{ben} = S/E_1 = 0.64$$

- Repeat the procedure outlined above to find, respectively, R_2 , E_3 , R_3 etcetera, until $R_n \leq 0.05$. Following this procedure, $N_{ts} \approx 3$.

- The unknowns R_1 and E_2 can be calculated from two equations, e.g.:

$$\text{overall} \quad F + E_2 = E_1 + R_1$$

$$\text{pyridine} \quad xF_{pyr} \cdot F + xE_{2pyr} \cdot E_2 = xE_{1pyr} \cdot E_1 + xR_{pyr} \cdot R_1$$

With $xR_{pyr} \approx 0.24$ and $xE_{2pyr} \approx 0.10$ (read from the diagram) it follows that $E_2 = 0.12 \text{ kg s}^{-1}$ ($R_1 = 0.06 \text{ kg s}^{-1}$).

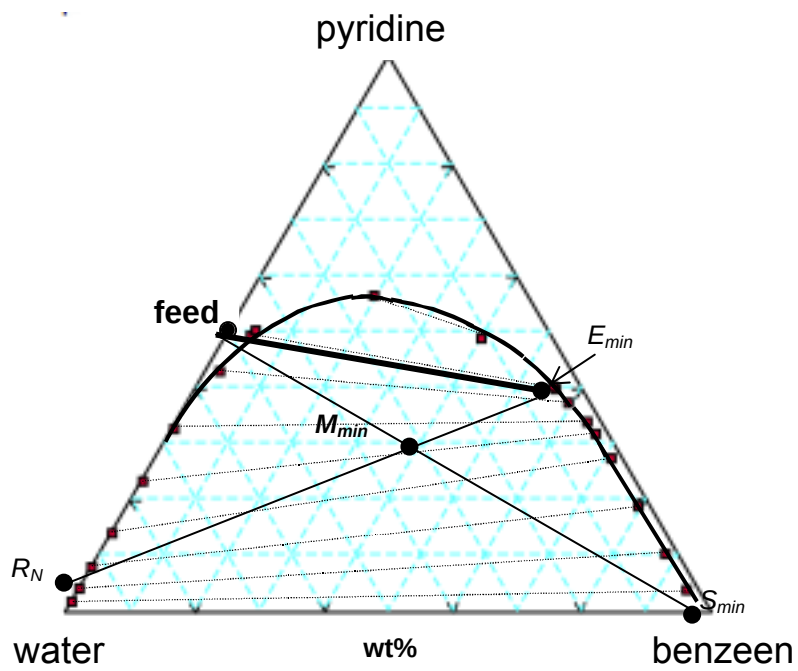


Fig. 5.9.2A

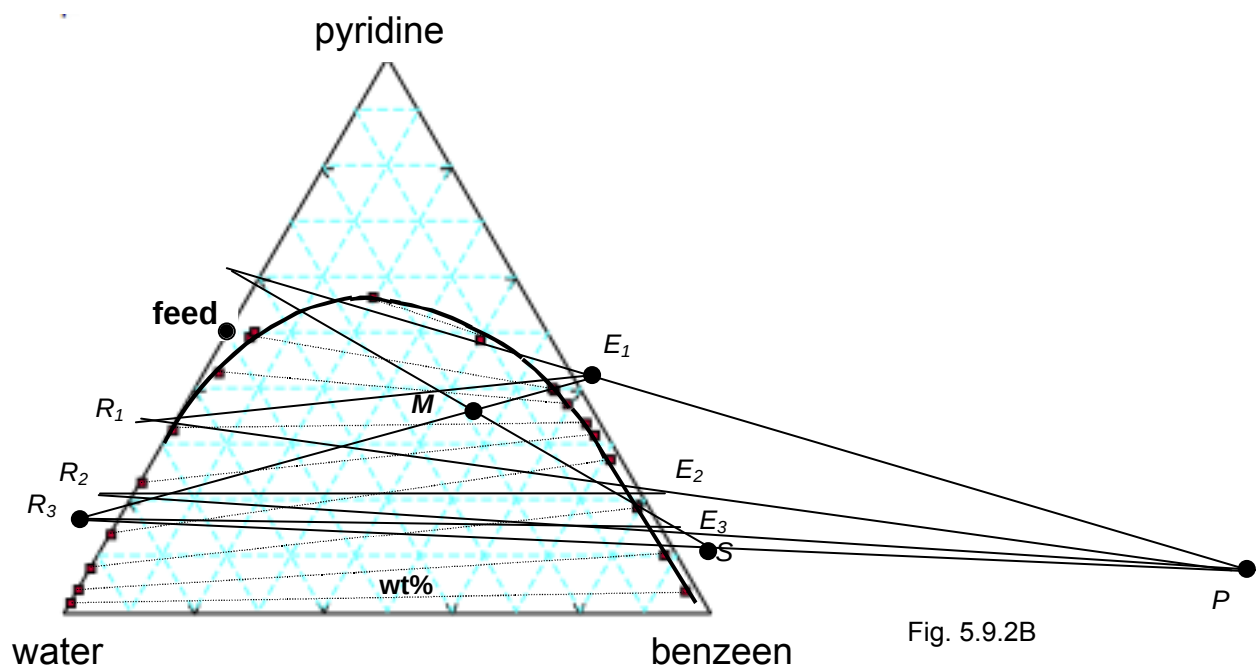


Fig. 5.9.2B

Chapter 5: LIQUID-LIQUID EXTRACTION (continued)

Exercise 5.3

Phenol is a toxic chemical that is used in the chemical industry for the production of resins. It has a solubility in water of approximately 8 g/ 100g water, and for environmental reasons it is important to treat process waters. Consider a 1000 kg aqueous process water stream with 8 wt% phenol that is to be treated. Because of its immiscibility with water, heptane is considered as solvent. The heptane is phenol free. The distribution coefficient of phenol at low concentrations is constant and has a value of $K_D = 0.2$ (wtfr. phenol in heptane/wtfr. phenol in water).

Calculate the solvent requirements for removing 99% of the phenol in:

- A single batch extraction
- Two crosscurrent stages using equal amounts of heptane
- Two countercurrent stages
- An infinite number of crosscurrent stages
- An infinite number of countercurrent stages

Exercise 5.6

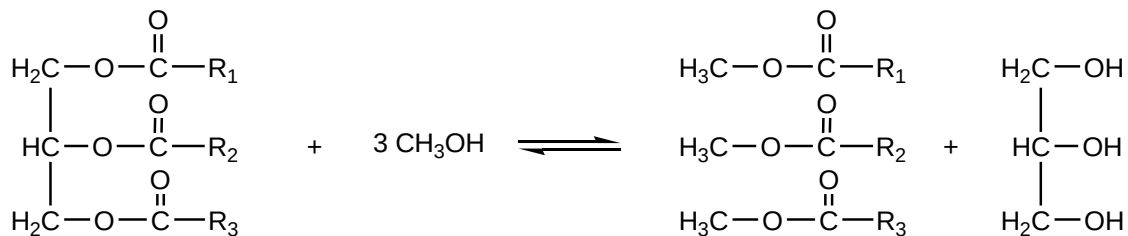
Under the umbrella of “green chemistry” also chemicals produced by fermentation are considered. This means that instead of fossil oil as feedstock, micro-organisms are applied to transform a biobased feedstock into valuable chemicals. For example organic waste can be used to make new chemistry out of waste. One example is the production of small carboxylic acids, volatile fatty acids. The smallest of them is formic acid with formula HCOOH . With extraction, the formic acid can be removed from the fermentation broth. In this exercise, octanol is applied. The ternary equilibrium data is given in the table:

Raffinate phase weight fractions			Extract phase weight fractions		
octanol	water	Formic acid	octanol	Water	Formic acid
0.0001	0.9998	0	0.667	0.333	0
0.0006	0.975	0.025	0.596	0.361	0.043
0.0017	0.948	0.05	0.521	0.393	0.086
0.0041	0.918	0.078	0.442	0.431	0.127
0.009	0.884	0.107	0.358	0.477	0.465
0.02	0.838	0.142	0.265	0.537	0.198

- Using this data, construct the ternary diagram, and determine for a 10 wt% formic acid in water solution, what would be the maximum concentration of formic acid that you can obtain in the extract phase when you use pure octanol to recover 90% of the formic acid.
- Determine the minimum solvent to feed ratio
- How many stages are required in an extraction process with a realistic solvent to feed ratio?

Exercise 5.10

In the past decade, due to governmental regulations, a large number of biodiesel production facilities has been built in Europe. In a typical process triglycerides from plant oils are converted into fatty acid methyl esters (FAME, or biodiesel), see the reaction scheme. From the scheme you can see that also glycerol is formed as byproduct of the biodiesel.



Such transesterification reactions are typically equilibrium-limited and therefore in batch processes typically a sixfold (molar basis) of methanol is applied. The process is essentially biphasic and the excess of the methanol distributes over the glycerol phase and the biodiesel phase. Most of the methanol is in the polar phase, but a water wash is necessary to remove unconverted methanol from the biodiesel phase, see process scheme.

- In Figure 2, the ternary diagram for biodiesel/water/methanol is given. Explain, on the basis of the ternary diagram, whether or not you think water is a suitable solvent to wash methanol from biodiesel.
- A new factory should produce 50 tons of biodiesel per hour, furthermore:
 - The product stream contains 1/9 weight fraction of methanol
 - The water used for washing initially does not contain any methanol
 - The aqueous extract contains 50 wt% methanol
 - The maximum allowed raffinate concentration is 1 wt% methanol

How many m³ wash water does the plant need per day?

- How many extraction stages are necessary to reach the desired purity of the raffinate?
- Which separation technology do you advise to remove the methanol from the wash water?

Exercise 5.3

Given Feed: 8 wt% phenol, 92 wt% water $T = 25^{\circ}\text{C}$
 Solvent: 100 wt% heptane Isothermal operation
 $K = 0.2$ (wt fraction phenol in heptane / phenol in water)
 Water and heptane immiscible

Find The solvent/feed ratio for 99% phenol removal for a number of process configurations

Thoughts Weight ratios have to be used because the concentrations are rather large. When using weight ratio's, the value of $K = 0.2$ can still be used

Phenol in = 80 kg per 1000 kg feed, phenol out = $(1 - 0.99) \cdot 80 = 0.8$ kg per unit time.

$$X_{out}/X_{feed} = (0.8/920)/(80/920) = 0.01.$$

The extraction factor is defined in Eq. 5.1, single batch in Eq. 5.9, crosscurrent operation in Eqs. 5.16 and 5.17 and countercurrent operation in Eq. 5.23a.

Solution In all cases, $X_{out}/X_{feed} = 0.01$ and $K = 0.2$

The solvent-to-feed ratio follows from the extraction factor:

$$E = K \cdot (S/F) \quad \leftrightarrow \quad (S/F) = E/K$$

a) $\frac{X_{out}}{X_{feed}} = \frac{1}{1+E} = 0.01$

thus $E = 99$ and $S/F = E/K = 99/0.2 = 495$

b) Two crosscurrent stages, equal amounts of heptane.
 In each stage, half the total amount of heptane is used. (Eq. 5.16)

$$\frac{X_{out,2}}{X_{feed}} = \frac{1}{(1+E/2)^2} = 0.01$$

thus $(1+E/2)^2 = 1/0.01 = 100$, $E = 18$ and $S/F = 18/0.2 = 90$

c) Two countercurrent stages (Eq. 5.23a): $\frac{X_{out,2}}{X_{feed}} = \frac{1}{1+E+E^2} = 0.01$

$E = 9.46$ and $S/F = 9.46/0.2 = 47.3$

d) Infinite number of cross-current stages (Eq. 5.17):

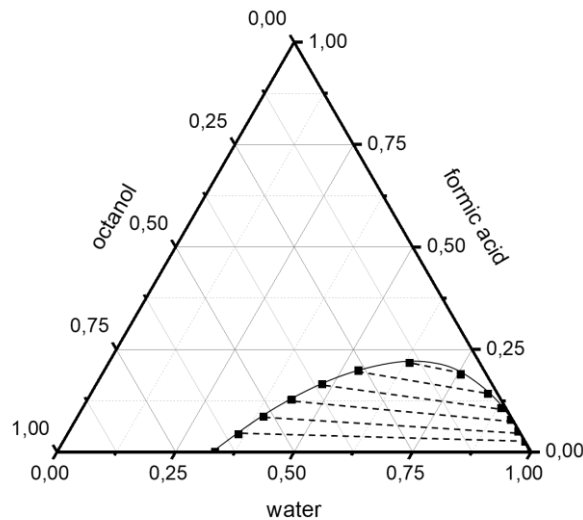
$$\frac{X_{\infty}}{X_{feed}} = \frac{1}{\exp(E)} = 0.01; \quad E = 4.6 \text{ and } S/F = 23.0$$

e) For infinite number of stages you can take

$$\lim_{N \rightarrow \infty} \frac{1}{\sum_{n=0}^{\infty} E^n} = 1 - E = 0.01 \text{ it follows that } E = 0.99 \text{ and } S/F = 4.95$$

Exercise 5.6

Given The ternary data is given in the table. On every line in the table a tie-line is given. see the figure:

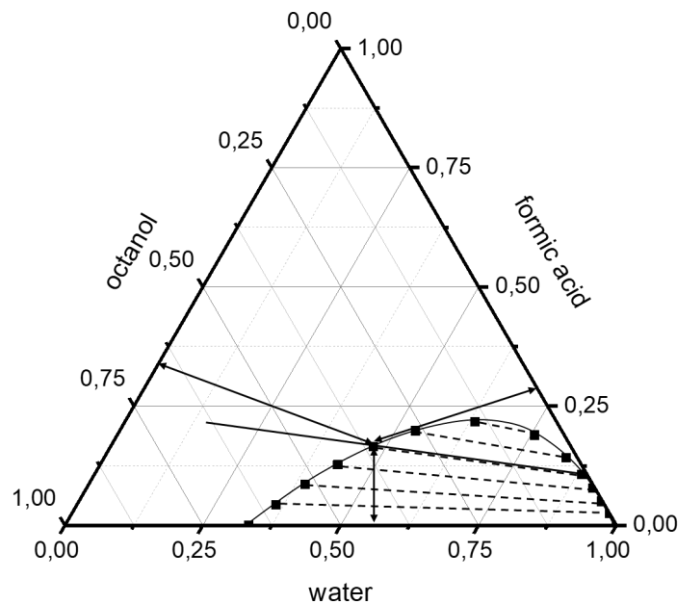


- Find*
- Construct the ternary diagram, and determine for a 10 wt% formic acid in water solution, what would be the maximum concentration of formic acid that you can obtain in the extract phase when you use pure octanol to recover 90% of the formic acid.
 - Determine the minimum solvent to feed ratio
 - How many stages are required in an extraction process with a realistic solvent to feed ratio?

Solution a) Draw the line from the formic acid side on the triangle at $x = 0.10$ parallel to the closest tieline to end up at the extract point at the minimum S/F ratio:

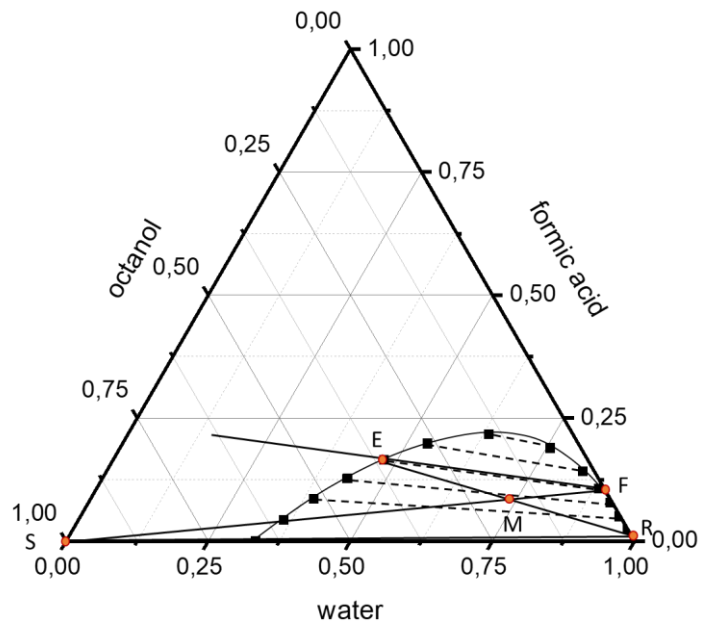
For that point, you read the composition by measuring the distances to the opposite side of the triangle. ($x_w = d_w / (d_w + d_{FA} + d_{oct})$ and $x_{FA} = d_{FA} / (d_w + d_{FA} + d_{oct})$ and $x_{oct} = d_{oct} / (d_w + d_{FA} + d_{oct})$)

$$\begin{aligned} x_w &= 0.48 \\ x_{FA} &= 0.16 \\ x_{oct} &= 0.36 \end{aligned}$$



- b) For this, you should consider what is the point S for octanol (pure) and the raffinate composition (90% extraction, thus 1% left).

You can then draw the cross from F to S and from R to E' (at $S/F_{\min}$):

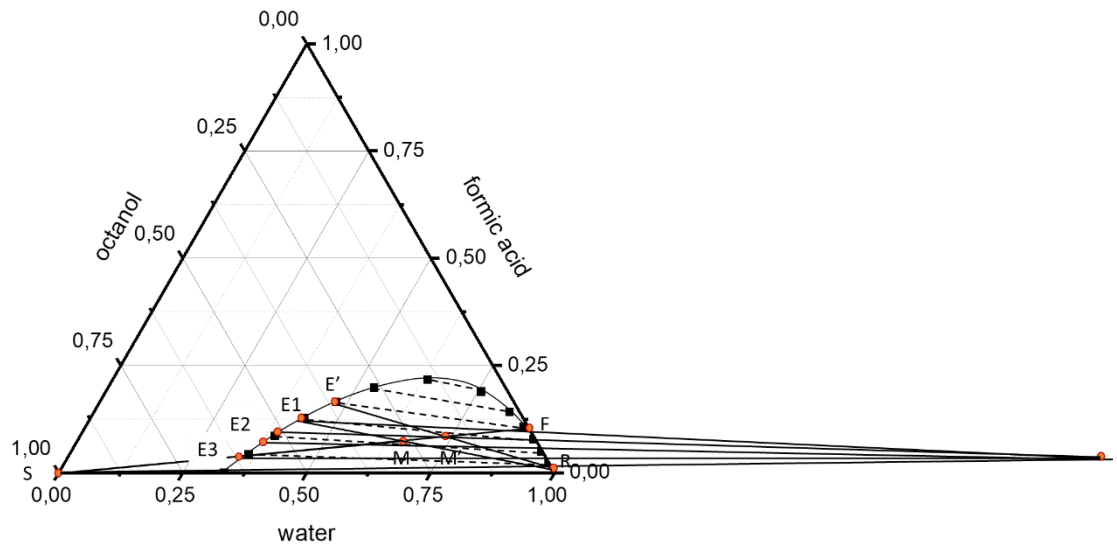


The minimum solvent to feed ratio follows as
 $\text{length}(FM)/\text{length}(SM) = 0.26$

- c) A realistic $S/F = 1.5 (S/F_{\min}) = 0.39$

Then you draw the new mixing point on SF, and E1 from R through M. next from E1 to R1 you go parallel to the closest tie-line.

Then from the working point, in which E1F and SR cross each other, you go through R1 to find E2, and after E3, the line parallel to the closest tieline goes through R. thus three stages are needed.

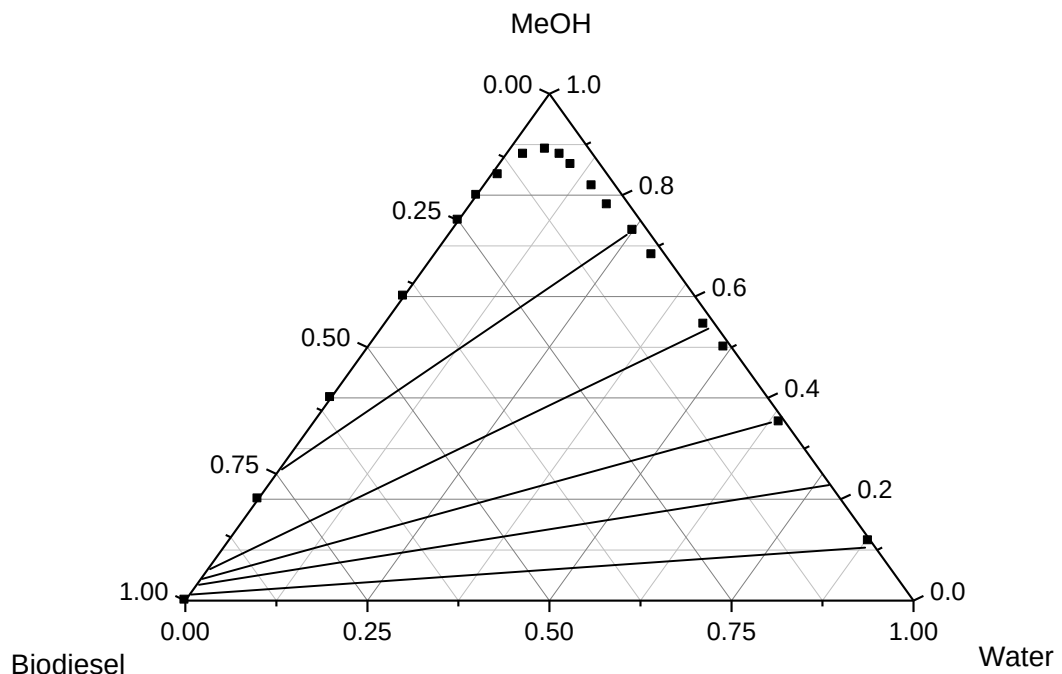


Exercise 5.10*Find*

- a) In Figure 2, the ternary diagram for biodiesel/water/methanol is given. Explain, on the basis of the ternary diagram, whether or not you think water is a suitable solvent to wash methanol from biodiesel.
- b) A new factory should produce 50 tons of biodiesel per hour, furthermore:
1. The product stream contains 1/9 weight fraction of methanol
 2. The water used for washing initially does not contain any methanol
 3. The aqueous extract contains 50 wt% methanol
 4. The maximum allowed raffinate concentration is 1 wt% methanol
- How many m³ wash water does the plant need per day?
- c) How many extraction stages are necessary to reach the desired purity of the raffinate?
- d) Which separation technology do you advise to remove the methanol from the wash water?

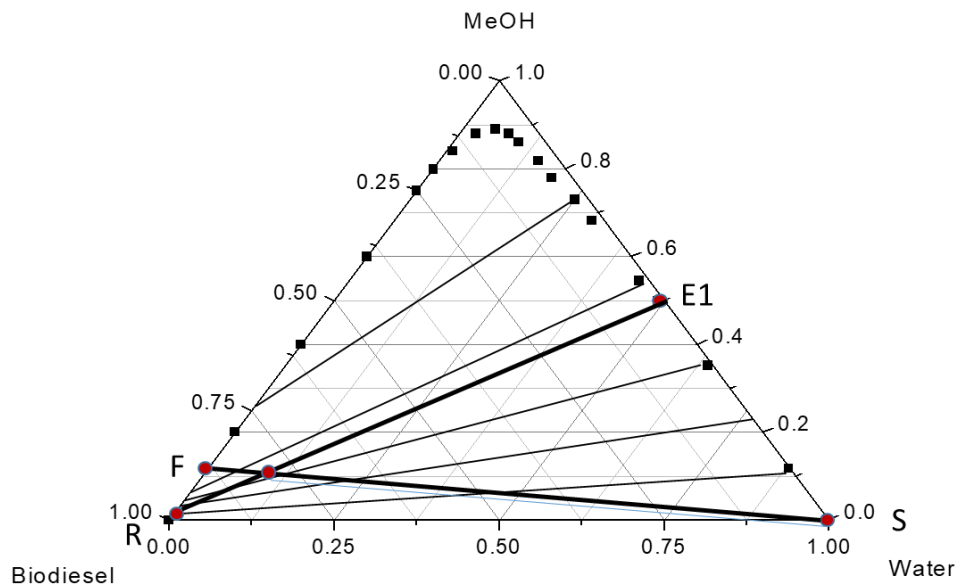
Solution

- a) Based on the tielines you can see whether a solvent is a good solvent or not. Furthermore, when the miscibility gap between the two phases is large, this is beneficial.



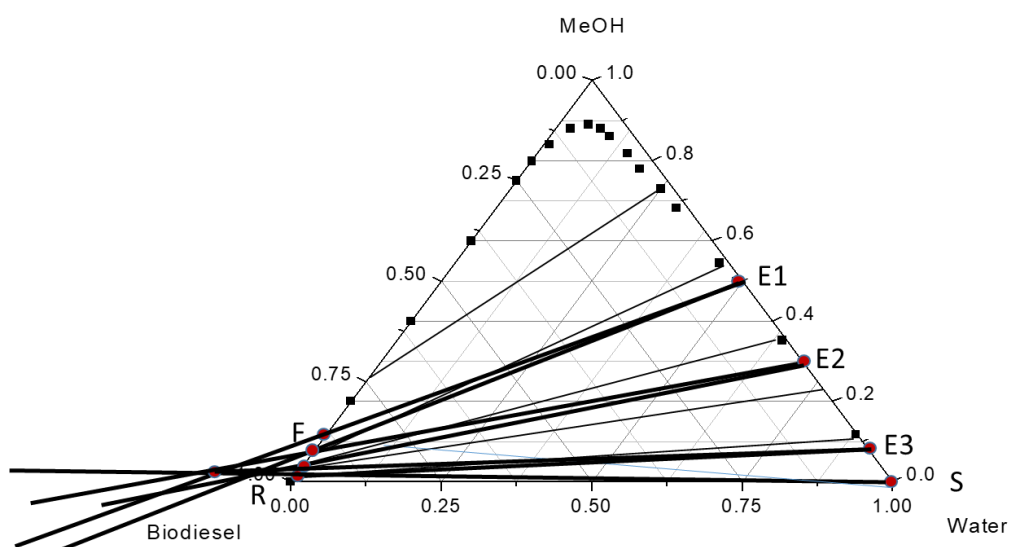
Since the tielines go relatively steep, much higher concentrations will be expected in water than in biodiesel and hence water would be a good solvent. Furthermore, water and biodiesel are almost completely immiscible.

- b) Based on the information given, you can draw the points F, E1, R and S, and the lines FS and E1R:



The ratio of the distances from F to the mixing point and S to the mixing point is the solvent to feed ratio: $SF = FM/SM = 0.09$. Since the feed rate is 50 ton per hour biodiesel, to which 11% methanol is added, the total feed is 55.5 ton/h. with a solvent to feed ratio of 0.09, this results in 5.1 t/h water.

- c) Use the E1F and SR crossing point as working point, then draw the stages, three stages are needed:



- d) Considering the difference in boiling point, distillation could be practiced

Chapter 6: ADSORPTION & ION EXCHANGE

Exercise 6.1

The following table gives the Langmuir isotherm constants for the adsorption of propane and propylene on various adsorbents.

Adsorbent	Adsorbate	q_m (mmol/g)	b (1/bar)
ZMS 4A	C ₃	0,226	9,770
	C ₃ ⁼	2,092	95,096
ZMS 5A	C ₃	1,919	100,223
	C ₃ ⁼	2,436	147,260
ZMS 13X	C ₃	2,130	55,412
	C ₃ ⁼	2,680	100,000
Activated Carbon	C ₃	4,239	58,458
	C ₃ ⁼	4,889	34,915

- Which component is most strongly adsorbed by each of the adsorbents
- Which adsorbent has the greatest adsorption capacity
- Which adsorbent has the greatest selectivity
- Based on equilibrium conditions, which adsorbent is the best for the separation

Exercise 6.2

The following data were obtained for the adsorption of toluene from water on activated carbon and water from toluene on activated alumina. Fit both sets to a Langmuir-type isotherm and compare the resulting isotherms with the experimental data.

Toluene (in water) on Activated Carbon		Water (in toluene) on Activated Alumina	
c (mg/l)	q (mg/g)	c (ppm)	q (g/100g)
0,01	12,5	25	1,9
0,02	17,1	50	3,1
0,05	23,5	75	4,2
0,1	30,3	100	5,1
0,2	39,2	150	6,5
0,5	54,5	200	8,2
1	70,2	250	9,5
2	90,1	300	10,9
5	125,5	350	12,1
10	165,0	400	13,3

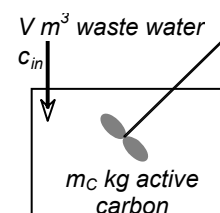
Exercise 6.3

An aqueous amount of V m³ contains 0.01 kg/m³ of nitrobenzene. We want to reduce this to 10⁻⁶ kg/m³ by adsorption on activated carbon. The solution is fed to an ideally stirred tank containing m_C kg adsorbent, see outline. At these low concentrations, the equilibrium adsorption isotherm appears to be linear:

$$q = K \cdot c$$

with q = amount adsorbed, kg/kg
 c = concentration of nitrobenzene, kg/m³
 K = Henry constant = 675 m³/kg¹

How many kg of activated carbon is required per m³ of treated water?



¹ In Eqs. 1.9 and 1.10 b_A and b'_A are Henry constants

Exercise 6.4

Nitrobenzene in an aqueous effluent should be reduced from its initial concentration of 0.02 kg/m^3 to a value of not more than $2 \cdot 10^{-5} \text{ kg/m}^3$. Nitrobenzene can be removed from water by adsorption on active carbon. The amount q of nitrobenzene adsorbed in equilibrium with its concentration c in water is given by the following Langmuir isotherm:

$$q(c) = \frac{510 \cdot c}{1 + 4550 \cdot c} \quad \text{with } c \text{ in kg/m}^3 \text{ and } q \text{ in kg/kg}$$

Two different process schemes (see outlines below) are considered to carry out the required reduction:

- a packed column (see process scheme **a** below), and
- a batch process in an ideally stirred tank (see process scheme **b** below)

The packed column contains W_{col} kg carbon and is fed with wastewater at a rate of $Q = 0.015 \text{ m}^3 \text{ s}^{-1}$.

The rate of convective transport through the bed is such that local adsorption equilibrium is established throughout the column. The concentration profile is flat (plug flow), as long as the column is not saturated, the outlet concentration is zero.

The length of the column $L = 2.0 \text{ m}$, its diameter $D = 0.6 \text{ m}$, the bed porosity $\varepsilon_{bed} = 0.6$ and the particle density $\rho_{part} = 800 \text{ kg m}^{-3}$.

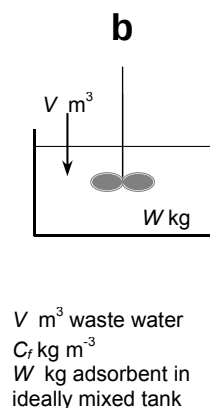
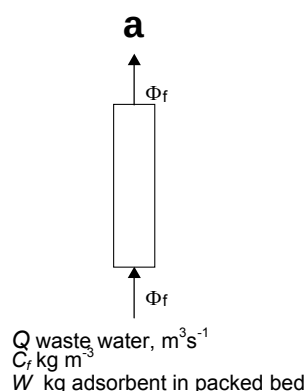
- a1. Calculate from a component balance the amount of carbon per unit volume of waste water (W_{col}/V in kg/m^3)
- a2. How much waste water (V in m^3) is purified when the column is about to break through?

After saturation of the column, it is regenerated with a water flow of the same size in which the concentration of nitrobenzene amounts to $2 \cdot 10^{-5} \text{ kg m}^{-3}$.

- a3. How long does it take to regenerate the column?

In the second process, an ideally stirred tank, $V \text{ m}^3$ waste water (as calculated in part a1) is treated. This batch is treated with W_{batch} kg active carbon. After equilibration the resulting concentration of nitrobenzene should be $2 \cdot 10^{-5} \text{ kg/m}^3$.

- b1. Calculate the excess amount of carbon to be used in the batch process compared to that required in the continuous process, in other words, calculate W_{batch} / W_{col} .
- b2. Comment on the answer of part b1.



Exercise 6.5

Derive the expression for the time t_{sat} required to saturate a freshly regenerated column (Eq. 6.22) from the mass balance over the entire column as given in Eq. 6.23.

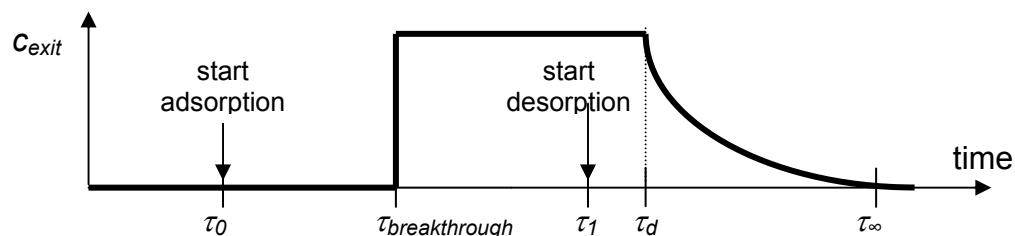
Exercise 6.6

A couple of students got an assignment to determine experimentally the residence time of CO_2 during adsorption and desorption in a packed bed column. They had to compare the results with a theoretical model based on local adsorption equilibrium.

A column ($L = 1$ m, internal diameter $D = 5$ mm) was filled with activated carbon and its exit provided with a CO_2 -detector. The temperature was kept constant at 20°C .

The column was pretreated by flushing with pure Helium. At time τ_0 a CO_2 -He mixture, containing 4 vol% CO_2 , was fed to the column.

Some time after saturation, at time τ_1 , the feed was switched to pure He. The concentration CO_2 in the effluent was measured continuously and is given schematically (not to scale) in the plot below:

**Additional data**

The particle density of the carbon $\rho_{part} = 790 \text{ kg/m}^3$ and the bed porosity $\varepsilon_{bed} = 0.40$. The adsorption of CO_2 at 20°C is given by a Langmuir-type adsorption isotherm:

$$q = q_m \frac{b \cdot c_{\text{CO}_2}}{1 + b \cdot c_{\text{CO}_2}}$$

where monolayer capacity $q_m = 1.39 \text{ mol/kg}$, the adsorption constant $b = 0.068 \text{ m}^3/\text{mol}$ and the concentration c is in mol m^{-3} .

In all experiments (flushing with He, adsorption with He/ CO_2 , desorption with He) the total gas flow Φ_V was maintained at $4 \cdot 10^{-6} \text{ m}^3/\text{s}$ at 20°C and 1 bar. In this equipment and at these conditions, adsorption and desorption are isothermal processes. $R = 8.31 \text{ J/mol K}$.

The following problems are to be addressed:

- Explain the form of the breakthrough curve qualitatively.
- Calculate the breakthrough time, e.g. $\tau_{breakthrough} - \tau_0$, using Eq. 6.22.
- Some students calculated the breakthrough time from the ratio of the maximum amount to be adsorbed and the flow rate of carbon dioxide:

$$\tau_{breakthrough} - \tau_0 = \frac{W_s \times q(c_f)}{Q \cdot c_f}$$

When calculated properly, this gives the same result as in part b. Explain.

- Calculate the desorption time, $\tau_\infty - \tau_1$.

Exercise 6.8

A commercial ion-exchange resin is made of 88 wt% styrene (MW = 0.104 kg/mol) and 12 wt% divinyl benzene (MW = 0.1302 kg/mol). Estimate the maximum ion-exchange capacity in equivalents/kg resin when an sulfonic acid group (MW = 0.0811 kg/mol) has been attached to each benzene ring.

Exercise 6.9

A continuous stream of a soil/water mixture ($0.3 \text{ m}^3/\text{s}$) containing 15 mol/m^3 of a heavy metal M^+ is treated with an ion exchange resin to remove the heavy metal by exchange against the sodium Na^+ ions present on the resin. The concentration M^+ has to be reduced from 15 to 1 mol/m^3 . The concentration M^+ on the incoming regenerated ion exchange resin equals 200 mol/m^3 . The regeneration liquid contains $3000 \text{ mol/m}^3 \text{ Na}^+$ and no M^+ .

- a. Determine the equilibrium diagrams for extraction and regeneration when the total ion concentrations in the resin, soil/water mixture and regeneration solution are given by:

Resin: $[C^+] = [\text{Na}^+] + [M^+] = 2400 \text{ mol/m}^3$

Soil/water: $[C^+] = [\text{Na}^+] + [M^+] = 30 \text{ mol/m}^3$

Regeneration liquid: $[C^+] = [\text{Na}^+] + [M^+] = 3000 \text{ mol/m}^3$

and the equilibrium follow ideal behavior with an equilibrium constant $K = 5$.

- b. Determine the minimal required ion exchanger stream
- c. Determine the number of equilibrium stages and concentration M^+ in the outgoing ion exchange resin for 1.2 times the minimal ion exchanger stream.
- d. Determine the minimal value of the regeneration liquid stream to obtain a regenerated resin with 200 mol/m^3 residual M^+
- e. How many equilibrium stages are required with 1.2 times the minimal regeneration stream

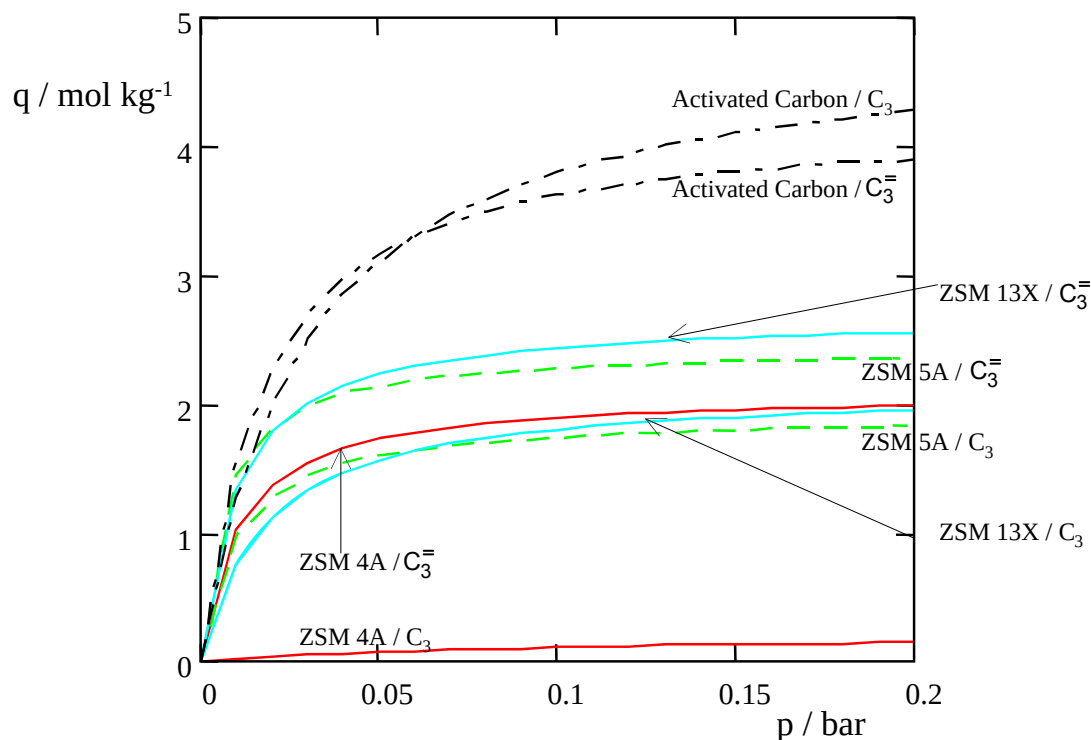
SOLUTIONS

Exercise 6.1

Given Monolayer capacities and adsorption constants of a number of adsorbent – adsorbate combinations.

Find The best adsorbent for the $C_3 - C_3^=$ separation

Thoughts The strongest interaction can be found by comparing the values of adsorption constants (see Eq. 6.8), but what is the definition of the ‘best’ adsorbent? It is helpful, to plot the 8 adsorption isotherms in a single graph:



- Solution**
- The system propene – ZSM 5A has the highest b -value, hence the highest heat of adsorption (see Eq. 6.8).
 - Activated carbon has the highest value of the monolayer capacity, q_m . It can accommodate the largest number of molecules on its internal surface area.
 - d . The intrinsic selectivity is given by the separation factor, which is defined in Chapter 1, Eq. 1.10. It should be noted that the parameter b in Eqs. 1.9 and 1.10 represents the slope of a Henry or linear isotherm. At sufficiently low pressures, the Langmuir isotherm transforms into a linear isotherm with slope $q_m \cdot b$. With this notation, the separation factor equals the ratio of the initial slopes in the Langmuir plots above. ZSM 4A shows a ratio of about 10, much larger than for other zeolites, and is the best adsorbent for the propene – propane separation.

Exercise 6.2

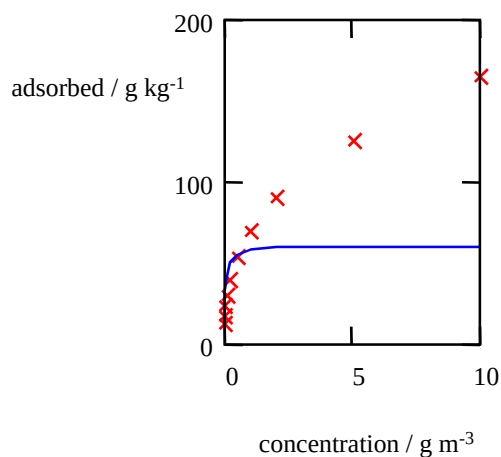
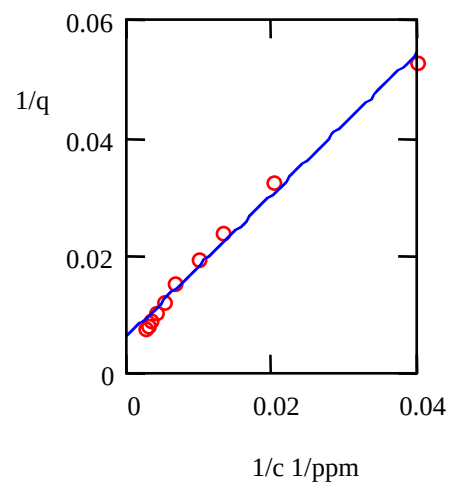
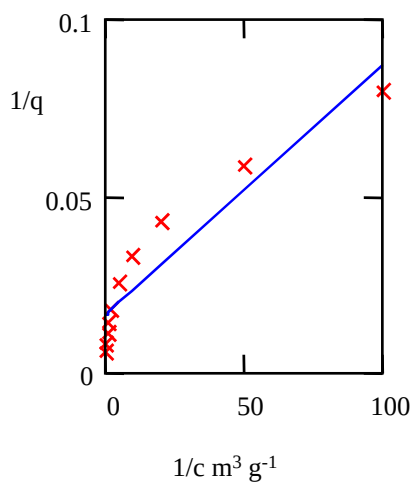
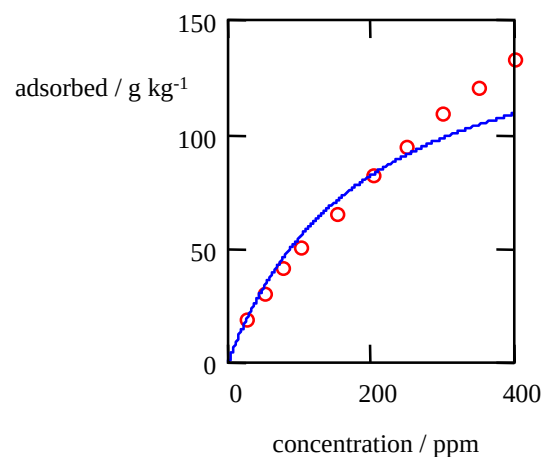
Given Adsorption data for adsorption of traces toluene in water on activated carbon and for traces water in toluene on activated alumina

Find Do these two different systems obey Langmuir type of adsorption?

Thoughts Checking experimental data against a linear model is easy. The Langmuir equation is not a linear one but it can be linearized easily. The reciprocal of Eq. 6.7 gives linear relation between $1/q$ and $1/c$: $\frac{1}{q} = \frac{1}{c} \cdot \frac{1}{q_m b} + \frac{1}{q_m}$. Thus, in case of Langmuir adsorption, plotting $1/q$ as a function of $1/c$ would result in a straight line with intercept $1/q_m$ and slope $1/b \cdot q_m$.

Solution For both systems a least square method is applied to all data points of both systems, see the lower two graphs. q_m is calculated from the reciprocal intercept, then the value of b from the slope. With these two parameters, the Langmuir isotherms are calculated, as shown in the upper two graphs. Note that in both cases the amount adsorbed is expressed as g / kg.

Adsorption of toluene on activated carbon does not obey the Langmuir model, whereas the adsorption data of water in the lower concentration range show a linear Langmuir plot. Symbols represent experimental data, solid lines are the calculated (regression) lines.

Toluene on activated carbon**Water on activated alumina**

Exercise 6.3

Given $c_{in} = 0.01 \text{ kg m}^{-3}$, $c_{\infty} = 10^{-6} \text{ kg m}^{-3}$, $q = K \cdot c$ with $K = 675 \text{ m}^3 \text{ kg}^{-1}$

Find The amount of carbon per unit volume solution to ensure the required equilibrium concentration.

Thoughts The total amount of nitrobenzene initially present equals the amount present after equilibrium

Solution The nitrobenzene mass balance reads:

$$V \cdot c_{in} + m_c \cdot q_{in} = V \cdot c_{\infty} + m_c \cdot q_{\infty}$$

with

$$q_{in} = 0 \text{ and } q_{\infty} = 675 \cdot c_{\infty}$$

The required ratio is calculated from

$$\frac{m_c}{V} = \frac{c_{in} - c_{\infty}}{q_{\infty}} = \frac{c_{in} - c_{\infty}}{675 \cdot c_{\infty}} = 14.8 \frac{\text{kg}}{\text{m}^3}.$$

Exercise 6.4

Given Langmuir adsorption isotherm with $q_m \cdot b = 510 \text{ m}^3/\text{kg}$ and $b = 4550 \text{ m}^3/\text{kg}$

Plug flow in packed column: $c_{in} = 0.02 \text{ kg/m}^3$, $c_{out} = 0 \text{ kg/m}^3$, $Q = 0.015 \text{ m}^3/\text{s}$,
 $\varepsilon_{bed} = 0.6$, $\rho_{part} = 800 \text{ kg/m}^3$, $D_{column} = 0.6 \text{ m}$, $L = 2.0 \text{ m}$,
 $W \text{ kg carbon adsorbent}$.

Batch adsorption in CISTR $c_{in} = 0.02 \text{ kg/m}^3$, $c_{\infty} = 2 \cdot 10^{-5} \text{ kg/m}^3$, $W \text{ kg carbon adsorbent}$.

Find a. The amount of carbon per unit volume solution, the amount of waste water treated until breakthrough and the regeneration time of a saturated column
 b. How much more adsorbent is required in a batch process?

Thoughts a. The total amount of nitrobenzene before equals that after equilibration.
 The amount of waste water to be treated till saturation of the column is the volume flow rate of the waste water times the residence (breakthrough) time τ of nitrobenzene and depends on the velocity v of nitrobenzene through the column, the residence time $\tau = L/v$.
 During desorption, all concentrations travel through the column, each with a velocity given by Eq. 6.20. Lower concentration travel slower than higher concentrations (favorable isotherm).

Solution a1. All nitrobenzene fed to the column just until break through is adsorbed on the active carbon. The component balance reads

$$V \cdot c_{in} + W \cdot q(0) = V \cdot c_{out} + W \cdot q(c_{in}) \text{ or}$$

$$W/V = c_{in} / q(c_{in}) = 0.02 \cdot (1 + 4550 \cdot 0.02) / (510 \cdot 0.02) = 0.02 / 0.111 = 0.180 \text{ kg /m}^3$$

a2. The amount of waste water treated until saturation of the column follows from volume flow rate and breakthrough time: $V = Q \cdot \tau$, with τ from Eq. 6.22;

$$\text{superficial carrier velocity } u = Q / 0.25\pi D^2 = 0.0531 \text{ m/s};$$

$$q(c_{in}) = 0.111 \text{ kg/kg and } \Delta q / \Delta c \text{ (shock wave)} = (0.111 - 0) / (c_{in} - 0) = 5.54$$

$$\tau = 66898 \text{ s (= 18.6 h) and } V = 0.015 \cdot 66898 = 1003 \text{ m}^3.$$

Alternative calculation:

$$W = 0.25\pi D^2 L (1 - \varepsilon_{bed}) \rho_{part} = 181 \text{ kg and (applying part a1) } V = W / 0.180 = 1003 \text{ m}^3$$

a3. Velocity of the very last trace $v_{c=0} \approx \frac{u_{carrier}}{(1 - \varepsilon_{bed}) \rho_{part} \left. \frac{dq}{dc} \right|_{c=0}}$ (Eq. 6.20)

$$\text{with } \left. \frac{dq}{dc} \right|_c = \frac{q_m b}{(1 + bc)^2} \text{ (derived from the Langmuir equation, Eq. 6.7) and } \left. \frac{dq}{dc} \right|_{c=0} = q_m b.$$

For the part with $c = 0$ at the inlet, it takes $\tau = L/v_{c=0}$ to travel to the exit of the column. $v_{c=0} = 3.25 \cdot 10^{-7}$ m/s, all other concentrations travel faster. The total time τ to regenerate the column is $L/v_{c=0} = 6.15 \cdot 10^6$ s = 71 days: the desorption takes an extremely long period of time, resulting from a very high value of the adsorption constant (steep initial part of adsorption isotherm). This carbon adsorbent is not feasible for regeneration.

- b1. Nitrobenzene initially present in waste water = nitrobenzene after equilibration: in solution + nitrobenzene adsorbed

$$V \cdot c_{in} = V \cdot c_{\infty} + W \cdot q(c_{\infty}) \text{ or}$$

$$W/V = (c_{in} - c_{\infty}) \cdot (1 + b \cdot c_{\infty}) / q_m \cdot b \cdot c_{\infty} = 1.98 \cdot 10^{-3} / 9.35 \cdot 10^{-3} = 2.14 \text{ kg/m}^3$$

- b2. The result calculated above is $2.14 / 0.180 = 11.8$ times more than the amount required in a column set-up to treat the same amount of waste water. Unlike the adsorbent in the column set-up, which is in equilibrium with the *feed concentration*, the adsorbent in the batch process is in equilibrium with the *final concentration*, which is much lower. Hence the resulting amount adsorbed is lower and more adsorbent is necessary to remove the same amount of nitrobenzene as in the continuous process.

Exercise 6.5

Derive $t_{sat} = \frac{L}{u_{carrier}} (1 - \varepsilon_{bed}) \rho_{part} \frac{q^*}{c_f}$ (Eq. 6.22), the time required to saturate a column with $c = c_f$

Initial condition: $c = 0$ and $q = 0$ for $0 \leq z \leq L$

Start from mass balance over entire column, assume plug flow.

Q = volume flow rate, c_f = feed concentration, q^* = amount adsorbed in equilibrium with c_f , W_s = weight of adsorbent

Thought Saturating a column is a non-stationary process, the mass balance in words being:
 $\text{in} = \text{accumulation} + \text{out}$

Solution During saturation no adsorbable material leaves the column. For plug flow, the mass balance reads

$$t_{sat} \cdot Q \cdot c_f = W_s \cdot q^* + 0$$

By definition (see Fig. 6.3)

$$W_s = V_{part} \cdot \rho_{part} = V_{bed} \cdot (1 - \varepsilon_{bed}) \cdot \rho_{part}$$

and

$$V_{bed} / Q = L / u_{carrier} \text{ (see section 6.3.2)}$$

hence

$$t_{sat} = \frac{W_s}{Q} \cdot \frac{q^*}{c_f} = \frac{L}{u_{carrier}} (1 - \varepsilon_{bed}) \rho_{part} \frac{q^*}{c_f}$$

Exercise 6.6

<i>Given</i>	$L = 1 \text{ m}$	$D = 5 \text{ mm}$	$T = 20^\circ\text{C}$
	$\varepsilon_{bed} = 0.40$	$q_m = 1.39 \text{ mol m}^{-3}$	$b = 0.068 \text{ m}^3/\text{mol}$
	$c_{in} = 4 \text{ vol\% CO}_2$	$Q = 4 \cdot 10^{-6} \text{ m}^3/\text{s}$	$\rho_{part} = 790 \text{ kg/m}^3$
	$R = 8.31 \text{ J/mol K}$		

- Find*
- Explain the rectangular shape of the exit concentration with time
 - Calculate the breakthrough time with Eq. 6.22
 - The same answer is obtained from $\Delta\tau = W_{carbon} \cdot q(c_{in}) / Q \cdot c_{in}$
 - Calculate the desorption time

- Thoughts**
- Langmuir-type is a favorable adsorption isotherm
 - In Eq. 6.18 $u_{carrier}$ = superficial velocity = Q/A with $A = \pi D^2/4$
 - Try to convert Eq. 6.22 into $W_{carbon} \cdot q(c_{in}) / Q \cdot c_{in}$
 - See part a3 in exercise 6.4

Solution

- If the feed enters as plug flow, the form of this front remains the same: all concentrations travel with the same velocity because $\Delta q / \Delta c = (q(c_{in}) - 0) / (c_{in} - 0) = \text{constant}$. If any other concentration profile enters the column, higher concentrations (smaller dq/dc) will catch up with lower concentrations (larger dq/dc) until a shock wave has formed. From that moment on, plug flow occurs

Bonus: the time required to transform an inlet profile into a shock wave usually is relatively short. Imagine a linear rise in inlet concentration, from 0 until c_{in} in say 1 s. At a certain distance z from the inlet, the feed concentration C_{in} , which entered 1 s after the lowest concentration, would overtake the lowest concentration. This takes time τ .

$$\tau = \frac{z}{v_{c=0}} = 1 + \frac{z}{v_{c=c_{in}}}$$

With $c_{in} = 1.643 \text{ mol m}^{-3}$, $v_{c=0} = 4.51 \text{ mm s}^{-1}$ and $v_{c=c_{in}} = 5.56 \text{ mm s}^{-1}$ it follows that $z = 2.4 \text{ mm}$ and $\tau = 5.3 \text{ s}$.

These values are small compared to $L = 1000 \text{ mm}$ and residence time $L/v_{c=0} = 222 \text{ s}$. Thus, the shockwave is built up fast; plug flow exists in the entire column, except for the small entrance effect.

- $\tau_{\text{breakthrough}} - \tau_0 = L / v_{\text{shockwave}}$, with $v_{\text{shockwave}}$ from Eq. 6.22.
 $c_{in} = 1.64 \text{ mol m}^{-3}$ (ideal gas law) and $\Delta q / \Delta c = (q(c_{in}) - 0) / (c_{in} - 0) = 0.140/1.64$
 $\tau_{\text{breakthrough}} - \tau_0 = L / v_{\text{shockwave}} = 1 / 5 \cdot 10^{-3} = 199.8 \text{ s}$
- Neglecting the accumulation of CO_2 in the gas phase between the particles, meaning

$\varepsilon_{bed} \ll (1 - \varepsilon_{bed}) \rho_p \frac{\Delta q}{\Delta c}$, Eq. 6.21 can be written as

$$\frac{L}{v_{\text{CO}_2}} = \frac{\frac{1}{4} \pi D^2}{\frac{1}{4} \pi D^2} \cdot \frac{L(1 - \varepsilon_{bed}) \rho_{part}}{u_{carrier}} \cdot \frac{\Delta q}{\Delta c} = \frac{W}{Q} \cdot \frac{q_{c=c_{in}} - 0}{c_{in} - 0}$$

- The total desorption time equals $\tau_{\infty} - \tau_1 = L / v_{c=0} = 222 \text{ s}$, see part a3 of exercise 6.4.

Exercise 6.8

Given $M_{\text{styrene}} = 0.104 \text{ kg/mol}$ $M_{\text{divinylbenzene}} = 0.1302 \text{ kg/mol}$ $M_{\text{sulfonicacid}} = 0.0811 \text{ kg/mol}$

Find The ion-exchange capacity of a resin with the highest possible sulfon-group content

Thoughts Calculate the number of moles sulfuric acid required to provide every aromatic ring with a sulfon group, starting from sulfon-free polymer. Express the result on a weight-basis: kg dry ion-exchange resin (polymer plus sulfon groups)

Solution Benzene in 1 kg dry polymer before sulfonation:
 $0.88/0.104 + 0.12/0.130 = 9.385 \text{ mol benzene / kg dry polymer}$.
 Complete saturation with sulfuric acid requires that amount of moles, corresponding to
 $9.385 \cdot 0.081 = 0.760 \text{ kg sulfon-groups}$.
 The weight after sulfonation is $1.000 + 0.760 = 1.760 \text{ kg resin}$.
 This amount contains 9.385 mol of groups available for ion-exchange or $9.385/1.760 = 5.33$
 eq. per unit weight of dry resin.

Exercise 6.9

Given Feed $L = 0.3 \text{ m}^3 \text{ s}^{-1}$ with $c_{M,f} = 15 \text{ mol m}^{-3}$ and $c_{Na,f} = 15 \text{ mol m}^{-3}$; the required exit concentration $c_{M,exit} = 1 \text{ mol m}^{-3}$.
 The initial sodium concentration in the regeneration liquid $c_{Na,rlf} = 3000 \text{ mol m}^{-3}$, and $c_{M,rlf} = 0$; in the regenerated ion-exchange resin these concentrations are $c_{Na,sf} = 2200 \text{ mol m}^{-3}$ and $c_{M,sf} = 200 \text{ mol m}^{-3}$, respectively.
 The ion-exchange equilibrium constant $K = 5$.
 The stream of ion-exchange resin (V in $\text{m}^3 \text{ s}^{-1}$) and regeneration liquid ($\underline{L}$ in $\text{m}^3 \text{ s}^{-1}$) are chosen as 1.2 times the minimum values.

Find a. The equilibrium diagrams for extraction and regeneration
 b. The minimum amount of ion-exchange resin per unit volume feed
 c-e. Various process conditions

Thoughts a. The definition equation of the ion-exchange equilibrium contains both the concentration of the metal M in the ion-exchange resin, $c_{M,s}$ and that in the aqueous phase, $c_{M,l}$.
 Note that the sum of the ion concentrations in the liquid (and in the solid as well) is constant, since each M^+ -ion exchanges with one Na^+ -ion.
 b. Given the feed flow L , the amount of resin per unit time follows from the slope of the operating line, L/V .

Solution a. Equilibrium diagram for extraction

$$c_{M,l} + c_{Na,l} = c_{M,f} + c_{Na,f} = 30 \text{ mol m}^{-3}$$

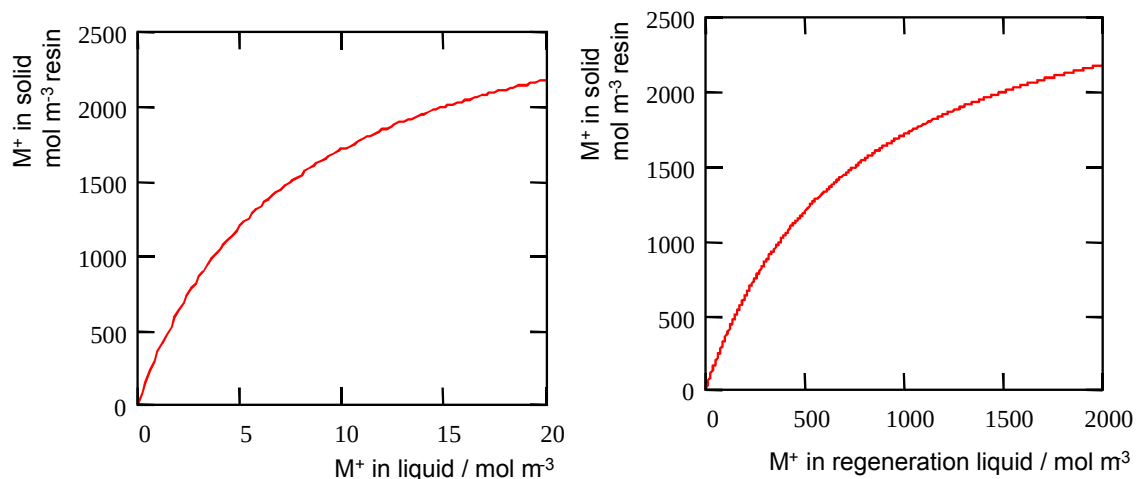
$$c_{M,s} + c_{Na,s} = c_{M,sf} + c_{Na,sf} = 200 + 2200 = 2400 \text{ mol m}^{-3}$$

$$K = \frac{c_{M,s} \cdot c_{Na,l}}{c_{M,l} \cdot c_{Na,s}} = \frac{c_{M,s} \cdot (30 - c_{M,l})}{c_{M,l} \cdot (2400 - c_{M,s})} = 5; \quad c_{M,s} = \frac{2400 \cdot K \cdot c_{M,l}}{30 + (K - 1) \cdot c_{M,l}}$$

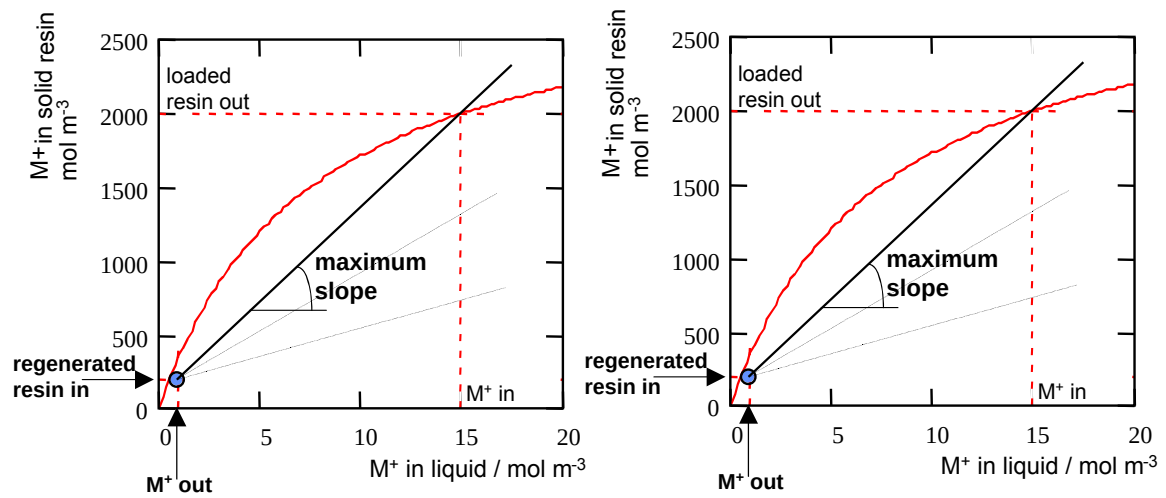
Similarly, for the regeneration:

$$K = \frac{c_{M,s} \cdot c_{Na,rl}}{c_{M,rl} \cdot c_{Na,s}} = \frac{c_{M,s} \cdot (3000 - c_{M,rl})}{c_{M,rl} \cdot (2400 - c_{M,s})} = 5; \quad c_{M,s} = \frac{2400 \cdot K \cdot c_{M,rl}}{30 + (K - 1) \cdot c_{M,rl}}$$

Both equilibrium equations are plotted in the two graphs below.



b. Three operating lines are drawn, all going through the point, defined by the top of the extraction column where the fresh, regenerated resin is fed and the purified solution is withdrawn. The maximum slope of the operating line is given by a second point, the point on the equilibrium line at the concentration of the metal in the feed, at 15 mol m^{-3} . The maximum slope is $L/V_{min} = (2000 - 200) / (15 - 1) = 128.6$; with $L = 0.3$ it follows that $V_{min} = 0.3/128.6 = 2.333 \cdot 10^{-3} \text{ m}^3 \text{ s}^{-1}$.



Chapter 6: ADSORPTION & ION EXCHANGE (continued)

Exercise 6.7

An aqueous waste stream contains small amounts of acetone (1) and propionitrile (2).

These contaminants can be captured by adsorption on active carbon. The respective adsorption isotherms of both contaminants at 20°C are of the Langmuir type::

$$q_1 = \frac{0.190 \cdot c_1}{1 + 0.146 \cdot c_1} \quad \text{and} \quad q_2 = \frac{0.173 \cdot c_2}{1 + 0.0961 \cdot c_2}$$

with q in mol kg⁻¹ and c in mol m⁻³. Simultaneous adsorption of acetone and propionitrile from the waste stream can be described by the 'extended Langmuir' model.

- Calculate $q_{1,m}$ and $q_{2,m}$
- Calculate the equilibrium loading of both contaminants when the waste stream is led through an active carbon column and contains 40 mol acetone m⁻³ and 34 mol propionitrile m⁻³.
- Calculate the ratio of the velocities by which both contaminants flow through the column.

Exercise 6.7

Given adsorption isotherms
 $C_{\text{acetone},\text{in}} = 40 \text{ mol m}^{-3}$ $C_{\text{propionitrile},\text{in}} = 34 \text{ mol m}^{-3}$

Find a) $q_{1,m}$ and $q_{2,m}$
 b) equilibrium loading of both contaminants
 c) velocity ratio of both contaminants

Solution a) $q = \frac{q_{\max} B c}{1 + B c}$ so

$$q_{m,1} = \frac{q_{\max} B}{B} = \frac{0.190}{0.146} = 1.301$$

$$q_{m,2} = \frac{q_{\max} B}{B} = \frac{0.173}{0.0961} = 1.800$$

b) Extended Langmuir formula (Eq 6.13 & Eq 6.14):

$$q_1 = \frac{q_{\max,1} B_1 c_1}{1 + B_1 c_1 + B_2 c_2} = \frac{0.190 \times 40}{1 + 0.146 \times 40 + 0.0961 \times 34} = 0.751 (\text{mol/kg})$$

$$q_2 = \frac{q_{\max,2} B_2 c_2}{1 + B_1 c_1 + B_2 c_2} = \frac{0.173 \times 34}{1 + 0.146 \times 40 + 0.0961 \times 34} = 0.582 (\text{mol/kg})$$

c)
$$v_c = \frac{u_{\text{carrier}}}{\varepsilon_{\text{bed}} + (1 - \varepsilon_{\text{bed}}) \rho_{\text{part}} \frac{\Delta q}{\Delta c}} \quad (\text{Eq 6.23})$$

Neglect contribution ε_{bed} , then (Eq 6.24):

$$\frac{v_1}{v_2} = \frac{\left(\frac{\Delta q}{\Delta c} \right)_2}{\left(\frac{\Delta q}{\Delta c} \right)_1} = \frac{\left(\frac{0.582}{34} \right)}{\left(\frac{0.751}{40} \right)} = 0.912$$

Chapter 7: DRYING OF SOLIDS

Exercise 7.1

Air of 20°C and 1 bar with a relative humidity of 80% is heated to 50°C. The heated air flows along a flat tray filled with completely wetted material. The heat transfer coefficient $h = 35 \text{ W/m}^2\text{K}$ and the heat of evaporation $\Delta H_v = 2.45 \cdot 10^6 \text{ J kg}^{-1}$.

Calculate r_c , the constant drying rate.

Exercise 7.2

A wet solid with a moisture content $w = 0.5 \text{ kg kg}^{-1}$ dry occupies 0.1 m^2 on a flat tray. A laboratory experiment shows that the critical moisture content $w_c = 0.2 \text{ kg kg}^{-1}$ dry and the constant drying rate $r_c = 0.36 \text{ g s}^{-1} \text{ m}^{-2}$.

The moisture content of 2 kg of the solid (dry basis) is reduced from 0.4 to 0.2 kg kg^{-1} dry under similar conditions.

How long will that take?

Exercise 7.3

A wet solid is dried with air at 60°C and a humidity $H_f = 0.010 \text{ kg H}_2\text{O/kg dry air}$. The air flows parallel to the flat, wet surface of the solid at a velocity $v = 5 \text{ m s}^{-1}$. Under these conditions the heat transfer coefficient h is given by $h = 14.3 (\rho v)^{0.8}$, where ρ is the density of the moist air in $\text{kg moist air per unit volume moist air}$.

The surface area available for heat exchange with air is 0.25 m^2 and the heat of evaporation $\Delta H_v = 2.45 \cdot 10^6 \text{ J kg}^{-1}$.

Calculate the total amount of water evaporating per unit time.

Exercise 7.4

Water is dispersed in droplets of 0.1 mm radius; $T = 300\text{K}$ and $\gamma_w = 0.073 \text{ N m}^{-1}$.

Calculate the relative change in saturation vapor pressure

Exercise 7.5

Air at 20°C and 1 bar with a relative humidity of 80% is available for drying a porous solid containing cylindrical pores with a diameter of 0.8 nm. The heat of evaporation $\Delta H_v = 44045 \text{ J mol}^{-1}$.

To what temperature should the air be heated at least to avoid that the pores remain filled with water?¹

Exercise 7.6

Wet air of 30°C and 2 bar with a relative humidity ($H_{rel} = p_{\text{water}}/p_{\text{water}}^0$) of 75% has to be dried at a rate of 0.2 kg s^{-1} . Drying is achieved by adsorption of water on porous alumina. Regeneration takes place at 60°C. The adsorption isotherms are given in the form of Henry's law:

$$\frac{\text{kg water adsorbed}}{\text{kg dry alumina}} = K \times H_{rel} \quad \text{with } K(30^\circ\text{C}) = 0.350 \text{ and } K(60^\circ\text{C}) = 0.121$$

The process is conducted in a packed bed with a length of 4 m and diameter of 0.5 m. At the start of the drying process the water vapor pressure increases linearly until the maximum value of 75% relative humidity is reached.

¹ Hint: the saturated vapor pressure obeys *Clapeyron*

The bed porosity $\varepsilon_{bed} = 0.32$ for a complete filling with the alumina particles of 2 mm diameter. The particle porosity $\varepsilon_{part} = 0.58$. Furthermore it is given that the saturated vapor pressure of water, p_{water}^0 equals 0.0418 bar at 30°C and 0.199 bar at 60°C. $M_{water} = 0.018$ kg/mol and $M_{air} = 0.029$ kg/mol.

- a. Calculate the maximum kilograms of dry alumina the adsorption vessel can contain.
- b. When the first trace of water is detected at the column exit, only 70% of the column is saturated with water. Draw the concentration profile in the adsorption vessel (relative humidity as function of the place).
- c. How long did it take the first trace of water to exit the column?
- d. Calculate the total amount of water at breakthrough.
- e. How long does it take to completely regenerate the column by heating the inlet air to 60°C and reducing the pressure to 1 bar.

SOLUTIONS

Exercise 7.1

Given $H_r = 80\%$

$\Delta H_v = 2.45 \times 10^6 \text{ J/kg}$

Find r_C , the constant drying rate

$T_{feed} = 50^\circ\text{C}$

$h = 35 \text{ W/m}^2\text{K}$

Thoughts Eq. 7.20b seems to be the appropriate equation to apply. This equation requires the calculation of the wet-bulb temperature T_{wb} .

Increasing the temperature from 20 to 80°C does only change the saturation pressure of water, not the partial pressure of water vapor.

Note: the heat of evaporation is given as J/kg.

Solution

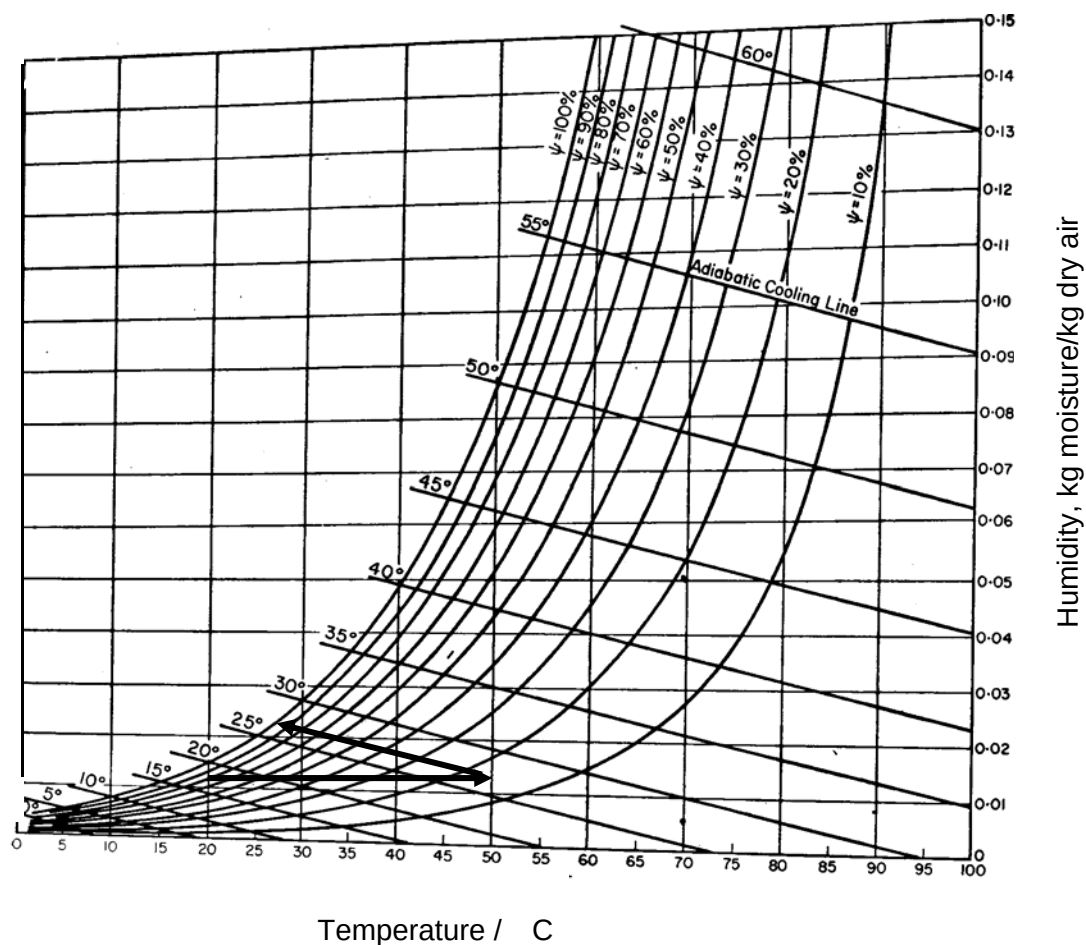
The humidity is constant, the relative humidity decreases.

Use the psychrometric chart (Fig. 7.3), draw a horizontal line (starting from the intersection of the curve representing 80% relative humidity and the vertical line at 20°C, see psychrometric chart below) to the point where $T = 50^\circ\text{C}$.

Now draw a straight line, starting from that point and parallel to the adiabatic cooling lines, to the saturation line (100%). Read the wet-bulb temperature: $T_{wb} = 26^\circ\text{C}$.

Apply Eq. 7.20b:

$$r_C = \frac{35 \times (50 - 26)}{2.45 \times 10^6} = 3.43 \times 10^{-4} \frac{\text{kg}}{\text{s m}^2}$$



Exercise 7.2

Given $r_C = 3.6 \cdot 10^{-4} \text{ kg / m}^2\text{s}$ $W = 2 \text{ kg dry solid}$
 $X_C = 0.2 \text{ kg / kg dry (critical moisture content)}$ $X_0 = 0.5 \text{ kg / kg dry}$
 $A = 0.1 \text{ m}^2$

Find t_C , drying time to reduce the moisture content from 0.4 to 0.2 kg / kg dry.

Thoughts Eq. 7.23 is applicable. Timing starts at $X = 0.4$ whereas $X_0 = 0.5 \text{ kg / kg dry}$, so the initial drying period A'B in Figs. 7.7 and 7.8, where surface temperature and rate of evaporation are adjusting to reach the stationary state, is (probably) excluded.

Solution
$$t_C = \frac{2}{0.1 \times 0.36 \cdot 10^{-3}} (0.4 - 0.2) = 11111 \text{ s} = 3.09 \text{ h}$$

Exercise 7.3

Given $T_{\text{feed}} = 60^\circ\text{C}$ $h = 14.3 (\rho v)^{0.8} \text{ W/m}^2\text{K}$
 $P_{\text{tot}} = 1.01325 \cdot 10^5 \text{ Pa}$ $A = 0.25 \text{ m}^2$
 $M_w = 0.018 \text{ kg/mol}$ $M_{\text{air}} = 0.029 \text{ kg/mol}$
 $H_f = 0.010 \text{ kg / kg dry}$ $v = 5.0 \text{ m/s}$
 $\Delta H_v = 2.45 \cdot 10^6 \text{ J/kg}$

Find the total amount of water evaporating per unit time

Thoughts This is similar to exercise 7.1, except that the heat transfer coefficient h has to be calculated from the air speed v and density of moist air ρ . The latter is found from the weight of moist air and the total volume occupied by that amount. It is convenient to start from 1 kg dry air, then calculate the weight of moisture in that amount of air, and finally calculate the volume of air and moisture, assuming the ideal gas law to hold.

Solution The volume of dry gas $V_{\text{air}} = \frac{R \cdot T_f}{M_{\text{air}} \cdot P_{\text{tot}}} = \frac{8.31 \times 333}{0.029 \times 101325} = 0.942 \frac{\text{m}^3}{\text{kg dry air}},$

the volume of water vapor therein $V_w = H_f \frac{R \cdot T_f}{M_w \cdot P_{\text{tot}}} = 0.015 \frac{\text{m}^3}{\text{kg dry air}}.$

$$\rho_{\text{moist air}} = \frac{1 + H_f}{V_{\text{air}} + V_w} = \frac{1.010}{0.9569} = 1.055 \frac{\text{kg}}{\text{m}^3}$$

$$h = 14.3 \times (1.055 \times 5.0)^{0.8} = 54.1 \frac{\text{W}}{\text{m}^2\text{K}}.$$

The wet bulb temperature, determined similarly as in exercise 7.1 amounts to 29°C .

The resulting drying rate is calculated to $A \cdot \frac{h \cdot (T_f - T_{wb})}{\Delta H_v} = 0.25 \times \frac{54.1 \times 31}{2.45 \cdot 10^6} = 1.71 \cdot 10^{-4} \frac{\text{kg}}{\text{s}}$

Exercise 7.4

Given $r_{\text{droplet}} = 0.1 \mu\text{m}$ $\gamma_w = 0.073 \text{ N/m}$
 $R = 8.31 \text{ J/mol.K}$ $T = 300\text{K}$
 $\rho_w = 1000 \text{ kg/m}^3$ $M_w = 18 \cdot 10^{-3} \text{ kg/mol}$

Find relative change in saturation pressure of water droplets at 300K

Thoughts Eq. 7.17a is applicable. Droplets are spherical so $r_1 = r_2 = r_{\text{droplet}}$.

Note the difference between Eq. 7.17a and Eq. 7.17b.

The molar volume V_m of water is calculated from its density and molecular weight.

Solution
$$\frac{p_{\text{sat}}}{p_{\text{sat}}^\infty} = \exp\left(\frac{2 \times 0.073}{10^{-7}} \times \frac{0.018}{1000} \times \frac{1}{8.31 \times 300}\right) = 1.0107$$

Exercise 7.5

<i>Given</i>	$r = 0.4 \text{ nm}$	$H_r = 80\%$
	$R = 8.31 \text{ J/mol.K}$	$T = 293\text{K}$
	$\Delta H_{vap} = 44045 \text{ J/mol}$	$\rho_w = 1000 \text{ kg/m}^3$
	$M_w = 18 \cdot 10^{-3} \text{ kg/mol}$	$\gamma_w = 0.073 \text{ N/m}$

Find the lowest temperature to evaporate the liquid water from the pores

Thoughts The vapor pressure of water, p_w , of the moist air is given through the relative humidity, Eq. 7.5:

$$H_r = \frac{p_w}{p_{sat}^0(293)} \cdot 100.$$

Increasing the temperature increases the saturation pressure of water, thus decreasing the relative humidity. The humidity should be sufficiently low (the temperature sufficiently high) that Eq. 7.19 is obeyed. Unlike p_w , which does not change with temperature, the saturation pressure of water increases exponentially with temperature. According to the Clapeyron-equation:

$$p_{sat}^0(T) = C \cdot \exp\left(-\frac{\Delta H_{vap}}{RT}\right) \text{ but also } p_{sat}^0(293) = C \cdot \exp\left(-\frac{\Delta H_{vap}}{R \cdot 293}\right),$$

where C is some constant, independent of T . Further, $p_w = 0.8 \cdot p_{sat}^0(293)$

Solution The highest temperature at which the liquid water is in equilibrium with the vapor pressure in the feed gas follows from Eq. 7.19:

$$\frac{p_w}{p_{sat}^0(T)} = \frac{0.8 \times \exp(-\Delta H_{vap} / R \cdot 293)}{\exp(-\Delta H_{vap} / R \cdot T)} = \exp\left(-\frac{2\gamma}{r} \times \frac{V_m}{R \cdot T}\right).$$

Solving for T gives $T = 333.2\text{K}$. Thus, at a temperature just slightly higher than 60°C the pores will empty².

Exercise 7.6

<i>Given</i>	$\Phi_{air} = 0.20 \text{ kg s}^{-1}$	$T_f = 30^\circ\text{C}$	$P_f = 2 \text{ bar}$
	$R = 8.31 \text{ J/mol.K}$	$K_{30} = 0.350$	$K_{60} = 0.121$
	$L = 4 \text{ m}$	$D = 0.5 \text{ m}$	$\rho_s = 3500 \text{ kg m}^{-3}$
	$\varepsilon_{bed} = 0.32$	$\varepsilon_{part} = 0.58$	$d_p = 2 \text{ mm}$
	$H_{rel} = 75\%$	$p_w^0(30) = 0.0418 \text{ bar}$	$p_w^0(60) = 0.199 \text{ bar}$
	$M_w = 0.018 \text{ kg mol}^{-1}$	$M_{air} = 0.029 \text{ kg mol}^{-1}$	

- Find*
- the weight of adsorbent in column
 - The concentration profile at the time of breakthrough
 - the adsorption time till breakthrough
 - the total amount of water adsorbed at breakthrough
 - the time required to regenerate the column at 60°C

- Thoughts*
- Fig. 6.3 shows the key how to calculate the amount of adsorbent to be contained in the vessel.
 - b, c All concentrations travel with the same speed through the column, because the slope of the adsorption isotherm is constant.
 - apply adsorption isotherm to the two sections (saturated, LUB-part)
 - use Eq. 6.26 with $dq/dc = K_{60}$

² N.B. A very thin water film at the pore surface, in equilibrium with p_w at that temperature, will remain

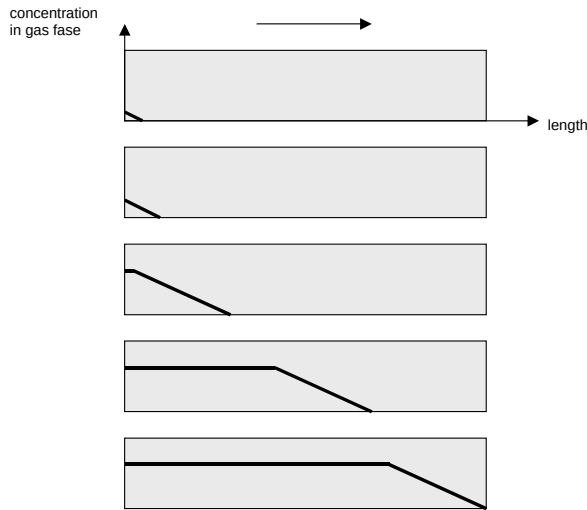
Solution a. $W_{alumina} = \rho_s \cdot V_{alumina}$ with $V_{alumina} = V_{vessel} \cdot (1 - \varepsilon_{tot})$

$$1 - \varepsilon_{tot} = (1 - \varepsilon_{bed}) \cdot (1 - \varepsilon_{part}) \rightarrow \varepsilon_{tot} = 1 - (1 - 0.32) \cdot (1 - 0.58) = 0.714$$

$$V_{alumina} = \frac{\pi}{4} \cdot 0.5^2 \cdot 4 \cdot (1 - 0.714) = 0.224 \text{ m}^3$$

$$W_{alumina} = 3500 \cdot 0.224 = 785.1 \text{ kg}$$

b.



c.

$$t_{breakthrough} = \frac{\text{length}}{\text{velocity water}}, \quad v_{water} = \frac{v_{air}}{(1 - \varepsilon_{tot}) \cdot \rho_s \cdot \frac{dq_w}{dc_w}}$$

$$v_{air} = \frac{(1 - y_w) \Phi_{air}}{M_{air}} \cdot \frac{RT_f}{P_f} \cdot \frac{1}{\frac{\pi}{4} D^2} = 0.254 \text{ m/s (superficial velocity)}$$

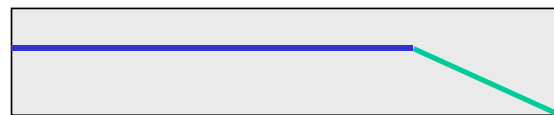
$$y_w = \frac{p_w^0}{P_f} \cdot H_{rel} = 0.0106$$

$$\frac{dq_w}{dc_w} = K' \quad \text{with } K' = \frac{K}{M_w} \cdot \frac{RT}{p_w^0} = 11.5 \text{ m}^3/\text{kg}$$

$$v_{water} = \frac{0.254}{(1 - 0.714) \cdot 3500 \cdot 11.5} = 2.205 \cdot 10^{-5} \text{ m/s}$$

$$t_{breakthrough} = \frac{4}{2.205 \cdot 10^{-5}} = 181422 \text{ s} = 50.4 \text{ h}$$

d.



70% with
equilibrium load

30% with half load on average

$$\text{Total water load} = 0.70 \cdot W_{alumina} \cdot K_{30} \cdot H_{rel} + (0.30 \cdot W_{alumina} \cdot K_{30} \cdot H_{rel})/2 = 175 \text{ kg water}$$

$$e. \quad v_{water} = \frac{v_{air}}{(1-\varepsilon_{tot}) \cdot \rho_s \cdot K'_{60}} \text{ with } v_{air} = \frac{RT_{reg}}{M_{air}P_f} \cdot \frac{(1-y_w)\phi_{air}}{\frac{\pi}{4}D^2} = 0.279 \text{ m/s (superficial velocity)}$$

$$K'_{60} = \frac{K}{M_w} \cdot \frac{RT_{reg}}{\rho_w^0} = 0.935 \text{ m}^3/\text{kg} \text{ and } v_{water} = \frac{0.279}{(1-0.714) \cdot 3500 \cdot 0.935} = 2.99 \cdot 10^{-3} \text{ m/s}$$

$$t_{breakthrough} = \frac{4}{2.99 \cdot 10^{-4}} = 13403 \text{ s} \approx 3.7 \text{ h}$$

Chapter 7: DRYING OF SOLIDS (continued)

Exercise 7.7

Silicagel is stored at room temperature (20°C) in an environment with a relative humidity of 60%. The adsorption isotherm of this material is given in figure 6.5. Before use the material is dried at 40°C in lucht with a wet bulb temperature of 18°C. The air velocity is such that the heat transfer coefficient $h = 30 \text{ W K}^{-1}\text{m}^{-2}$. The critical moisture content amounts 18 wt% and the heat of evaporation of water is $2.45 \cdot 10^6 \text{ J kg}^{-1}$. Entrance effects can be neglected.

- Determine the relative humidity of the drying air.
- Calculate the constant drying velocity in wt% per second per unit of drying area.
- How long does it take to reduce the moisture content to 18 wt% ?

Exercise 7.8

Filtration of a crystal suspension yields a cake (69.4 ton/hr) that contains 10 wt% water. The residual water is removed from the filter cake in a belt dryer until a maximum moisture content of 0.1 wt%. This process can be modelled as a flat plate with a drying area of 2500 m² with air of 80 °C and a relative humidity of 10 % flowing over it. Laboratory experiments have shown that the drying rate over the whole drying trajectory is constant.

The density of this air is 1.3 kg/m³.

The heat of evaporation of water equals $2.45 \cdot 10^6 \text{ J/kg}$.

- Calculate the specific drying rate?
- What should be the minimal value of the heat transfer coefficient.
- Estimate the minimal velocity of the air stream.

Exercise 7.9

A produced calcium sulphate filter cake (351 kg/hr) needs to be dried from 30 wt% water per kg dry product (CaSO_4) till 5 wt%. This is done by distributing the wet cake in thin layers on several trays in a tray dryer. Air of 95 °C with a humidity of 0.04 kg moisture per kg dry air is led over these trays with a velocity of 2 m/s. Each tray has an area of 4 m² and the layers are sufficiently thin to assume a constant drying velocity of the whole drying trajectory.

$2.45 \cdot 10^6 \text{ J/kg}$ water and 1 kg/m³ can be taken for the heat of evaporation of water and the density of the air, respectively.

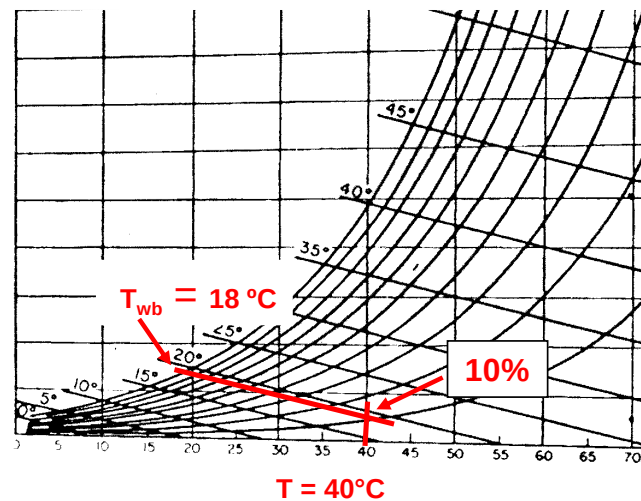
- a. How high should the drying rate be to keep up with the rate of filter cake production.
- b. How many trays of 4 m² are required?
- c. When recycling part of the air from the dryer, till which temperature should the from the dryer with water vapor saturated drying air be cooled to reach again the initial moisture content of 0.04 kg water per kg dry air?

Exercise 7.7

Given $H_r = 60\%$ $T_f = 40\text{ }^\circ\text{C}$ $T_{wb} = 18\text{ }^\circ\text{C}$ $\phi_{\text{moist}} = \phi_{\text{vocht}} = 0.18$
 $\Delta H_v = 2.45 \times 10^6\text{ J/kg}$ $h = 35\text{ W/m}^2\text{K}$ $W_{\text{moist}} = W_{\text{vocht}}$

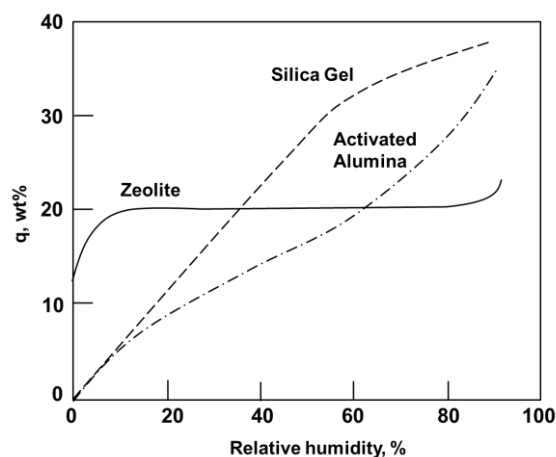
Find a) Relative humidity of the drying air
 b) Constant drying velocity in wt%.
 c) Time to reduce the moisture content to 18 wt%

Solution a) Go in the psychrometric chart (Fig. 7.3) from $18\text{ }^\circ\text{C}$ parallel to the adiabatic saturation line (allowed in case of air-water) to $40\text{ }^\circ\text{C}$. There you intersect with the line with 10% relative humidity



$$b) \quad r_c = \frac{h(T_f - T_{wb})}{\Delta H_{\text{vap}}} = \frac{30(40 - 18)}{2.45 \times 10^6} = 2.7 \times 10^{-4} \left(\frac{\text{kg}}{\text{m}^2\text{s}} \right)$$

c) At 60% relative humidity the diagram in Fig 6.5 RH gives an initial humidity of 32 wt%



Initial humidity 32 wt% $\rightarrow$ initial weight

$$\phi_{\text{vocht}} = \frac{w_{\text{vocht}}}{w_{\text{vocht}} + w_{\text{silica}}} \rightarrow \frac{w_{\text{vocht}}}{w_{\text{silica}}} = \frac{\phi_{\text{vocht}}}{1 - \phi_{\text{vocht}}}$$

$$\text{Initial: } W_0 = \frac{w_{\text{vocht}}}{w_{\text{silica}}} = \frac{\phi_{\text{vocht}}}{1 - \phi_{\text{vocht}}} = \frac{0.32}{1 - 0.32} = 0.470 \left(\frac{\text{kg water}}{\text{kg silica}} \right)$$

$$\text{Critical: } W_{cr} = \frac{w_{\text{vocht}}}{w_{\text{silica}}} = \frac{\phi_{\text{vocht}}}{1 - \phi_{\text{vocht}}} = \frac{0.18}{1 - 0.18} = 0.220 \left(\frac{\text{kg water}}{\text{kg silica}} \right)$$

$$\text{Drying time: } t_C = \frac{w_0}{A} \frac{(W_0 - W_{cr})}{r_C} = \frac{1}{1} \frac{(0.47 - 0.22)}{2.7 \times 10^{-4}} = 926 \text{ (s)} = 15.4 \text{ (min)}$$

w_0 = weight of dry solid = 1 kg silica

A = evaporating surface r_C is per m^2 , so A = 1 m^2

Exercise 7.8

Given $H_r = 10\%$ $T_f = 80\text{ }^\circ\text{C}$ $W_0 = 0.10$ $W_{\text{end}} = 0.01$
 $A = 2500\text{ m}^2$ $\Delta H_v = 2.45 \times 10^6\text{ J/kg}$ $\rho_{\text{air}} = 1.3\text{ kg/m}^3$

Find a) Specific drying rate?
 b) Minimal value of the heat transfer coefficient.
 c) Minimal velocity of the air stream

Solution a) Mass flow cake 69.4 ton/hr

Moisture content (10%) 6.94 ton/hr
 To be reduced to 0.1 wt%, 0.0694 ton/hr.
 To evaporate 6.87 ton/hr

Drying rate = $r_c A = 6870/3600 = 1.91\text{ kg/s}$

Specific drying rate $r_c = 1.91/2500 = 7.64 \times 10^{-4}\text{ kg/m}^2\text{s}$

b) Specific drying rate: $r_c = \frac{h(T_f - T_{wb})}{\Delta H_{\text{vap}}} \left(\frac{\text{kg}}{\text{m}^2\text{s}} \right)$

Psychrometric chart (Fig. 7.3), starting point $80\text{ }^\circ\text{C}$ and $\Psi=10\%$
 Line parallel to the adiabatic line and then straight down.

$T_{wb} = 47\text{ }^\circ\text{C}$

$$h = \frac{r_c \Delta H_{\text{vap}}}{(T_f - T_{wb})} = \frac{7.64 \times 10^{-4} \cdot 2.45 \times 10^6}{(80 - 47)} = 56.7\text{ (W / m}^2\text{K)}$$

c) $h = 12(\rho v)^{0.8} = 12(1.3 \times v)^{0.8} \frac{\text{J}}{\text{m}^2\text{s}} \rightarrow v = 5.3\text{--}5.4\text{ m/s}$

Exercise 7.9

<i>Given</i>	$H = 0.04 \text{ kg/kg}$	$T_f = 95 \text{ }^\circ\text{C}$	$W_o = 0.30$
	$A_{\text{Tray}} = 4 \text{ m}^2$	$v = 2 \text{ m/s}$	$W_{\text{end}} = 0.05$
	$\Delta H_v = 2.45 \times 10^6 \text{ J/kg}$		$\rho_{\text{air}} = 1 \text{ kg/m}^3$

- Find*
- The drying rate needed to keep up with the rate cake production.
 - Number trays of 4 m² required
 - Temperature to which the saturated drying air should be cooled

Solution a) Cake production (precipitaatproductie) = 351 kg/hr

$$r_{\text{vap}} = \text{precipitaatproductie} \times (W_o - W_{\text{end}}) = \frac{351}{3600} \times (0.30 - 0.05) = 0.0244 \text{ (kg/s)}$$

(3)

$$\text{b) } r_c = \frac{h(T_f - T_{\text{wb}})}{\Delta H_{\text{vap}}} \left(\frac{\text{kg}}{\text{m}^2 \text{s}} \right)$$

$$h = 12(\rho v)^{0.8} = 12(1 \times 2)^{0.8} = 20.9 \frac{\text{J}}{\text{m}^2 \text{s}}$$

Psychrometric chart (Fig. 7.3), start at 0.04 kg/kg and 95 °C
Line parallel to adiabatic line and then straight down

$$T_{\text{wb}} = 45 \text{ }^\circ\text{C}$$

$$r_c = \frac{h(T_f - T_{\text{wb}})}{\Delta H_{\text{vap}}} = \frac{20.9(95 - 45)}{2.45 \times 10^6} = 4.265 \times 10^{-4} \text{ (kg/m}^2 \text{s)}$$

$$A = \frac{r_{\text{vap}}}{r_c} = \frac{0.0244}{4.265 \times 10^{-4}} = 57.2 \text{ m}^2$$

$$\text{Number of plates} = A/4 = 15$$

- c) From 45 °C follow the saturated line down until a residual moisture of 0.04 is reached. Moisture content will remain constant when heating. .

Alternative is to determine the intersection of the 0.04 kg/kg line with the saturation line..

Reading from diagram (Fig. 7.3) yields in both cases 36/37 °C.

Chapter 8: CRYSTALLIZATION & PRECIPITATION

Exercise 8.1

Determine the maximum yield of anhydrous sugar crystals deposited at equilibrium in the following situations. Initially 100 kg of water is present.

- A saturated sugar solution is cooled from 80 °C to 20 °C.
- A saturated sugar solution at 80 °C has half of its water evaporated and the solution is then cooled to 20 °C.

Temperature:	20°C	80°C	
Solubility:	2.04	3.62	(kg anhydrous salt/kg water)

Exercise 8.2

A solution of 80 wt% of naphthalene in benzene is cooled to 30°C to precipitate naphthalene crystals. The solubility of naphthalene at 30°C is 45 wt% in solution. The feed rate is 5000 kg solution / h.

- Calculate the maximum production rate of crystals
- Calculate the maximum yield in kg crystals per unit weight of solute fed

Exercise 8.3

A cooling crystallizer is used to crystallize sodium acetate, $\text{NaC}_2\text{H}_3\text{O}_2$ (MW = 0.082 kg/mol), from a saturated aqueous solution. The stable form of the crystals is a hydrate with 3 moles of water. The feed is initially saturated at 40°C while the crystallizer is cooled to 0°C until equilibrium conditions are established.

If the anhydrous salt is dissolved in 100 kg of water/h, how many kg of crystals/h are collected?

Temperature:	0	10	20	30	40	60	(°C)
Solubility:	0.363	0.408	0.465	0.545	0.655	1.39	(kg anhydrous salt/kg water)

Exercise 8.4

Copper sulfate is crystallized as $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ by combined evaporative/cooling crystallization. 1000 kg/h of water is mixed with 280 kg/h of anhydrous copper sulfate (MW = 0.160 kg/mol) at 40°C. The solution is cooled to 10°C and 38 kg/h of water is evaporated in the process. How many kg/h of crystals can be collected theoretically?

Temperature:	10°C	40°C	
Solubility:	17.4	28.5	(g anhydrous salt/100 g water)

Exercise 8.5

Calculate the maximum (theoretical) yield of pure crystals that could be obtained from a saturated solution of sodium sulfate ($M_{anh} = 0.142$ kg/mol) with 0.25 kg anhydrous salt/kg free water by

- isothermal evaporation of 25% of the free water
- isothermal evaporation of 25% of the original water
- cooling to 10°C and assuming 2% of the original water lost by evaporation; $X_C = 0.090$ kg anhydrous salt / kg solute-free solvent
- adding 0.75 kg ethanol / kg free water; $X_C = 0.030$ kg anhydrous salt / kg solute-free solvent
- adding 0.75 kg ethanol / kg free water; $X_C = 0.068$ kg $\text{Na}_2\text{SO}_4 \cdot 10\text{aq}$ / kg solute-free solvent

Exercise 8.6

Assuming that $G \propto \Delta c$, determine whether or not the Arrhenius equation is valid for $(\text{NH}_4)_2\text{SO}_4$ crystallization. If it is valid, determine ΔE .

Note: c^* depends on T and ammonium sulfate crystallizes as an anhydrate

Given	Temperature:	30	60	90	°C
	Solubility:	78	88	99	g anhydrous salt/100 g water
	s :	0,05	0,05	0,01	-
	G :	5,0	8,0	0,6	10^{-7} m/s

Exercise 8.7

Estimate the supersaturation s of an aqueous solution of K_2SO_4 at 30°C required to get a heterogeneous nucleation rate of 1 nucleus per second. Eq. 8.16 is applicable with an apparent interfacial surface tension γ of $2 \cdot 10^{-3}$ Pa.s and a pre-exponential coefficient $A = 10^{25}$. The density of the K_2SO_4 crystals is 2662 kg/m^3 , its molecular weight 0.174 kg/mol .

Exercise 8.8

Potash alum was crystallized from its aqueous solution in a laboratory scale continuous MSMPR crystallizer of 10 l capacity at steady state, supersaturation being achieved by cooling. The size analysis of the produced crystals resulting from a steady-state sample taken from the crystallizer operated at means residence time of 900 s, is given in the table below. Assume that the crystal density is 1770 kg/m^3 and the volume shape factor is 0.47.

Sieve Analysis Data:	Standard sieve Size (μm)	Weight on the sieve (g/kg of water)
	850	2,3
	710	16,3
	500	20,9
	355	28,9
	250	18,0
	180	10,7
	125	3,7
	90	1,5
	63	0,6
	45	

The steps to be taken are:

- Calculate the population density distribution of the product crystals at mean size L over a size interval ΔL
- Determine the growth and nucleation rate graphically from the population density plot on a semilog scale.

Exercise 8.9

A sodium acetate crystallizer receives a saturated solution at 60°C. This solution is seeded with 0.2 mm average size crystals and cooled to 10°C. Use a basis of 100 kg of entering water to which 0.25 kg seeds is added. The sodium acetate crystallizes as the trihydrate.

- What is the mean size of the product crystals L_p ?
- If the residence time is 21 hours, what linear growth rate G is required?

Temperature:	10	60	(°C)
Solubility:	40,8	139	(g anhydrous salt/100 g water)

Exercise 8.10

Design a crystallizer to produce potassium sulfate crystals assuming an MSMPR unit operating continuously at steady state.

The following specifications and data are available:

relative nucleation kinetics:	B	$= 1 \times 10^{19} M_T G^2$ (no/s kg water)
production rate:	P	$= 1000$ kg/h
product dominant size:	L_D	$= 490$ μm
magma concentration:	M_T	$= 0.100$ kg/kg water
crystal density:	ρ_c	$= 2660$ kg/m ³
shape factors:	k_v	$= 0.525$
outlet concentration	C_{out}	$= 0.1167$ kg/kg water
density of product solution	ρ_{soln}	$= 1090$ kg/m ³

SOLUTIONS

Exercise 8.1

Given Temperature: 20°C 80°C
 Solubility: 2.04 3.62 (kg anhydrous sugar/kg water)

Find a. the yield of sugar crystals when a saturated solution is cooled from 80°C to 20°C.
 b. the yield of sugar crystals when a saturated solution is cooled from 80°C to 20°C and half of its water is evaporated.

Base calculations on anhydrous sugar.

Thoughts The problem is easily solved by applying Eq. 8.8 with $\varphi_E = 0$ and $\varphi_E = 0.5$, respectively. A more versatile approach (which will also work in more complex situations, e.g. when hydrates are involved), would be to derive both the water and the sugar balance (Eqs. 8.6 and 8.7). The solubility data suggest to work on a solute-free basis.

Solution a. sugar balance: sugar in = crystals produced + sugar in solution¹
 or $X_F \cdot F' = C + X_C \cdot S'$

water balance: $F' = S'$

where F' and S' = solute-free solvent per unit time;

$X_F = 3.62$ and $X_C = 2.04$ kg anhydrous sugar / kg water.

Solving for C gives $C / F' = X_F - X_C = 1.58$ kg anhydrous sugar / kg water fed.

The maximum yield = $\frac{\text{sugar crystals produced}}{\text{sugar in feed}} = \frac{C}{X_F \cdot F'} = 0.436$

b. sugar balance: $X_F \cdot F' = C + X_C \cdot S'$

water balance: water in feed = water evaporated + water in solution

or $F' = 0.5 \cdot F' + S'$

Solving for C gives

$C / F' = X_F - 0.5 \cdot X_C = 2.60$ kg anhydrous sugar crystals / kg water fed.

The maximum yield = $\frac{C}{X_F \cdot F'} = 0.718$

Exercise 8.2

Given $F = 5000$ kg/h, $X_F = 80$ wt% naphthalene and $X_C (30^\circ\text{C}) = 45$ wt% in solution

Find a. maximum production rate
 b. maximum yield (kg crystals / unit weight of solute fed)

Thoughts Derive the naphthalene and benzene balances.
 Note that now X_F and X_C are weight fractions of the *total* feed and *total* solution, respectively

Solution a. naphthalene balance: naphthalene in = crystals produced + naphthalene in solution
 or $X_F \cdot F = C + X_C \cdot S$

benzene balance: $(1 - X_F) \cdot F = (1 - X_C) \cdot S$

both F and S refer to solution (solvent + solute): $X_F = 0.80$ and $X_C = 0.45$ kg/kg solution.

Solving for C gives $C = 3182$ kg crystals / h

b. The (maximum) yield = crystals produced (loss free) / naphthalene fed = $C / X_F \cdot F = 0.795$

¹ Note that here S means solvent, not solid, and C means solid crystals, not concentration.

Exercise 8.3

Given $F' = 100 \text{ kg water/h}$ $T_f = 40^\circ\text{C}$ $T_p = 0^\circ\text{C}$
 $M_{Ac} = 0.082 \text{ kg mol}^{-1}$ $M_{3aq} = 3 \cdot 0.018 = 0.054 \text{ kg mol}^{-1}$

The stable form of sodium acetate crystals is a hydrate with 3 moles of water.

Temperature: 0 10 20 30 40 60 $^\circ\text{C}$

Solubility: 0.363 0.408 0.465 0.545 0.655 1.39 kg anhydrous salt/kg water

Find The product flow of crystals that are collected after cooling to 0°C .

Thoughts Derive the water and acetate balances on a solute-free basis (see exercise 8.1)

Solution water balance: $F' = C \cdot M_{3aq} / M_{Ac} + S'$
 acetate balance (anhydrous): $X_F \cdot F' = C + X_C \cdot S'$
 where $C = \text{kg acetate} / \text{h}$ (note that the actual product carries 3 aq / mol salt)
 $X_F = 0.655$ and $X_C = 0.363 \text{ kg anhydrous acetate / kg water}$.
 Solving for C gives $C = 38.4 \text{ kg crystals (waterfree basis) / h}$.
 The product stream in kg crystals.3aq / h = $C \cdot (M_{3aq} + M_{Ac}) / M_{Ac} = 63.6 \text{ kg / h}$.

Exercise 8.4

Given $F' = 1000 \text{ kg water/h}$ $\text{CuSO}_4.0\text{aq} = 280 \text{ kg/h}$ $T_f = 40^\circ\text{C}$
 $V = 38 \text{ kg water evaporated/h}$ $T_p = 10^\circ\text{C}$
 $M_{anh} = 0.160 \text{ kg mol}^{-1}$ $M_{5aq} = 5 \cdot 0.018 = 0.090 \text{ kg mol}^{-1}$

The stable form of copper sulfated crystals is a hydrate with 5 moles of water

Temperature: 10 $^\circ\text{C}$ 40 $^\circ\text{C}$

Solubility: 17.4 28.5 g anhydrous salt/100 g water

Find The product flow of crystals collected.

Thoughts Derive the sulfate and water balances (see Eqs. 8.6 & 8.7)

Solution water balance: $F' = V + C \cdot M_{5aq} / M_{anh} + S'$
 sulfate balance (anhydrous): $280 = C + X_C \cdot S'$
 where $C = \text{kg anhydrous sulfate / h}$;
 $X_C = 0.174 \text{ kg anhydrous sulfate / kg water}$.
 Solving for C gives $C = 124.8 \text{ kg crystals (waterfree basis) / h}$.
 The product stream in kg crystals.5aq / h = $C \cdot (M_{5aq} + M_{anh}) / M_{anh} = 195.0 \text{ kg / h}$.
 Check that the feed concentration at 40°C is just below the saturation value.
 (280 kg $\text{CuSO}_4.0\text{aq/h}$ + 1000 kg $\text{H}_2\text{O/h}$ $\rightarrow$ 28.0 g $\text{CuSO}_4/100 \text{ g H}_2\text{O}$)

Exercise 8.5

Given $X_F = 0.25$ kg anhydrous water/kg free water.
 The stable form of sodium sulfate crystals is a hydrate with 10 moles of water, $n_{aq} = 10$.
 $M_{anh} = 0.142$ kg mol⁻¹, $M_w = 0.018$ kg mol⁻¹.

Find Calculate the maximal (theoretical) yield of crystals from a saturated aqueous solution of sodium sulfate by:

- isothermal evaporation of 25 % of the free water
- Isothermal evaporation of 25% of the original water
- cooling to 10 °C and assuming 2 % of original water lost by evaporation; at 10°C $X_C = 0.09$ kg anhydrous salt/kg solute-free solvent
- adding 0.75 kg ethanol / kg free water; $X_C = 0.030$ kg anhydrous salt/kg solute-free solvent after adding that much of ethanol
- adding 0.75 kg ethanol / kg free water; $X_C = 0.068$ kg hydrated salt/kg solute-free solvent after adding that much of ethanol

Thoughts The weight of crystals precipitated is calculated from the difference between the amount fed to the crystallizer and the amount left in the saturated solution after precipitation. Hence, deriving the appropriate mass-balances, similar to the overall balance in Eq. 8.6, can solve this problem. Note that *bound water* is the part of the solvent required to crystallize all of the solute, the remainder is *free water*. Note also that in this exercise, while X_C is expressed per kg of *solute-free* solvent, X_F is expressed per unit weight of *free* water. Therefore, in addition to F' and S' (solute-free solvent per unit time, see Eq. 8.4), it is convenient to introduce F'' and S'' in kg *free* water per unit time.

Solution The feed to the crystallizer amounts to F'' kg of *free* water per unit time. *Bounded* water amounts to $\frac{M_{aq} \cdot 10}{M_{salt}} = 1.268$ kg water / kg salt.0aq. Then, this feed contains $X_F \cdot F''$ anhydrous salt binding $X_F \cdot F'' \cdot \frac{M_w \cdot n_{aq}}{M_{anh}} = X_F \cdot F'' \cdot 1.268$ kg water per unit time and the total amount of water fed equals $1.317 \cdot F''$.

- a. Salt balance:

$$X_F \cdot F'' = C + X_F \cdot S''$$

where C = kg crystals(anh) precipitated and S'' = kg free water (both per unit time).

Water balance:

In per unit time = free water + water bounded to $X_F \cdot F''$ kg salt.0aq

Out per unit time = evaporated + water in precipitated crystals + free water in solution + water in solution bounded to $X_F \cdot S'' = X_F \cdot F'' - C$ kg salt.0aq

or

$$F'' + X_F \cdot F'' \cdot 1.268 = 0.25 \cdot F'' + C \cdot 1.268 + S'' + X_F \cdot S'' \cdot 1.268$$

Solving these two equations for C gives the maximum theoretical $yield = \frac{C}{X_F F''} = 0.25$ The

product stream of hydrated crystals amounts to

$$C + C \cdot 1.268 = 0.142 \text{ kg crystals.10aq / kg free water fed.}$$

- b. The water balance now reads

$$F'' + X_F \cdot F'' \cdot 1.268 = 0.25 \cdot (F'' + X_F \cdot F'' \cdot 1.268) + C \cdot 1.268 + S'' + X_F \cdot S'' \cdot 1.268$$

and with the salt balance as in previous part

$$X_F \cdot F'' = C + X_F \cdot S''$$

$$C \text{ (and } S'') \text{ is solved giving } Yield = \frac{C}{X_F F''} = 0.329$$

and the product stream of hydrated crystals amounts to

$$C \cdot 2.268 = 0.187 \text{ kg crystals.10aq / kg free water fed.}$$

- c. Because X_C is expressed per unit weight of solute-free solvent, the anhydride balance now reads:

$$X_F \cdot F'' = C + X_C \cdot S'$$

where S' = total water in solution (solute-free base) per unit time.

Water balance:

$$F'' + X_F \cdot F'' \cdot 1.268 = 0.02 \cdot (F'' + X_F \cdot F'' \cdot 1.268) + C \cdot 1.268 + S'$$

$$\text{Solving for } C \text{ gives } Yield = \frac{C}{X_F F''} = 0.604$$

and the product stream of hydrated crystals amounts to

$$2.268 \cdot C = 0.343 \text{ kg crystals.10aq / kg free water fed.}$$

- d. Addition of 0.75 kg ethanol/kg free water decreases the solubility of the sulfate to $X_C = 0.030$ kg salt (anh) / kg solute-free solution (water + ethanol). The solvent balance (water + ethanol) now reads

$$F'' + X_F \cdot F'' \cdot 1.268 + 0.75 \cdot F'' = C \cdot 1.268 + S'$$

The salt balance is the same as in part c:

$$X_F \cdot F'' = C + X_C \cdot S'$$

$$\text{Solving for } C \text{ gives } Yield = \frac{C}{X_F F''} = 0.782$$

and the product stream of hydrated crystals amounts to

$$2.268 \cdot C = 0.443 \text{ kg crystals.10aq / kg free water fed.}$$

- e. Expressing the solubility in kg hydrated crystals / kg solute-free solution changes the anhydrous salt balance, the amount of hydrated salt in solution has to be converted to an anhydrous basis:

$$\text{kg anhydrous salt/unit time: } X_F \cdot F'' = C + X_C \cdot S' \cdot \frac{0.142}{0.142 + 10 \cdot 0.018}$$

The solvent (water + ethanol) balance is the same as given in part d:

$$\text{kg solvent/unit time: } F'' + X_F \cdot F'' \cdot 1.268 + 0.75 \cdot F'' = C \cdot 1.268 + S'$$

Solving for C and conversion to hydrated crystals per unit weight of free water fed gives exactly the same result as in part d.

Exercise 8.6

Given	Temperature:	30	60	90	°C
	Solubility:	78	88	99	g anhydrous salt/100 g water
	s (Eq. 8.13):	0,05	0,05	0,01	-
	G:	5,0	8,0	0,6	10 ⁻⁷ m/s

ammonium sulfate crystallizes as anhydrate

- Find**
- determine whether or not the Arrhenius equation is valid
 - if so, determine ΔE .

Thoughts If the Arrhenius equation is valid (Eq. 8.27), a plot of $\ln(k_G)$ against $1/T$ will result in a straight line. Assuming $G \propto \Delta c$, an expression for k_G can be found by rewriting Eq. 8.29 and elimination of c^* . Note that the exponential factor n is unknown beforehand and should be determined by trial and error.

Solution a. From Eqs. 8.13, 8.27 and 8.29 the following expression for k_G is derived:

$$\left. \begin{array}{l} \text{Eq. 8.29 } G = k_G (c - c^*)^n \\ \text{Eq. 8.13 } s = \frac{c - c^*}{c^*} \\ \text{Eq. 8.27 } k_G = k_G^0 \exp\left[\frac{-\Delta E}{RT}\right] \end{array} \right\} k_G = \frac{G}{\left(c - \frac{c}{s+1}\right)^n} = k_G^0 \exp\left[\frac{-\Delta E}{RT}\right]$$

- b. Taking $n = 1$ (+ in graph below) does not give a straight line when $\ln(k_G)$ is plotted as a function of $1/T$

Taking $n = 2$ (o in graph below) a linear dependence is obtained $\rightarrow \Delta E = 8.23 \text{ kJ/mol}$

MathCad calculation:

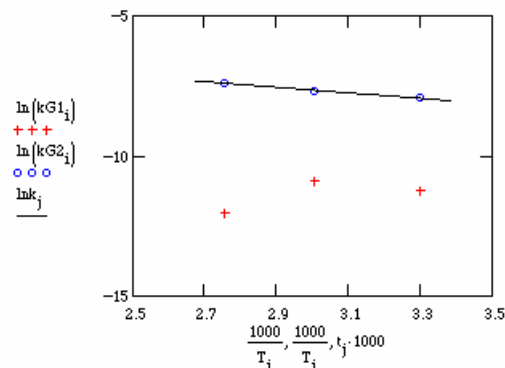
$$G := \begin{bmatrix} 5 \cdot 10^{-7} \\ 8 \cdot 10^{-7} \\ 6 \cdot 10^{-8} \end{bmatrix} \quad c := \begin{bmatrix} 0.78 \\ 0.88 \\ 0.99 \end{bmatrix} \quad s := \begin{bmatrix} 0.05 \\ 0.05 \\ 0.01 \end{bmatrix} \quad R := 8.314 \quad T := \begin{bmatrix} 303 \\ 333 \\ 363 \end{bmatrix}$$

$$i := 0..2 \quad kG1_i := \frac{G_i}{c_i - \frac{c_i}{s_i + 1}} \quad kG2_i := \frac{G_i}{\left(c_i - \frac{c_i}{s_i + 1}\right)^2} \quad x_i := \frac{1}{T_i}$$

$$y2_i := \ln(kG2_i) \quad j := 0..100 \quad t_j := x_2 \cdot 0.97 + \frac{1}{100} (x_0 \cdot 1.05 - x_2)$$

$$m := \text{slope}(x, y2) \quad a := \text{intercept}(x, y2) \quad \ln k_j := a + m \cdot t_j$$

$$m = -989.99 \quad \Delta E := -m \cdot R \quad \Delta E = 8.23 \cdot 10^3 \text{ J/mol}$$



Exercise 8.7

Given $\gamma = 2 \cdot 10^{-3} \text{ Pa.s}$ $T = 30^\circ\text{C}$
 $N_{av} = 6.02 \cdot 10^{23}$ $\rho_{salt} = 2662 \text{ kg/m}^3$
 $k = R / N_{av}$ $M_{salt} = 0.174 \text{ kg/mol}$

Find the value of supersaturation s to give a nucleation rate of 1 nucleus / s.

Thoughts Eq. 8.16 is applicable, taking $A \approx 10^{25} \text{ nuclei / s cm}^3$ (see text on top of page 8.16).
 On dissociation K_2SO_4 gives 2K^+ and 1SO_4^{2-} hence $v = 3$; molecular volume $v_m = M_{salt} / \rho_{salt} \cdot N_{av}$

Solution Solve $1 = 10^{25} \cdot \exp\left(-\frac{16\pi \cdot v_m^2 \cdot \gamma^3}{3 \cdot 3^2 \cdot (k \cdot 303)^3 \cdot s^2}\right)$ for s , giving $s = 0.00645$

Note that at slightly higher values of s , B^0 is increasing tremendously.

Exercise 8.8

Given MSMPR crystallizer
 $V = 10 \text{ l}$ $\tau = 900 \text{ s}$ $k_v = 0.47$ $\rho_c = 1770 \text{ kg/m}^3$

The sieve analysis data is given in the shaded columns below - sieve size and W (the weight on the sieve).

Find a. the population density distribution over a size interval ΔL
 b. the growth and nucleation rate graphically

Thoughts The population density n , is defined as the number of crystals (ΔN) in the size range of L to $L + \Delta L$.
 The nucleation and growth rate can then be found by a plot of $\ln(n)$ against L to give a straight line according to Eq. 8.37.

Solution a. The population density is given by (Eqs. 8.31 and 8.32):

$$n = \frac{\Delta N}{\Delta L} = \frac{W}{k_v \rho_c L^3 \Delta L}$$

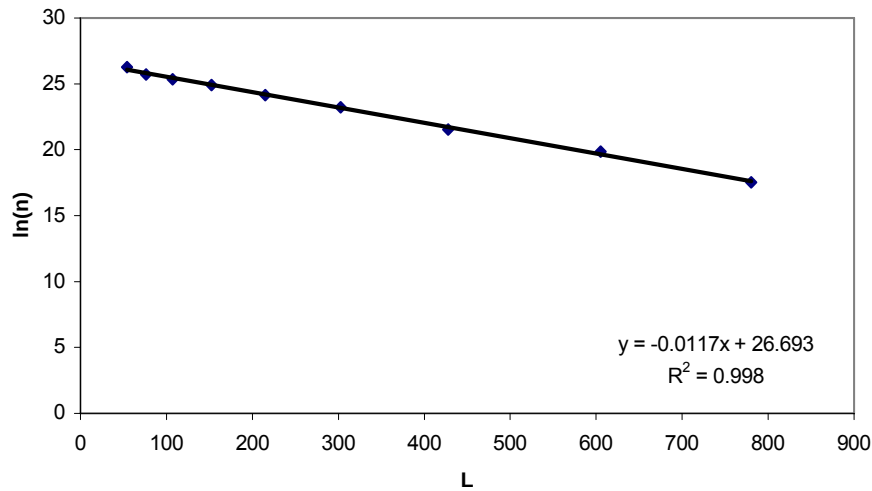
Population densities n calculated as a function of average crystal sizes L :

Sieve size [μm]	L [μm]	ΔL [μm]	W [g/kg]	n no./m kg	$\ln(n)$
850	780	140	2.3	4.16E+07	17.54396
710	605	210	16.3	4.21E+08	19.85895
500	427.5	145	20.9	2.22E+09	21.51973
355	302.5	105	28.9	1.20E+10	23.20421
250	215	70	18	3.11E+10	24.16054
180	152.5	55	10.7	6.59E+10	24.91199
125	107.5	35	3.7	1.02E+11	25.35109
90	76.5	27	1.5	1.49E+11	25.72833
63	54	18	0.6	2.54E+11	26.26243
45					

b. Combine Eqs. 8.37 and 8.38 to give: $n = \frac{B^0}{G} \exp\left[\frac{-L}{G\tau}\right] \Rightarrow \ln(n) = \ln\left[\frac{B^0}{G}\right] - \frac{L}{G\tau}$

Plotting of $\ln(n)$ against L gives a straight line with slope of $1/G\tau = 0.0117$ and intercept $\ln(B^0/G) = 26.693$, see graph below.

Thus, as $\tau = 900$ seconds: $G = 1 / 0.0117 \cdot 900 = 0.095 \mu\text{m/s}$
and $B^0 = 3.91 \cdot 10^{11} \cdot 0.095 \cdot 10^{-6} = 3.72 \cdot 10^4 \text{ no/s kg.}$



Exercise 8.9

Given $F' = 100 \text{ kg}$ $T_F = 60^\circ\text{C}$
 $\text{Seed} = 0.25 \text{ kg (in the form of trihydrate)}$ $T_P = 10^\circ\text{C}$
 $L_{\text{seed}} = 0.2 \text{ mm}$ $M_{\text{NaAc}} = 0.082 \text{ kg/mol}$

The stable form of sodium acetate crystals is a hydrate with 3 moles of water

Temperature: 10 60 °C
 Solubility: 40,8 139 g anhydrous salt/100 g water

Find a. the mean size of the product crystals.
 b. the required linear growth rate G if the residence time is 21 hrs.

Thoughts a. The mass of a crystal is proportional to L_{crystal}^3 (Eq. 8.23a).
 The total mass $\text{Seed} \propto n \cdot k_V \cdot \rho \cdot L_{\text{seed}}^3$ where n = total number of seed crystals.
 Analogously for the total mass of product crystals: $P \propto n \cdot k_V \cdot \rho \cdot L_{\text{product}}^3$
 Hence find the ratio P / Seed from appropriate mass balances.
 b. When the linear growth rate is independent of crystal size and at constant supersaturation, the simple ΔL -law of Eq. 8.26 applies: $\Delta G = \Delta L / \Delta t$

Solution a. Mass balance over water in the system:
 Water fed (F') + crystal water in seed crystals = water in saturated solution (L) +
 crystal water in product crystals (P), or

$$F' + \text{Seed} \cdot \frac{3M_{\text{water}}}{M_{\text{anh.salt}} + 3M_{\text{water}}} = L + P \cdot \frac{3M_{\text{water}}}{M_{\text{anh.salt}} + 3M_{\text{water}}}$$

Mass balance over anhydrous salt:

Salt in feed + salt in seed crystals = salt in solution + salt in product crystals

$$F' \cdot x_F + \text{Seed} \cdot \frac{M_{\text{salt}}}{M_{\text{salt}} + 3M_{\text{water}}} = L \cdot x_P + P \cdot \frac{M_{\text{salt}}}{M_{\text{salt}} + 3M_{\text{water}}}$$

Solving for L and P with $x_F = 1.39$ and $x_P = 0.408$ in kg anhydrous salt / kg water gives $L = 11.6$ kg water and $P = 223.0$ kg crystals (anhydrous).

$$\frac{\text{Seed}}{P} = \left(\frac{L_{\text{seed}}}{L_{\text{product}}} \right)^3 \text{ or } L_{\text{product}} = L_{\text{seed}} \cdot \left(\frac{P}{\text{Seed}} \right)^{1/3} = 1.93 \text{ mm}$$

b. Follows for the linear growth rate

$$G = \frac{\Delta L}{\Delta \tau} = \frac{L_{\text{product}} - L_{\text{seed}}}{\Delta \tau} = \frac{1.93 - 0.2}{21} = 0.082 \text{ mm/h}$$

Exercise 8.10

Given	nucleation kinetics:	B^0	$= 1 \times 10^{19} M_T G^2$ (no/s kg water) (see Eq. 8.19)
	production rate:	P	$= 1000$ kg crystals/h
	product dominant size:	L_D	$= 490 \mu\text{m}$
	magma concentration:	M_T	$= 0.100$ kg crystals/kg water
	crystal density:	ρ_c	$= 2660$ kg/m ³
	shape factors:	k_v	$= 0.525$
	outlet concentration	C_{out}	$= 0.1167$ kg/kg water
	density of product solution	ρ_{soln}	$= 1090$ kg/m ³

Design an MSMPR crystallizer

Thoughts Designing a crystallizer means to estimate the residence time τ of the magma (solid – saturated mixture), the crystallizer volume V and required feed flow rate Q .

Eq. 8.41 relates residence time τ and magma concentration M_T . The initial population density n_0 can be eliminated with Eq. 8.38, the nucleation rate B^0 with Eq. 8.19 and the crystal growth rate G with Eq. 8.40. Production rate P and residence time τ determine the capacity of the crystallizer; the density of crystals and the density of the saturated solution determine the total volume required.

Solution Solving Eq. 8.41:

$$M_T = 6\rho_c k_v \cdot n_0 \cdot (G\tau)^4 = 6\rho_c k_v \cdot \frac{B^0}{G} \cdot (G\tau)^4 =$$

$$6\rho_c k_v \cdot 10^{19} M_T G \cdot (G\tau)^4 = 6\rho_c k_v \cdot 10^{19} M_T \frac{L_D}{3\tau} \cdot \left(\frac{L_D}{3} \right)^4$$

for τ gives $\tau = \underline{9740 \text{ s}}$

A production rate of P kg crystals/h requires a feed flow rate $Q = \frac{P}{3600 \cdot M_T} = \underline{2.78 \text{ kg water/s}}$.

The capacity of the crystallizer = $Q \cdot \tau = 27.056$ ton water.

1 kg H₂O corresponds to $(1 + C_{\text{out}})$ kg solution; the volume of the saturated solution equals

$$\frac{(1 + C_{\text{out}}) \cdot 27056}{\rho_{\text{soln}}} \frac{\text{kg solution}}{\text{kg solution/m}^3 \text{ solution}} = 27.72 \text{ m}^3$$

The crystals occupy $27056 \cdot M_T / \rho_c = 1.02 \text{ m}^3$.

Hence the total volume = $27.72 + 1.02 \approx \underline{29 \text{ m}^3}$.

Chapter 8: CRYSTALLIZATION & PRECIPITATION (continued)

Exercise 8.12

Molasses contains calcium which is being removed by precipitation. The feed amounts 15 m³/hr molasses with a density of 1400 kg/m³ and contains 0.5 wt% Ca²⁺ (MW=40 g/mol) of which 98% needs to be removed by precipitation as CaSO₄ (MW=136 g/mol, density=2600 kg/m³) with concentrated sulfuric acid (H₂SO₄, MW=98 g/mol). The solubility product of CaSO₄ is given by:

$$K_s^{\text{CaSO}_4} = [\text{Ca}^{2+}][\text{SO}_4^{2-}] = 5 \times 10^{-5} \frac{\text{mol}^2}{\text{kg}^2}$$

- Calculate the concentration [SO₄²⁻] required to achieve the desired Ca²⁺ removal.
- How much (kg/hr) sulfuric acid needs to be dosed.
- Calculate the resulting slurry concentration (vol%) of CaSO₄ particles

Exercise 8.13

A crystallization plant produces 250.000 ton dry salt per year. The feed consists of saturated brine with a concentration of 300 kg/m³ NaCl (MW=58 kg/kmol, density 2170 kg/m³) and a density of 1200 kg/m³. This saturation concentration can be considered as temperature independent. Salt is crystallized from the saturated brine in a 3-step crystallizer in which 20% of the water from the initial feed stream is evaporated in each step. The crystallization plant operates 8000 hrs/year.

- Calculate the yield of the crystallization plant.
- Calculate the crystal production rate C (kg/hr)
- Determine the brine feed stream (kg/hr)

We now consider the first stage of the crystallization plant, which can be considered a MSMPR-crystallizer. The nucleation rate Bo in number of nuclei per unit of time and per m³ solution depends on the growth rate G and the Magma concentration M_T (kg/m³ solution) as follows:

$$Bo = 1.6 \cdot 10^{18} \cdot M_T G^2$$

The residence time of the slightly supersaturated solution in the first stage needs to be selected such that the dominant crystal size equals 0.5 mm. Volume form factor kv=0.56. NaCl crystallizes as anhydrate (0 aq).

- Calculate the required residence time τ in the crystallizer.
- Calculate the needed slurry filled volume of the first stage to obtain the required residence time.
- Calculate the volume of the crystals in the first stage crystallizer.

Exercise 8.12

Given $F = 15 \text{ m}^3 \text{ hr}^{-1}$ $C_{\text{Ca}^{2+}, \text{in}} = 0.5 \text{ wt}\%$
 $\text{MW}_{\text{Ca}^{2+}} = 40 \text{ g/mol}$ $\text{MW}_{\text{CaSO}_4} = 136 \text{ g/mol}$ $\text{MW}_{\text{H}_2\text{SO}_4} = 98 \text{ g/mol}$
 $\rho_{\text{molasses}} = 1400 \text{ kg/m}^3$ $\rho_{\text{CaSO}_4} = 2600 \text{ kg/m}^3$

Find a) Concentration $[\text{SO}_4^{2-}]$ required for desired Ca^{2+} removal.
 b) Required dosage (kg/hr) sulfuric acid
 c) Resulting slurry concentration (vol%) of CaSO_4 particles

Solution a) 98% removal means $0.5 \text{ wt}\% \text{ Ca}^{2+} \rightarrow 0.01 \text{ wt}\%$.

In concentrations this means:

$$\frac{0.005 \frac{\text{kg}}{\text{kg}}}{40 \times 10^{-3} \frac{\text{kg}}{\text{mol}}} = 0.125 \frac{\text{mol}}{\text{kg}} \quad \text{moet naar} \quad \frac{0.0001 \frac{\text{kg}}{\text{kg}}}{40 \times 10^{-3} \frac{\text{kg}}{\text{mol}}} = 0.025 \frac{\text{mol}}{\text{kg}}$$

Required SO_4^{2-} concentration follows from the solubility product:

$$[\text{SO}_4^{2-}] = \frac{K_s^{\text{CaSO}_4}}{[\text{Ca}^{2+}]} = \frac{5 \times 10^{-5}}{0.025} = 0.02 \frac{\text{mol}}{\text{kg}}$$

- b) Amount of H_2SO_4 to be added:
 - for CaSO_4 precipitation = $0.125 - 0.025$ (mol/kg)
 - to reach final concentration $\text{SO}_4^{2-} = 0.02$ (mol/kg)

Total: $0.125 - 0.025 + 0.02 = 0.1425$ (mol/kg)

Recalculation to kg/hr:

$$0.1425 \frac{\text{mol}}{\text{kg}} \cdot 98 \times 10^{-3} \frac{\text{kg}}{\text{mol}} \cdot 15 \frac{\text{m}^3}{\text{hr}} \cdot 1400 \frac{\text{kg}}{\text{m}^3} = 293 \frac{\text{kg H}_2\text{SO}_4}{\text{hr}}$$

- c) Resulting slurry concentration

Precipitate = $0.125 - 0.0025 = 0.1225$ mol/kg molasses

$$= 0.1225 \cdot 136 \times 10^{-3} \text{ (kg/mol)} = 0.0167 \text{ kg/kg molasses}$$

$$= 0.0167 \cdot 1400 \text{ (kg/m}^3\text{)} = 23.38 \text{ kg/m}^3 \text{ molasses}$$

$$= 23.38 / 2600 \text{ (kg/m}^3\text{)} = 0.0090 \text{ m}^3 \text{ CaSO}_4/\text{m}^3 \text{ molasses}$$

$$= 0.9 \text{ vol}\%$$

Exercise 8.13

Given $F = 250.000 \text{ ton/yr}$ 8000 hrs/yr $C_{\text{NaCl}} = 300 \text{ kg/m}^3$
 $L_D = 0.5 \text{ mm}$ $k_v = 0.56$ Anhydrate $MW_{\text{NaCl}} = 58 \text{ g/mol}$
 $\rho_{\text{NaCl}} = 2170 \text{ kg/m}^3$ $\rho_{\text{Brine}} = 1200 \text{ kg/m}^3$

Find

- Yield of the crystallization plant
- Crystal production rate C (kg/hr)
- Brine feed stream (kg/hr)
- Required residence time τ in the crystallizer.
- Needed slurry filled volume of the first stage
- Volume of the crystals in the first stage crystallizer

Solution a) Equation 8.54:

$$\text{Yield} = 1 - \frac{X_C}{X_F} (1 - \Phi_E) = 1 - 1 \cdot (1 - \Phi_E) = \Phi_E$$

20% evaporation/stage, total 60%.

$$\Phi_E = \text{Yield} = 60\% = 0.60$$

b) $C = 250.000 \text{ ton}/8000 \text{ hr} = 31250 \text{ kg/hr}$

c) Brine feed $= F'(1 + X_F)$

$$C = F'(X_F - X_C(1 - \Phi_E))$$

1 m³ feed contains 300 kg NaCl and has a density of 1200 kg/m³

1 m³ feed thus consists of 300 kg NaCl and 900 kg water

$$X_F = X_C = \frac{300 \text{ kg zout}}{900 \text{ kg water}} = 0.333 \frac{\text{kg zout}}{\text{kg water}}$$

$$F' = \frac{C}{(X_F - X_C(1 - \Phi_E))} = \frac{31250}{(0.333 - 0.333(1 - 0.60))} = 156406 \frac{\text{kg water}}{\text{uur}}$$

$$\text{Brine feed} = F'(1 + X_F) = 208490 \text{ kg brine/hr}$$

d) $B_o = 1.6 \times 10^{18} M_T G^2$

Introducing Eq. 8.47 eliminates M_T :

$$B_o = 1.6 \times 10^{18} (6\rho_c k_v) n_0 (G\tau)^4 G^2$$

Introducing Eq. 8.44 eliminates n_0

$$B_o = 1.6 \times 10^{18} (6\rho_c k_v) \left(\frac{B_o}{G} \right) (G\tau)^4 G^2$$

B_o cancels out

$$0 = 1.6 \times 10^{18} (6\rho_c k_v) G^5 \tau^4$$

Rewriting Eq. 8.46 for L_D gives

$$G = \frac{L_D}{3\tau}$$

and eliminates G

$$\tau = 1.6 \times 10^{18} (6\rho_c k_v) \left(\frac{L_D}{3} \right)^5$$

$$\tau = 1.6 \times 10^{18} (6 \times 2170 \times 0.56) \left(\frac{1.0 \times 10^{-3}}{3} \right)^5 = 1500(s)$$

- e) First stage evaporates 20% water from the feed, $\Phi_E = 0.60/3$

The residence time of the slurry is determined by the volumetric flow of the slurry that leaves the first stage. This slurry consist of saturated brine (density 1200 kg/m³) and crystals (density 2170 kg/m³)

Saturated brine flow follows from $F'(1-\Phi_E)(1+X_C)$
 $= 156406(1-0.6/3)(1+0.333) = 145942$ kg brine/hr
 using a density of 1200 kg/m³ yields = 121.6 m³/hr

The crystal mass produced follows from:

$C = F'(X_F - X_C(1-\Phi_E))$
 $= 156406(0.333 - 0.333(1-0.6/3)) = 10417$ kg salt/hr
 using a density of 2170 kg/m³ yields = 4.80 m³/uur

Total volume flow leaving the crystallizer $Q = 126.40$ m³/uur

Residence time = 1500 (s) =

$$V = Q \cdot \tau = 126.40 \cdot 1500/3600 = 52.7 \text{ m}^3$$

- f) First stage evaporates only 20%.
 Yield then 1/3 of total crystal production = 31250/3 = 10417 kg/uur

$$V_{\text{kristallen}} = \frac{\text{Productie} \times \tau}{\text{kristaldichtheid}} = \frac{\frac{10417}{3600} \times 1500}{2170} = 2.00 \text{ m}^3$$

Chapter 9: SEDIMENTATION & SETTLING

Exercise 9.1

Calculate the terminal settling velocity v_∞ of a $70\text{ }\mu\text{m}$ diameter sphere (density 2600 kg/m^3) in water (density 1000 kg/m^3 , viscosity $1 \times 10^{-3}\text{ Ns/m}^2$).

Exercise 9.2

Design a gravity settling tank separator to handle $15\text{ m}^3/\text{h}$ of an aqueous suspension containing $0.118\text{ vol\%}$ of copper oxide. Laboratory tests have shown that the unhindered solids settling rate $v_\infty = 3.54 \times 10^{-4}\text{ m/s}$. The density of copper oxide is 5600 kg/m^3 .

Exercise 9.3

A settling tank is used to separate ion exchange particles ($\rho_s = 1200\text{ kg/m}^3$, $d_p = 1\text{ mm}$) from an effluent stream ($\rho = 1000\text{ kg/m}^3$, $\eta = 0.001\text{ Pa.s}$, $Q = 0.3\text{ m}^3/\text{s}$).

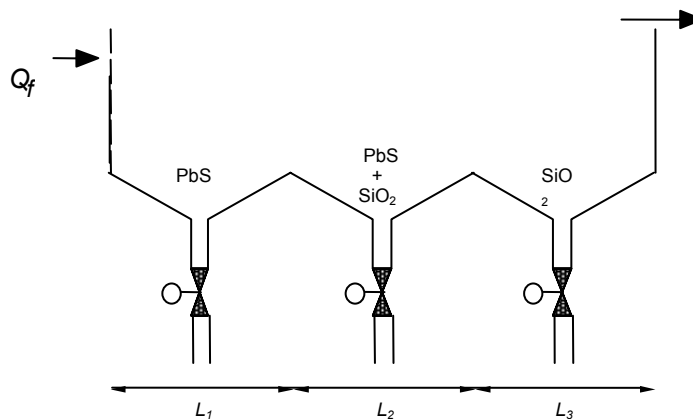
Calculate the required area of this settling tank operating under laminar flow conditions.

Exercise 9.4

Calculate the diameter D of a settling tank to separate light oil from water. The oil is the dispersed phase. Oil mass flow rate $Q_{oil} = 1000\text{ kg/h}$, $\rho_{oil} = 900\text{ kg/m}^3$, $\eta_{oil} = 0.003\text{ Pa.s}$. Water mass flow rate $Q_{water} = 5000\text{ kg/h}$, $\rho_{water} = 1000\text{ kg/m}^3$, $\eta_{water} = 0.001\text{ Pa.s}$. The droplet diameter $d_p = 150\text{ }\mu\text{m}$.

Exercise 9.5

This exercise concerns the separation of minerals by density difference. A water stream Q_f ($0.05\text{ m}^3\text{ s}^{-1}$) contains a mixture of spherical lead sulfide particles and spherical quartz (sand) particles. The quartz and lead sulfide particles should be separated to the best extend. However, this separation is complicated by the fact that smaller heavy particles can have the same settling velocity as larger but lighter particles. As shown in the drawing below the whole settler consists of three settling chambers, each with its own length L . It can be assumed that a uniform horizontal flow exists in which the particles can settle unhindered. The values of L_1 , L_2 and L_3 have been chosen in such a way that no quartz is collected in the first compartment and no lead sulfite in the last compartment. The width of all three settling compartments is the same (say 5.0 m).



$\rho_{PbS} = 7500\text{ kg m}^{-3}$ and $\rho_{quarz} = 2650\text{ kg m}^{-3}$. Both minerals are present in particle sizes ranging from $10.0\text{ }\mu\text{m}$ to $25.0\text{ }\mu\text{m}$. The density ρ_f of water at 20°C equals 998 kg m^{-3} and its viscosity $\eta_f = 1.005 \times 10^{-3}\text{ kg m}^{-1}\text{ s}^{-1}$. The gravitational acceleration $g = 9.81\text{ m s}^{-2}$.

- Calculate the smallest particle diameter of lead sulfide in the first chamber.
- Calculate value of $L_1 + L_2$ to ensure that no lead sulfide particles settle in the last chamber.

Exercise 9.6

Compare the gravitational and centrifugal sedimentation rate at 5000 rev/min of yeast cells ($d_p = 8 \mu\text{m}$, $\rho_s = 1050 \text{ kg/m}^3$) in water ($\rho_L = 1000 \text{ kg/m}^3$, $\eta_L = 10^{-3} \text{ Pa.s}$).

The centrifuge bowl has an inner radius of $r_c = 0.2 \text{ m}$.

Exercise 9.7

If a centrifuge is 0.9 m in diameter and rotates at 20 Hz, at what speed should a laboratory centrifuge of 150 mm diameter be run if it is to duplicate plant conditions?

Exercise 9.8

A continuous tube centrifuge with a bowl 1.5 m long and 0.75 m diameter operating with a pool depth of 0.1 m at 1800 rpm is clarifying an aqueous suspension at a rate of $5.4 \text{ m}^3/\text{min}$. All particles of diameter greater than $10 \mu\text{m}$ are being removed. Calculate the efficiency of this machine. The solid and liquid densities are 2800 and 1000 kg/m^3 , respectively and the liquid viscosity $\eta = 0.001 \text{ Pa.s}$.

Exercise 9.9

A centrifuge basket 600 mm long and 100 mm internal diameter has a discharge weir 25 mm diameter.

What is the maximum volumetric flow of liquid through the centrifuge such that when the basket is rotated at 200 Hz all particles of diameter greater than $1 \mu\text{m}$ are retained on the centrifuge wall?

Solid density $\rho_s = 2600 \text{ kg/m}^3$, liquid density $\rho_L = 1000 \text{ kg/m}^3$, liquid viscosity $\eta_L = 0.01 \text{ poise}$.

Exercise 9.10

A low-concentration suspension of clay in water is to be separated by centrifugal sedimentation. Pilot runs on a laboratory tubular bowl centrifuge ($L = 0.2 \text{ m}$, $r_2 = 0.0220 \text{ m}$, $r_1 = 0.011 \text{ m}$) operating at 20000 rev/min indicate that satisfactory overflow clarity (50% collection efficiency) is obtained at a throughput of $8 \cdot 10^{-6} \text{ m}^3/\text{s}$.

- a. If the separation is to be carried out in the plant using a tubular centrifuge with $L = 0.734 \text{ m}$, internal radius $R = 0.0521 \text{ m}$ and $R - r_1 = 0.0295 \text{ m}$, what production flow rate could be expected when operating at 15000 rev/min?
- b. What is the effective cut size?

Densities and liquid viscosity as in the previous exercise.

Exercise 9.11

Estimate the diameter of hydrocyclones ($H = 4D$) required for the separation of polypropylene particles ($d_p = 10^{-4} \text{ m}$, $\rho_p = 900 \text{ kg/m}^3$) from butane ($\eta_G = 10^{-4} \text{ Pa.s}$, $\rho_G = 600 \text{ kg/m}^3$). The inlet velocity is limited to 3 m/s to limit the pressure drop.

How many cyclones should be arranged in parallel to handle a stream of 600 kg slurry/s ?

SOLUTIONS

Exercise 9.1

Given $d_p = 70 \mu\text{m}$ (sphere) $\rho_S = 2600 \text{ kg/m}^3$ $\rho_L = 1000 \text{ kg/m}^3$ $\eta_L = 1 \cdot 10^{-3} \text{ Ns/m}^2$

Find v_∞ , the terminal settling velocity of this particle

Thoughts To calculate the terminal settling velocity v_∞ of a spherical particle, Eq. 9.9 with the appropriate expression for C_D should be used (Eq. 9.10, 9.12 or 9.13). The easiest solution is to assume that one equation is valid and check the Re number afterwards.

Solution For $Re < 1$, the combination of Eqs. 9.9 and 9.10 gives Eq. 9.11:

$$v_\infty = \frac{d_p^2}{18\eta_L} (\rho_p - \rho_L) g = \frac{(70 \cdot 10^{-6})^2}{18 \cdot 10^{-3}} (2600 - 1000) \cdot 9.8 = 4.27 \cdot 10^{-3} \text{ m/s}$$

Check Reynolds number: $Re = \frac{\rho_L \cdot v_\infty \cdot d_p}{\eta_L} = 0.3 < 1 \rightarrow$ Eq. 9.11 is valid so $v_\infty = 4.27 \cdot 10^{-3} \text{ m/s}$

A more general approach is to eliminate v_∞ from Eqs. 9.9 and 9.15, resulting in Eq. 9.14,

$$C_D Re^2 = \frac{4}{3} \frac{d_p^3 \rho_L (\rho_S - \rho_L) g}{\eta^2} \quad (\text{valid for any value of } Re)$$

and to solve for C_D and Re with either Eq. 9.10 or Eq. 9.12 or Eq. 9.13:

- Assume $Re < 1$ and solve Eqs. 9.14 and 9.10: $\begin{pmatrix} C_D \\ Re \end{pmatrix} = \begin{pmatrix} 80 \\ 0.3 \end{pmatrix}$ hence $Re < 1$ is OK and v_∞ follows from Eq. 9.15.
- Assume $1 < Re < 5$ and solve Eqs. 9.14 and 9.12: $\begin{pmatrix} C_D \\ Re \end{pmatrix} = \begin{pmatrix} 83.2 \\ 0.5 \end{pmatrix}$, Re is outside valid range, otherwise v_∞ would follow from Eq. 9.15.
- Assume $5 < Re < 500$ and solve Eqs. 9.14 and 9.13: $\begin{pmatrix} C_D \\ Re \end{pmatrix} = \begin{pmatrix} 27.8 \\ 0.5 \end{pmatrix}$, Re is outside valid range, otherwise v_∞ would follow from Eq. 9.15.

Exercise 9.2

Given aqueous suspension, gravity settling

$$v_\infty = 3.54 \cdot 10^{-4} \text{ m/s}$$

$$Q = 15 \text{ m}^3/\text{h}$$

$$\rho_S = 5600 \text{ kg/m}^3$$

$$c_{Cu} = 0.118 \text{ vol\%}$$

Find The required tank area A

Thoughts The minimal required area can be calculated from Eq. 9.22. As the gravitational solids settling rate is given, the values for concentration and density are not necessary to solve the problem.

Solution Volumetric flow rate $Q = 15 \text{ m}^3/\text{h} = 4.167 \cdot 10^{-3} \text{ m}^3/\text{s}$

$$\text{Use Eq. 9.22 to calculate the required tank area: } A \geq \frac{Q}{v_\infty} = \frac{15/3600}{3.54 \cdot 10^{-4}} = 11.8 \text{ m}^2$$

Note:

Probably, the sedimentation is unhindered at this low concentration. Thus, the particle diameter d_p can be calculated from the known settling rate, v_∞ .

$$\text{If } Re < 1 \text{ then Eq. 9.11 applies and } v_\infty = \frac{d_p^2}{18\eta_L} (\rho_p - \rho_L) g.$$

Solving for d_p gives $d_p = 11.9 \mu\text{m}$ and $Re = 4.2 \cdot 10^{-3} < 1$ (Re OK).

Exercise 9.3

Given $d_p = 1 \text{ mm}$ $\rho_S = 1200 \text{ kg/m}^3$ $\rho_L = 1000 \text{ kg/m}^3$
 $\eta = 1 \cdot 10^{-3} \text{ Pa}\cdot\text{s}$ $Q_V = 18 \text{ m}^3/\text{min}$

Find A , the required tank area for this separation

Thoughts The terminal settling velocity v_∞ has to be calculated from Eq. 9.9 with the appropriate expression for C_D (see Exercise 9.1). The area A follows from Eq. 9.22.:

Solution First assume that $Re < 1$ and use Eq. 9.11:

$$v_\infty = \frac{d_p^2}{18\eta} (\rho_S - \rho_L) g = \frac{(1 \cdot 10^{-3})^2}{18 \cdot 10^{-3}} (1200 - 1000) \cdot 9.8 = 0.109 \text{ m/s}$$

Check Re :

$$Re = \frac{\rho_L \cdot v_\infty \cdot d_p}{\eta_L} = 109 \rightarrow \text{Eq. 9.11 is not valid.}$$

Now assume that $5 < Re < 500$ and use Eq. 9.14 with $C_D = 18.5 \cdot Re^{-0.6}$ (Eq. 9.13).

$$\text{Solve for } C_D \text{ and } Re: \left(\frac{C_D}{Re} \right) = \left(\frac{2.2}{34.4} \right), \text{ } Re \text{ OK and } v_\infty = \frac{Re \cdot \eta}{\rho_L \cdot d_p} = 0.0344 \text{ m s}^{-1}.$$

$$\text{The required tank area with Eq. 9.22: } A_{min} = \frac{Q_V}{v_\infty} = \frac{18/60}{0.0344} = 8.7 \text{ m}^2$$

Exercise 9.4

Given $\rho_{oil} = 900 \text{ kg/m}^3$ $\eta_{oil} = 0.003 \text{ Pa}\cdot\text{s}$ $Q_{oil} = 1000 \text{ kg/h}$
 $\rho_{water} = 1000 \text{ kg/m}^3$ $\eta_{water} = 0.001 \text{ Pa}\cdot\text{s}$ $Q_{water} = 5000 \text{ kg/h}$
 $d_p = 150 \text{ }\mu\text{m}$ (oil in water dispersion)

Find D , diameter of a settling tank to separate oil from water

Thoughts To calculate the required tank area, the volumetric flow of the continuous phase and the settling rate of the dispersed phase (the oil droplet) have to be calculated. For the settling rate, Eq. 9.11 may be applied (check Re). The hindered settling rate is smaller by a factor $\varepsilon^2 \cdot f(\varepsilon)$, as given in Eq. 9.16. The volume fraction ε of the fluid is calculated from Eq. 9.17. Finally, Eq. 9.22 is used to evaluate the minimum tank area.

Solution The unhindered terminal droplet velocity (Eq. 9.11):

$$v_\infty = \frac{d_p^2}{18\eta} (\rho_{oil} - \rho_{water}) g = \frac{(1.5 \cdot 10^{-4})^2}{18 \cdot 10^{-3}} (900 - 1000) \cdot 9.8 = -1.225 \cdot 10^{-3} \text{ m/s}$$

Note that the velocity of the oil droplets is directed upwards (oil is lighter than water).

$$\text{Check the } Re \text{ number: } Re = \frac{\rho_{oil} \cdot |v_\infty| \cdot d_p}{\eta_{water}} = 0.18 \rightarrow \text{Eq. 9.11 is valid.}$$

$$\text{The volume fraction of the fluid } \varepsilon = \frac{Q_{water} / \rho_{water}}{Q_{water} / \rho_{water} + Q_{oil} / \rho_{oil}} = 0.818,$$

the settling can be considered to be hindered.

$$\text{Combining Eqs. 9.16 and 9.18 with } n = 2.65 \text{ gives } v_s / v_\infty = \varepsilon^2 \cdot \varepsilon^{2.65} = 0.393^1$$

and the hindered settling rate $v_s = 0.482 \cdot 10^{-3} \text{ m/s}$.

From this settling rate, the tank area can be calculated:

$$A \geq \frac{Q_{water} / \rho_{water}}{v_s} = 2.9 \text{ m}^2. \text{ For a cylindrical tank, } D_{min} = 1.9 \text{ m.}$$

¹ with $n = 2.5$ it follows that $v_s = -5 \cdot 10^{-4} \text{ m/s}$; with Eqs. 9.16 and 9.19 v_s would be $-3.7 \cdot 10^{-4} \text{ m/s}$

Exercise 9.5

<i>Given</i>	$\rho_{PbS} = 7500 \text{ kg/m}^3$	$\rho_{quarz} = 2650 \text{ kg/m}^3$	$\rho_{water} = 998 \text{ kg/m}^3$
	$g = 9.81 \text{ m/s}^2$	$\eta_{water} = 0.001 \text{ Pa}\cdot\text{s}$	$w = 5.0 \text{ m}$
	$Q_V = 0.05 \text{ m}^3/\text{s}$	$d_{min} = 10 \text{ }\mu\text{m}$	$d_{max} = 25 \text{ }\mu\text{m}$

- Find*
- Smallest particle size of heavier species in the first chamber
 - Minimum value of $L_1 + L_2$ to prevent settling of the heavier species in the last chamber

- Thoughts*
- The velocity of the smallest leadsulfide particles (with diameter d_x) in the first chamber equals the velocity of the largest quartz particles in the second chamber: $v_{PbS}(d_x) = v_{quarz}(d_{max})$. An expression to relate unhindered, stationary settling velocity with density and diameter is available (Eq. 9.11). Don't forget to check the boundary condition, $Re < 1$.
 - The settling time of the smallest PbS particles equals the residence time in the direction of the flow along a distance $L_1 + L_2$.

- Solution*
- Eq. 9.11 is applicable only for $Re < 1$. Check this for the *largest* leadsulfide particles:

$$Re_{max} = \frac{\rho_{water} \cdot v_{PbSmax} \cdot d_{max}}{\eta_{water}} \text{ with}$$

$$v_{PbSmax} = \frac{g \cdot (\rho_{PbS} - \rho_{water}) \cdot d_{max}^2}{18 \cdot \eta_{water}} = 2.204 \cdot 10^{-3} \text{ m/s}$$

Hence, $Re = 0.055$, this is the maximum value, thus Eq. 9.11 is applicable to *all* particles.

Combining $v_{PbS}(d_x) = v_{quarz}(d_{max})$ and Eq. 9.11 leads to

$$(\rho_{PbS} - \rho_{water}) \cdot d_x^2 = (\rho_{quarz} - \rho_{water}) \cdot d_{max}^2$$

Solving for d_x gives $d_x = 12.6 \text{ }\mu\text{m}$.

- The settling time of the smallest PbS particles $\tau_{\downarrow} = \frac{H}{v_{PbS}(d_{min})}$ (H is the settling height)

$$\text{The residence time } \tau_{\rightarrow} = \frac{L_1 + L_2}{u} = \frac{(L_1 + L_2) \cdot w \cdot H}{Q_{water}} \text{ (} w \text{ is the width of the settling chambers)}$$

In the limiting situation that the smallest PbS particles just reach the end of the trajectory $L_1 + L_2$, see Eq. 9.22 with $A = (L_1 + L_2) \cdot w$, the settling time balances the residence time:

$$\tau_{\downarrow} = \tau_{\rightarrow}$$

With $v_{PbS}(d_{min}) = 3.54 \cdot 10^{-4} \text{ m/s}$ follows $L_1 + L_2 = 28.2 \text{ m}$.

Exercise 9.6

<i>Given</i>	$d_p = 8 \text{ }\mu\text{m}$	$\rho_p = 1050 \text{ kg/m}^3$	$\rho_L = 1000 \text{ kg/m}^3$
	$\eta = 1 \cdot 10^{-3} \text{ Pa}\cdot\text{s}$	$r_{c(entrifuge)} = 0.2 \text{ m}$	$\omega = 5000 \text{ rev/min}$

- Find* The gravitational and the centrifugional stationary sedimentation velocity v_{∞} .

- Thoughts* Eq. 9.31 applies to centrifugal sedimentation and can be used at $Re < 1$; Eq. 9.11 can be used to calculate the gravitational sedimentation rate at $Re < 1$.

- Solution* Gravitational sedimentation rate:

$$v_{g\infty} = \frac{d_p^2}{18\eta} (\rho_S - \rho_L) g = \frac{(8 \cdot 10^{-6})^2}{18 \cdot 10^{-3}} (1050 - 1000) \cdot 9.8 = 1.74 \cdot 10^{-6} \text{ m/s}$$

$$\text{Check: } Re = \frac{\rho_L \cdot v_{g\infty} \cdot d_p}{\eta} = 1.5 \cdot 10^{-5} < 1, \text{ Eq. 9.11 is valid.}$$

Sedimentation rate in the centrifuge:

$$\omega = 5000 \text{ rev/min} = 83.3 \text{ rev/s} = 523.4 \text{ rad/s}$$

$$v_{c\infty} = \frac{d_p^2}{18\eta} (\rho_S - \rho_L) \cdot r_c \omega^2 = \frac{(8 \cdot 10^{-6})^2}{18 \cdot 10^{-3}} (1050 - 1000) \cdot 0.2 \cdot (523.4)^2 = 9.75 \cdot 10^{-3} \text{ m/s}$$

$$\text{Check } Re = \frac{\rho_L \cdot v_{c\infty} \cdot d_p}{\eta} = 0.078 < 1, \text{ Eq. 9.31 is valid.}$$

Note that the sedimentation rate in the centrifuge is $\frac{v_{c\infty}}{v_{g\infty}} = \frac{r_c \omega^2}{g} \approx 5600$ times faster compared to gravitational sedimentation.

Exercise 9.7

Given Large centrifuge:
 $D = 0.9 \text{ m}; \omega = 20 \text{ Hz}$

Lab-scale centrifuge:
 $D = 150 \text{ mm}$

Find The rotational speed required in the lab centrifuge to duplicate plant conditions

Thoughts To duplicate plant conditions, the centrifuges should produce the same acceleration: Eq. 9.27.

$$\text{Solution } r_1 \cdot \omega_1^2 = r_2 \cdot \omega_2^2 \text{ hence } \omega_2 = \omega_1 \cdot \sqrt{\frac{r_1}{r_2}} = 20 \cdot \sqrt{\frac{0.45}{0.075}} = 49 \text{ Hz}$$

Exercise 9.8

Given Tube centrifuge:

$L = 1.5 \text{ m}$	$d_{\text{cut-off}} = 10 \text{ }\mu\text{m}$	pool depth = 0.1 m
$D = 0.75 \text{ m}$	$\omega = 1800 \text{ rpm}$	$Q_V = 5.4 \text{ m}^3/\text{min}$
$\eta_L = 0.001 \text{ Pa}\cdot\text{s}$	$\rho_L = 1000 \text{ kg/m}^3$	$\rho_S = 2800 \text{ kg/m}^3$

Find The efficiency of this machine compared to gravitational settling

Thoughts To calculate the efficiency, the Sigma concept is used. In Eq. 9.35, the left-hand side represents the process parameters, or the desired area (the required 'performance' of the machine); this is known as the 'process' Sigma. The right-hand side represents the actual performance of the machine, or the machine Sigma. The ratio of the actual 'area' and the area required by the process can be used as a measure of efficiency.

Solution First, use Eq. 9.11 to calculate the terminal settling velocity (under gravity), v_{∞} :

$$v_{\infty} = \frac{d_p^2}{18\eta} (\rho_S - \rho_L) g = \frac{(10 \cdot 10^{-6})^2}{18 \cdot 10^{-3}} (2800 - 1000) \cdot 9.8 = 9.8 \cdot 10^{-5} \text{ m/s}$$

$$Q_V = 5.4 \text{ m}^3/\text{min} = 0.09 \text{ m}^3/\text{s} \text{ and } \Sigma_g = \frac{Q_V}{v_{\infty}} = \frac{0.09}{9.8 \cdot 10^{-5}} = \mathbf{918 \text{ m}^2}$$

For the machine sigma, use the right-hand side of Eq. 9.35:

$$r_2 = \text{centrifuge radius} = D/2 = 0.375 \text{ m}$$

$$r_1 = r_2 - \text{pool depth} = 0.275 \text{ m}$$

$$\omega = 1800 \text{ rpm} = 30 \text{ r/s} = 188.5 \text{ rad/s}$$

$$\text{Centrifuge volume } V_C: V_C = L\pi(r_2^2 - r_1^2) = 1.5 \cdot \pi \cdot (0.375^2 - 0.275^2) = 0.306 \text{ m}^3$$

$$\Sigma_{\text{machine}} = \frac{V_C \cdot \omega^2}{g \cdot \ln\left(\frac{r_2}{r_1}\right)} = \frac{0.306 \cdot 188.5^2}{9.8 \cdot \ln(0.375/0.275)} = \mathbf{3580 \text{ m}^2}$$

→ the centrifuge is $3580 / 918 = 3.90$ times more efficient than gravitational settling.

Exercise 9.9

Given	$L = 0.6 \text{ m}$	$D = 0.1 \text{ m}$	weir = 25 mm
	$\omega = 200 \text{ Hz}$	$d_{\max} = 1 \text{ }\mu\text{m}$	$\rho_S = 2600 \text{ kg/m}^3$
	$\eta_L = 0.01 \text{ poise}$	$\rho_L = 1000 \text{ kg/m}^3$	

Find The maximum volumetric flow Q to reach this separation

Thoughts The volume of the centrifuge can be calculated from the given dimensions. The maximum flow Q is obtained if the centrifuge works at 100% efficiency. Eq. 9.34 or 9.35 can then be used directly to calculate the flow rate. Note that the viscosity of the liquid is given in poise.

Solution $\eta_L = 0.01 \text{ poise} = 0.01 \text{ g/s}\cdot\text{cm} = 0.001 \text{ kg/s}\cdot\text{m} = 0.001 \text{ N}\cdot\text{s/m}^2 \equiv 0.001 \text{ Pa}\cdot\text{s}$.

$$r_2 = 100/2 \text{ mm} = 0.05 \text{ m}$$

$$r_1 = 25/2 \text{ mm} = 0.0125 \text{ m}$$

$$V_C = L\pi(r_2^2 - r_1^2) = 0.6 \cdot \pi \cdot (0.05^2 - 0.0125^2) = 4.42 \cdot 10^{-3} \text{ m}^3$$

$$\omega = 200 \text{ Hz} = 200 \cdot 2\pi \text{ rad/s} = 1256.6 \text{ rad/s}$$

$Q_{\max}$ follows from Eqs. 9.34 or 9.35 with a cut-off diameter of $1 \text{ }\mu\text{m}$:

$$Q_{\max} = V_C \left[\frac{d_{\max}^2}{18\eta_L} (\rho_S - \rho_L) \cdot g \right] \frac{\omega^2}{g \cdot \ln \frac{r_2}{r_1}} =$$

$$= 4.42 \cdot 10^{-3} \cdot \frac{(10^{-6})^2}{18 \cdot 10^{-3}} (2600 - 1000) \cdot 9.8 \cdot \frac{1256.6^2}{9.8 \cdot \ln \frac{0.05}{0.0125}} = 4.47 \cdot 10^{-4} \text{ m}^3/\text{s}$$

Exercise 9.10

Given	$L_{\text{lab}} = 0.2 \text{ m}$	$r_{2,\text{lab}} = 0.022 \text{ m}$	$r_{1,\text{lab}} = 0.011 \text{ m}$	$\omega_{\text{lab}} = 20000 \text{ rev/min}$
	$\eta_L = 0.01 \text{ poise}$	$\rho_L = 1000 \text{ kg/m}^3$	$\rho_S = 2600 \text{ kg/m}^3$	$Q_{V,\text{lab}} = 8 \cdot 10^{-6} \text{ m}^3\text{s}^{-1}$
	$L_{\text{plant}} = 0.734 \text{ m}$	$R = 0.0521 \text{ m}$	$R - r_1 = 0.0295 \text{ m}$	$\omega_{\text{plant}} = 15000 \text{ rev/min}$
	50% collection efficiency			

Find a. The maximum volumetric flow $Q_{V,\text{plant}}$ to reach this separation

b. Calculate the effective cut size

Thoughts a. The maximum flow is obtained if the centrifuge works at 50% efficiency. Eq. 9.39 can then be used directly to calculate the flow rate.

b. The cut size depends on the Stokes terminal velocity v_∞ , hence combine Eqs. 9.11 and 9.39.

Note that the viscosity of the liquid is given in poise, see previous exercise.

Solution a. $\Sigma_{\text{lab}}^{50\%} = \frac{V_{C,\text{lab}} \cdot \omega_{\text{lab}}^2}{g \cdot \ln \left(\frac{2r_{2,\text{lab}}^2}{r_{1,\text{lab}}^2 + r_{2,\text{lab}}^2} \right)}$ with $V_{C,\text{lab}} = L_{\text{lab}} \cdot \pi(r_{2,\text{lab}}^2 - r_{1,\text{lab}}^2) = 2.28 \cdot 10^{-4} \text{ m}^3$ and

$$\omega_{\text{lab}} = \frac{20000}{60} \cdot 2\pi = 2094 \frac{\text{rad}}{\text{s}} \Rightarrow \Sigma_{\text{lab}}^{50\%} = \frac{2.28 \cdot 10^{-4} \cdot 2090^2}{9.8 \cdot \ln \left(\frac{2 \cdot 0.022^2}{0.011^2 + 0.022^2} \right)} = 217 \text{ m}^2$$

$$\Sigma_{plant}^{50\%} = \frac{V_{C,plant} \cdot \omega_{plant}^2}{g \cdot \ln \left(\frac{2R^2}{r_1^2 + R^2} \right)} \quad \text{with } V_{C,plant} = 5.08 \cdot 10^{-3} \text{ m}^3 \text{ and } \omega_{plant} = 1571 \frac{\text{rad}}{\text{s}}$$

$$\Rightarrow \Sigma_{plant}^{50\%} = \frac{5.08 \cdot 10^{-3} \cdot 1571^2}{9.8 \cdot \ln \left(\frac{2 \cdot 0.0521^2}{0.0226^2 + 0.0521^2} \right)} = 2455 \text{ m}^2$$

$$Q_{V,plant} = Q_{V,lab} \cdot \frac{\Sigma_{plant}}{\Sigma_{lab}} = 9.05 \cdot 10^{-5} \text{ m}^3 \text{s}^{-1}, \text{ about 10-times that of the lab centrifuge.}$$

- b. The cut size depends on the Stokes' terminal velocity v_∞ and the terminal velocity determines the throughput Q_V :

$$\text{Eq. 9.11} \quad v_\infty = \frac{d_{cut}^2}{18 \cdot \eta_w} \cdot (\rho_s - \rho_L) \cdot g$$

$$\text{Eq. 9.39} \quad v_\infty = \frac{Q_{V,plant}}{2 \Sigma_{plant}}$$

$$\text{Eliminate } v_\infty \text{ and solve for } d_{cut} \Rightarrow d_{cut} = 0.15 \text{ } \mu\text{m} \text{ (check that } \text{Re} < 1)$$

Exercise 9.11

Given Hydrocyclone:

H	$= 4D$	v_{in}	$< 3 \text{ m/s}$
d_p	$= 10^{-4} \text{ m}$	ρ_s	$= 900 \text{ kg/m}^3$
η_L	$= 10^{-4} \text{ Pa.s}$	ρ_L	$= 600 \text{ kg/m}^3$

- Find**
- the diameter of the cyclone
 - the number of cyclones to process 600 kg/s slurry

- Thoughts**
- The performance of a hydrocyclone can be estimated with Eq. 9.53. The inlet area can be calculated from the cyclone diameter using the 'rule-of-thumb' (see context of Eq. 9.49).
 - Calculate the flow in one cyclone (Eq. 9.49). By converting the total mass flow to a total volumetric flow, the number of required cyclones follows from the ratio of total flow and capacity of one cyclone.

- Solution**
- For $d_0 = 0.1 \text{ mm}$ and $H = 4D$, the cyclone diameter can be calculated:

$$d_0 = \sqrt{\frac{3\eta D^2}{2\pi v_f(\rho_s - \rho)H}}$$

$$D = \frac{d_0^2 8\pi v_f(\rho_s - \rho)}{3\eta} = \frac{(1 \cdot 10^{-4})^2 \cdot 8 \cdot \pi \cdot 3 \cdot (900 - 600)}{3 \cdot 10^{-4}} = 0.754 \text{ m}$$

- The maximum volumetric flow rate that can be treated in one cyclone is:

$$A_{in} = \frac{D^2}{6} = 0.095 \text{ m}^2$$

$$Q = v_f \cdot A_{in} = 3 \cdot 0.095 = 0.28 \text{ m}^3/\text{s}$$

The minimum density of the slurry is the butane density, 600 kg/m³; the total volumetric flow rate of 600 kg slurry/s equals $600 \frac{\text{kg}}{\text{s}} / 600 \frac{\text{kg}}{\text{m}^3} = 1.0 \text{ m}^3 \text{s}^{-1}$.

Number of cyclones: $1.0 / 0.28 = 4$ cyclones.

Chapter 9: SEDIMENTATION & SETTLING (continued)**Exercise 9.12**

As a last step produced fruit juices need to be clarified by removing the very fine particles (10 kg/m^3 fruit juice). Assume that all particles have the same size. Before the final filtration the particles are concentrated by sedimentation. In the laboratory the following sedimentation rates have been measured:

time(hr)	0	1	2	3	6	10
h(cm)	35	29	23	17	13	11

where h is the height of the interface between clear juice and the slurry layer.

- Calculate the size of the particle in the slurry
- How large should be the area of a settling tank when $0.01 \text{ m}^3/\text{s}$ fruit juice needs to be processed?

As alternative a centrifuge is used that is constructed in such a way the the clear juice stream leaves in axial direction and while a part of the juice with the particles is discharged radial. The two radii r_1 and r_2 equal 0.2 respectively 0.4 m , while $L = 1.4 \text{ m}$ (compare Fig. 9.13). This centrifugal sedimentation yields a slurry with 100 kg solids per m^3 slurry.

$$\rho_s = 1050 \text{ kg m}^{-3} \quad \rho_L = 1000 \text{ kg m}^{-3} \quad \eta = 10^{-3} \text{ Pa.s} \quad g = 9.81 \text{ m s}^{-2}$$

- Which rpm is required to separate all particles with this centrifuge?
- Calculate the volume flow ($\text{m}^3 \text{ s}^{-1}$) of the resulting slurry?

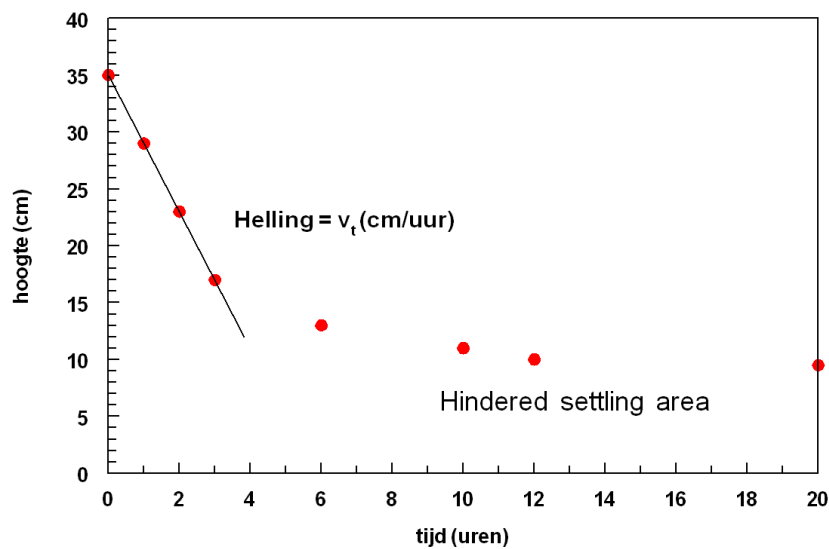
Exercise 9.12

Given $F = 0.01 \text{ m}^3 \text{ s}^{-1}$ $C_{\text{part,in}} = 10 \text{ kg/m}^3$ $C_{\text{slurry,out}} = 100 \text{ kg/m}^3$
 $r_1 = 0.2 \text{ m}$ $r_2 = 0.4 \text{ m}$ $L = 1.4 \text{ m}$
 $\rho_s = 1050 \text{ kg/m}^3$ $\rho_L = 1000 \text{ kg/m}^3$
 $\eta = 10^{-3} \text{ Pa.s}$ $g = 9.81 \text{ m s}^{-2}$

Find

- Size of the particle in the slurry
- Area of a settling tank for 0.01 m³/s fruit juice
- rpm required to separate all particles with the centrifuge
- Volume flow (m³ s⁻¹) of the resulting slurry

Solution a) Graph of height vs time yields:



Initially linear settling velocity, hereafter hindered settling/compression.

$$v_t = (35-17) \text{ cm} / 3 \text{ uur} = 1.667 \cdot 10^{-5} \text{ (m/s)}$$

$$d = \sqrt{\frac{18\eta v_t}{(\rho_s - \rho)g}} = \sqrt{\frac{18 \times 10^{-3} \times 1.667 \cdot 10^{-5}}{(1050 - 1000)9.8}} = 24.73 \text{ } \mu\text{m}$$

$$\text{Check: } Re = \frac{\rho v_\infty d_p}{\eta} = \frac{1000 \cdot 1.667 \times 10^{-5} \cdot 24.73 \times 10^{-6}}{10^{-3}} = 0.0004$$

(<<1, dus OK)

$$\text{b) } A = \frac{Q}{v_t} = \frac{0.01}{1.667 \times 10^{-5}} = 599 \text{ m}^2$$

(3)

$$\begin{aligned} \text{c)} \quad \omega &= \sqrt{\frac{Q g \ln\left(\frac{r_2}{r_1}\right)}{v_\infty V_c}} = \sqrt{\frac{0.01 \times 9.8 \ln\left(\frac{0.4}{0.2}\right)}{1.667 \times 10^{-5} \cdot 0.5278}} = 87.91 \left(\frac{\text{rad}}{\text{s}}\right) \\ V_c &= \pi h (r_u^2 - r_i^2) = \pi \cdot 1.4 (0.4^2 - 0.2^2) = 0.5278 \text{ m}^3 \\ \omega &= \frac{\omega}{2\pi} = \frac{87.91}{2\pi} = 13.99 \text{ s}^{-1} = 840 \text{ rpm} \end{aligned}$$

$$\text{d)} \quad Q_{uit} = Q_f \frac{Cs_f}{Cs_{uit}} = 0.01 * \frac{10}{100} = 0.001 \frac{\text{m}^3}{\text{s}}$$

Chapter 10: FILTRATION

Exercise 10.1

A slurry is filtered with a laboratory leaf filter with a filtering surface area of 0.05 m^2 to determine the specific cake and cloth resistance using a vacuum giving a pressure difference of 0.7 bar. The volume of filtrate collected in the first 5 min was 250 cm^3 and, after a further 5 min, an additional 150 cm^3 was collected. The filtrate viscosity is 10^{-3} Pa.s , the slurry contains 5 vol% of solids with a density of 3000 kg m^{-3} .

Calculate the specific cake resistance α and the cloth resistance R_M .

Exercise 10.2

The data given in the table below were obtained from the constant pressure period of a pilot scale plate and frame filter press.

The mass of dry cake per unit volume of filtrate amounts to 125 kg/m^3 , filter area $A = 2.72 \text{ m}^2$, viscosity $\eta = 10^{-3} \text{ Pa.s}$, pressure difference $\Delta P = 3 \text{ bar}$.

time:	92	160	232	327	418	472	538	(s)
filtrate volume V:	0.024	0.039	0.054	0.071	0.088	0.096	0.106	(m^3)

Calculate the cake resistance α .

Exercise 10.3

Calculate the specific cake resistance α and the medium resistance R_M when the same slurry data apply from the following constant rate data obtained on the same pilot scale plate and frame filter press:

V:	0.016	0.032	0.040	0.056	0.064	0.072	0.088	0.096	0.114	(m^3)
ΔP :	0.9	1.2	1.35	1.7	1.8	1.85	2.3	2.4	2.7	(bar)

Exercise 10.4

Laboratory filtrations conducted at constant pressure drop on a slurry of CaCO_3 in water gave the data shown in the following table:

Filtrate Volume	Test 1 (0.45 bar)	Test 2 (1.10 bar)	Test 3 (1.95 bar)	Test 4 (2.50 bar)	Test 5 (3.40 bar)
V (l)	t (s)	t (s)	t (s)	t (s)	t (s)
0.5	17	7	6	5	4
1.0	41	19	14	12	9
1.5	72	35	24	20	16
2.0	108	53	37	30	25
2.5	152	76	52	43	35
3.0	202	102	69	57	46
3.5		131	89	73	59
4.0		163	110	91	74
4.5			134	111	89
5.0			160	133	107
5.5				157	
6.0				183	

The filter area $A = 440 \text{ cm}^2$, the mass of solid per unit volume of filtrate was 23.5 g/L , viscosity $\eta = 10^{-3} \text{ Pa.s}$ and the temperature 25°C . Evaluate the quantities α and R_M as a function of pressure drop ΔP , and fit an empirical equation to the results for α .

Exercise 10.5

Calculate the relationship between the average specific resistance and the filtration pressure from the following data, obtained from a series of constant pressure filtration experiments:

Filtration pressure ΔP :	70	104	140	210	400	800	(kPa)
Specific resistance α :	1.4	1.8	2.1	2.7	4.0	5.6	($\times 10^{11} \text{ m/kg}$)

Exercise 10.6

A slurry, containing 0.1 kg of solid (solid density $\rho_S = 2500 \text{ kg m}^{-3}$) per kilogram of water, is fed to a rotary drum filter with length $L = 0.6 \text{ m}$ and diameter $D = 0.6 \text{ m}$. The drum rotates at a speed of one revolution in 6 min and 20 per cent of the filtering surface is in contact with the slurry at any instant. Specific cake resistance $\alpha = 2.8 \times 10^{10} \text{ m/kg}$ and medium resistance $R_M = 3.0 \times 10^9 \text{ m}^{-1}$.

Liquid density $\rho_L = 1000 \text{ kg m}^{-3}$, liquid viscosity $\eta_L = 0.001 \text{ kg m}^{-1} \text{ s}^{-1}$.

- Determine the filtrate and dry solids production rate when filtering with a pressure difference of 65 kN/m^2 .
- Calculate the thickness of the cake produced when it has a porosity $\varepsilon = 0.5$.

Exercise 10.7

A rotary drum filter with 30 per cent submergence is to be used to filter an aqueous slurry of CaCO_3 containing 230 kg of solids per cubic meter of water. The pressure drop is to be 0.45 bar .

Liquid density and viscosity as in previous exercise, solid density ρ_S as in Exercise 10.4.

The specific cake resistance $\alpha = 1.1 \times 10^{11} \text{ m/kg}$ and medium resistance $R_M = 6.0 \times 10^9 \text{ m}^{-1}$.

Calculate the filter area required to filter 40 ltr/min of slurry when the filter cycle time is 5 min .

Exercise 10.8

Calculate the dry solids production from a 10 m^2 rotating vacuum filter operating at 68 kPa vacuum and the following conditions:

cake resistance $\alpha = 1 \times 10^{10} \text{ m/kg}$, medium resistance $R_M = 1 \times 10^{10} \text{ 1/m}$, drum speed = 1 rpm , fraction submerged $f = 0.3$, solids concentration $x_S = 0.1 \text{ kg solids/kg slurry}$, cake moisture $3.5 \text{ kg wet cake/kg dry cake}$, liquid density $\rho_L = 1000 \text{ kg/m}^3$, liquid viscosity $\eta_L = 0.001 \text{ Pa.s}$

Exercise 10.9

Calculate the filtration time, required area, operational speed and cake thickness required to produce $5 \text{ m}^3/\text{h}$ of filtrate with a 1 m wide horizontal belt filter operating at a pressure difference $\Delta P = 60 \text{ kPa}$. The slurry has the following properties: specific cake resistance $\alpha = 5 \times 10^9 \text{ m/kg}$, slurry solids concentration $c_S = 350 \text{ kg/m}^3$, solids density $\rho_S = 2000 \text{ kg/m}^3$, liquid density $\rho_L = 1000 \text{ kg/m}^3$ and viscosity $\eta_L = 0.001 \text{ Pa.s}$. The cake porosity $\varepsilon = 0.43$.

Exercise 10.10

It is proposed to use an existing horizontal belt filter to separate phosphoric acid from a slurry containing gypsum at 30% w/w. Cake formation at a constant pressure difference of $\Delta P = 50$ kPa is to be followed by displacement washing and deliquoring. Of the total 9 m belt length 1.5 m is available for the filtration stage. Calculate the solids production rate (kg/s).

Filter data: width 2 m, linear velocity 0.1 m/s, medium resistance $R_M = 2 \cdot 10^9$ 1/m
Cake properties: $\alpha_{av} = 7.1 \cdot 10^8 \Delta P^{0.46}$ m/kg, $\varepsilon_{av} = 0.84 \Delta P^{-0.054}$, solid density $\rho_S = 2350$ kg/m³
Filtrate: density $\rho_L = 1390$ kg/m³, viscosity $\eta_L = 0.001$ Pa.s

Exercise 10.11

A tank filter is operated at a constant rate of 25 L/min from the start of the run until the pressure drop $\Delta P = 3.5$ bar, and then at a constant pressure drop of 3.5 bar until a total of 5 m³ of filtrate is obtained.

Given: specific cake resistance $\alpha = 1.8 \cdot 10^{11}$ m/kg, medium resistance $R_M = 1.0 \cdot 10^{10}$ m⁻¹, solids concentration $c_S = 150$ kg/m³ filtrate, viscosity $\eta_L = 0.001$ Pa.s.

Calculate the total filtration time required.

Exercise 10.12

Calculate the washing rate of a filter cake 0.025 m thick deposited on a centrifuge basket (0.635 m diameter, 0.254 m height) rotating at 20 rps. The cake porosity $\varepsilon = 0.53$, its specific resistance $\alpha = 6 \times 10^9$ m/kg. A medium with a resistance of $R_M = 1 \cdot 10^8$ m⁻¹ is used to line the perforate basket. Solid density $\rho_S = 2000$ kg/m³, liquid density $\rho_L = 1000$ kg/m³ and liquid viscosity $\eta_L = 0.001$ Pa.s. Assume the cake is incompressible.

- What time is required for the passage of two void volumes of wash if there is almost no supernatant liquid layer over the cake.
- How does the washing rate change when a 5 cm thick supernatant liquid layer is present?

SOLUTIONS

Exercise 10.1

$$cS := 0.05 \frac{\text{m}^3}{\text{m}^3}$$

$$\rho S := 3000 \frac{\text{kg}}{\text{m}^3}$$

$$A := 0.05 \text{ m}^2$$

$$\eta := 0.001 \text{ Pa} \cdot \text{sec}$$

$$\Delta P_f := 0.7 \cdot 10^5 \text{ Pa}$$

$$t := \begin{bmatrix} 5 \\ 10 \end{bmatrix} \text{ min}$$

$$V_f := \begin{bmatrix} 250 \\ 400 \end{bmatrix} \text{ mL} \quad k := 0..1$$

according to Eq. 10.11 $y_k := \frac{t_k}{V_{f,k}}$

$$y = \begin{bmatrix} 1.200 \cdot 10^6 \\ 1.500 \cdot 10^6 \end{bmatrix} \cdot \text{m}^{-3} \cdot \text{sec}$$

calculate slope slp
from the 2 data points $\text{slp} := \frac{y_1 - y_0}{V_{f,1} - V_{f,0}}$

$$\text{slp} = 2.000 \cdot 10^9 \cdot \text{m}^{-6} \cdot \text{sec}$$

specific cake resistance α from slope slp:

$$\alpha := \text{slp} \cdot \frac{2 \cdot A^2 \cdot \Delta P_f}{\eta \cdot (\rho S \cdot cS)}$$

$$\alpha = 4.67 \cdot 10^{12} \cdot \text{kg}^{-1} \cdot \text{m}$$

filter medium resistance RM from intercept intcpt $\text{intcpt} := y_1 - \text{slp} \cdot V_{f,1}$

$$\text{intcpt} = 7.000 \cdot 10^5 \cdot \text{m}^{-3} \cdot \text{sec}$$

or

$$\text{intcpt} := y_0 - \text{slp} \cdot V_{f,0}$$

$$\text{intcpt} = 7.000 \cdot 10^5 \cdot \text{m}^{-3} \cdot \text{sec}$$

$$\text{RM} := \text{intcpt} \cdot \frac{A \cdot \Delta P_f}{\eta}$$

$$\text{RM} = 2.45 \cdot 10^{12} \cdot \text{m}^{-1}$$

Exercise 10.2

$$c := 125 \frac{\text{kg}}{\text{m}^3} \quad \eta := 0.001 \text{ Pa} \cdot \text{sec} \quad A := 2.72 \text{ m}^2 \quad \Delta P_f := (3 - 0) \cdot 10^5 \text{ Pa}$$

$$t := \begin{bmatrix} 92 \\ 160 \\ 232 \\ 327 \\ 418 \\ 472 \\ 538 \end{bmatrix} \text{ sec}$$

$$V := \begin{bmatrix} 0.024 \\ 0.039 \\ 0.054 \\ 0.071 \\ 0.088 \\ 0.096 \\ 0.106 \end{bmatrix} \text{ m}^3$$

$$t_{\text{div}} V_k := \frac{t_k}{V_k}$$

Apply Eq. 10.11 to filtration at constant ΔP :

$$\text{slp} := \text{slope}(V, t_{\text{div}} V) \quad \text{slp} = 1.47 \cdot 10^4 \cdot \text{m}^{-6} \cdot \text{sec}$$

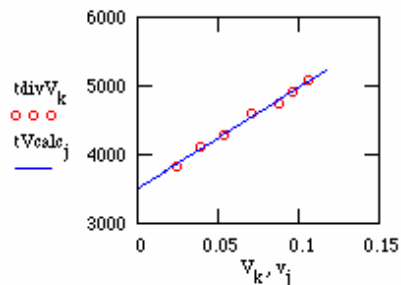
$$\text{intcpt} := \text{intercept}(V, t_{\text{div}} V) \quad \text{intcpt} = 3.51 \cdot 10^3 \cdot \text{m}^{-3} \cdot \text{sec}$$

linearized form

$$tV_{\text{calc},j} := \text{slp} \cdot v_j + \text{intcpt}$$

$$\alpha := \text{slp} \cdot \frac{2 \cdot A^2 \cdot \Delta P_f}{\eta \cdot c} \quad \alpha = 5.23 \cdot 10^{11} \cdot \text{kg}^{-1} \cdot \text{m}$$

$$\text{RM} := \text{intcpt} \cdot \frac{A \cdot \Delta P_f}{\eta} \quad \text{RM} = 2.86 \cdot 10^{12} \cdot \text{m}^{-1}$$



Exercise 10.3 as in previous exercise: $c := 125 \frac{\text{kg}}{\text{m}^3}$ $\eta := 0.001 \text{ Pa} \cdot \text{sec}$ $A := 2.72 \text{ m}^2$

$$\Delta P := \begin{bmatrix} 0.9 \\ 1.2 \\ 1.35 \\ 1.7 \\ 1.8 \\ 1.85 \\ 2.3 \\ 2.4 \\ 2.7 \end{bmatrix} \text{ atm} \quad V := \begin{bmatrix} 0.016 \\ 0.032 \\ 0.04 \\ 0.056 \\ 0.064 \\ 0.072 \\ 0.088 \\ 0.096 \\ 0.114 \end{bmatrix} \text{ m}^3$$

At constant filtration rate $\Phi v = V/t$, Eq. 10.13 is to be applied,
a plot of ΔP versus V will give a straight line

$$a2 := \text{slope}(V, \Delta P) \quad a2 = 1.88 \cdot 10^6 \cdot \text{kg} \cdot \text{m}^{-4} \cdot \text{sec}^{-2}$$

$$b2 := \text{intercept}(V, \Delta P) \quad b2 = 6.19 \cdot 10^4 \cdot \text{kg} \cdot \text{m}^{-1} \cdot \text{sec}^{-2}$$

The filtration rate Φv is calculated from the previous exercise by extrapolation the filtration data to $\Delta P=3 \text{ bar}$:

$$V_{\text{at}_3\text{bar}} := \frac{\Delta P_f - b2}{a2} \quad V_{\text{at}_3\text{bar}} = 0.13 \cdot \text{m}^3$$

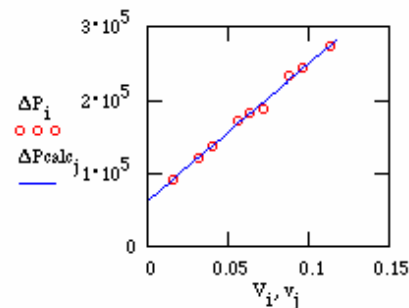
$$\Phi v := \frac{1}{(\text{intcpt} + \text{slp} \cdot V_{\text{at}_3\text{bar}})} \quad \Phi v = 1.86 \cdot 10^{-4} \cdot \text{m}^3 \cdot \text{sec}^{-1}$$

α and R_M from slope and intercept, respectively

$$\Delta P_{\text{calc}_j} := a2 \cdot v_j + b2$$

$$\alpha := a2 \cdot \frac{A^2}{\eta \cdot c \cdot \Phi v} \quad \alpha = 5.97 \cdot 10^{11} \cdot \text{kg}^{-1} \cdot \text{m}$$

$$R_M := b2 \cdot \frac{A}{\eta \cdot \Phi v} \quad R_M = 9.04 \cdot 10^{11} \cdot \text{m}^{-1}$$



Exercise 10.4

$cS := 23.5 \frac{\text{kg}}{\text{m}^3}$ $\eta := 0.001 \text{ Pa} \cdot \text{sec}$ $A := 0.0440 \text{ m}^2$ $V_0 := 0.5 \text{ liter}$

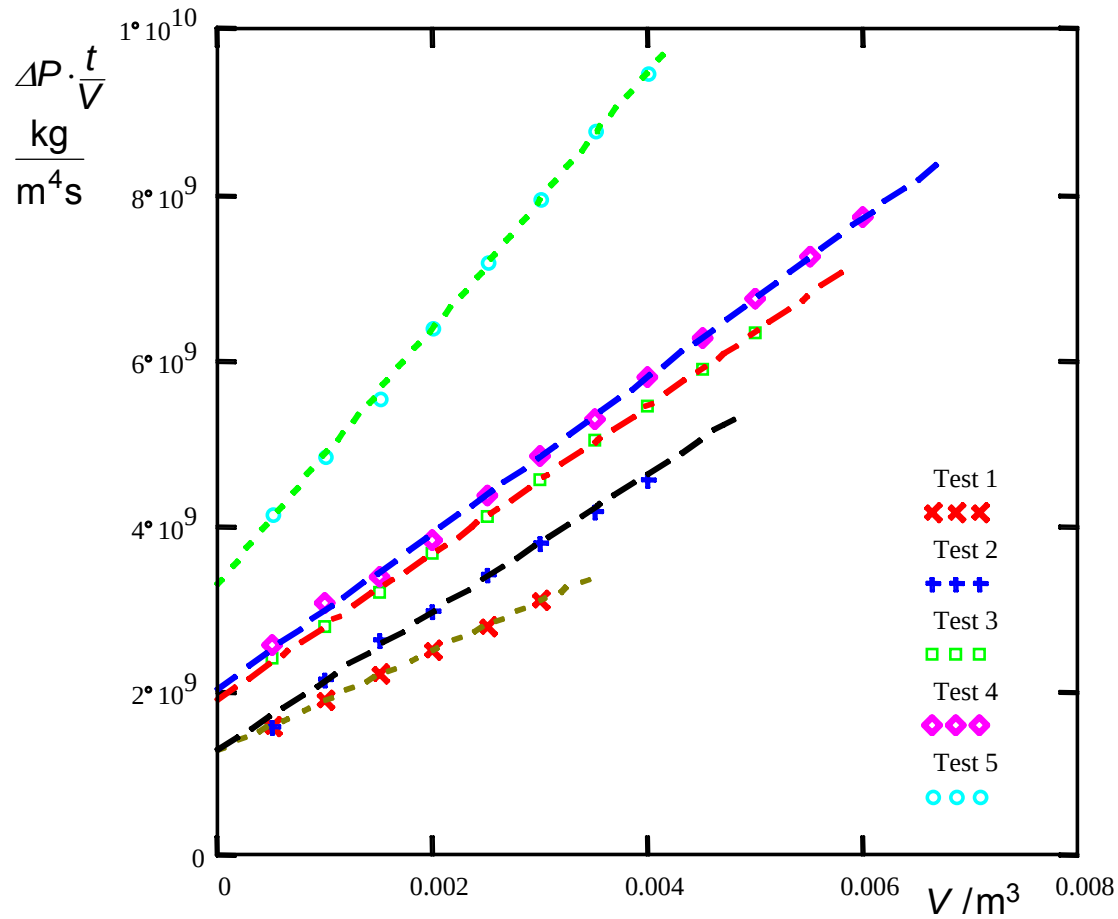
$$V(k) := V_0 \cdot (k + 1)$$

$$\Delta P := \begin{bmatrix} 0.45 \\ 1.1 \\ 1.95 \\ 2.5 \\ 3.4 \end{bmatrix} \text{ atm}$$

$$\text{tf1} := \begin{bmatrix} 17 \\ 41 \\ 72 \\ 108 \\ 152 \\ 202 \end{bmatrix} \text{ sec} \quad \text{tf2} := \begin{bmatrix} 7 \\ 19 \\ 35 \\ 53 \\ 76 \\ 102 \\ 131 \\ 163 \end{bmatrix} \text{ sec} \quad \text{tf3} := \begin{bmatrix} 6 \\ 14 \\ 24 \\ 37 \\ 52 \\ 69 \\ 89 \\ 110 \\ 134 \\ 160 \end{bmatrix} \text{ sec} \quad \text{tf4} := \begin{bmatrix} 5 \\ 12 \\ 20 \\ 30 \\ 43 \\ 57 \\ 73 \\ 91 \\ 111 \\ 133 \\ 157 \end{bmatrix} \text{ sec} \quad \text{tf5} := \begin{bmatrix} 4 \\ 9 \\ 16 \\ 25 \\ 35 \\ 46 \\ 59 \\ 74 \\ 89 \\ 107 \end{bmatrix} \text{ sec}$$

Compressible cake, plot $\Delta P \cdot \frac{t}{V}$ versus filtrate volume V , according to Eq. 10.14

In the graph at the following page a plot at each pressure difference is given. The table summarizes the calculated values of α_{av} and R_M .



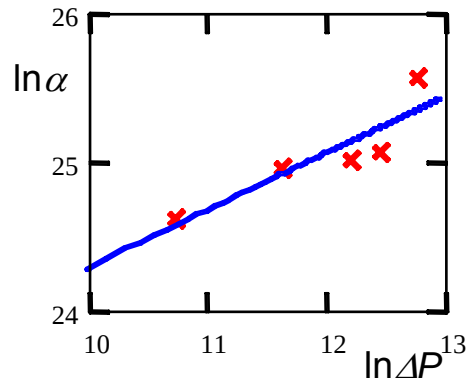
$$\begin{aligned}
 x1_{k1} &:= V(k1) & y1_{k1} &:= \frac{\Delta P_0 \cdot t_{f1_{k1}}}{x1_{k1}} & s11 &:= \text{slope}(x1, y1) & \text{intcpt1} &:= \text{intercept}(x1, y1) \\
 s11 &= 6.05 \cdot 10^{11} \cdot \text{kg} \cdot \text{m}^{-7} \cdot \text{sec}^{-1} & \alpha c_0 &:= s11 \cdot \frac{A^2}{\eta \cdot cS} & \alpha c_0 &= 4.98 \cdot 10^{10} \cdot \text{kg}^{-1} \cdot \text{m} \\
 \text{intcpt1} &= 1.261 \cdot 10^9 \cdot \text{kg} \cdot \text{m}^{-4} \cdot \text{sec}^{-1} & RM1 &:= \text{intcpt1} \cdot \frac{A}{\eta} & RM1 &= 5.55 \cdot 10^{10} \cdot \text{m}^{-1}
 \end{aligned}$$

Test	slope $\times 10^{11} \text{ kg/m}^7 \text{ s}$	α $\times 10^{10} \text{ m/kg}$	intercept $\times 10^9 \text{ kg/m} \cdot \text{s}$	R_M $\times 10^{10} \text{ 1/m}$
1	6.05	4.98	1.26	5.55
2	8.37	6.89	1.26	5.53
3	8.88	7.32	1.88	8.29
4	9.46	7.79	2.00	8.80
5	15.5	12.8	3.29	14.5

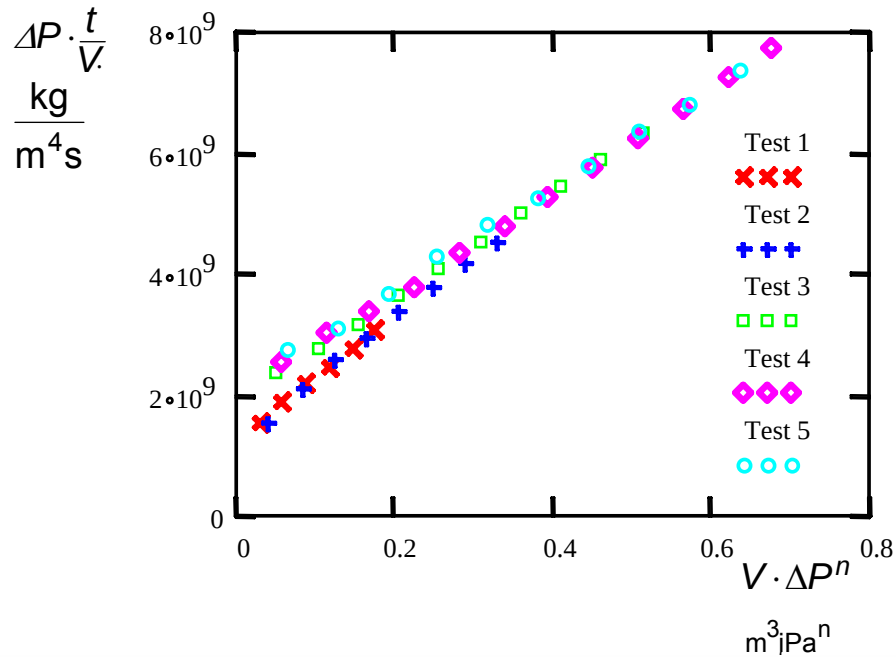
Find α_0 and n by plotting $\ln(\alpha)$ versus $\ln(\Delta P)$, see Eq. 10.15

$$\begin{aligned}
 n &:= \text{slope}(xln, yln) & \ln \alpha_0 &:= \text{intercept}(xln, yln) & \alpha_{calc_j} &:= \left[\exp(\ln \alpha_0) \cdot \left(\frac{\Delta p_j}{P_{ref}} \right)^n \right] \cdot \alpha_{ref} \\
 n &= 0.38 & \ln \alpha_0 &= 20.51 & \alpha_0 &:= \exp(\ln \alpha_0) \cdot \alpha_{ref} & \alpha_0 &= 8.11 \cdot 10^8 \cdot \text{kg}^{-1} \cdot \text{m}
 \end{aligned}$$

A plot of $\ln(\alpha)$ vs $\ln(\Delta P)$ is presented in the following figure:



Closer inspection of Eqs. 10.14 and 10.15 suggests plotting $\Delta P \cdot \frac{t}{V}$ vs $V \cdot \Delta P^n$ to get a unified plot



Exercise 10.5

$$i := 0..5 \quad \Delta P := \begin{bmatrix} 70000 \\ 104000 \\ 140000 \\ 210000 \\ 400000 \\ 800000 \end{bmatrix} \text{ Pa} \quad \alpha := \begin{bmatrix} 1.4 \\ 1.8 \\ 2.1 \\ 2.7 \\ 4 \\ 5.6 \end{bmatrix} \frac{10^{11} \cdot \text{m}}{\text{kg}} \quad \alpha_{\text{ref}} := 1 \frac{\text{m}}{\text{kg}} \quad \Delta P_{\text{ref}} := 1 \text{ Pa}$$

N.B. ref-values are necessary to ensure dimensionless log-arguments

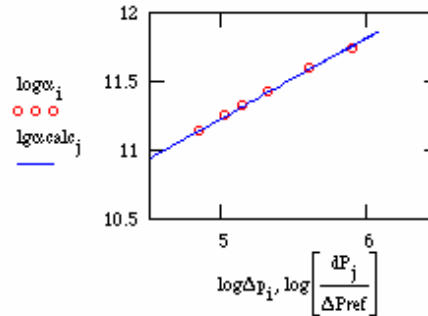
compressible cake: apply Eq. 10.15 $\log \alpha_i := \log \left(\frac{\alpha_i}{\alpha_{\text{ref}}} \right) \quad \log \Delta p_i := \log \left(\frac{\Delta P_i}{\Delta P_{\text{ref}}} \right)$

$$n := \text{slope}(\log \Delta p, \log \alpha) \quad n = 0.574$$

$$\log \alpha_0 := \text{intercept}(\log \Delta p, \log \alpha) \quad \log \alpha_0 = 8.37 \quad \alpha_0 := 10^{\log \alpha_0} \quad \alpha_0 \cdot \alpha_{\text{ref}} = 2.36 \cdot 10^8 \cdot \text{kg}^{-1} \cdot \text{m}$$

A plot of $\log(\alpha)$ against $\log(\Delta P)$:

$$\log \alpha_{\text{calc},j} := \log \alpha_0 + n \cdot \log \left(\frac{dP_j}{\Delta P_{\text{ref}}} \right)$$



Exercise 10.6

$$\begin{aligned} w_f S &:= 0.1 \frac{\text{kg}}{\text{kg}} & \rho S &:= 2500 \frac{\text{kg}}{\text{m}^3} & s_{\text{cake}} &:= 0.5 & \rho L &:= 1000 \frac{\text{kg}}{\text{m}^3} \\ L_{\text{filter}} &:= 0.6 \text{ m} & D_{\text{filter}} &:= 0.6 \text{ m} & \alpha &:= 2.8 \cdot 10^{10} \frac{\text{m}}{\text{kg}} & R_M &:= 3 \cdot 10^9 \frac{1}{\text{m}} \\ t_C &:= 6 \cdot 60 \text{ sec} & \phi &:= 0.2 & \Delta P &:= 65000 \text{ Pa} & \eta L &:= 1 \cdot 10^{-3} \cdot \text{kg} \cdot \text{m}^{-1} \cdot \text{sec}^{-1} \\ cS &:= w_f S \cdot \rho L & cS &= 100 \cdot \text{kg} \cdot \text{m}^{-3} \end{aligned}$$

a) first calculate filter area A , filtration time t_F and filtrate volume V

$$\text{according to Eq. 10.27} \quad t_F := \phi \cdot t_C \quad t_F = 72 \cdot \text{sec}$$

$$A_{\text{tot}} := \pi \cdot D_{\text{filter}} \cdot L_{\text{filter}} \quad A_{\text{tot}} = 1.13 \cdot \text{m}^2$$

$$\begin{aligned} \text{given} \quad \frac{\alpha \cdot \eta \cdot cS}{2 \cdot \phi \cdot A_{\text{tot}}} \cdot V^2 + \eta \cdot R_M \cdot V &= t_F \cdot \Delta P \cdot A & V &:= \text{find}(V) \\ V &= 5.5308 \cdot 10^{-3} \cdot \text{m}^3 \end{aligned}$$

$$\text{Filtrate production rate} \quad \frac{V}{t_C \cdot \phi \cdot A_{\text{tot}}} = 6.79 \cdot 10^{-5} \cdot \text{m} \cdot \text{sec}^{-1}$$

$$\text{dry solids production rate} \quad \frac{cS \cdot V}{t_C \cdot \phi \cdot A_{\text{tot}}} = 6.79 \cdot 10^{-3} \cdot \text{kg} \cdot \text{m}^{-2} \cdot \text{sec}^{-1}$$

b) the solid deposition amounts to $cS \cdot V$

$$cS \cdot V = 0.55 \cdot \text{kg}$$

volume solid

$$\frac{cS \cdot V}{\rho S} = 2.21 \cdot 10^{-4} \cdot \text{m}^3$$

volume cake

$$\frac{cS \cdot V}{\rho S \cdot s_{\text{cake}}} = 4.42 \cdot 10^{-4} \cdot \text{m}^3$$

thickness cake

$$\frac{cS \cdot V}{\rho S \cdot s_{\text{cake}} \cdot (\phi \cdot A_{\text{tot}})} = 1.96 \cdot 10^{-3} \cdot \text{m}$$

Exercise 10.7

$$\begin{aligned}
 cS &:= 230 \frac{\text{kg}}{\text{m}^3} & \Delta P &:= 45000 \text{ Pa} & f &:= 0.3 & tC &:= 5 \text{ min} \\
 \alpha &:= 1.1 \cdot 10^{11} \frac{\text{m}}{\text{kg}} & RM &:= 6 \cdot 10^9 \frac{1}{\text{m}} & & & tC &= 300 \cdot \text{sec} \\
 \Phi_{\text{slurry}} &:= 40 \frac{\text{liter}}{\text{min}} & \Phi_{\text{slurry}} &= 6.67 \cdot 10^{-4} \cdot \text{m}^3 \cdot \text{sec}^{-1} & & & \rho S &:= 2700 \frac{\text{kg}}{\text{m}^3} \\
 \eta_L &:= 0.01 \text{ poise} & \eta_L &= 1 \cdot 10^{-3} \cdot \text{kg} \cdot \text{m}^{-1} \cdot \text{sec}^{-1} & & & &
 \end{aligned}$$

Eq. 10.23 correlates filtrate volume V , filtration time t_F and filtration (submerged) area A for given values of parameters in a continuous vacuum filter

Each unit volume water in the slurry contains cS kg solids with a volume of $\frac{cS}{\rho S} = 0.085 \frac{\text{m}^3}{\text{m}^3} \frac{\text{solid}}{\text{water}}$
 hence 1 m³ water fed corresponds to $\left(1 + \frac{cS}{\rho S}\right)$ m³ slurry

The filtration time $t_F := f \cdot tC$ and the filtrate volume $V := \frac{\Phi_{\text{slurry}}}{1 + \frac{cS}{\rho S}} \cdot t_F$ $V = 0.0553 \cdot \text{m}^3$ and
 $\frac{V}{t_F} = 6.14 \cdot 10^{-4} \cdot \text{m}^3 \cdot \text{sec}^{-1}$

$$\begin{aligned}
 \text{given} \quad & \frac{\alpha \cdot \eta_L \cdot cS}{2 \cdot A} \cdot V^2 + \eta_L \cdot RM \cdot V = t_F \cdot A \cdot \Delta P & \text{Eq. 10.23} \\
 & \text{the (submerged) filter area} & A := \text{find}(A) & A = 3.13 \cdot \text{m}^2 \\
 & \text{the total filter area} & A_{\text{tot}} := \frac{A}{f} & A_{\text{tot}} = 10.4 \cdot \text{m}^2
 \end{aligned}$$

Note that in this solution the holdup of water in the filter cake is not accounted for.
 Taking the moisture in the cake into account will slightly decrease the filtrate volume.

Recalculate the total filter area in case the pores in the cake are completely filled with water $\rho_L := 1000 \frac{\text{kg}}{\text{m}^3}$
 resulting in a moisture content $xM := 0.6$ in kg water / kg dry cake

The non-stationary volume balance of water reads: accumulation in cake equals feed stream - filtrate rate

$$\begin{aligned}
 \text{The volume of water fed per unit time} \quad & \frac{\Phi_{\text{slurry}}}{1 + \frac{cS}{\rho S}} = 6.14 \cdot 10^{-4} \cdot \text{m}^3 \cdot \text{sec}^{-1} \\
 \text{water contained in the cake per unit time} = \text{accumulation in cake} \quad & cS \cdot \frac{\Phi_{\text{slurry}}}{1 + \frac{cS}{\rho S}} \cdot \frac{xM}{\rho_L} = 8.48 \cdot 10^{-5} \cdot \text{m}^3 \cdot \text{sec}^{-1} \\
 \text{Hence the corrected filtration rate follows from}
 \end{aligned}$$

$$\text{given} \quad cS \cdot \frac{\Phi_{\text{slurry}}}{1 + \frac{cS}{\rho S}} \cdot \frac{xM}{\rho_L} = \frac{\Phi_{\text{slurry}}}{1 + \frac{cS}{\rho S}} - \frac{V}{t_F} \quad V := \text{find}(V) \quad V = 0.0477 \cdot \text{m}^3$$

$$\begin{aligned}
 \text{given} \quad & \frac{\alpha \cdot \eta_L \cdot cS}{2 \cdot A} \cdot V^2 + \eta_L \cdot RM \cdot V = t_F \cdot A \cdot \Delta P \\
 & \text{The (submerged) filter area} & A := \text{find}(A) & A = 2.70 \cdot \text{m}^2 \\
 & \text{The total filter area} & A_{\text{tot}} := \frac{A}{f} & A_{\text{tot}} = 9.0 \cdot \text{m}^2
 \end{aligned}$$

Exercise 10.8

$$\begin{aligned} \Delta P &:= (10^5 - 68000) \text{ Pa} & A_{\text{tot}} &:= 10 \text{ m}^2 & \alpha &:= 1 \cdot 10^{10} \frac{\text{m}}{\text{kg}} & R_M &:= 1 \cdot 10^{10} \frac{1}{\text{m}} \\ f &:= 0.3 & t_C &:= 1 \text{ min} & t_C &= 60 \cdot \text{sec} \\ x_S &:= 0.1 \frac{\text{kg}}{\text{kg}} \frac{\text{solid}}{\text{slurry}} & x_M &:= (3.5 - 1) \frac{\text{kg}}{\text{kg}} \frac{\text{moist}}{\text{dry_cake}} & \rho_S &:= 2000 \frac{\text{kg}}{\text{m}^3} \\ \rho_L &:= 1000 \frac{\text{kg}}{\text{m}^3} & \eta_L &:= 0.01 \text{ poise} & \eta_L &= 1 \cdot 10^{-3} \cdot \text{kg} \cdot \text{m}^{-1} \cdot \text{sec}^{-1} \end{aligned}$$

As in the previous exercise, the filtration rate $\frac{V}{t_F}$ is defined in Eq. 10.23 with:

$$t_F := f \cdot t_C \quad t_F = 18 \cdot \text{sec} \quad \text{and} \quad A := f \cdot A_{\text{tot}} \quad A = 3.00 \cdot \text{m}^2$$

Unlike the previous solution, here Φ_{slurry} has to be calculated from $\frac{V}{t_F}$, requiring the value of c_S (kg S / m³ water)

$$\begin{aligned} c_{S\text{in}} &:= \frac{x_S}{\left(\frac{1 - x_S}{\rho_L} \right)} \frac{\text{kg}_S}{\text{m}^3 \text{ water_in}} & c_{S\text{in}} &= 111.11 \cdot \text{kg} \cdot \text{m}^{-3} \\ \text{given} \quad \frac{\alpha \cdot \eta_L \cdot c_{S\text{in}}}{2 \cdot A} \cdot V^2 + \eta_L \cdot R_M \cdot V &= t_F \cdot A \cdot \Delta P & V &:= \text{find}(V) & V &= 0.073 \cdot \text{m}^3 \\ & & \text{and} \quad \frac{V}{t_F} &= 4.07 \cdot 10^{-3} \cdot \text{m}^3 \cdot \text{sec}^{-1} \end{aligned}$$

Now calculate Φ_{slurry} from the water volume balance: accumulation in cake equals $\text{water fed} / \text{time} - \text{filtration rate}$

$$\begin{aligned} \text{given} \quad c_{S\text{in}} \cdot \frac{\Phi_{\text{slurry}}}{1 + \frac{c_{S\text{in}}}{\rho_S}} \cdot \frac{x_M}{\rho_L} &= \frac{\Phi_{\text{slurry}}}{1 + \frac{c_{S\text{in}}}{\rho_S}} - \frac{V}{t_F} & \Phi_{\text{slurry}} &:= \text{find}(\Phi_{\text{slurry}}) & \Phi_{\text{slurry}} &= 5.95 \cdot 10^{-3} \cdot \text{m}^3 \cdot \text{sec}^{-1} \\ \text{with} \quad \rho_{\text{slurry}} &:= \frac{1}{\frac{x_S}{\rho_S} + \frac{1 - x_S}{\rho_L}} & \rho_{\text{slurry}} &= 1.053 \cdot 10^3 \cdot \text{kg} \cdot \text{m}^{-3} \end{aligned}$$

$$\text{follows the solids production rate} \quad x_S \cdot \rho_{\text{slurry}} \cdot \Phi_{\text{slurry}} = 0.627 \cdot \text{kg} \cdot \text{sec}^{-1}$$

Alternatively, the solids production rate follows from the *water fed* times the solids concentration in kg per unit volume *water fed*, or

$$\frac{\Phi_{\text{slurry}}}{1 + \frac{c_{S\text{in}}}{\rho_S}} \cdot c_{S\text{in}} = 0.627 \cdot \text{kg} \cdot \text{sec}^{-1} \quad \text{also} \quad \left[\frac{V}{t_F} + c_{S\text{in}} \cdot \frac{\Phi_{\text{slurry}}}{1 + \frac{c_{S\text{in}}}{\rho_S}} \cdot \frac{x_M}{\rho_L} \right] \cdot c_{S\text{in}} = 0.627 \cdot \text{kg} \cdot \text{sec}^{-1}$$

Exercise 10.9

$$\begin{aligned} \Delta P &:= 60000 \text{ Pa} & \alpha &:= 5 \cdot 10^9 \frac{\text{m}}{\text{kg}} & R_M &:= 0 \frac{1}{\text{m}} & \text{width} &:= 1 \text{ m} \\ \epsilon_{\text{cake}} &:= 0.43 & c_S &:= 350 \frac{\text{kg}}{\text{m}^3} \frac{\text{solid}}{\text{slurry}} & \rho_S &:= 2000 \frac{\text{kg}}{\text{m}^3} & \rho_L &:= 1000 \frac{\text{kg}}{\text{m}^3} \\ \eta_L &:= 0.01 \text{ poise} & \eta_L &= 1 \cdot 10^{-3} \cdot \text{kg} \cdot \text{m}^{-1} \cdot \text{sec}^{-1} & \Phi_{\text{filtrate}} &:= 5 \frac{\text{m}^3}{\text{hr}} & \Phi_{\text{filtrate}} &= 1.39 \cdot 10^{-3} \cdot \text{m}^3 \cdot \text{sec}^{-1} \end{aligned}$$

Unlike the previous exercise, where cS is expressed in kg S / m³ water, here the solids concentration is expressed in kg S / m³ *slurry*, hence now 1 m³ slurry corresponds to $\left(1 - \frac{cS}{\rho S}\right)$ m³ *water fed*

and the cake formation rate equals $\Phi_{slurry} \cdot \frac{cS}{\rho S}$ in m³ solid per unit time.

The non-steady state volume balance for water reads :

$$\text{accumulation of water in cake} = \text{water fed} - \text{filtrate rate}$$

or

$$\text{given } \Phi_{slurry} \cdot \frac{cS}{\rho S} \cdot \frac{s_{cake}}{1 - s_{cake}} = \Phi_{slurry} \cdot \left(1 - \frac{cS}{\rho S}\right) - \Phi_{filtrate} \quad \Phi_{slurry} := \text{find}(\Phi_{slurry})$$

$$\Phi_{slurry} = 2.004 \cdot 10^{-3} \cdot \text{m}^3 \cdot \text{sec}^{-1}$$

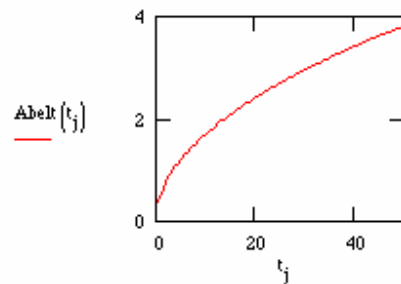
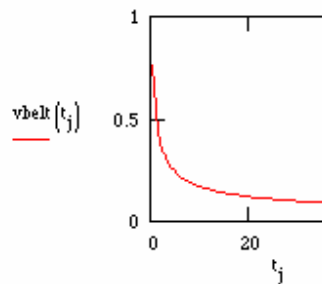
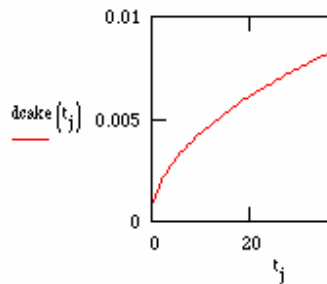
$$\text{solids deposition rate} \quad \Phi_{slurry} \cdot \frac{cS}{\rho S} = 3.51 \cdot 10^{-4} \cdot \text{m}^3 \cdot \text{sec}^{-1}$$

$$\text{cake formation rate} \quad \Phi_{slurry} \cdot \frac{cS}{\rho S} \cdot \frac{1}{1 - s_{cake}} = 6.15 \cdot 10^{-4} \cdot \text{m}^3 \cdot \text{sec}^{-1}$$

rearrangement of Eq. 10.23

$$\text{with } \Phi_{filtrate} := \frac{V}{tF} \text{ and } cS := \frac{cS}{1 - \frac{cS}{\rho S}} \quad (\text{in kg S/m}^3 \text{ water}) \text{ gives filtration area as a function of } tF:$$

$$Abelt(tF) := \Phi_{filtrate} \cdot \sqrt{\frac{\alpha \cdot \eta L \cdot cS}{2 \cdot \Delta P}} \cdot tF \text{ and } vbelt(tF) := \frac{Abelt(tF)}{tF \cdot \text{width}} \quad dcake(tF) := \Phi_{slurry} \cdot \frac{cS}{\rho S} \cdot \frac{1}{1 - s_{cake}} \cdot \frac{tF}{Abelt(tF)}$$



A high belt speed $vbelt$ requires a low filtration area $Abelt$ but a thin cake layer $dcake$. Choosing either of these parameters fixes the value of the others. In practise the cake thickness is chosen such that it can be removed easily.

Exercise 10.10

$$\alpha_0 := 7.1 \cdot 10^8 \cdot \frac{\text{m}}{\text{kg}} \quad s_{\alpha} := 0.46 \quad RM := 2 \cdot 10^9 \cdot \frac{1}{\text{m}}$$

$$s_0 := 0.84 \quad s_{\epsilon} := -0.054 \quad \Delta P := 50000 \text{ Pa} \quad \Delta P_{\text{pref}} := 1 \text{ Pa}$$

$$L_{\text{tot}} := 9 \text{ m} \quad L_{\text{filt}} := 1.5 \text{ m} \quad \text{width} := 2 \text{ m} \quad vbelt := 0.1 \cdot \frac{\text{m}}{\text{sec}}$$

$$xS := 0.3 \cdot \frac{\text{kg}}{\text{kg}} \cdot \frac{\text{solid}}{\text{slurry}} \quad \rho S := 2350 \cdot \frac{\text{kg}}{\text{m}^3}$$

$$\eta L := 0.01 \text{ poise} \quad \eta L = 1 \cdot 10^{-3} \cdot \text{kg} \cdot \text{m}^{-1} \cdot \text{sec}^{-1} \quad \rho L := 1390 \cdot \frac{\text{kg}}{\text{m}^3}$$

Again, Eq. 10.23 relates filtrate volume V and filtration time t_F , given filtration surface area A , pressure drop ΔP and specific resistances. The following values are calculated directly from the given data:

$$A := L_{\text{filt}} \cdot \text{width} \quad A = 3 \cdot \text{m}^2 \quad t_F := \frac{L_{\text{filt}}}{v_{\text{belt}}} \quad t_F = 15 \cdot \text{sec}$$

$$\alpha_{\text{av}} := \alpha_0 \cdot \left(\frac{\Delta P}{P_{\text{ref}}} \right)^{\text{sc}} \quad \alpha_{\text{av}} = 1.03 \cdot 10^{11} \cdot \text{kg}^{-1} \cdot \text{m} \quad \varepsilon_{\text{av}} := \varepsilon_0 \cdot \left(\frac{\Delta P}{P_{\text{ref}}} \right)^{\text{sc}} \quad \varepsilon_{\text{av}} = 0.468$$

Note that cS in Eq. 10.23 is expressed in kg S per unit volume filtrate
1 kg of slurry contains

$$xS = 0.3 \cdot \text{kgS} \quad \text{and} \quad (1 - xS) = 0.7 \cdot \text{kg}_{\text{liquid}} \quad \text{of which} \quad \frac{xS}{\rho_S} \cdot \frac{\varepsilon_{\text{av}}}{1 - \varepsilon_{\text{av}}} = 1.12 \cdot 10^{-4} \cdot \text{kg}^{-1} \cdot \text{m}^3 \quad \text{will be contained in } xS \text{ kg cake after filtration of } (1-xS) \text{ kg liquid}$$

hence

$$cS := \frac{xS}{\frac{1 - xS}{\rho_L} - \frac{xS}{\rho_S} \cdot \frac{\varepsilon_{\text{av}}}{1 - \varepsilon_{\text{av}}}} \quad cS = 767 \cdot \text{kg} \cdot \text{m}^{-3} \quad \frac{\text{kgS}}{\text{m}^3 \text{ filtrate}}$$

the non-stationary water volume balance: accumulation in cake equals *liquid fed / time* - filtration rate

$$\text{given} \quad \frac{\alpha_{\text{av}} \cdot \eta_L \cdot cS}{2 \cdot A^2 \cdot \Delta P} \cdot V^2 + \frac{\eta_L \cdot RM}{A \cdot \Delta P} \cdot V = t_F \quad V := \text{find}(V) \quad V = 0.0130 \cdot \text{m}^3$$

$$\text{The solid production rate amounts to} \quad cS \cdot \frac{V}{t_F} = 0.66 \cdot \text{kg} \cdot \text{sec}^{-1}$$

$$\text{Check:} \quad \text{filtration rate} \quad \frac{V}{t_F} = 8.67 \cdot 10^{-4} \cdot \text{m}^3 \cdot \text{sec}^{-1}$$

+

$$\text{liquid feed} \quad \frac{V}{t_F} \cdot \left(1 + \frac{cS}{\rho_S} \cdot \frac{\varepsilon_{\text{av}}}{1 - \varepsilon_{\text{av}}} \right) = 1.12 \cdot 10^{-3} \cdot \text{m}^3 \cdot \text{sec}^{-1}$$

$$\frac{V}{t_F} \cdot \left(1 + \frac{cS}{\rho_S} \cdot \frac{\varepsilon_{\text{av}}}{1 - \varepsilon_{\text{av}}} \right) \cdot \rho_L = 1.55 \cdot \text{kg} \cdot \text{sec}^{-1}$$

$$\text{solid feed} \quad \frac{xS}{1 - xS} \cdot \left[\frac{V}{t_F} \cdot \left(1 + \frac{cS}{\rho_S} \cdot \frac{\varepsilon_{\text{av}}}{1 - \varepsilon_{\text{av}}} \right) \cdot \rho_L \right] = 0.66 \cdot \text{kg} \cdot \text{sec}^{-1}$$

Exercise 10.11

$$\Delta P_{\text{max}} := 350000 \cdot \text{Pa} \quad \alpha := 1.8 \cdot 10^{11} \frac{\text{m}}{\text{kg}} \quad RM := 10^{10} \frac{1}{\text{m}}$$

$$cS := 150 \frac{\text{kg}}{\text{m}^3} \frac{\text{solid}}{\text{filtrate}} \quad \Phi_{\text{filtrate}} := 25 \frac{\text{liter}}{\text{min}} \quad \Phi_{\text{filtrate}} = 4.17 \cdot 10^{-4} \cdot \text{m}^3 \cdot \text{sec}^{-1}$$

$$\eta_L := 0.01 \text{ poise} \quad \eta_L = 1 \cdot 10^{-3} \cdot \text{kg} \cdot \text{m}^{-1} \cdot \text{sec}^{-1} \quad V_{\text{tot}} := 5 \text{ m}^3 \quad \text{Assume } A := 6 \text{ m}^2$$

First part at constant filtration rate, apply Eq. 10.13 to find the amount filtrate accumulated at $\Delta P := \Delta P_{\text{max}}$

$$\text{given} \quad \frac{\alpha \cdot \eta_L \cdot cS}{2 \cdot A} \cdot V_{\text{cr}} + \eta_L \cdot RM = \Delta P_{\text{max}} \cdot \frac{A}{\Phi_{\text{filtrate}}} \quad V_{\text{cr}} := \text{find}(V_{\text{cr}}) \quad V_{\text{cr}} = 2.24 \cdot \text{m}^3$$

$$\tau_{\text{cr}} := \frac{V_{\text{cr}}}{\Phi_{\text{filtrate}}} \quad \tau_{\text{cr}} = 5.37 \cdot 10^3 \cdot \text{sec}$$

Second part at constant $\Delta P := \Delta P_{\max}$, apply Eq. 10.11 to find the time τ_{cp} to accumulate $(V_{\text{tot}} - V_{cr})$ m³ filtrate

$$V_{cp} := V_{\text{tot}} - V_{cr} \quad V_{cp} = 2.76 \cdot \text{m}^3$$

$$\text{given} \quad \frac{\alpha \cdot \eta L \cdot cS}{2 \cdot A} \cdot V_{cp} + \eta L \cdot RM = \frac{\tau_{cp}}{V_{cp}} \cdot A \cdot \Delta P_{\max} \quad \tau_{cp} := \text{find}(\tau_{cp}) \quad \tau_{cp} = 8.20 \cdot 10^3 \cdot \text{sec}$$

$$\text{The total time required amounts to} \quad \tau_{cr} + \tau_{cp} = 1.36 \cdot 10^4 \cdot \text{sec} \quad \text{or} \quad \frac{\tau_{cr} + \tau_{cp}}{3600 \text{ sec}} = 3.77 \text{ hr}$$

Exercise 10.12

$$\begin{aligned} d_{\text{cake}} &:= 0.025 \text{ m} & \epsilon &:= 0.53 & \alpha &:= 6 \cdot 10^9 \frac{\text{m}}{\text{kg}} & RM &:= 10^8 \frac{1}{\text{m}} & N &:= 20 \frac{1}{\text{sec}} \\ \rho_L &:= 1000 \frac{\text{kg}}{\text{m}^3} & \rho_S &:= 2000 \frac{\text{kg}}{\text{m}^3} & \eta &:= 0.001 \text{ Pa} \cdot \text{sec} & D_{\text{basket}} &:= 0.635 \text{ m} & H_{\text{basket}} &:= 0.254 \text{ m} \\ \text{Apply Eq. 10.4X with} & r_0 &:= \frac{D_{\text{basket}}}{2} & r_C &:= r_0 - d_{\text{cake}} & r_0 &= 0.3175 \cdot \text{m} & r_C &= 0.2925 \cdot \text{m} \\ & \omega &:= N \cdot 2 \cdot \pi & \omega &= 125.66 \cdot \text{sec}^{-1} \end{aligned}$$

No liquid on top of the filter cake, hence $r := r_C$

$$Q(r) := \frac{\frac{\rho_L \cdot \omega^2}{2} \cdot (r_0^2 - r^2)}{\left[\frac{\eta \cdot \alpha \cdot (1 - \epsilon) \cdot \rho_S}{2 \cdot \pi \cdot H_{\text{basket}}} \right] \cdot \ln\left(\frac{r_0}{r_C}\right) + \frac{\eta \cdot RM}{2 \cdot \pi \cdot r_0 \cdot H_{\text{basket}}}} \quad Q(r_C) = 4.15 \cdot 10^{-4} \cdot \text{m}^3 \cdot \text{sec}^{-1}$$

$$\text{a) Interstitial volume} \quad V_{\epsilon} := H_{\text{basket}} \cdot \pi \cdot (r_0^2 - r_C^2) \cdot \epsilon \quad V_{\epsilon} = 6.45 \cdot 10^{-3} \cdot \text{m}^3$$

$$\tau := \frac{2 \cdot V_{\epsilon}}{Q(r_C)} \quad \tau = 31.1 \cdot \text{sec}$$

$$\text{b) } \delta L := 0.05 \text{ m} \quad r_F := r_C - \delta L \quad r_F = 0.2425 \cdot \text{m} \quad Q(r_F) = 1.14 \cdot 10^{-3} \cdot \text{m}^3 \cdot \text{sec}^{-1}$$

$$\text{change in washing rate} = \frac{Q(r_F)}{Q(r_C)} = 2.75 \text{ times}$$

Chapter 10: FILTRATION (continued)

Exercise 10.13

To evaluate the feasibility of filtration a titanium dioxide slurry is filtered in a laboratory setup with an area of 0.01 m^2 and a constant pressure difference of 1 bar. The solid concentration equals $50 \text{ kg dry titanium dioxide per m}^3$ filtrate. The liquid viscosity is 0.001 Pa.s . A typical measuring series yields:

Time(s):	60	300	540	900
Filtrate volume *103 m3:	0.25	0.59	0.79	1.03

- a. Determine the specific cake resistance α

The same titanium dioxide slurry is filtered with a horizontal belt filter at a constant pressure difference $\Delta P = 60 \text{ kPa}$ and a belt velocity of 0.1 m s^{-1} . The filter area of 2 m^2 uses the same filter cloth as used in the laboratory trials. The medium resistance R_M can be neglected compared to the specific cake resistance. The belt width equals $w=0.5 \text{ m}$

- b. Calculate the production capacity of this belt filter in $\text{kg dry titanium dioxide per hour}$.

Exercise 10.14

A slurry that contains $100 \text{ kg dry solid particles per m}^3$ of slurry has been evaluated in a laboratory filter with an area of 0.050 m^2 . These tests were performed with a constant pressure difference of 0.6 bar. After each half liter of filtrate volume the time was recorded. A representative series is:

time (s)	45	101	180	265
Filtrate volume (liter)	0.5	1.0	1.5	2.0

- a. Determine the specific cake resistance α (m kg^{-1}) and the filter resistance R_M (m^{-1})

$$\rho_s = 1050 \text{ kg m}^{-3} \quad \rho_L = 1000 \text{ kg m}^{-3} \quad \eta = 10^{-3} \text{ Pa.s} \quad g = 9.81 \text{ m s}^{-2}$$

The large scale filtration of this slurry is executed under constant rate conditions in a pressure filter. The maximum overpressure of the filter housing amounts 2 bar. The filtration starts with a pressure difference of 0.6 bar on a clean filter medium with the same properties as used in the laboratory tests. The filter area $A = 4 \text{ m}^2$.

- b. Calculate the initial filtration rate?
 c. Calculate the filtrate amount at the moment the overpressure equals 2 bar.
 d. How long does it take to reach this 2 bar overpressure?

Exercise 10.13

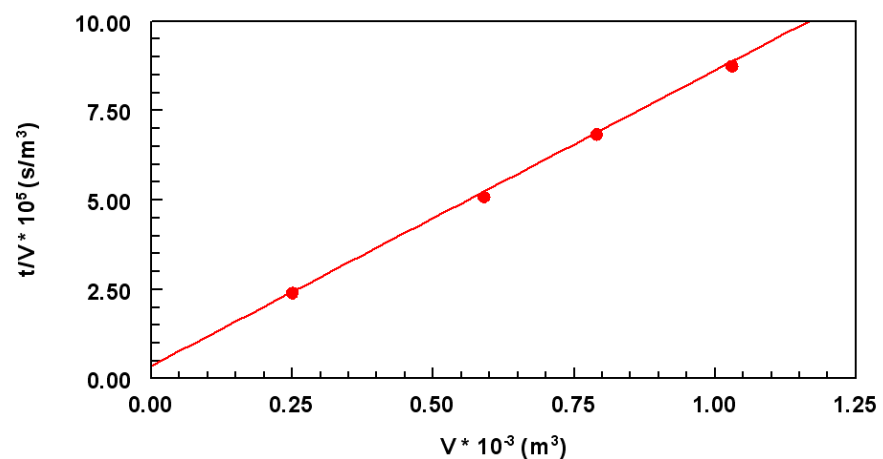
Given $C_S = 50 \text{ kg m}^{-3}$ $A_{\text{LAB}} = 0.010 \text{ m}^2$ $\Delta P_{\text{LAB}} = 1 \text{ bar}$
 $A_{\text{Large}} = 2 \text{ m}^2$ $v_B = 0.1 \text{ m s}^{-1}$ $\Delta P_{\text{Belt}} = 0.6 \text{ bar}$ $w = 0.5 \text{ m}$
 $\rho_S = 1050 \text{ kg/m}^3$ $\rho_L = 1000 \text{ kg/m}^3$
 $\eta = 10^{-3} \text{ Pa.s}$ $g = 9.81 \text{ m s}^{-2}$

Find a) Specific cake resistance α
 b) Belt filter production capacity

Solution a) Constant pressure filtration

$$\frac{t}{V} = \frac{\eta c \alpha}{2A^2 \Delta P} V + \frac{\eta R_M}{A \Delta P} \quad \text{graph of } t/V \text{ vs } V \text{ gives } \alpha \text{ and } R_M$$

t(s)	V(*10 ³ m ³)	t/V (*10 ⁵ s/m ³)
60	0.25	2.40
300	0.59	5.08
540	0.79	6.83
900	1.03	8.74



Slope: $\frac{\eta c \alpha}{2A^2 \Delta P} = \frac{(10 - 0.35) \times 10^5}{1.17 \times 10^{-3}} = 8.25 \times 10^8 \text{ (s)}$

$$\alpha = \frac{2A^2 \Delta P}{\eta c} = 8.25 \times 10^8 \frac{2 \cdot 0.01^2 \cdot 10^5}{10^{-3} \cdot 50} = 3.3 \times 10^{11} \text{ (m/kg)}$$

b) Neglect R_m

$$\left(\frac{\alpha \eta c}{2\Delta P A^2}\right) V^2 - t_F = 0 \rightarrow V = \sqrt{\left(\frac{2\Delta P A^2 t_F}{\alpha \eta c}\right)} \quad (\text{m}^3)$$

$$z_F = \frac{A}{w} = \frac{2}{0.5} = 4 \quad (\text{m})$$

$$t_F = \frac{z_F}{v_B} = \frac{4}{0.1} = 40 \quad (\text{s})$$

Filtrate production:

$$V = \sqrt{\left(\frac{2\Delta P A^2 t_F}{\alpha \eta c}\right)} = \sqrt{\frac{2 \cdot 60 \times 10^3 \cdot 2^2 \cdot 40}{3.3 \times 10^{11} \cdot 10^{-3} \cdot 50}} = 3.4 \times 10^{-2} \quad (\text{m}^3)$$

Dry production capacity:

$$w_r = \frac{cV}{t_F} = \frac{50 \cdot 3.4 \times 10^{-2}}{40} \times 3600 = 153 \quad (\text{kg/uur})$$

Exercise 10.14

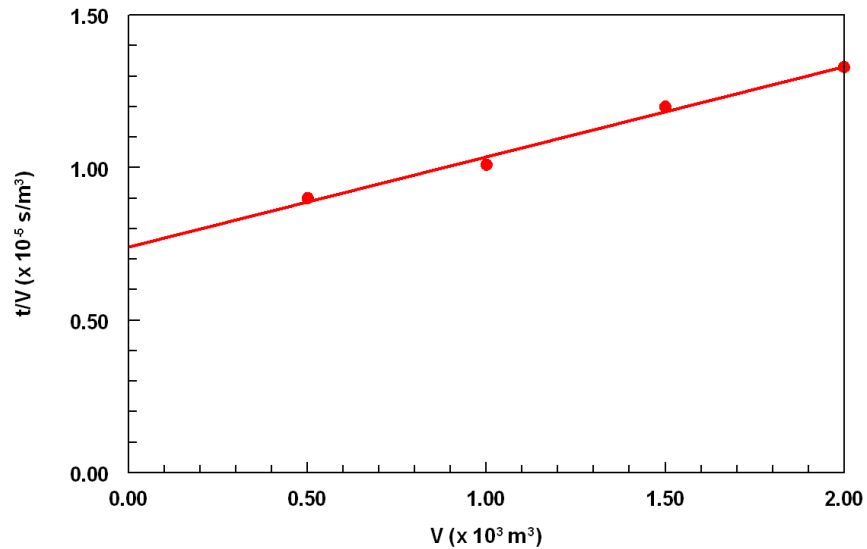
Given $C_S = 100 \text{ kg m}^{-3}$ $A_{\text{LAB}} = 0.050 \text{ m}^2$ $\Delta P_{\text{LAB}} = 0.6 \text{ bar}$
 $A_{\text{Large}} = 4 \text{ m}^2$ $\rho_S = 1050 \text{ kg/m}^3$ $\rho_L = 1000 \text{ kg/m}^3$
 $\eta = 10^{-3} \text{ Pa.s}$ $g = 9.81 \text{ m s}^{-2}$

Find a) Specific cake resistance α and the filter resistance R_m
 b) Initial filtration rate
 c) Filtrate amount at overpressure of 2 bar
 d) Time to reach this 2 bar overpressure

Solution a) Constant pressure difference:

$$\frac{t}{V} = \frac{\eta c \alpha}{2A^2 \Delta P} V + \frac{\eta R_m}{A \Delta P} \quad \text{graph of } t/V \text{ vs } V \text{ yields } \alpha \text{ and } R_m$$

$t(\text{s})$	$V(*10^3 \text{ m}^3)$	$t/V (*10^5 \text{ s/m}^3)$
45	0.50	0.90
101	1.00	1.01
180	1.50	1.20
265	2.00	1.33



$$\text{Slope: } \frac{\eta c \alpha}{2A^2 \Delta P} = \frac{(1.33 - 0.74) \times 10^5}{2.00 \times 10^{-3}} = 2.95 \times 10^7 \text{ (s)}$$

$$\alpha = 2.95 \times 10^7 \frac{2 \cdot 0.05^2 \cdot 6 \times 10^4}{10^{-3} \cdot 100} = 8.85 \times 10^{10} \text{ (m/kg)}$$

$$\text{Intercept: } \frac{\eta R_m}{A \Delta P} = 0.74 \times 10^5 \text{ (s/m}^3\text{)}$$

$$R_m = 0.74 \times 10^5 \frac{0.05 \cdot 6 \times 10^4}{10^{-3}} = 2.22 \times 10^{11} \text{ (1/m)}$$

b) Initial filtration rate

$$\frac{dV}{dt} = \frac{A \Delta P}{\eta R_m} = \frac{4 \cdot 0.6 \times 10^5}{10^{-3} \cdot 2.22 \times 10^{11}} = 1.081 \times 10^{-3} \left(\frac{\text{m}^3}{\text{s}} \right) = \text{constant}$$

c)

$$\Delta P_{\max} = \frac{\eta c \alpha}{A^2} \frac{dV}{dt} V + \frac{\eta R_m}{A} \frac{dV}{dt}$$

$$= \frac{10^{-3} \cdot 100 \cdot 8.85 \times 10^{10}}{4^2} 1.081 \times 10^{-3} V + \frac{10^{-3} \cdot 2.22 \times 10^{11}}{4} 1.081 \times 10^{-3} = 2 \times 10^5$$

gives $V = 0.234 \text{ m}^3$

d) $t = \frac{V}{\frac{dV}{dt}} = \frac{0.234}{1.081 \times 10^{-3}} = 216 \text{ (s)}$

Chapter 11: MEMBRANE FILTRATION

Exercise 11.1

Show that permeability can be expressed in m^2s^{-1} .

Exercise 11.2

The concentration of a solute at both sides of a membrane with thickness $30\mu\text{m}$ is 0.030 kmol m^{-3} and $0.0050\text{ kmol m}^{-3}$. The equilibrium constant $K = 1.5$ and $D_A = 7.0 \cdot 10^{-11}\text{ m}^2\text{ s}^{-1}$ in the membrane. The mass transfer coefficients at both liquid sides can be considered as infinite.

Calculate the flux and the concentrations at the membrane interfaces inside the membrane.

Exercise 11.3

Repeat the previous exercise in case that the mass transfer coefficient at the product side amounts to $2 \cdot 10^{-5}\text{ m s}^{-1}$.

Exercise 11.4

Calculate the osmotic pressure of a solution containing $0.1\text{ mol NaCl kg H}_2\text{O}$ at 25°C . The density of pure water at 25°C amounts to 997.0 kg m^{-3} .

Compare the result with the experimental value given in Table 11.1.

Exercise 11.5

The permeability of a membrane is experimentally determined by measuring the flow of an aqueous NaCl solution in a reverse osmosis module at a given pressure difference.

The inlet concentration $c_{in} = 10\text{ kg /m}^3$ solution with a density of 1004 kg/m^3 solution. The product concentration $c_{out} = 0.39\text{ kg/m}^3$ solution with a density of 997 kg/m^3 solution.

The measured flow rate is $1.92 \cdot 10^{-8}\text{ m}^3\text{ solution/s}$ at a pressure difference of 54.4 atm , the membrane area $A = 0.0020\text{ m}^2$.

Calculate the permeances for water and sodium chloride.

SOLUTIONS

Exercise 11.1

Show The dimension of permeability can be $\text{m}^2 \text{s}^{-1}$.

Thoughts Pick a flux equation such as Eq. 11.3c, with Δc as the driving force. Then the proportionality constant $P_{M,i}$ plays a role similar to that of the coefficient of diffusion.

Solution $[P_{M,i}] = \frac{[N] \cdot [\delta]}{[c]} = \frac{\text{mol}}{\text{m}^2 \cdot \text{s}} \cdot \text{m} \cdot \frac{\text{m}^3}{\text{mol}} = \frac{\text{m}^2}{\text{s}}$. Compare with permeance in exercise 11.5.

Exercise 11.2

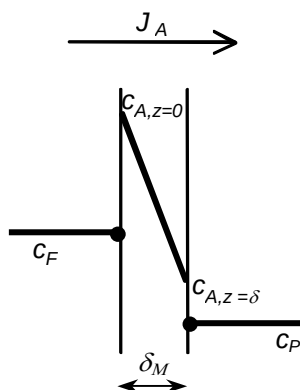
Given $c_F = 0.030 \text{ kmol s}^{-1}$ $c_P = 0.0050 \text{ kmol s}^{-1}$ $\delta = 30 \text{ }\mu\text{m}$
 $K_A = 1.5$ $D_A = 7.0 \cdot 10^{-11} \text{ m}^2 \text{ s}^{-1}$ $k_1 = k_2 = \infty$

Find The flux J_A and the concentrations at the interfaces inside the membrane, $c_{A,z=0}$ and $c_{A,z=\delta}$, respectively.

Thoughts The concentration gradient is depicted in Fig. 11.7; Eqs. 11.3c and 11.12 is applicable (Δc driving force).

Solution The relevant transport equation is given by Eq. 11.12:

$$J_A = \frac{D_A}{\delta_M} (c_{A,z=0} - c_{A,z=\delta}) = \frac{K_A D_A}{\delta_M} (c_F - c_P) = \frac{1.5 \cdot 7 \cdot 10^{-11}}{30 \cdot 10^{-6}} \cdot (0.03 - 0.0050) = 8.75 \cdot 10^{-8} \text{ kmol s}^{-1} \text{ m}^{-2}$$



$$c_{A,z=0} = K_A \cdot c_F = 45 \text{ mol m}^{-3};$$

$$c_{A,z=\delta} = K_A \cdot c_P = 7.5 \text{ mol m}^{-3}.$$

Exercise 11.3

Given $c_F = 0.030 \text{ kmol m}^{-3}$ $c_P = 0.0050 \text{ kmol m}^{-3}$ $\delta_M = 30 \text{ }\mu\text{m}$
 $K_A = 1.5$ $D_A = 7.0 \cdot 10^{-11} \text{ m}^2 \text{ s}^{-1}$
 $k_F = \infty$ $k_P = 2 \cdot 10^{-5} \text{ m s}^{-1}$.

Find The flux J_A and the concentrations at the interfaces inside the membrane, $c_{A,z=0}$ and $c_{A,z=\delta}$, respectively.

Thoughts On the feed side simply apply Eq. 11.11. On the product side, see the derivation of the kinetic expression in Eq. 4.6, where also a concentration gradient contributes to the rate of mass transfer.

Solution Feed side: $c_{A,z=0} = K_A \cdot c_F = 45 \text{ mol m}^{-3}$.

No accumulation of matter takes place in the interface between membrane surface and product phase, hence the rate of mass transfer through the membrane equals that through the stagnant film at the product side. The rate expressions according to the film theory:

$$J_A = \frac{D_A}{\delta_M} (c_{A,z=0} - c_{A,z=\delta}) = k_P (c_{P,z=\delta} - c_P) \text{ or, with Eq. 11.11a}$$

$$J_A = \frac{K_A D_A}{\delta_M} (c_F - c_{P,z=\delta}) = k_P (c_{P,z=\delta} - c_P)$$

Eliminate $c_{P,z=\delta}$ by summation of the following two expressions

$$c_F - c_{P,z=\delta} = J_A \frac{\delta_M}{K_A D_A}$$

$$c_{P,z=\delta} - c_P = \frac{J_A}{k_P}$$

giving

$$c_F - c_P = J_A \left(\frac{\delta_M}{K_A D_A} + \frac{1}{k_P} \right) \text{ or}$$

$$J_A = \frac{c_F - c_P}{\frac{\delta_M}{K_A D_A} + \frac{1}{k_P}} = k_{OL} (c_F - c_P) \text{ with } \frac{1}{k_{OL}} = \frac{1}{\frac{\delta_M}{K_A D_A} + \frac{1}{k_P}}$$

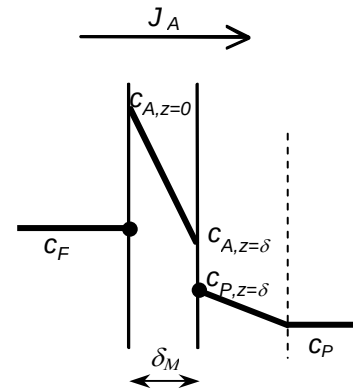
$$J_A = 2.98 \cdot 10^{-6} \cdot (0.030 - 0.005) = 7.45 \cdot 10^{-8} \text{ kmol s}^{-1} \text{ m}^{-2}$$

According to the film theory:

$$J_A = k_P (c_{P,z=\delta} - c_P) = 7.45 \cdot 10^{-8} \text{ kmol m}^{-2} \text{ s}^{-1}$$

Hence, $c_{P,z=\delta} = 8.72 \text{ mol m}^{-3}$ and

$$c_{A,z=\delta} = K \cdot c_{P,z=\delta} = 13.0 \text{ mol m}^{-3}.$$



Exercise 11.4

Given $c_{NaCl} = 0.1 \text{ mol / kg H}_2\text{O}$

$$\rho_w = 997.0 \text{ kg m}^{-3}.$$

Find Osmotic pressure

Thoughts Apply the Van't Hoff relation $\Pi_{osm} \approx cRT$ (Eq. 11.1) with c in mol ions per unit volume of pure solvent. Note that 1 mole NaCl dissociates into 1 mole Na^+ plus 1 mole Cl^- . Assume that the density of this very diluted solution equals that of pure water.

Solution $c = \frac{2 \cdot 0.1}{1/997.0} \cdot 8.31 \cdot 298 = 4.938 \text{ bar} = 4.88 \text{ atm}.$

The experimental value is slightly less: 4.56 atm.

Exercise 11.5

Given flow rate = $1.92 \cdot 10^{-8} \text{ m}^3 \text{ s}^{-1}$

$c_{in} = 10.0 \text{ kg / m}^3 \text{ solution}$

$c_{out} = 0.39 \text{ kg / m}^3 \text{ solution}$

$M_{salt} = 0.0585 \text{ kg mol}^{-1}$

$$\Delta p = 54.4 \text{ atm}$$

$$\rho_{solution \text{ in}} = 1004 \text{ kg/m}^3$$

$$\rho_{solution \text{ out}} = 997 \text{ kg/m}^3$$

$$A = 2.0 \cdot 10^{-3} \text{ m}^2$$

Find The permeances for water and sodium chloride at 25°C.

Thoughts Eq. 11.03b is applicable for water whereas Eq. 11.3c defines the solute flux.

The water flow has to be converted into a water flux [$\text{kg s}^{-1} \text{ m}^{-2}$] and the osmotic pressures at both sides of the membrane must be calculated (Van 't Hoff: $\pi = cRT$). Note that the concentration of salt at the product side is sufficiently low to assume that $\rho_{\text{pure water}} = \rho_{\text{solution out}}$.

From the water flux, the salt flux can be calculated by noting that the product concentration is defined by the ratio of the solute flux and the solvent flux.

Solution Calculation of the water flux

$$1.92 \cdot 10^{-8} \text{ m}^3 \text{ soln} / \text{s} \Rightarrow \frac{1.92 \cdot 10^{-8} \times 997}{2 \cdot 10^{-3}} \frac{\text{m}^3 \text{ solution}}{\text{s}} \cdot \frac{1}{\text{m}^2} \cdot \frac{\text{kg solvent}}{\text{m}^3 \text{ solution}} \approx 9.57 \cdot 10^{-3} \frac{\text{kg water}}{\text{s} \cdot \text{m}^2}$$

Calculation of osmotic pressure

The concentration c to be applied in the Van 't Hoff equation is expressed as mol NaCl per m^3 pure water. 1 m^3 product solution contains $0.39 / 0.0585$ mol NaCl and $(997 - 0.39) /$

997 m^3 pure water. Hence, $c_{\text{product}} = \frac{0.39 / 0.0585}{(997 - 0.39) / 997} = 6.67 \cdot 10^{-3} \text{ mol NaCl} / \text{m}^3 \text{ H}_2\text{O}$ and

the $\pi_{\text{product}} = 2 \cdot 6.67 \cdot 8.31 \cdot 298 = 0.33 \cdot 10^5 \text{ Pa} = 0.33 \text{ atm}$.

Similarly, 1 m^3 feed solution contains $10 / 0.0585$ mol NaCl / m^3 solution and $1004 - 10 =$

994 kg pure H_2O with a volume of $994 / 997 \text{ m}^3$. Hence, $c_{\text{feed}} = \frac{10 / 0.0585}{(1004 - 10) / 997} = 171.5$

$\text{mol NaCl} / \text{m}^3 \text{ H}_2\text{O}$ and $\pi_{\text{feed}} = 2 \cdot 171.5 \cdot 8.31 \cdot 298 = 8.49 \cdot 10^5 \text{ Pa} = 8.38 \text{ atm}$.

It follows for the permeance of water (Eq. 11.3b)

$$9.57 \cdot 10^{-3} / [54.4 - (8.32 - 0.33)] = 0.207 \cdot 10^{-3} \text{ kg s}^{-1} \text{ m}^{-2} \text{ atm}^{-1}.$$

Calculation of the solute flux

$$c_{\text{out}} = \frac{N_{\text{solute}}}{N_{\text{water}} / \rho_{\text{water}}}. \quad N_{\text{solute}} = 0.39 \cdot \frac{9.57 \cdot 10^{-3}}{997} = 3.74 \cdot 10^{-6} \frac{\text{kg NaCl}}{\text{s} \cdot \text{m}^2}$$

The permeance of NaCl becomes (Eq. 11.3c): $3.74 \cdot 10^{-6} / (10 - 0.39) = 0.390 \cdot 10^{-6} \text{ m s}^{-1}$.

Chapter 11: MEMBRANE FILTRATION (continued)

Exercise 11.6

Ultrafiltration is used to remove proteins from whey. For this purpose dead-end experiments have been performed using a flat plat UF membrane with an area of 25x25 cm². To determine the clean membrane resistance some clean water ($\eta = 1.10^{-3}$ Pa.s) filtration experiments have been conducted where the pressure is measured for various flowrates. The following data were obtained:

Flowrate (l/h)	3.0	3.5	4.5	5.0
TMP (bar)	0.62	0.75	0.88	1.00

- a. Determine the clean membrane resistance R_M .

A subsequent experiment in which whey is filtered for 20 mins at a constant flux van 50 l/h/m², resulting in the formulation of the following fouling model:

$$\frac{dR}{dt} = kJ, R(0) = R_M$$

where R (1/m), $k = 41.5$ (m²/m³), J (m/s) en t (s).

- b. Calculate the energy consumption of the pump that has been used during this experiment. Pump efficiency $\eta_P = 0.7$
- c. Determine the pump power required for pilot plant scale filtration test that contains a membrane area of 40 m² and operates at an average trans membrane pressure of 0.5 bar. Pump efficiency $\eta_P = 0.7$

Exercise 11.7

For an UF membrane clean water experiments have yielded the following results:

Flux (l/h/m ²)	30	40	50	60	70
TMP (Pa)	4100	5500	7000	8000	9700

- a. Calculate the clean membrane resistance.

In a second experiment river water is filtered over the membrane at constant flux (=60 l/h/m²) during 15 min. (=900 sec), providing the following data:

Filtration time (sec)	60	180	300	420	540	660	780	900
Total resistance (m ⁻¹) x10 ¹¹	5.30	5.98	6.75	7.61	8.58	9.67	10.9	12.3

The filtration behaviour can be described by an exponential model or a quadratic model (for constant flux filtration):

$$R_t = R_M \exp(kJt)$$

$$R_t = R_M + kJt^2$$

- b. Which of the two models would be preferable to use, explain?
 c. Calculate the power consumption of the pump for constant flux filtration when $\eta = 0.001$ Pa.s (viscosity (river)water), $A = 0.05$ m² (Membrane area), $\eta_P = 0.7$ (-) (pump efficiency)

Exercise 11.6

Given $\eta = 10^{-3}$ Pa.s $\eta_P = 0.7$

Find a) Clean membrane resistance R_M
 b) Energy consumption of the pump
 c) Pump power required for pilot plant scale filtration test

Solution a) Transform flow into flux (m³/m²/s) and pressure in Pa
 Plot Flux against pressure.
 Darcy's equation reads:

$$J = \frac{\Delta P}{\eta R}$$

Slope depends on $(\eta R_M)^{-1}$

$$\eta = 1.0 \text{ Pa.s} \rightarrow R_M = 4.55 \times 10^{12} \text{ (1/m)}$$

b) Equations needed:

Pump equation for power: $P = \frac{1}{\eta_P} \Delta P Q$

Darcy's equation: $J = \frac{\Delta P}{\eta R}$ Note that: $J = \frac{Q}{A}$

Fouling model: $\frac{dR}{dt} = kJ$, $R(0) = R_M \Rightarrow R(t) = R_M \exp(kJt)$

Integration over time transforms power to energy consumption:

$$E = \int_0^t P dt$$

Combining the pump equation with Darcy's equation:

$$P = \frac{\eta}{A \eta_P} R J^2$$

Combining this with the fouling model:

$$P = \frac{\eta}{A\eta_p} R_M \exp(kJt) J^2$$

Integration over time:

$$E = \frac{\eta J^2}{A\eta_p} R_M \int_0^t \exp(kJt) dt \Rightarrow E = \frac{\eta J}{kA\eta_p} R_M \exp(kJt)$$

$$E = \frac{\eta J}{kA\eta_p} R_M \exp(kJt) = \frac{1.0 * 1.4 * 10^{-5}}{41.5 * (0.25 * 0.25) * 0.7} * 4.55 * 10^9 * \exp(41.5 * 1.4 * 10^{-5} * 1200) \sim 7000 Nm$$

c) Use the pump equation:

$$P = \frac{1}{\eta_p} \Delta P Q = \frac{1}{0.7} * 0.5 * 10^5 * (1.4 * 10^{-5} * 40) = 80 \frac{Nm}{s}$$

Exercise 11.7

Given $\eta = 10^{-3} \text{ Pa.s}$ $A = 0.05 \text{ m}^2$ $\eta_p = 0.7 \text{ (-)}$

Find a) Clean membrane resistance R_M
 b) Preferred filtration model
 c) Power consumption of the pump for constant flux filtration

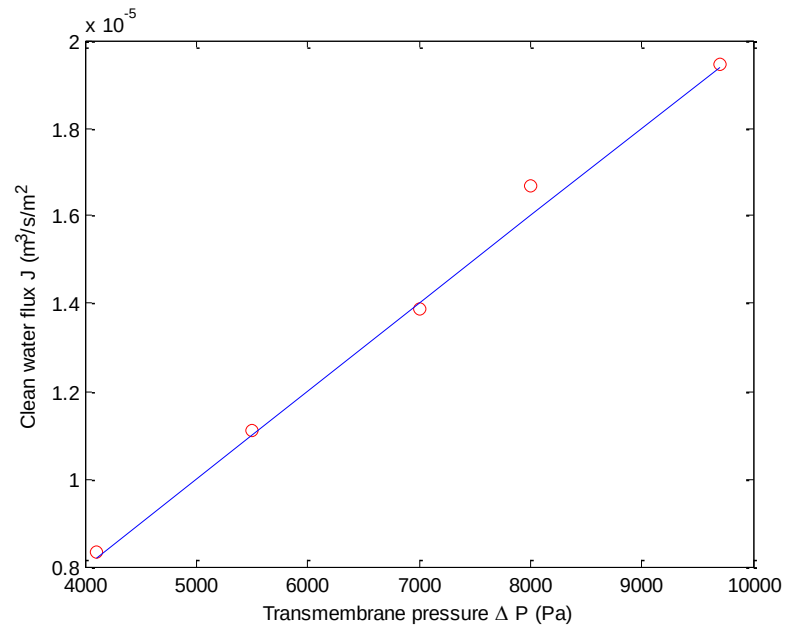
Solution a) Determine clean water flux:

Clean water flux (l/h/m ²)	Transmembraandruk (Pa)
30	4100
40	5500
50	7000
60	8000
70	9700

Use Darcy's equation: $J = \frac{\Delta P}{\eta R_t}$

Filtration with clean water means: $R_M = R_t$

Plot J against ΔP and determine slope: $\frac{1}{\eta R_M}$



$$\frac{1}{\eta R_M} = 2.10^{-9} \Rightarrow R_M = 5.10^{11} (1/m)$$

- b) Determine fouling resistance as function of filtration time for constant flux ($J = 60 \text{ l/h/m}^2$),

Flux (l/h/m ²)	Resistance (1/m)
60	5.30918E+11
180	5.98609E+11
300	6.74929E+11
420	7.60981E+11
540	8.58003E+11
660	9.67396E+11
780	1.09074E+12
900	1.2298E+12

For the two possible fouling models:

$$R_t = R_M \exp(kJt)$$

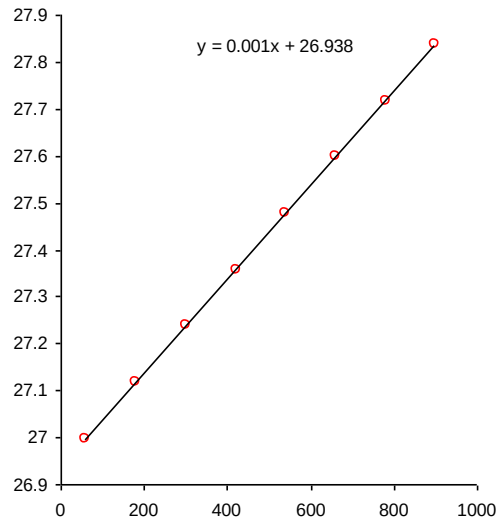
$$R_t = kJt^2 + R_M$$

determine k from the experimental data.

Exponential model:

$$\ln R_t = kJt + \ln R_M, \text{ plot } \ln R_t \text{ tegen } t$$

Slope equals kJ and the intercept $\ln R_M$



From intercept: $\ln R_M = 26.938 \Rightarrow R_M = 5.10^{11} (1/m)$

From slope:

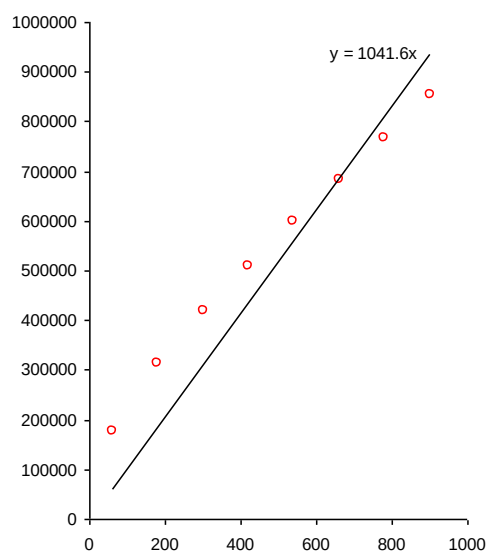
$$kJ = 0.001 \Rightarrow k = 0.001 / 1.67 \cdot 10^{-5} = 59.88 (m^3 / m^2)$$

$$J = 60 \text{ l/h/m}^2 = 1.67 \times 10^{-5} \text{ m/s}$$

Quadratic model:

$$\sqrt{R_t - R_M} = \sqrt{kJt}, \text{ plot } \sqrt{R_t - R_M} \text{ tegen } t$$

Slope equals $\sqrt{kJ}$



From slope:

$$\sqrt{kJ} = 1041.6 \Rightarrow k = 1041.6^2 / 1.67 \cdot 10^{-5} = 6.5 \cdot 10^{10} (m^3 / m^2 s)$$

Exponential model represents data better than quadratic model.

- c) Combination of the pump equation, Darcy's equation and flux equation yields:

$$\left. \begin{aligned} P &= \frac{1}{\eta_p} \Delta P Q \\ Q &= JA \\ J &= \frac{\Delta P}{\eta R_t} \end{aligned} \right\} P = \frac{A\eta}{\eta_p} R_t J^2$$

Power consumption can be determined by introducing the fouling model and integration over time:

$$\left. \begin{aligned} P &= \frac{1}{\eta_p} \frac{A}{\eta} R_t J^2 \\ R_t &= R_M \exp(kJt) \\ E &= \int_0^{t_F} P dt \end{aligned} \right\} E = \frac{1}{\eta_p} \frac{A}{\eta} R_M \int_0^{t_F} \exp(kJt) J^2 dt$$

$$E = \frac{1}{\eta_p} \frac{A}{\eta} R_M \int_0^{t_F} \exp(kJt) J^2 dt = \frac{1}{\eta_p} \frac{A}{\eta} R_M \frac{J}{k} \exp(kJt) \Big|_0^{t_F} = \frac{1}{\eta_p} \frac{A}{\eta} R_M \left(\frac{J}{k} \exp(kJt_F) - 1 \right)$$

$$\frac{1}{\eta_p} \frac{A}{\eta} R_M \left(\frac{J}{k} \exp(kJt_F) - \frac{J}{k} \exp(kJ0) \right) = 2.9 \cdot 10^{-11} \text{ Nm}$$

Chapter 12: SEPARATION SELECTION METHODS

Exercise 12.1

The vent gas from several polymer plants contains air, paraffins and olefins (monomers). The composition is given by (1 bar, 25°C):

Air	91	mol%			
Butane	1,5	mol %	1-Butene	3	mol %
Pentane	1,0	mol %	1-Pentene	2	mol %
Hexane	0,5	mol %	1-Hexene	1	mol %

Within the R&D department of the company it is explored which techniques may be technically and economically feasible to recover the three olefins (1-butene, 1-pentene en 1-hexene) in pure state. The company has decided to approach you as a consultant and prepared several questions for the first meeting. As a first step they are considering the purification of the air by simultaneous recovery of the olefins and the paraffins.

- a. What separation methods are, in your opinion, applicable for this simultaneous olefin and paraffin recovery from the vent gas?

After recovery the olefin/paraffin needs to be separated in two fractions, the paraffins (butane, pentane, hexane) and the olefins. In principle, this is possible by selective absorption of the olefins.

- b. Can you determine which two key components need to be separated (selectivity>1) in order to achieve a full olefin/paraffin separation?
- c. What is the minimal required difference in affinity (activity coefficients) of the absorption liquid towards both key components to achieve this full olefin/paraffin separation. In other words achieve a selectivity = $(x_1/y_1) / (x_2/y_2) > 1$?

A possible alternative for absorption is extraction with a solvent that has a higher affinity for olefins compared to paraffins.

- d. What are the two key components in order to obtain a full olefin/paraffin separation when extraction with a highly polar solvent is applied?
- e. Which of the solvents below are in principle suitable (selectivity>1) for a complete separation of the olefin and paraffin fractions?

	C_4	$C_4^=$	C_5	$C_5^=$	C_6	$C_6^=$
Vapor pressure(25°C, bar)	2,4	3,0	0,68	0,85	0,20	0,24
<i>Distribution Coefficient</i> (concentration in solvent/concentration in feed)						
NMP	0,10	0,12	0,08	0,08	0,06	0,05
DMSO	0,05	0,15	0,04	0,12	0,03	0,10
DEG	0,02	0,25	0,01	0,20	0,01	0,15

Exercise 12.2

In a large chemical plant methylcyclohexane is separated from toluene by distillation. For the construction of a new plant it is considered whether the use of extractive distillation or extraction could provide economically more attractive options. A detailed evaluation requires firstly the characterization of the current separation between methylcyclohexane and toluene.

- Calculate from the given data the relative volatility for the separation of methylcyclohexane and toluene by distillation. Consider the mixture to behave (almost) ideal.
- Which of the given solvents might be feasible to enhance the relative volatility sufficiently to make extractive distillation economically attractive?
- The same question for extraction. Assume that the solvents and the methylcyclohexane/toluene mixture are (almost) immiscible.

Besides methylcyclohexane (45%) and toluene (45%) the feed also contains 5% of cyclohexane as well as benzene.

- What would be the optimal arrangement of separation steps for distillation, for extractive distillation and for extraction to separate this mixture into its four pure constituents? What distillative separation might become problematic and why?
- Which of the arrangements seems, considering the number of operations (columns), the most economical solution.
- Why is the counting of the number of operations sufficient to get a first impression about the most economical route? What effects are not taken into account?

	<i>Benzene</i>	<i>Cyclo- hexane</i>	<i>Methyl- cyclohexane</i>	<i>Toluene</i>
<i>Vapor pressure</i> (80°C, bar)	1,01	0,99	0,54	0,39
<i>Activity coefficient at infinite dilution</i>				
NMP	1,5	7	8	2
DMSO	4	25	30	4
DEG	7	41	60	10
NMF	1,5	10	10	2,5

SOLUTIONS**Exercise 12.1**

Given The composition of the vent gas at 1 bar and 25°C:

Air	91	mol%			
Butane	1,5	mol%	1-Butene	3	mol%
Pentane	1,0	mol%	1-Pentene	2	mol%
Hexane	0,5	mol%	1-Hexene	1	mol%

The vapor pressures of the pure components are given as:

	C_4	$C_4^=$	C_5	$C_5^=$	C_6	$C_6^=$
Vapor pressure (25°C, bar)	2,4	3,0	0,68	0,85	0,20	0,24

The distribution coefficients (concentration in solvent/concentration in feed) of the components in three solvents:

	C_4	$C_4^=$	C_5	$C_5^=$	C_6	$C_6^=$
NMP	0,10	0,12	0,08	0,08	0,06	0,05
DMSO	0,05	0,15	0,04	0,12	0,03	0,10
DEG	0,02	0,25	0,01	0,20	0,01	0,15

- Find*
- what separation methods are in your opinion applicable for the simultaneous olefin and paraffin recovery from the vent gas.
 - can you determine which two key components need to be separated (selectivity > 1) in order to achieve a full olefin/paraffin separation.
 - what is the minimal required difference in affinity (activity coefficients) of the adsorbing liquid towards both key components to achieve this full olefin/paraffin separation.
 - what are in your eyes the two key components in order to obtain a full olefin/paraffin separation when extraction with a highly polar solvent is applied.
 - which of the given solvents are in principle suitable (selectivity > 1) for a complete separation of the olefin and paraffin fractions.

Thoughts In this problem, absorptive and extractive separations are compared on basis of the expected selectivities as discussed in chapters 3 (absorption) and 5 (extraction).

Solution

- A sharp separation of the olefin/paraffin fraction from the vent gas is required. The separation methods that can be used to obtain a sharp separation are physical absorption, adsorption, cryogenic distillation and chemical adsorption (see section 12.4). At high flows of the vent gas, physical absorption and cryogenic distillation seem to be the most applicable separation methods.
- The olefin and paraffin fractions have to be separated between the key components which are the most difficult separable components of both fractions. During the absorption of the olefin fraction, the less soluble component will be 1-butene. The best soluble component of the paraffin fraction will be hexane, which is undesired as a product in the extracted olefin mixture.

(c) The selectivity of the absorption of 1-butene compared to hexane is given as:

$$\text{Selectivity} = \frac{\left(\frac{x_{1\text{-butene}}}{y_{1\text{-butene}}} \right)}{\left(\frac{x_{\text{hexane}}}{y_{\text{hexane}}} \right)} = \alpha'_{1\text{-butene,hexane}} = \frac{P_{1\text{-butene}}/P_{\text{hexane}}}{x_{1\text{-butene}}/x_{\text{hexane}}}$$

with Raoult's law (equation 3.2):
 $P_{1\text{-butene}} = \gamma_{1\text{-butene}}^{\infty} \cdot P_{1\text{-butene}}^0 \cdot x_{1\text{-butene}}$
 $P_{\text{hexane}} = \gamma_{\text{hexane}}^{\infty} \cdot P_{\text{hexane}}^0 \cdot x_{\text{hexane}}$

Substitution gives:

$$\text{Selectivity} = \frac{\gamma_{1\text{-butene}}^{\infty} \cdot P_{1\text{-butene}}^0}{\gamma_{\text{hexane}}^{\infty} \cdot P_{\text{hexane}}^0} > 1 \Rightarrow \frac{\gamma_{\text{hexane}}^{\infty}}{\gamma_{1\text{-butene}}^{\infty}} > \frac{P_{1\text{-butene}}^0}{P_{\text{hexane}}^0} = \frac{3.00}{0.20} = 15.0$$

Thus, the minimal required difference in affinity of the absorbing liquid towards the key components must be of a factor 15: $\gamma_{\text{hexane}}^{\infty} > 15 \cdot \gamma_{1\text{-butene}}^{\infty}$.

- (e) During the extraction of the olefin fraction, the less polar (due to the larger molecule) – and thus less soluble – component will be 1-hexene. The most polar – and thus best soluble – component of the paraffin fraction will be butane.
- (f) The selectivity of 1-hexene compared to butane during the extraction with a solvent that has a higher selectivity for olefins can be calculated by using Eq. 5.3:

$$\text{Selectivity} = \beta_{1\text{-hexene,hexane}} = \frac{K_{1\text{-butene}}}{K_{\text{hexane}}}$$

Suitable solvents are characterized by a selectivity above 1. For the three given solvents, these selectivities were calculated and summarized in the table below.

Solvent	$\beta_{1\text{-butene,hexane}}$	Remarks
NMP	$\frac{K_{1\text{-hexene}}}{K_{\text{butane}}} = \frac{0.05}{0.10} = 0.005$	not suitable
DMSO	$\frac{K_{1\text{-hexene}}}{K_{\text{butane}}} = \frac{0.10}{0.05} = 2.00$	suitable
DEG	$\frac{K_{1\text{-hexene}}}{K_{\text{butane}}} = \frac{0.15}{0.02} = 7.50$	best suitable of the three given solvents

Exercise 12.2

Given The vapor pressures of the pure components are given as:

	<i>Benzene</i>	<i>Cyclo-hexane</i>	<i>Methyl-cyclohexane</i>	<i>Toluene</i>
Vapor pressures (80°C, bar)	1,01	0,99	0,54	0,39

Activity coefficients at infinite dilution:

	<i>Benzene</i>	<i>Cyclo-hexane</i>	<i>Methyl-cyclohexane</i>	<i>Toluene</i>
MP	1,5	7	8	2
DMSO	4	25	30	4
DEG	7	41	60	10
NMF	1,5	10	10	2,5

- Find
- calculate the relative volatility for the separation of methylcyclohexane (MCH) and toluene (T) by distillation.
 - which of the given solvents might be feasible to enhance the relative volatility to make extractive distillation economically attractive.
 - which of the given solvents might be feasible to make extraction economically attractive.
 - which three arrangements of separation steps would be optimal when distillation, extractive distillation or extraction is chosen as the first step to separate this mixture into its four pure components. What distillative separation might become problematic and why?
 - which of the arrangements seems, considering the number of operations (columns), the most economical solution.
 - why is counting of the number of operations sufficient to get a first impression about the most economical route? What effects are not taken into account?

Thoughts Distillation (chapter 2), extractive distillation (chapter 2 and 3) and extraction (chapter 5) are being discussed in this problem on basis of the enhancement of the selectivities when different solvents are being used.

Solution (a) The relative volatility for ideal mixtures is given by Eq. 1.5:

$$\alpha_{M,T} = \frac{P_M^0}{P_T^0} = \frac{0.54}{0.39} = 1.38$$

(b) The relative volatility for non-ideal mixtures is given by Eq. 1.6:

$$\alpha'_{M,T} = \frac{\gamma_M}{\gamma_T} \cdot \frac{P_M^0}{P_T^0} \text{ with the enhancement factor } = \frac{\gamma_M}{\gamma_T}$$

The relative volatility will be enhanced with increasing enhancement factor. The calculated enhancement factors and separation factors ($\alpha'_{M,T}$) are summarized in the following table:

<i>Solvent</i>	<i>Enhancement factor</i>	$\alpha'_{M,T}$
<i>NMP</i>	$\frac{\gamma_M}{\gamma_T} = \frac{8}{2} = 4$	5.52
<i>DMSO</i>	$\frac{\gamma_M}{\gamma_T} = \frac{30}{4} = 7.5$	10.4
<i>DEG</i>	$\frac{\gamma_M}{\gamma_T} = \frac{60}{10} = 6$	8.28
<i>NMF</i>	$\frac{\gamma_M}{\gamma_T} = \frac{10}{2.5} = 4$	5.52

DMSO will be the most economically attractive solvent of these four, because the economical enhancement factor for DMSO is the largest. However, all solvents obey the requirement (see figure 12.4) that $\alpha'_{M,T} > 2$ for $\alpha_{M,T} = 1.38$.

- (c) The selectivity of the extraction process is given by Eq. 5.5:

$$\beta_{M,T} = \frac{\left(\frac{\gamma_M}{\gamma_T}\right)_{EXTRACT}}{\left(\frac{\gamma_M}{\gamma_T}\right)_{RAFFINATE}} \text{ with the enhancement factor} = \left(\frac{\gamma_M}{\gamma_T}\right)_{EXTRACT}$$

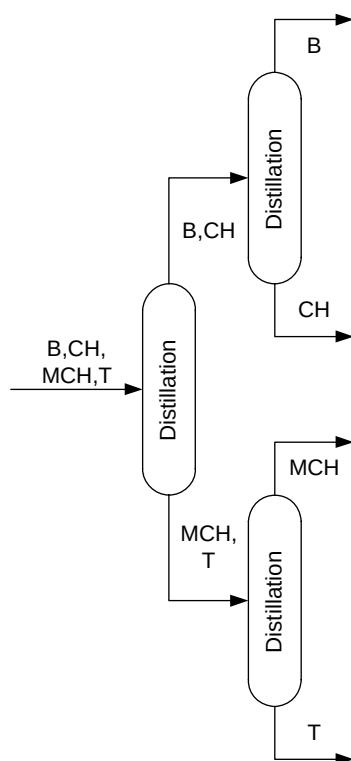
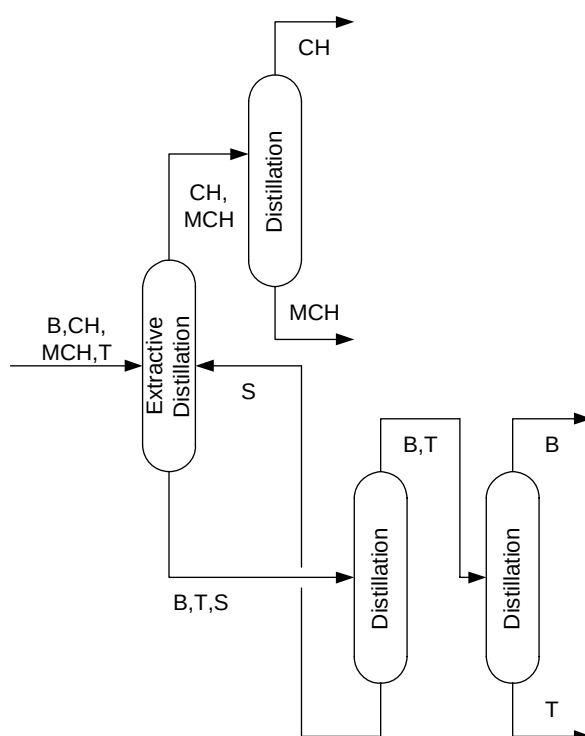
Assuming that $\left(\frac{\gamma_M}{\gamma_T}\right)_{RAFFINATE}$ is constant during the extraction, it may be stated

that the enhancement factor equals the factors as summarized in the table above. DMSO and DEG are economically attractive solvents, because their enhancement factors are > 5 (see Fig. 12.4 in *Fund. Ind. Sepns.*).

- (d) The optimal arrangements are shown in Figs. 12.1 to 12.3 on the next two pages. The distillative separation of benzene (B) from cyclohexane (CH) might become problematic due to the insufficient difference in boiling point of both components. The relative volatilities are almost equal:

$$\alpha_{B,C} = \frac{P_B^o}{P_C^o} = \frac{1.01}{0.99} = 1.02$$

- (e) The distillative separation, consisting of three columns seems the most economical solution when compared to extractive distillation and extraction.
- (f) Neglected are, for instance effects like the difficulties of the separations (column dimensions), energy requirements and product stability.

**Figure 12.1.** Distillation**Figure 12.2.** Extractive distillation

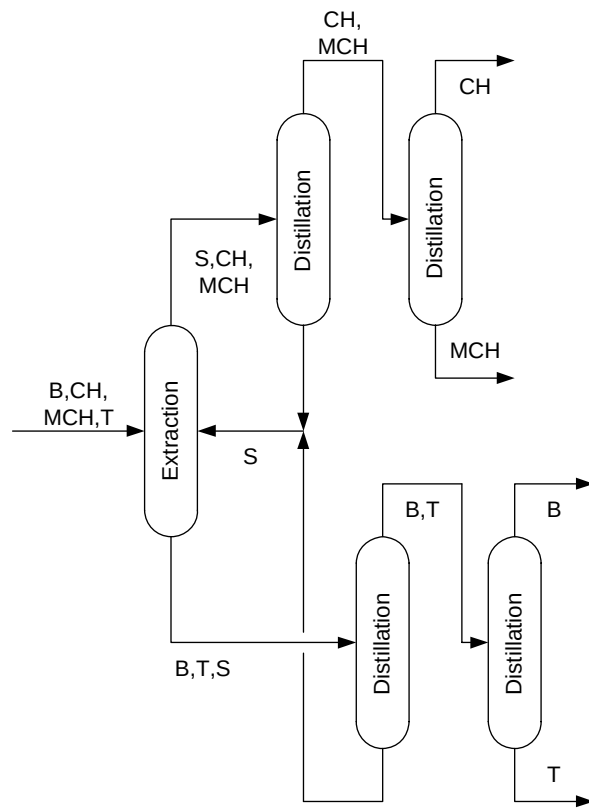


Figure 12.3. Extraction