

# **FLUID SEPARATIONS**

## **THERMISCHE VERFAHRENSTECHNIK II**

### **TUTORIALS**

#### **Summer Term 2023**

**Department of Biochemical and Chemical Engineering**  
**Laboratory of Fluid Separations**

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**Table of content**

<b>1</b>	<b>Membrane Separations .....</b>	<b>3</b>
1.1	Pervaporation	3
1.2	Reverse osmosis	6
<b>2</b>	<b>Crystallisation .....</b>	<b>7</b>
2.1	Crystal growth	7
2.2	Crystallization in the T-x-diagram	8
2.3	Freeze crystallization	9
<b>3</b>	<b>Vaporisation, condensation &amp; evaporation .....</b>	<b>10</b>
3.1	Single-stage evaporator (1)	10
3.2	Single-stage evaporator (2)	11
3.3	Single-stage co-current evaporator	12
3.4	Three-stage evaporation process	15
<b>4</b>	<b>Multistage distillation .....</b>	<b>18</b>
4.1	Multistage distillation using McCabe-Thiele method	18
4.2	Multistage distillation using h,w-diagram	20
4.3	Distillation column with side stream withdrawal	22
4.4	Distillation column with two feed streams & one side stream withdrawal	24
4.5	Discontinuous multistage distillation (1)	26
4.6	Discontinuous multistage distillation (2)	28
4.7	Distillation lines (1)	30
4.8	Distillation lines (2)	32
4.9	Distillation lines (3)	35
4.10	Separation of azeotropic mixtures	37
<b>5</b>	<b>Absorption .....</b>	<b>38</b>
5.1	Absorbent recirculation	38
5.2	Non-isothermal absorption	40
5.3	HTU-NTU concept	42

# 1 Membrane Separations

## 1.1 Pervaporation

An alcohol A is to be dewatered by pervaporation with a hydrophilic polymer membrane. Results for permeate fluxes and concentrations from investigations in a lab-scale pervaporation plant are shown in Figure 1-2 and Figure 1-3, depending on temperature and water content of the feed.

- 1.1.1 Draw the schematic profile of a membrane. Deduce the equation for description of the driving force for a pervaporation process.

The permeance  $Q_i$  is not constant, but changes in dependence of the mixture composition and temperature. In tasks 3.1.2 – 3.1.4 you shall develop step by step an equation to calculate the permeance  $Q_i$  of the membrane as a function of the mass fraction of water and the temperature.

- 1.1.2 Calculate the permeances  $Q_i$  [kmol/(m<sup>2</sup>·h·bar)] for alcohol and water for a feed mass fraction of water of 15 wt.-% based on the experimental results and the different temperatures.

- 1.1.3 Determine the dependency of the permeance  $Q_i$  on the mass fraction of water according to the following equation:

$$Q_i = Q_{i,0} \cdot w_0$$

- 1.1.4 Specify the influence of pervaporation temperature on the permeance using the Arrhenius term:

$$Q_{i,0} = Q_{i,T_0} \cdot \exp \left[ \frac{E_A}{R} \left( \frac{1}{T_0} - \frac{1}{T} \right) \right] \text{ with } E_A = 66330 \text{ kJ/kmol and } T_0 = 328.15 \text{ K}$$

Using the developed equation for calculation of  $Q_i$  in dependence of the mixture composition and temperature, you shall now design the pervaporation process. At the outlet of the pervaporation unit the water content shall be 3 wt.-%.

- 1.1.5 Calculate the membrane area needed for dewatering of the alcohol, considering the enthalpy of vaporization.
- 1.1.6 Draw the profile of temperature and concentration along a membrane module.
- 1.1.7 How does an increase of permeate pressure by factor 2 influence the permeate flux?

### Further information:

- Feed:
  - Mass flow:  $\dot{m}_0 = 1000 \text{ kg/h}$
  - Mass fraction of water in alcohol:  $w_{w,0} = 6 \text{ wt.-%}$
  - Temperature:  $T_0 = 85 \text{ °C}$

- Permeate pressure:  $P_P = 20 \text{ mbar}$
- Saturated vapour pressure:
 
$$\log_{10}(p_{\text{Sat,H}_2\text{O}}) = 5,08 - \frac{1659,8}{T[\text{K}] - 45,9} \quad \text{mit } p_{\text{sat,H}_2\text{O}} [=] \text{ bar}$$
- Vaporization enthalpy of water:  $\Delta h_v = 2310 \text{ kJ/kg}$
- Heat capacities for liquid streams:
  - $C_{P,F} = 2.6 \text{ kJ/(kg K)}$
  - $C_{P,R} = 2.4 \text{ kJ/(kg K)}$
  - $C_{P,P} = 4.2 \text{ kJ/(kg K)}$
- Assumption: Temperatures of retentate and permeate are equal:  
 $T_R \sim T_P$
- Molar masses:
  - $M_A = 65 \text{ kg/kmol}$
  - $M_W = 18 \text{ kg/kmol}$

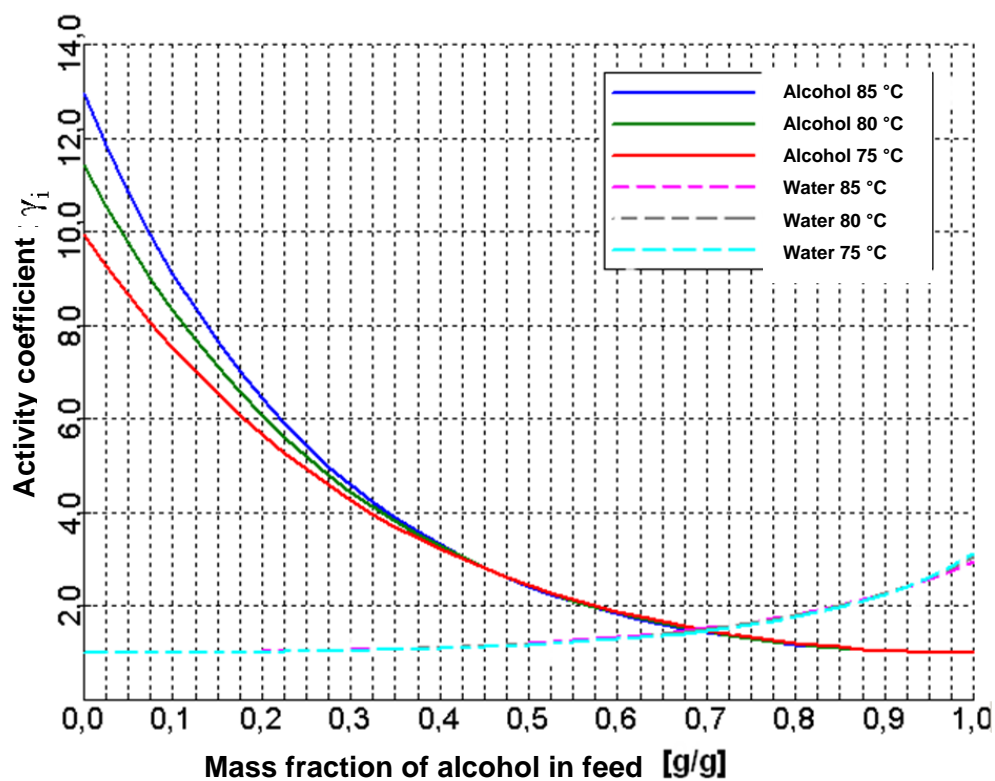


Figure 1-1: Dependency of activity coefficients on mass fractions of alcohol in binary mixtures of alcohol and water

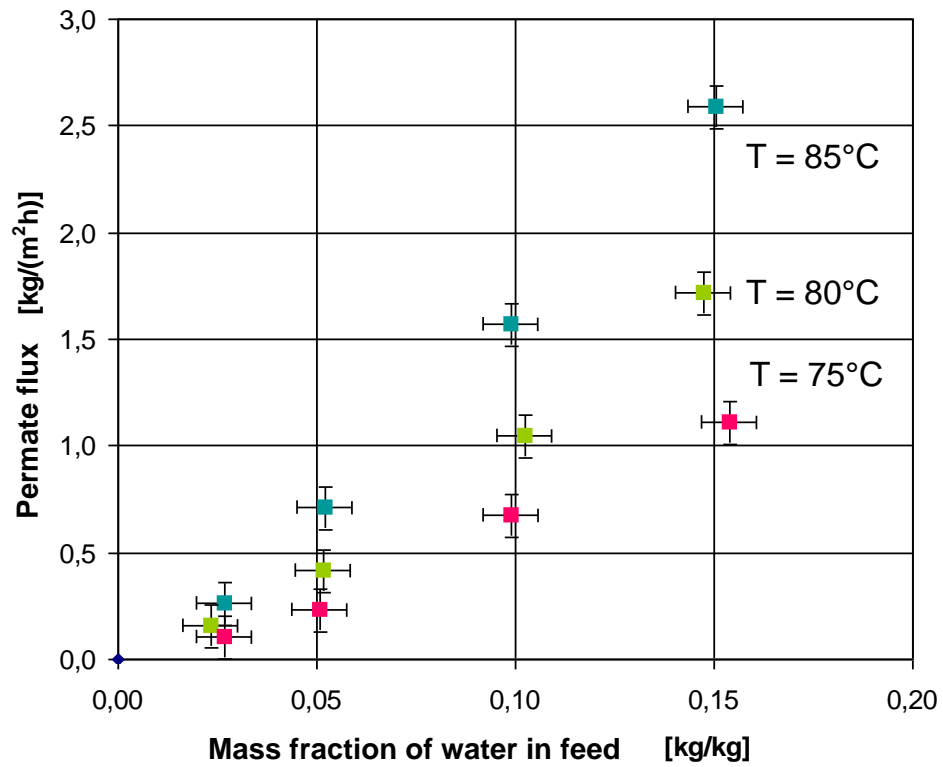


Figure 1-2: Permeate flux, dependent on temperature and mass fraction of water in feed

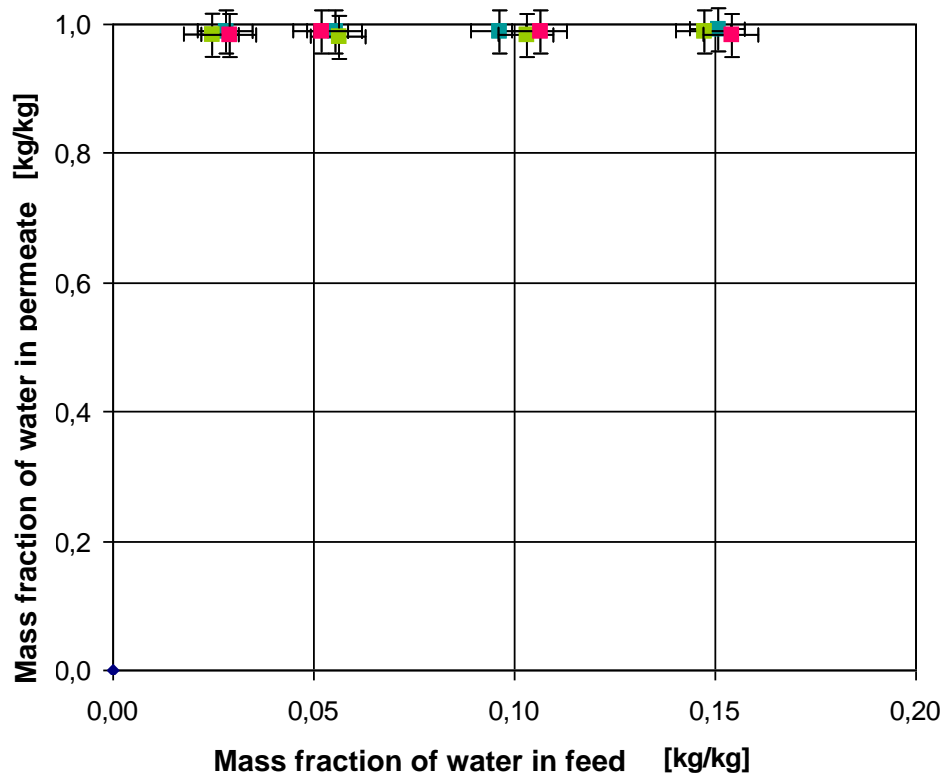


Figure 1-3: Mass fraction of water in permeate, dependent on temperature and mass fraction of water in feed

## 1.2 Reverse osmosis

On the island of Gran Canaria, the tourism industry and the dry climate are the reasons for frequently water shortage. A reverse osmosis plant has to be built up to produce drinking water from sea water. Turbines at the outlet of the membrane unit are used for energy recovery.

- 1.2.1 Calculate the retention of the membrane and the concentration of salt in the retentate.
- 1.2.2 Determine the pressure difference needed for reverse osmosis. The transmembrane pressure difference is minimum 1.1 times the minimal required one:  $\Delta P = 1.1 \cdot \Delta \pi$ .
- 1.2.3 Determine the membrane area  $A_M$  needed for this separation. How many spiral wound modules are needed considering fouling of the membrane?
- 1.2.4 Determine the specific energy consumption of the plant per  $\text{m}^3$  of produced drinking water.

### Further information:

#### Process streams:

- Plant capacity:  $\dot{m} = 36.000 \text{ t/d}$
- Salt concentration of sea water:  $c_{S,F} = 40.000 \text{ mg/l}$
- Max. permissible salt concentration in drinking water:  $c_{S,P} = 400 \text{ mg/l}$
- Temperature:  $T_{RO} = 20 \text{ }^\circ\text{C}$
- Simplification: Assume all salts can be seen as binary electrolytes with an average molar mass of  $M_{\text{Salt}} = 62.9 \text{ g/mol}$ . The influence of the salt on the density of water can be neglected.
- The mean salt concentration on the feed side can be determined by using the logarithmic mean.

#### Plant:

- Yield:  $Y = 45 \text{ } \%$
- Pressure loss along the plant (linear):  $\Delta p_{\text{friction}} = 5 \text{ bar}$

#### Membrane:

- Membrane area of one spiral wound module:  $A_w = 27.3 \text{ m}^2$
- Consideration of fouling:  $A_{M,\text{ideal}}/A_{M,\text{real}} = 0.8115$
- Membrane permeability:  $Q_{\text{H}_2\text{O}} = 0.2292 \text{ kmol}/(\text{bar} \cdot \text{m}^2 \cdot \text{h})$
- Pump efficiency:  $\eta_{\text{Pump}} = 0.8$
- Turbine efficiency:  $\eta_{\text{Turbine}} = 0.9$

## 2 Crystallisation

### 2.1 Crystal growth

For a stirred crystallization, dissolution and growth rates of potash alum-crystals ( $\text{KAl}(\text{SO}_4)_2 \cdot 12 \text{H}_2\text{O}$ ) have been determined experimentally for a tight fraction at a temperature of  $\vartheta = 25^\circ\text{C}$ . Figure 2-1 shows dissolution and growth rates depending on concentration-difference  $\Delta C = C - C_r$ .

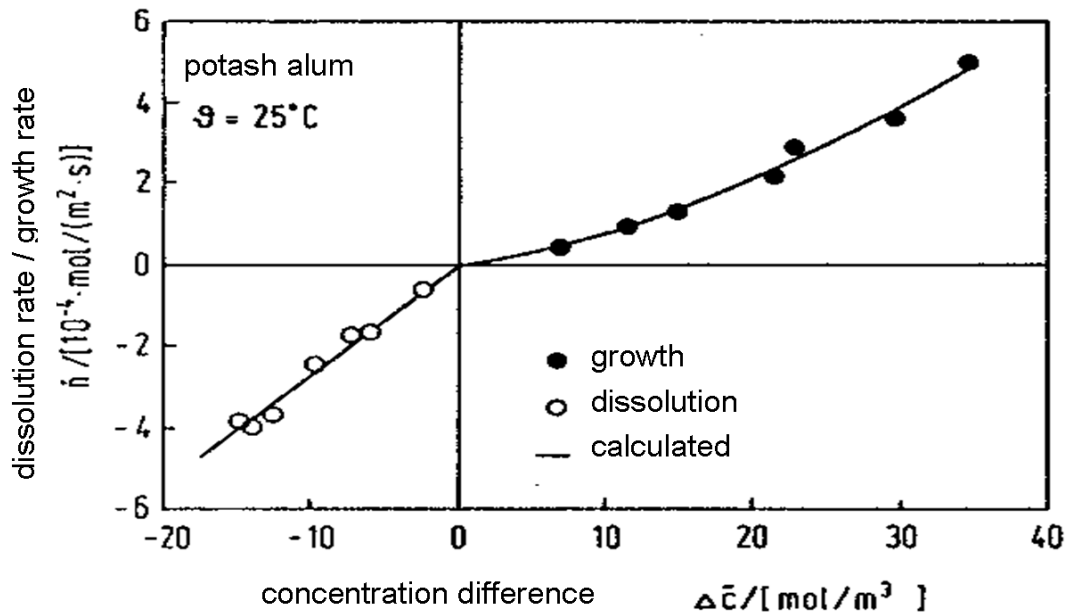


Figure 2-1: Dissolution and growth rates of potash alum-crystals

2.1.1 Calculate the overall mass transfer coefficient  $\beta_1$ .

2.1.2 Calculate the reaction constant  $k_r$ .

Remarks:

- The dissolution process is diffusion limited.
- Incorporation of molecules into the crystal lattice according to reaction of 2<sup>nd</sup> order.
- Use the information of Figure 2-1 for solution.

## 2.2 Crystallization in the T-x-diagram

In Figure 2-2 the phase diagram for the binary system H<sub>2</sub>O-salt A (abscissa ends with solvate (A · 2H<sub>2</sub>O)) is shown. From a feed solution ( $m_0 = 50$  kg) half of the initial mass of pure A has to be separated by crystallization ( $m_S^A = 0.5 \cdot m_0^A$ ).

2.2.1 Determine the mass fractions  $w_S^A$  for the solvate  $m_S$  and  $w_0^A$  for the starting mixture  $m_0$ .

2.2.2 Calculate the temperature required for this crystallization and the mass (kg) of the remaining solvent  $m_L$ .

Molar mass: Salt = 90 g/mol  
Water = 18 g/mol

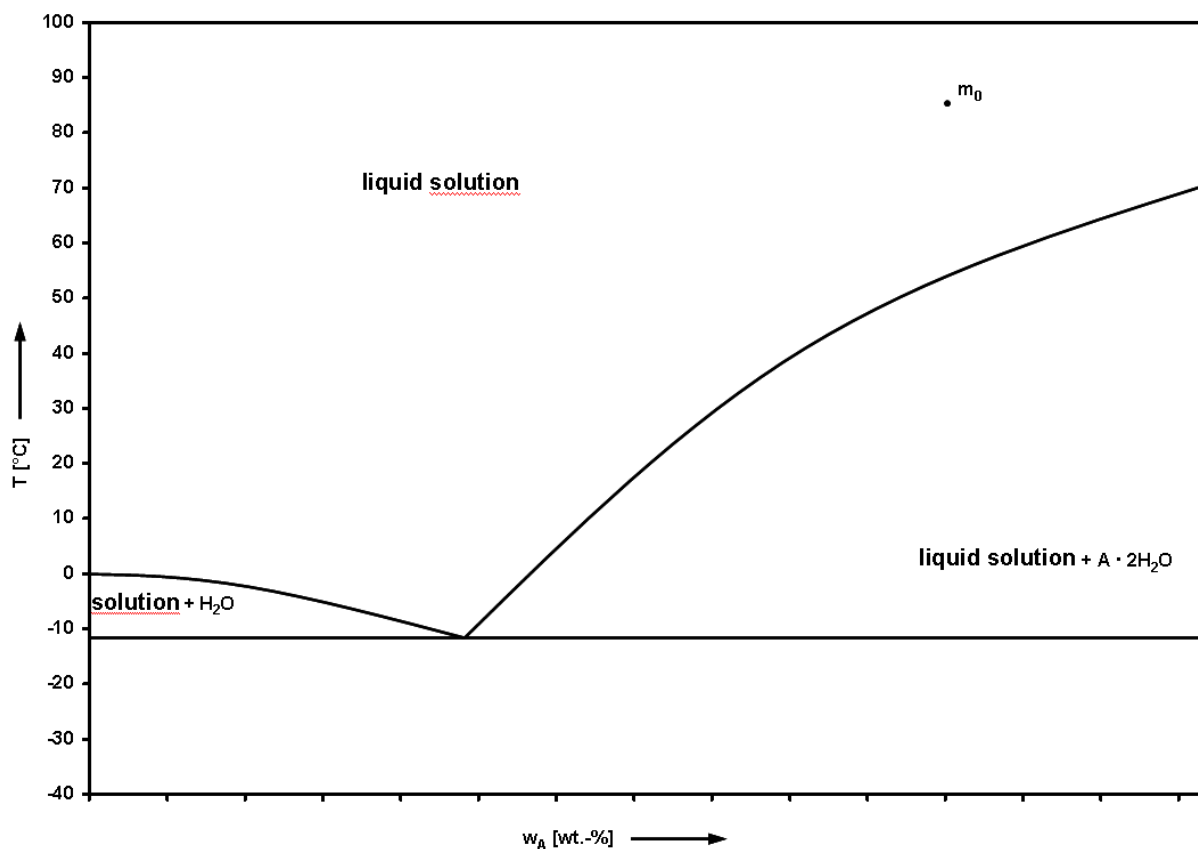


Figure 2-2: Phase diagram for the binary system H<sub>2</sub>O-Salt A.



## 2.3 Freeze crystallization

A cooling crystallizer is used to concentrate an aqueous solution of sodium chloride  $m_{L,0}$ . The solution ( $5^{\circ}\text{C}$ ) with a mass-fraction of  $w_0 = 0.029 \text{ kg NaCl/kg solution}$  shall be vaporized by flash evaporation at  $-10^{\circ}\text{C}$ . The solids loading at the outlet of the evaporator is  $\frac{\dot{m}_{S,1}}{\dot{m}_{L,1}} = 0,1 \text{ kg ice/kg solution}$ .

2.3.1 Draw a flow diagram and name all streams.

2.3.2 Calculate the vapour-stream  $\dot{m}_{V,1}$  and the exiting stream  $\dot{m}_{L,1}$ , in relation to the feed flow that has to be discharged of the freezer apparatus.

2.3.3 Determine the concentration  $w_1$  of the exiting liquid stream.

Additional information:

$$h_0 = 25 \text{ kJ/kg}$$

$$h_V = 2479 \text{ kJ/kg}$$

$$h_{S,1} = -335 \text{ kJ/kg}$$

$$h_{L,1} = -30 \text{ kJ/kg}$$

### 3 Vaporisation, condensation & evaporation

#### 3.1 Single-stage evaporator (1)

A saline solution with 0.13 w-% delivered at mass flow rate of 22 t/h is to be concentrated to 1.3 w-% of salt in a single-stage evaporator at ambient pressure. The evaporator is heated by saturated vapour at a pressure of 3.43 bar which is condensed to saturated liquid.

Additional data:

- Heat capacity of water:  $c_{P,\text{water}} = 4.2 \frac{\text{kJ}}{\text{kg K}}$
- Others: *Table 3.1: Steam table*

Assumptions:

- Considering the low concentration of the salt, the influence of the salt on the enthalpy and the boiling temperature of the solution can be neglected.
- The vapour does not contain any salt and can be considered saturated vapour.

3.1.1 Calculate the mass flow rate of water needed to evaporate to reach the required salt concentration in the solution!

3.1.2 How much heat steam is necessary, if the inlet temperature of the saline solution is set to (a) 95°C and (b) 20 °C respectively?

### 3.2 Single-stage evaporator (2)

A suspension of colloids in water at a total mass flow of 25 t/h is to be concentrated from 10 w-% to 50 w-% of solid in a single stage evaporator. Saturated vapour at 2 bar is available and is to be condensed to saturated liquid. The evaporator pressure is set to 0.136 bar.

#### Additional data

- Heat capacity of feed:  $c_{P,L,0} = 3.86 \frac{kJ}{kg K}$
- Heat capacity of water:  $c_{P,water} = 4.2 \frac{kJ}{kg K}$
- Overall heat transfer coefficient:  $k_v = 2.8 \frac{kW}{m^2 K}$
- Others: *Table 3.1: Steam table*

#### Assumptions:

- The elevation of the boiling temperature and the loss of heat can be disregarded.
- The vapour does not contain any salt and can be considered saturated steam.
- The heat capacity of the solid suspension can be assumed to be linearly depending on the solid weight fraction.

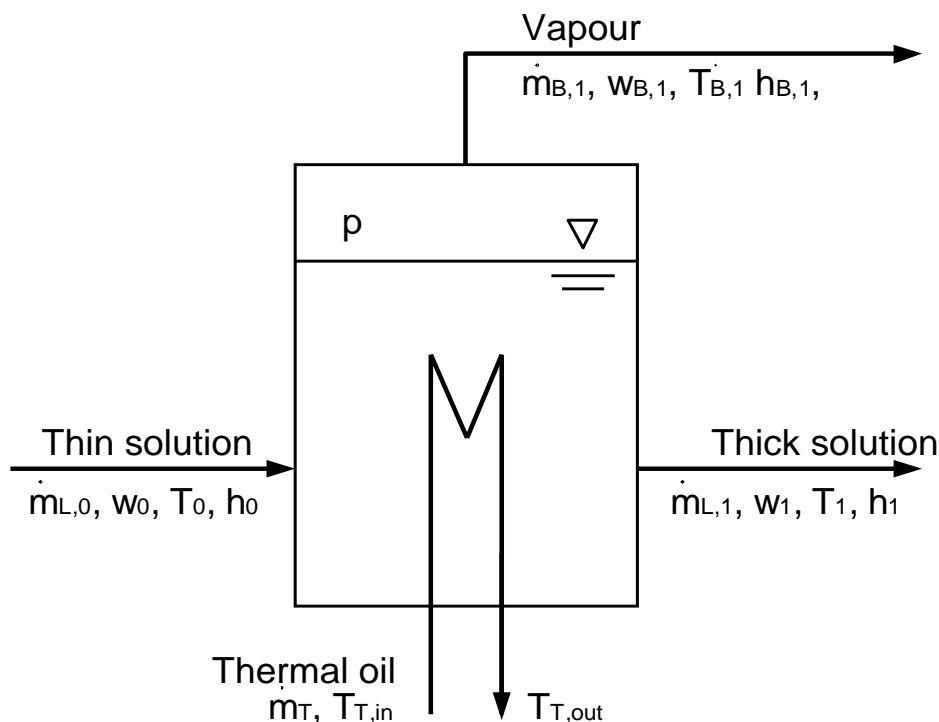
- 3.2.1 Calculate the total and the specific demand of heating steam (related to the produced vapour) for a feed temperature of 50.96 °C!
- 3.2.2 Which surface area for heating is necessary for the evaporator to achieve the required concentration of the solid?

### 3.3 Single-stage co-current evaporator

In a continuously operating evaporator, a saline solution (see *Figure 3-1*) is concentrated from 10 w-% to 50 w-%. The evaporator works at a pressure of 1.5 bar and is heated by thermal oil.

Additional data:

- Heat capacity of vapour:  $c_{p,B} = 2.0 \frac{kJ}{kg K}$
- Heat capacity of thermal oil:  $c_{p,T} = 2.5 \frac{kJ}{kg K}$
- Temperatures of thermal oil:  $T_{T,in} = 240^{\circ}C$ ,  $T_{T,out} = 160^{\circ}C$
- Specific enthalpy of thin solution:  $h_0 = 200 \frac{kJ}{kg}$
- Others:  $h,w$ -diagram *Figure 3-2* and *Table 3.1: Steam table*



*Figure 3-1: Balance scope of a continuous evaporator.*

- 3.3.1 Use the balance equations for the evaporator to determine the required specific mass of the produced vapour and the thick solution related to the thin solution ( $\dot{m}_{L,1}/\dot{m}_{L,0}$  and  $\dot{m}_{B,1}/\dot{m}_{L,0}$ )!
- 3.3.2 Determine the elevation of boiling temperatures at 1.5 bar (compared to pure water) for the thin and the thick solution, respectively!
- 3.3.3 Assuming an ideally mixed evaporator, what is the elevation of the boiling temperature in the evaporator?

- 3.3.4 Determine the total specific heat demand (related to the thin solution)!
- 3.3.5 Determine the specific heat quantity (related to the thin solution) that is required to reach the boiling temperature!
- 3.3.6 What mass flow of thermal oil is required (related to the thin solution)?

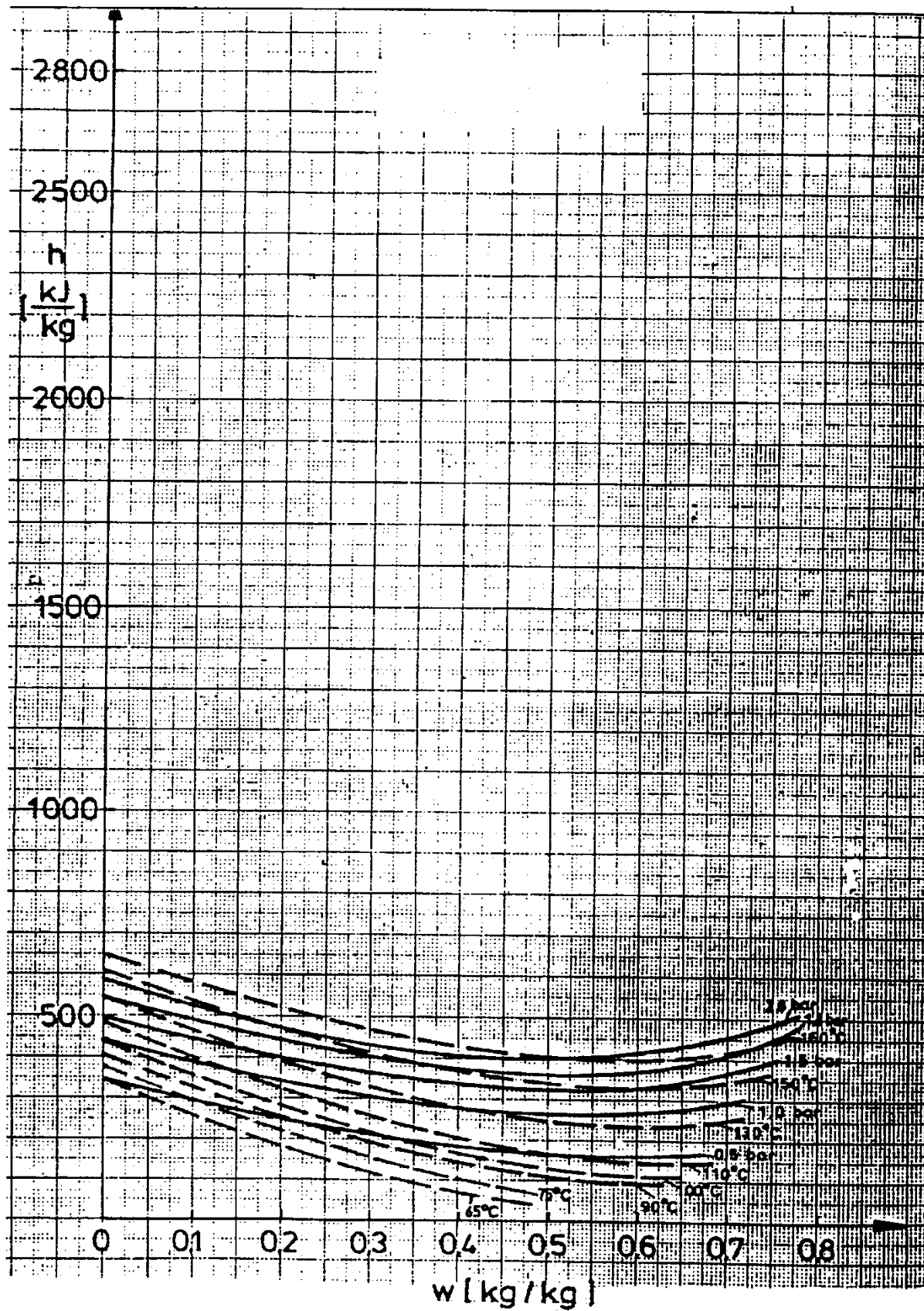


Figure 3-2:  $h,w$ -diagram of a saline solution.

### 3.4 Three-stage evaporation process

In a three-stage evaporation process, a saline solution is to be concentrated to 70 w-%. The evaporators are co-currently connected. The pressures in the stages are 1 bar, 0.5 bar and 0.1 bar. The first stage is fresh heated by heating steam at 2 bar, which can be condensed to saturated water.

Additional data:

- Specific enthalpy of thin solution:  $h_0 = 225 \frac{kJ}{kg}$
- Width of evaporation in last stage:  $\Delta w_3 = 0.17$
- Heat capacity of vapour:  $c_{p,B} = 1.88 \frac{kJ}{kg K}$
- Others: h,w-diagram *Figure 3-3* and  
*Table 3.1: Steam table*

Assumptions:

- The temperatures of the produced vapour in each stage and the vapour emerged from the previous throttle can be assumed to be equal for the calculations (from 3.4.2).

3.4.1 Sketch the temperature, pressure and concentration profile of the entire process!

3.4.2 Determine the changes in concentration for each stage of the process!

3.4.3 Which amount of fresh heating steam (related to the thin solution) is required for the process?

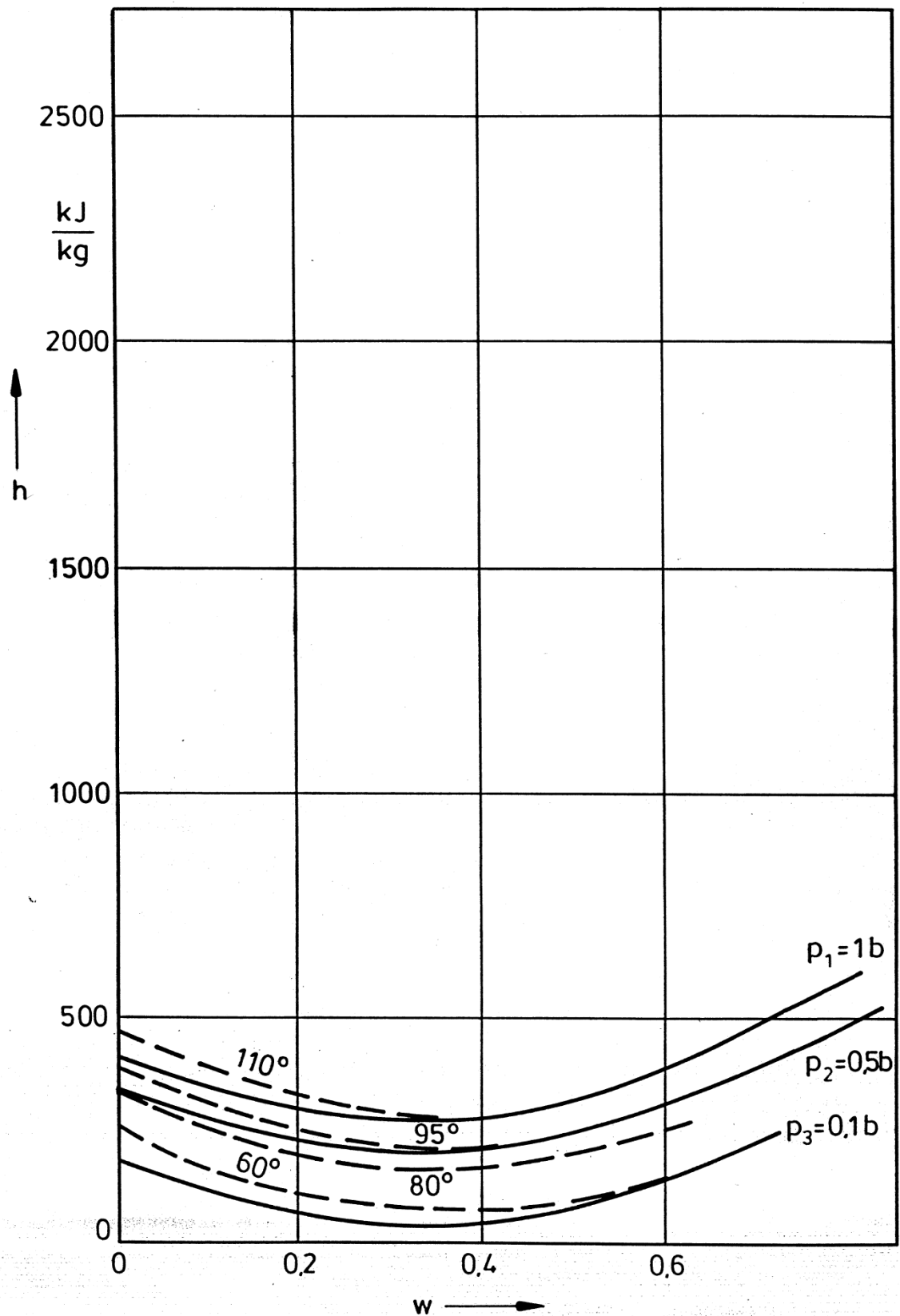


Figure 3-3:  $h,w$ -diagram of a saline solution.



Table 3.1: Steam table.

pressure		temperature	spec. volume of water	spec. volume of vapour	Density of water	Enthalpy of water	Enthalpy of vapour	Heat of vaporisation
p	p	t	v'	v''	ρ	h'	h''	r
kPa	bar	°C	dm³	dm³	kg/dm³	kJ/kg	kJ/kg	kJ/kg
1	0,01	6,9808	1,0001	129,200	0,0077	29,340	2514,4	2485,0
2	0,02	17,5130	1,0012	67,010	0,0149	73,460	2533,6	2460,2
3	0,03	24,1000	1,0027	45,670	0,0219	101,000	2545,6	2444,6
4	0,04	28,9830	1,0040	34,800	0,0287	121,410	2554,5	2433,1
5	0,05	32,8980	1,0052	28,190	0,0355	137,770	2561,6	2423,8
6	0,06	36,1830	1,0064	23,740	0,0421	151,500	2567,5	2416,0
7	0,07	39,0250	1,0074	20,530	0,0487	163,380	2572,6	2409,2
8	0,08	41,5340	1,0084	18,100	0,0552	173,860	2577,1	2403,2
9	0,09	43,7870	1,0094	16,200	0,0617	183,280	2581,1	2397,9
10	0,1	45,8330	1,0102	14,670	0,0681	191,830	2584,8	2392,9
20	0,2	60,0860	1,0172	7,650	0,1307	251,450	2609,9	2358,4
30	0,3	69,1240	1,0223	5,229	0,1912	289,300	2625,4	2336,1
40	0,4	75,8860	1,0265	3,993	0,2504	317,650	2636,9	2319,2
50	0,5	81,3450	1,0301	3,240	0,3086	340,560	2646,0	2305,4
60	0,6	85,9540	1,0333	2,732	0,3661	359,930	2653,6	2293,6
70	0,7	89,9590	1,0361	2,365	0,4229	376,770	2660,1	2283,3
80	0,8	93,5120	1,0387	2,087	0,4792	391,720	2665,8	2274,0
90	0,9	96,7130	1,0412	1,869	0,5350	405,210	2670,9	2265,6
100	1	99,6320	1,0434	1,694	0,5904	417,510	2675,4	2257,9
150	1,5	111,3700	1,0530	1,159	0,8628	467,130	2693,4	2226,2
200	2	120,2300	1,0608	0,885	1,1290	504,700	2706,3	2201,6
250	2,5	127,4300	1,0675	0,718	1,3920	535,340	2716,4	2181,0
300	3	133,5400	1,0735	0,606	1,6510	561,430	2724,7	2163,2
350	3,5	138,8700	1,0789	0,524	1,9080	584,270	2731,6	2147,4
400	4	143,6200	1,0839	0,462	2,1630	604,670	2737,6	2133,0
450	4,5	147,9200	1,0885	0,414	2,4170	623,160	2742,9	2119,7
500	5	151,8400	1,0928	0,375	2,6690	640,120	2747,5	2107,4
600	6	158,8400	1,1009	0,316	3,1700	670,420	2755,5	2085,0
700	7	164,9600	1,1082	0,273	3,6670	697,060	2762,0	2064,9
800	8	170,4100	1,1150	0,240	4,1620	720,940	2767,5	2046,5
900	9	175,3600	1,1213	0,215	4,6550	742,640	2772,1	2029,5
1000	10	179,8800	1,1274	0,194	5,1470	762,610	2776,2	2013,6
1100	11	184,0700	1,1331	0,177	5,6370	781,130	2779,7	1998,5
1200	12	187,9600	1,1386	0,163	6,1270	798,430	2782,7	1984,3
1300	13	191,6100	1,1438	0,151	6,6170	814,700	2785,4	1970,7
1400	14	195,0400	1,1489	0,141	7,1060	830,080	2787,8	1957,7
1500	15	198,2900	1,1539	0,132	7,5960	844,670	2798,9	1945,2
1600	16	201,3700	1,1586	0,124	8,0850	858,560	2791,7	1933,2
1700	17	204,3100	1,1633	0,117	8,5750	871,840	2793,4	1921,5
1800	18	207,1100	1,1678	0,110	9,0650	884,580	2794,8	1910,3
1900	19	209,8000	1,1723	0,105	9,5550	896,810	2796,1	1899,3
2000	20	212,3700	1,1766	0,100	10,0500	908,590	2797,2	1888,6
2500	25	223,9400	1,1972	0,080	12,5100	961,960	2800,9	1839,0
3000	30	233,8400	1,2163	0,067	15,0100	1008,400	2802,3	1793,9
4000	40	250,3300	1,2521	0,050	20,1000	1087,400	2800,3	1712,9
5000	50	263,9100	1,2858	0,037	25,3600	1154,500	2794,2	1639,7
6000	60	275,5500	1,3187	0,032	30,8300	1213,700	2785,0	1571,3
7000	70	285,7900	1,3513	0,027	36,5300	1267,400	2773,5	1506,0
8000	80	294,9700	1,3842	0,024	42,5100	1317,100	2759,9	1442,8
9000	90	303,3100	1,4179	0,021	48,7900	1363,700	2744,6	1380,9
10000	100	310,9600	1,4526	0,018	55,4300	1408,000	2727,7	1319,7
11000	110	318,0500	1,4887	0,016	62,4800	1450,600	2709,3	1258,7
12000	120	324,6500	1,5268	0,014	70,0100	1491,800	2689,2	1197,4
13000	130	330,8300	1,5672	0,013	78,1400	1532,000	2667,0	1135,0
14000	140	336,6400	1,6106	0,012	86,9900	1571,600	2642,4	1070,7
15000	150	342,1300	1,6579	0,010	96,7100	1611,000	2615,0	1004,0
20000	200	365,7000	2,0370	0,006	170,2000	1826,500	2418,4	591,9
22000	220	373,6900	2,6714	0,004	268,3000	2011,100	2195,6	184,5
22120	221,2	374,1500	3,1700	0,003	315,5000	2107,400	2107,4	0,0

## 4 Multistage distillation

### 4.1 Multistage distillation using McCabe-Thiele method

The liquid-vapour equilibrium of a binary azeotropic mixture consisting of component A and B is described in *Figure 4-1* as an x,y-diagram. The molar fraction of component A is plotted on the x-axis.

Additional data:

- Feed composition:  $x_{A,F} = 0,8$
- Reflux ratio:  $v = 1.5 * v_{\min}$
- Others: x,y-diagram in *Figure 4-1*

- 4.1.1 Which product compositions can be achieved with an infinite number of stages, if the feed composition is varied along the entire range of values? What simple process can be used to separate the mixture into the pure components? Outline qualitatively the process and the important points in an x,y-diagram!
- 4.1.2 Determine the required number of theoretical stages to separate the mixture into two fractions with 95 mol-% and 35 mol-% of component A. The feed is supplied as a saturated liquid and the reflux ratio is 1.5 times higher than the minimal reflux ratio.

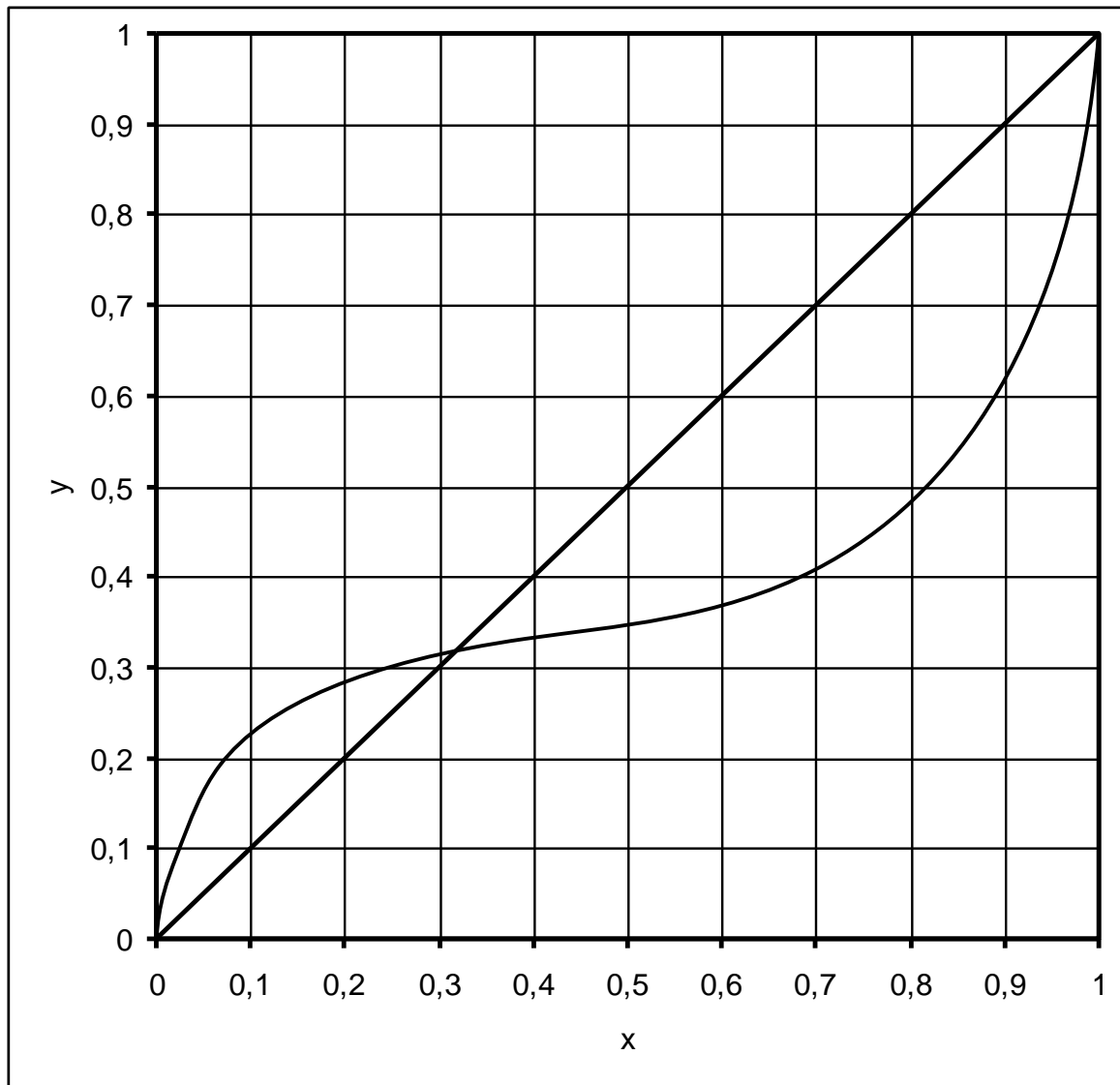


Figure 4-1:  $x,y$ -diagram of a binary azeotropic mixture

## 4.2 Multistage distillation using h,w-diagram

A solution of ammonia-water shall be concentrated at a pressure of 10 bar to a product purity of  $w_E = 98$  w-% ammonia. The solution is available as saturated liquid and with a concentration of  $w_F = 29$  w-%. After being totally condensed the product is transferred to further process steps as a liquid.

The reflux ratio is 23 % higher than the minimal required reflux ratio. The trays of the column have a constant separation efficiency of  $E = 60$  %.

### Additional data:

- Mass flow of feed:  $\dot{m}_F = 1$  t/h
- Others: h,w-diagram in *Figure 4-2*

- 4.2.1 Derive the mathematical expression for the intersection lines of the rectifying and the stripping section as well as for the main line and the reflux ratio using adequate balancing scopes.
- 4.2.2 Determine the real number of trays if the bottom product shall contain 7.6 w-% of ammonia (note: The dashed line in the diagram are a support to draw the wet steam isotherms).
- 4.2.3 What amount of heat is supplied to the evaporator and withdrawn from the condenser?
- 4.2.4 What is the difference in calculating and determining the number of theoretical stages if the condenser worked as a partial condenser (qualitatively)?

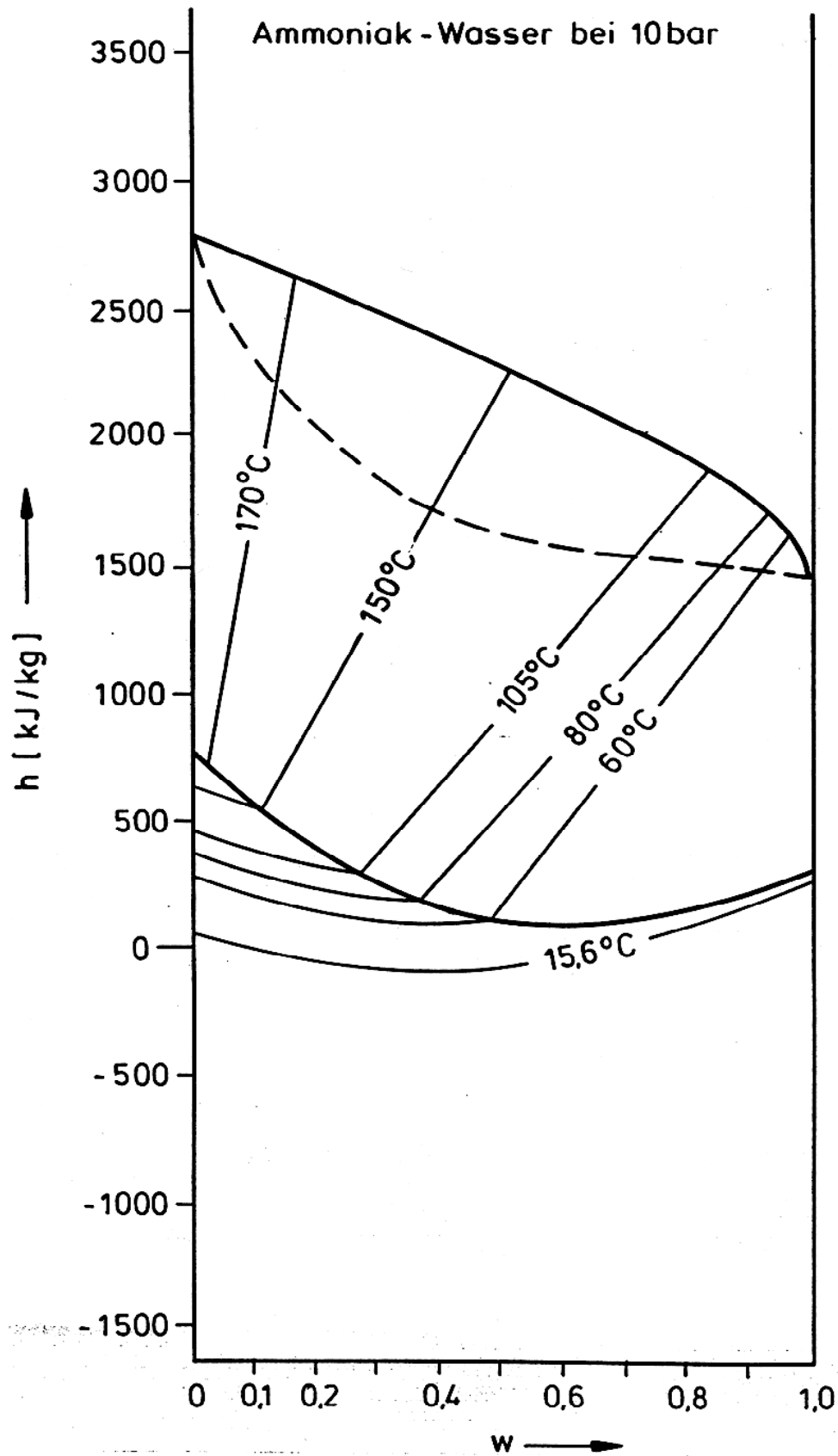


Figure 4-2:  $h,w$ -diagram for ammonia/water at 10 bar. Dashed line is an auxiliary line to draw the wet steam isotherms.

### 4.3 Distillation column with side stream withdrawal

A mixture of water and methanol with a mole fraction of 45 mol-% methanol shall be separated in a distillation column. The distillate has to contain at minimum 96 mol-% of methanol and the bottom product at minimum 3 mol-% methanol. In addition, for a further step of synthesis at the integrated facility a fraction with 70 mol-% methanol shall be withdrawn.

For the process a feed stream of 200 kmol/h as saturated liquid is available, from which a side stream at a molar flow rate of 20 kmol/h is to be withdrawn. With this information a first approximation is to be done.

#### Additional data:

- Reflux ratio:  $v = 1.5$
- Rectifying line:  $y = \frac{v}{v+1}x + \frac{x_E}{v+1}$
- Others: x,y-diagram at Figure 4-3

#### Additional data:

- Side stream is saturated liquid

4.3.1 Identify the molar flow rates and the compositions of all external streams!

4.3.2 In which section of the column is the side stream to be withdrawn?

4.3.3 Derive the equation for the operation line of the middle section of the column expressed only by external variables (in- and out-flowing streams, reflux ratio)!

4.3.4 Identify the required number of theoretical stages by using the x,y-diagram! At which theoretical stage is the side stream withdrawal located and at which stage is the feed supplied?

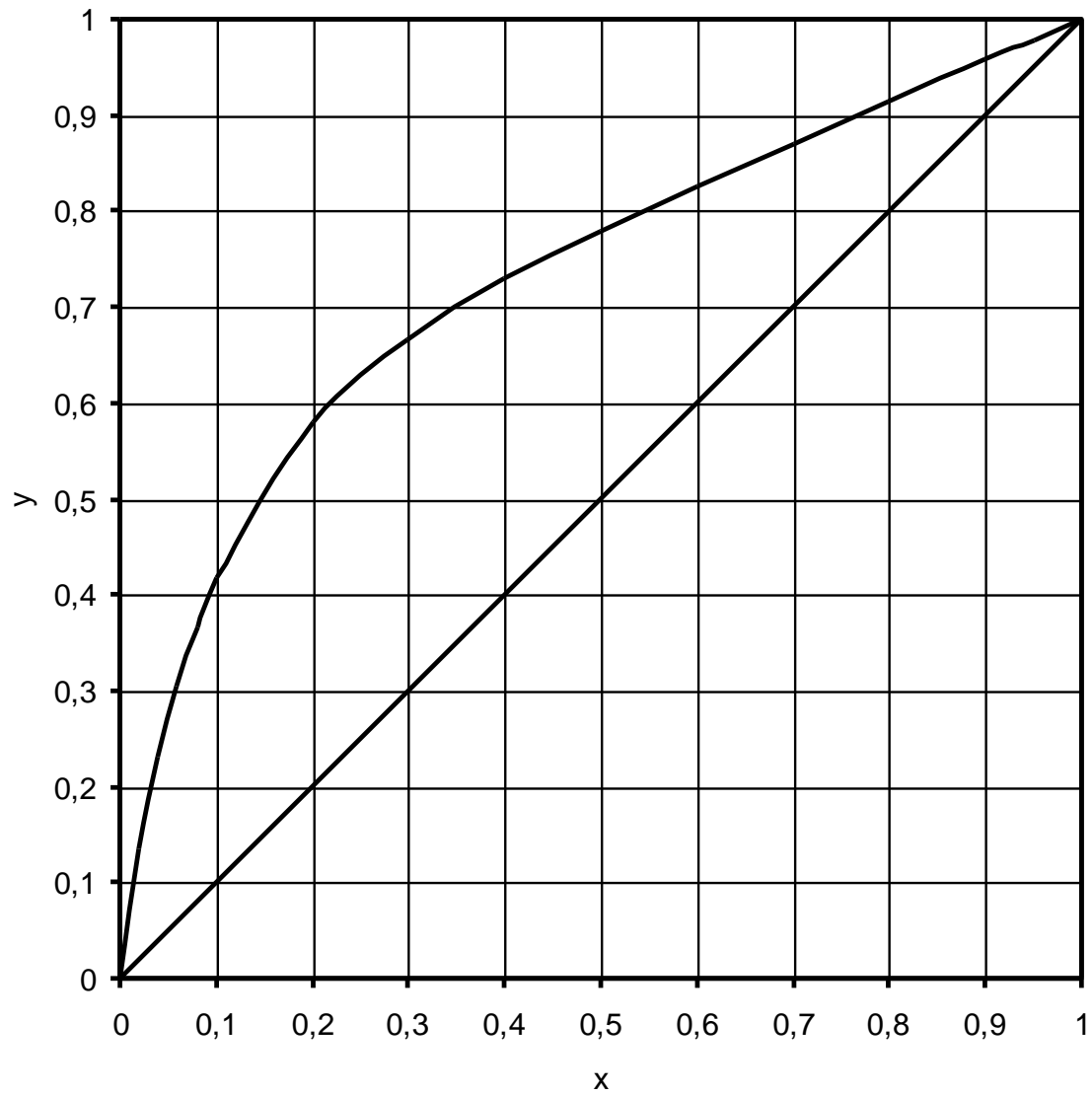


Figure 4-3: x,y-diagram methanol-water

#### 4.4 Distillation column with two feed streams & one side stream withdrawal

In your integrated facility two product streams containing methanol and water with different composition are available. Both product streams shall be supplied to a distillation column without being mixed before. In addition, there is a side stream withdrawal, which composition is useful for a further step of in the plant.

##### Additional data:

State of product stream 1:

- Molar stream:  $\dot{n}_{\text{product,stream.1}} = 200 \text{ kmol} / \text{h}$
- Mean composition:  $Z_{\text{product stream 1, MeOH}} = 43 \text{ Mol-\%}$
- Thermal condition: 80% liquid, 20% vapour

State of product stream 2:

- Mole stream:  $\dot{n}_{\text{product,stream.2}} = 100 \text{ kmol} / \text{h}$
- Mean composition:  $X_{\text{product stream 2, MeOH}} = 18 \text{ Mol-\%}$
- Thermal condition: saturated liquid

Side stream withdrawal requirements:

- Molar stream:  $\dot{n}_{\text{side,stream}} = 35 \text{ kmol} / \text{h}$
- Mean composition:  $X_{\text{side stream, MeOH}} = 67 \text{ Mol-\%}$
- Distillate requirement:  $x_{E,\text{MeOH}} \geq 96 \text{ mol-\%}$
- Bottom stream requirement:  $x_{B,\text{MeOH}} \leq 3 \text{ mol-\%}$
- Reflux ratio:  $v = 2,0$
- Rectifying line:  $y = \frac{v}{v+1} x + \frac{x_E}{v+1}$
- Intersection line:  $y = \frac{e}{e-1} x - \frac{1}{e-1} x_F$   
with  $e = 1 + \frac{h_{F,0} - h_F}{\Delta h^{LV}}$
- Others: x,y-diagram in *Figure 4-4*

4.4.1 Derive the equation of the operation line for a middle section of a column with two feed streams (no side stream) expressed by only external variables (in- and out-flowing streams, reflux ratio)!

Note: The following tasks can be solved independently from task 4.4.1.

4.4.2 Sketch the column with all supplied and withdrawn streams!

4.4.3 Identify the required number of theoretical stages by using the x,y-diagram! At which theoretical stages are the feed streams and the side stream to be positioned? (Hint: Start at the distillate point and draw the operating line using its gradient.)



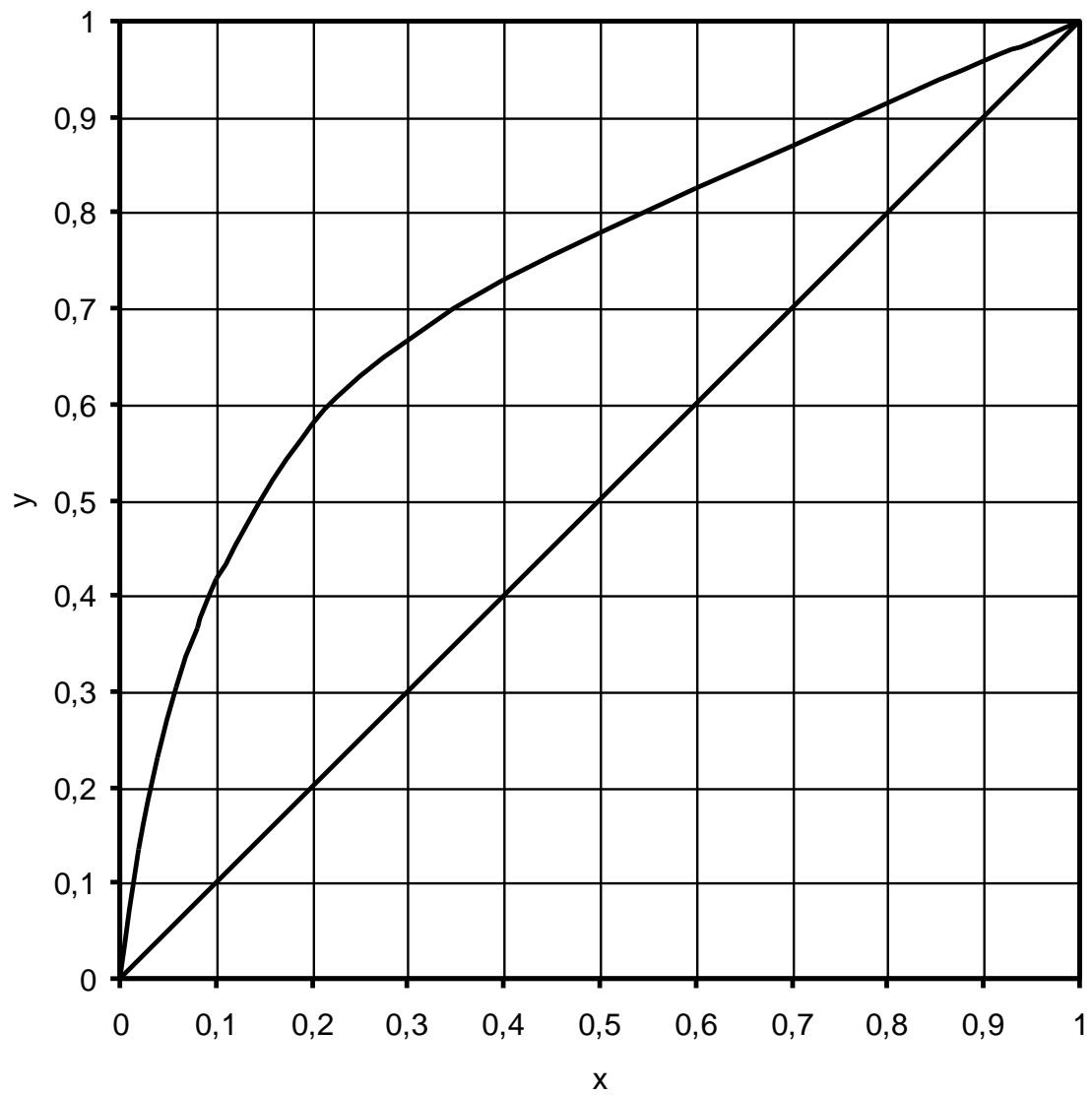


Figure 4-4: x,y-diagram methanol-water

## 4.5 Discontinuous multistage distillation (1)

An equimolar mixture of benzene (low boiler) and chlorobenzene at 40 kmol of are available for this process. However, you require chlorobenzene containing less than 6 mol-% benzene. To purify the liquid mixture a distillation column with two theoretical stages, an evaporator and a total condenser are available. You decide to use a discontinuous process working at ambient pressure with a constant reflux ratio of 1 to purify the liquid. The data of the vapour-liquid-equilibrium are available for a first rough calculation.

Additional data:

- Rayleigh-Equation: 
$$\frac{dn_B}{n_B} = \frac{dx_B}{x_E - x_B}$$
- Vapour-liquid-equilibrium for benzene and chlorobenzene at  $p = 1 \text{ atm}$ :

x	0	0.100	0.200	0.300	0.400	0.500	0.600	0.700	0.800	1
y	0	0.314	0.508	0.640	0.734	0.806	0.862	0.905	0.943	1

Assumptions:

- To determine the area below the graph in the auxiliary diagram (Figure 4-5) an approximation with four supporting points ( $x_E = \{0.95; 0.70; 0.50; 0.30\}$ ) from the McCabe-Thiele-Diagram is sufficient.

- 4.5.1 What is described by the Rayleigh-Equation and where is the difference between its use for a simple discontinuous distillation and for a discontinuous multistage distillation?
- 4.5.2 Transform the Rayleigh-Equation into a form that can be solved graphically using a McCabe-Thiele-Diagram! What is the essential assumption to use the McCabe-Thiele-Diagram for a discontinuous multistage distillation at a constant reflux ratio?
- 4.5.3 Calculate the quantity of bottoms product at the required purity, the quantity and the mean composition of the distillate and the yield of benzene in the distillate.

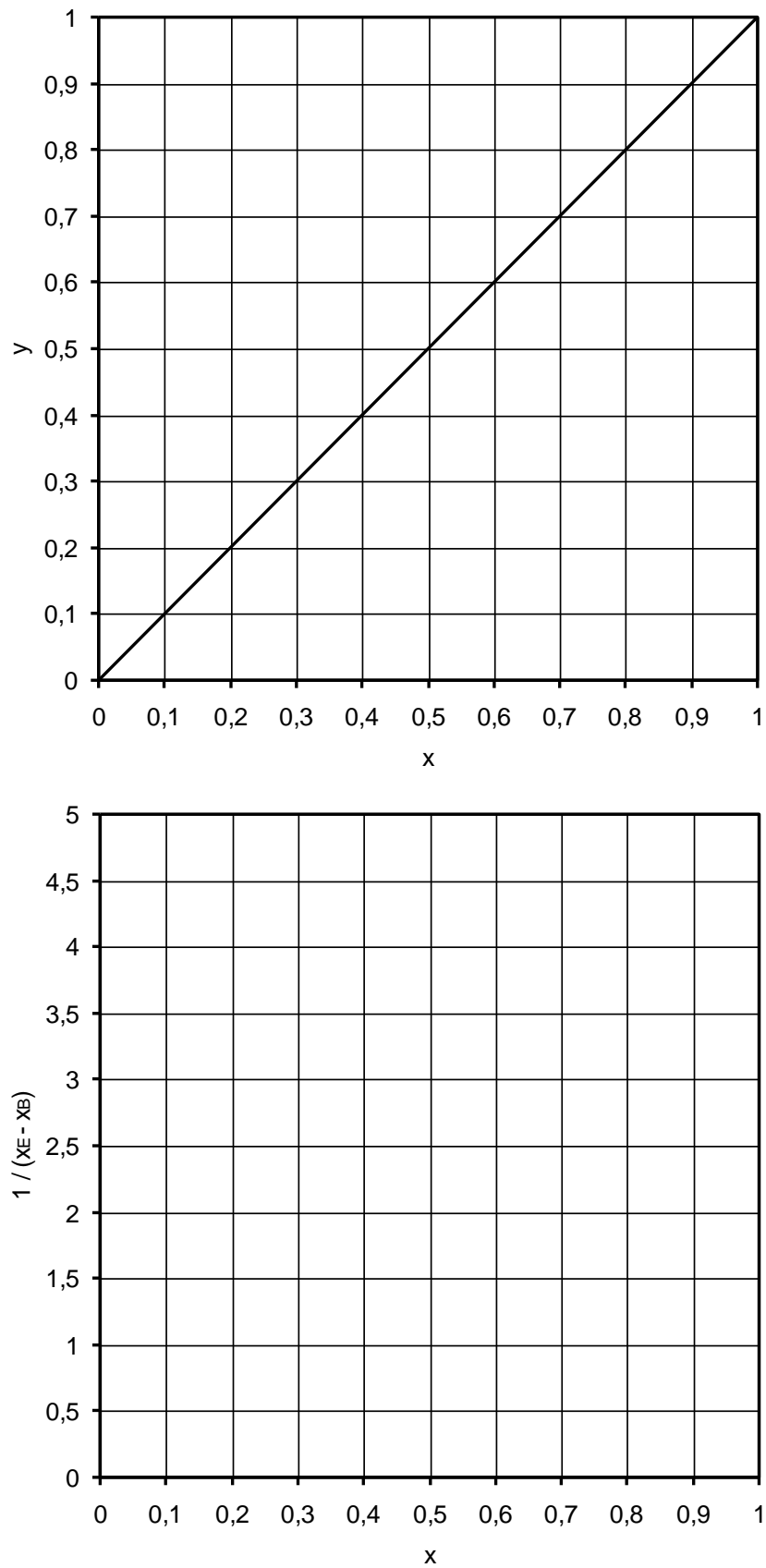


Figure 4-5:  $x, y$ -diagram and auxiliary diagram.

## 4.6 Discontinuous multistage distillation (2)

(Remark: This task is not relevant for the exam)

In your plant 10 kmol of acetone were pumped into a receiver tank filled with 10 kmol ethanol by accident. Therefore, it was decided to purify the mixture discontinuously in already existing distillation equipment that consists of a column with 6 theoretical stages, an evaporator and a total condenser.

In addition the maximal acceptable contamination of the distillate was chosen to be 10 mol-% alcohols and for the bottom to be 12 mol-% acetone. The mixture is separated at ambient pressure and at variable reflux ratio. The data of the vapour-liquid-equilibrium are available for a first rough calculation.

Additional data:

- Quantity of vapour (from Rayleigh-Equation): 
$$n_D = n_{B,0} (x_{B,0} - x_E) \int_{x_{B,0}}^{x_B} \frac{1+v}{(x_E - x_B)^2} dx_B$$
- Vapour-liquid-equilibrium for acetone and ethanol at  $p = 1 \text{ atm}$ :

x	0	0.05	0.10	0.20	0.30	0.40	0.50	0.60	0.70	0.80	0.90	1
y	0	0.16	0.25	0.42	0.51	0.60	0.67	0.72	0.79	0.87	0.93	1

Assumptions:

- The reflux is saturated liquid.
- The dependence of enthalpy of vaporisation on the concentration can be neglected.
- The loss of heat can be neglected.
- To determine the area below the graph in the auxiliary diagram (Figure 4-6) it is sufficient to use an approximation with five supporting points ( $v = \{2.75; 3.40; 4.50; 8.00; 11.35\}$ ) from the McCabe-Thiele-Diagram.

- 4.6.1 What are the product quantities (head and still at the bottom) for the required purities?
- 4.6.2 Which reflux ratios have to be chosen at the beginning and in the end of the discontinuous multistage distillation?
- 4.6.3 How much time do you need to separate the mixture into the required purities assuming a vapour production in the evaporator of 6 kmol vapour per hour? Calculate the quantity of vapour that is produced in the still during the discontinuous multistage distillation first. Use the diagrams in Figure 4-6 to solve the problem.

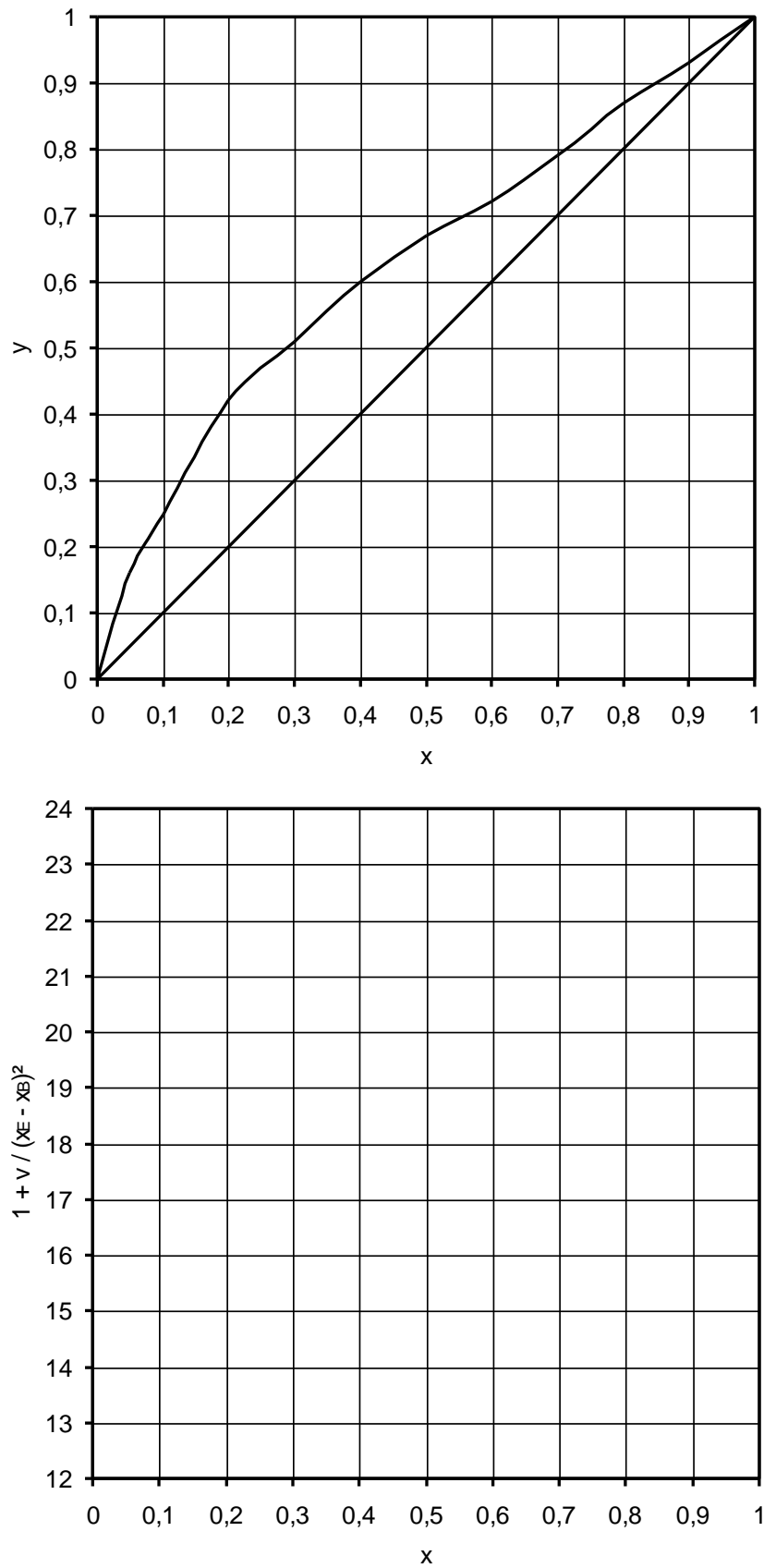


Figure 4-6:  $x,y$ -diagram and auxiliary diagram.

## 4.7 Distillation lines (1)

Within the process of an air fractionation it shall be checked, if it were possible to separate a mixture of nitrogen „1“ ( $x_{N,0} = 0.05$ ), argon „2“ ( $x_{A,0} = 0.36$ ) and oxygen „3“ at 1 bar using a multistage distillation unit. For a first evaluation the determination of the distillation lines for this composition is necessary. Constant mean separation factors can be assumed.

### Additional data:

- Boiling temperatures at  $p = 1$  bar:  
 $T_{S,N} = 77.5$  K  
 $T_{S,A} = 87.3$  K  
 $T_{S,O} = 90.3$  K
- Mean separation factors:  
 $\alpha_{N,O} = 4.032$   
 $\alpha_{A,O} = 1.339$

### Assumptions:

- The mixture can be assumed to be ideal.

- 4.7.1 Calculate the points of the distillation line through the composition of the mixture for 10 steps.
- 4.7.2 Draw the distillation line using a ternary diagram (Figure 4-7). Sketch the course of all distillation lines existing in the diagram.

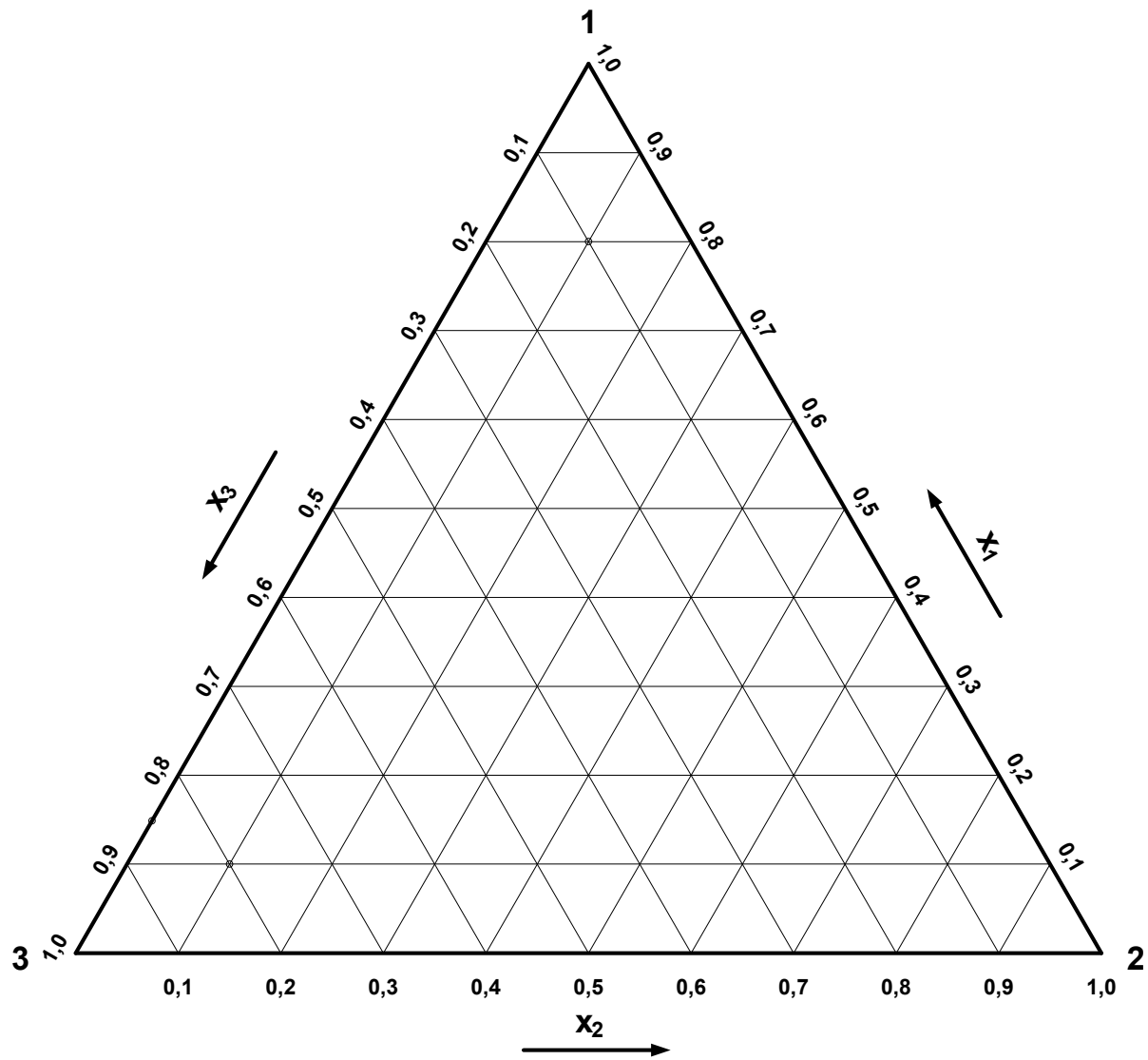


Figure 4-7: Ternary diagram.

## 4.8 Distillation lines (2)

For the evaluation of the separation possibilities for the ternary mixture consisting of benzene, 2-propanal and cyclohexane only the boiling point surface and the boiling points of the pure components are available. Your task is a feasibility study for the separation of this mixture.

### Additional data:

- Boiling points at  $p = 1$  bar:

$$T_{S,Benz.} = 80.1 \text{ }^{\circ}\text{C}$$

$$T_{S,2-Prop.} = 82.6 \text{ }^{\circ}\text{C}$$

$$T_{S,Cyclohex.} = 80.7 \text{ }^{\circ}\text{C}$$

- 4.8.1 Mark all azeotropic points in the boiling point surface for the mixture and characterise them (Figure 4-8). Draw the existing border distillation lines and their course and direction. Additionally, qualitatively sketch a few distillation lines and their course in each section of the boiling point surface (including binary edge of the triangle)!
- 4.8.2 Arrange the azeotropic points with decreasing temperatures. Transfer the boiling point surface from Figure 4-8 into a ternary diagram (Figure 4-9) and indicate the azeotropic points, the border distillation lines and its course as well. Furthermore, draw distillation lines and their course in each section of the diagram
- 4.8.3 How do you rate the separation possibilities of the mixture using multistage distillation?



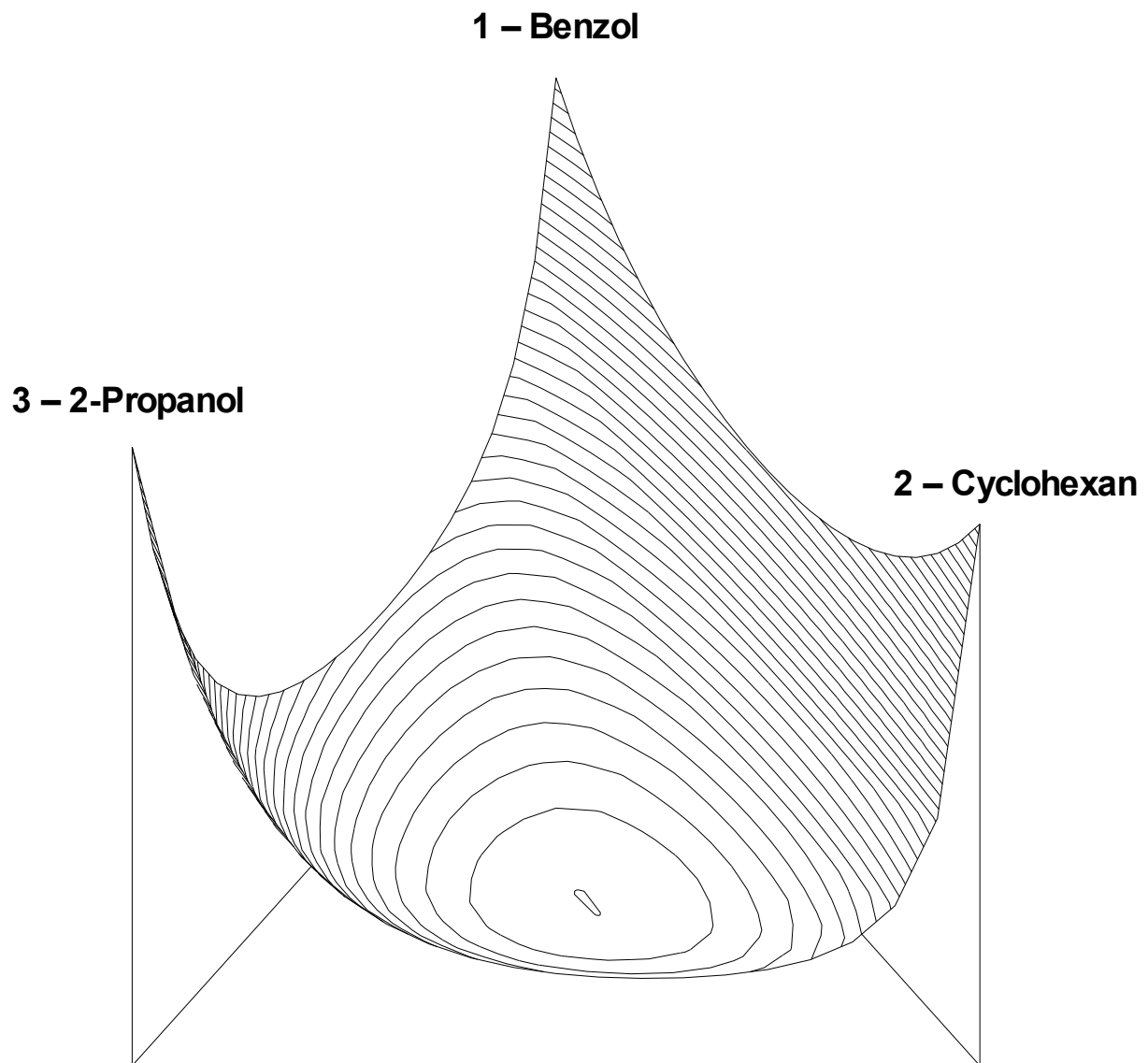


Figure 4-8: Boiling surface of the ternary mixture: benzene, cyclohexane and 2-propanol.

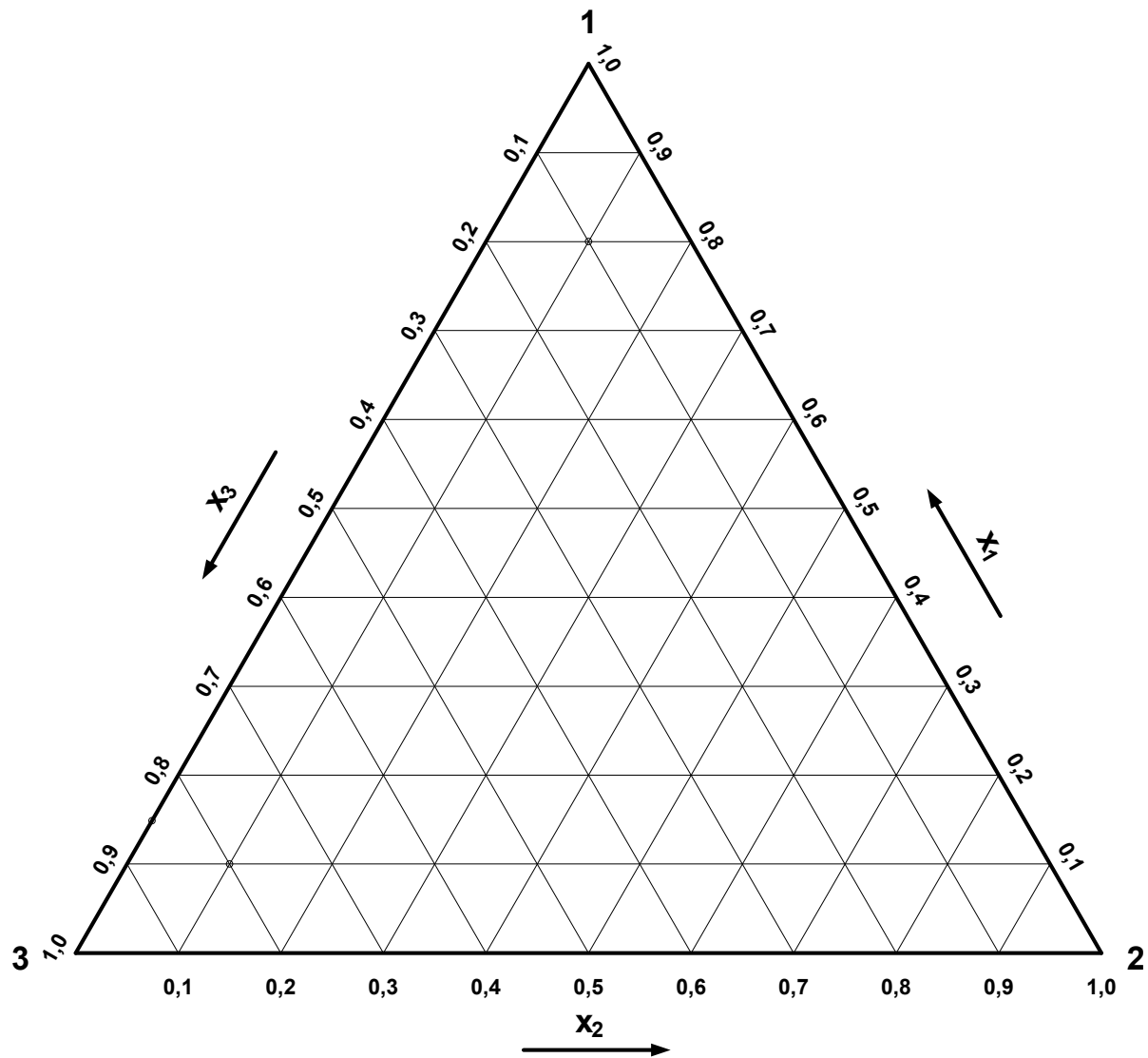


Figure 4-9: Ternary diagram.

## 4.9 Distillation lines (3)

It shall be estimated how a ternary mixture consisting of octane, 2-ethoxyethanol and ethylbenzene can be separated using a rectification unit at atmospheric pressure. The boiling temperatures, information about the two azeotropic points are available and indicated in a ternary diagram (Figure 4-10). For the first evaluation a very high reflux ratio can be assumed.

### Additional data:

- Boiling temperatures at  $p = 1$  bar:
  - $T_{S,\text{Oktan}} = 125.4\text{ }^{\circ}\text{C}$
  - $T_{S,2\text{-Ethoxyethanol}} = 134.9\text{ }^{\circ}\text{C}$
  - $T_{S,\text{Ethylbenzol}} = 136.2\text{ }^{\circ}\text{C}$
- Temperatures of the azeotropic points:
  - $T_{\text{Az-1}} = 117.0\text{ }^{\circ}\text{C}$
  - $T_{\text{Az-2}} = 128.1\text{ }^{\circ}\text{C}$
- Feed composition A:
  - $x_{1,F} = 0.15$
  - $x_{2,F} = 0.60$
- Feed composition B:
  - $x_{1,F} = 0.65$
  - $x_{2,F} = 0.10$
- Bottom composition A:
  - $x_{1,B} = 0.02$
  - $x_{2,B} = 0.70$
- Distillate composition B:
  - $x_{1,D} = 0.65$
  - $x_{2,D} = 0.30$
- Others:
  - Ternary diagram Figure 4-10

- 4.9.1 For each mixture mark the possible separation regions in the ternary diagram and assign them to the head- (D) and bottom (B) product! What information can generally be derived from separation regions?
- 4.9.2 The mixtures have to be separated with respect to their required purities. Assuming multistage distillation at a very high reflux ratio, what are the compositions in the head and the bottom? Calculate the molar “distillate to feed”-ratio!
- 4.9.3 What possibilities exist to obtain nearly pure ethylbenzene and 2-ethoxyethanol from the mixture? What possibilities do exist for this mixture to cross the border distillation line? Are there any other possibilities for mixtures with different properties? Give two examples!

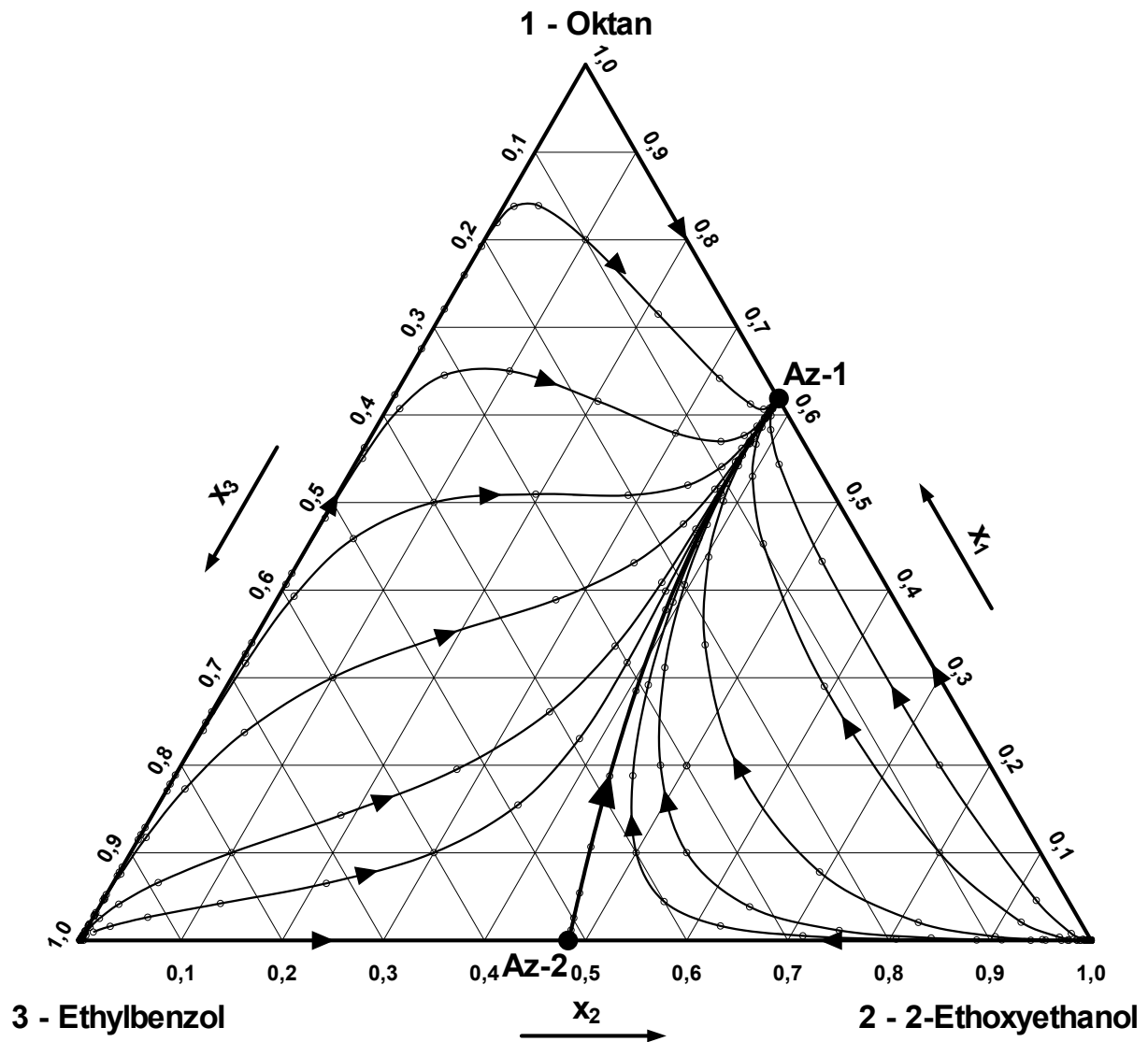


Figure 4-10: Ternary diagram with border distillation line and chosen distillation lines at 1 bar.

#### **4.10 Separation of azeotropic mixtures**

What types of azeotropic mixtures do you know? What are the possibilities to purify these mixtures in spite of their azeotropic points?

## 5 Absorption

### 5.1 Absorbent recirculation

In the rectisol process CO<sub>2</sub> should be removed from a gas stream consisting of CO<sub>2</sub>, CO and H<sub>2</sub> with methanol as the absorption solvent. The absorption is operated at -40°C and 30 bar. The concentration of CO<sub>2</sub> shall be reduced to 35% of the initial value to provide ideal conditions for a subsequent reaction unit.

5.1.1 Draw the column and name all required streams.

5.1.2 Calculate the minimal required amount of solvent.

5.1.3 Determine the mass loading of the liquid stream at the outlet of the column, assuming that the real amount of solvent is 1.2 times higher than the minimal one!

5.1.4 Calculate the height of the packing, which is required to fulfill the separation task.

The column available for CO<sub>2</sub> separation has an effective packing height of 5.5m. Therefore, the separation efficiency is higher than it is necessary. The loading of the gas stream at the outlet of the available column is  $0.05 \text{ mol CO}_2 / \text{mol} \frac{\text{molCO}_2}{\text{molwashingagent}}$ .

To reduce the separation efficiency of the column, a part of the solvent from the bottom of the column is recirculated and mixed with the fresh solvent. The total amount of fresh washing agent which enters absorption unit remains constant. The gas stream is constant too.

5.1.5 Draw a flow scheme of the changed process configuration and name all stream.

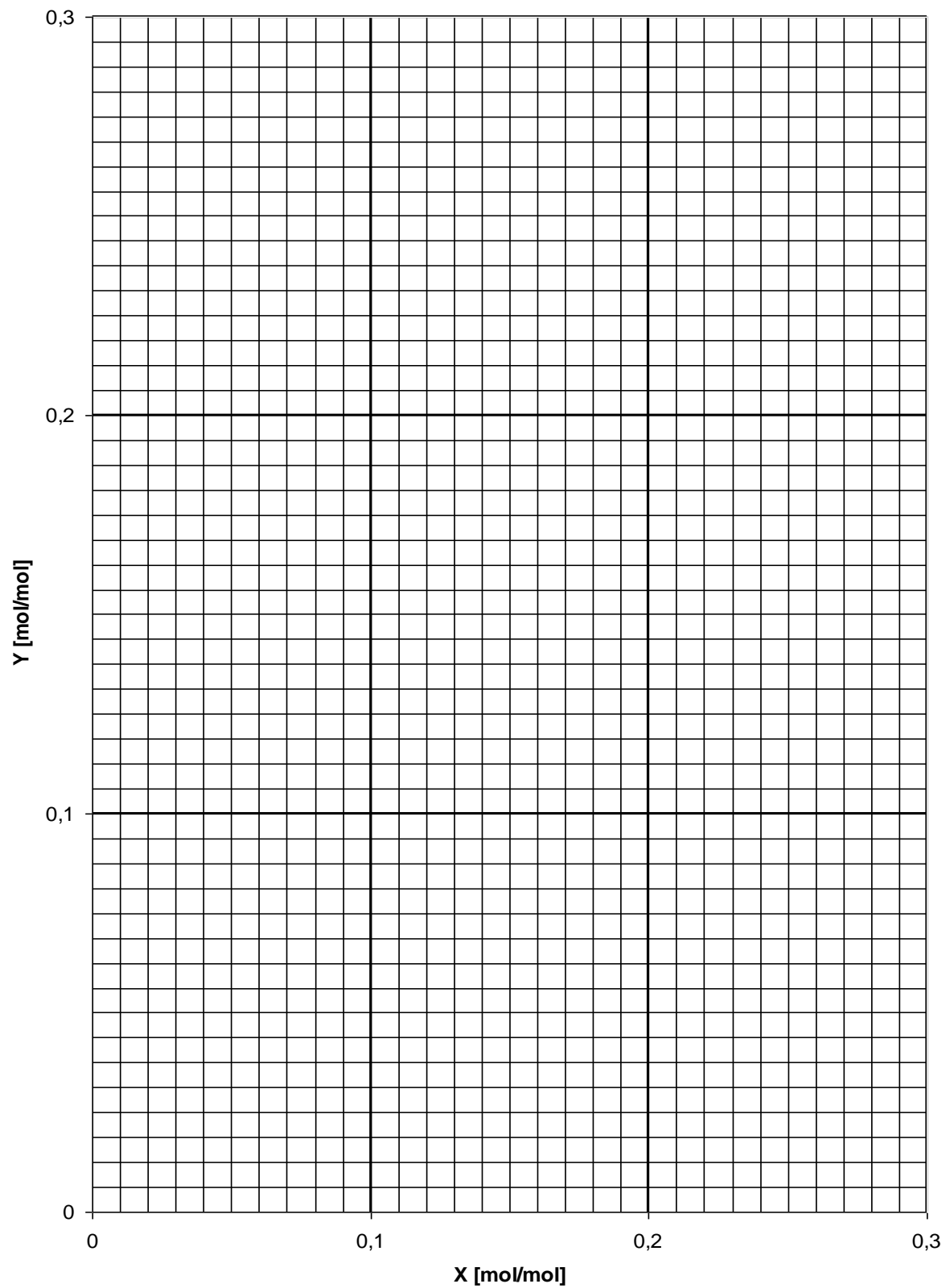
5.1.6 Calculate the loading of the washing agent at the outlet for a recirculation ratio of  $m_R = 0$ .

5.1.7 Check if the required gas composition from the previous parts (exercise 1.1.1 – 1.1.4) leaving the column will be reached with a recirculation ratio of  $m_R = 0.25$  using the available column with a packing height of 5.5 m.

5.1.8 How is the maximum recirculation ratio defined? Calculate!

#### Further information:

- Crude gas:
  - 1000 kmol/h
  - 63 mol.-% H<sub>2</sub>
  - 20 mol.-% CO<sub>2</sub>
  - 17 mol.-% CO
- Absorption solvent:
  - Loading of fresh solvent is negligible.
  - Non-volatile.
- CO and H<sub>2</sub> are insoluble in the washing agent and can be considered as inert.
- The gas stream shows ideal behaviour.
- Under the given conditions the equilibrium can be described by the linear relationship  $Y^{EQ} = 0.94 * X$
- HETP-value for the used packing is 0.61 m.

*Figure 5-1: X,Y-diagram*

## 5.2 Non-isothermal absorption

In a plate column air containing ammonia is washed with pure water. 95 mol% of the ammonia ( $\text{NH}_3$ ) entering the column shall be removed. The absorption column is operated at atmospheric pressure. Because of the development of heat the absorption is non-isothermal.

5.2.1 Derive the dependency of the liquid loading on the temperature.

Display the gas-sided concentration gradient  $(Y - Y^{\text{EQ}})_{\text{non-isothermal}}$  depending on the loading  $X$  of  $\text{NH}_3$  in the liquid phase in a diagram.

5.2.2 Draw the loading gradient  $(Y - Y^{\text{EQ}})_{\text{isothermal}}$  for an isothermal absorption into the same diagram and compare the results. Assume a temperature of the water of 20 °C at the inlet.

5.2.3 Determine real number of stages necessary for this absorption using the gas-sided Murphree efficiency.

### Process and property data:

- Composition of feed gas :  $c_1 = 4.76 \text{ Vol\% } \text{NH}_3$  ; rest: inert air
- Gas stream (inert)  $\dot{V}_G = 1.25 \text{ m}^3 \text{ air / s}$  (0 °C, 1.013 bar)
- Liquid stream  $\dot{m}_{L,\text{inert}} = 1.60 \text{ kg/s}$   $\dot{n}_L = 1,60 \frac{\text{kg}}{\text{s}}$
- Temperature of the washing solvent (inlet)  $T_0 = 20^\circ\text{C}$
- Absorption enthalpy  $\Delta H_S = 36.3 \text{ kJ/(mol absorb. NH}_3)$
- Molar heat capacity of water  $c_p = 75 \text{ kJ/(kmol K)}$   $\frac{\text{kJ}}{\text{kmol} \cdot \text{K}}$
- Gas side Murphree efficiency  $E_m = 60 \%$
- Molar mass of water  $M_{\text{H}_2\text{O}} = 18 \text{ kg/kmol}$   $\frac{\text{kg}}{\text{kmol}}$
- Molar volume of ideal gases (0°C, 1,013 bar):  $V_0 = 22.4 \text{ m}^3/\text{kmol}$   $\frac{\text{m}^3}{\text{kmol}}$

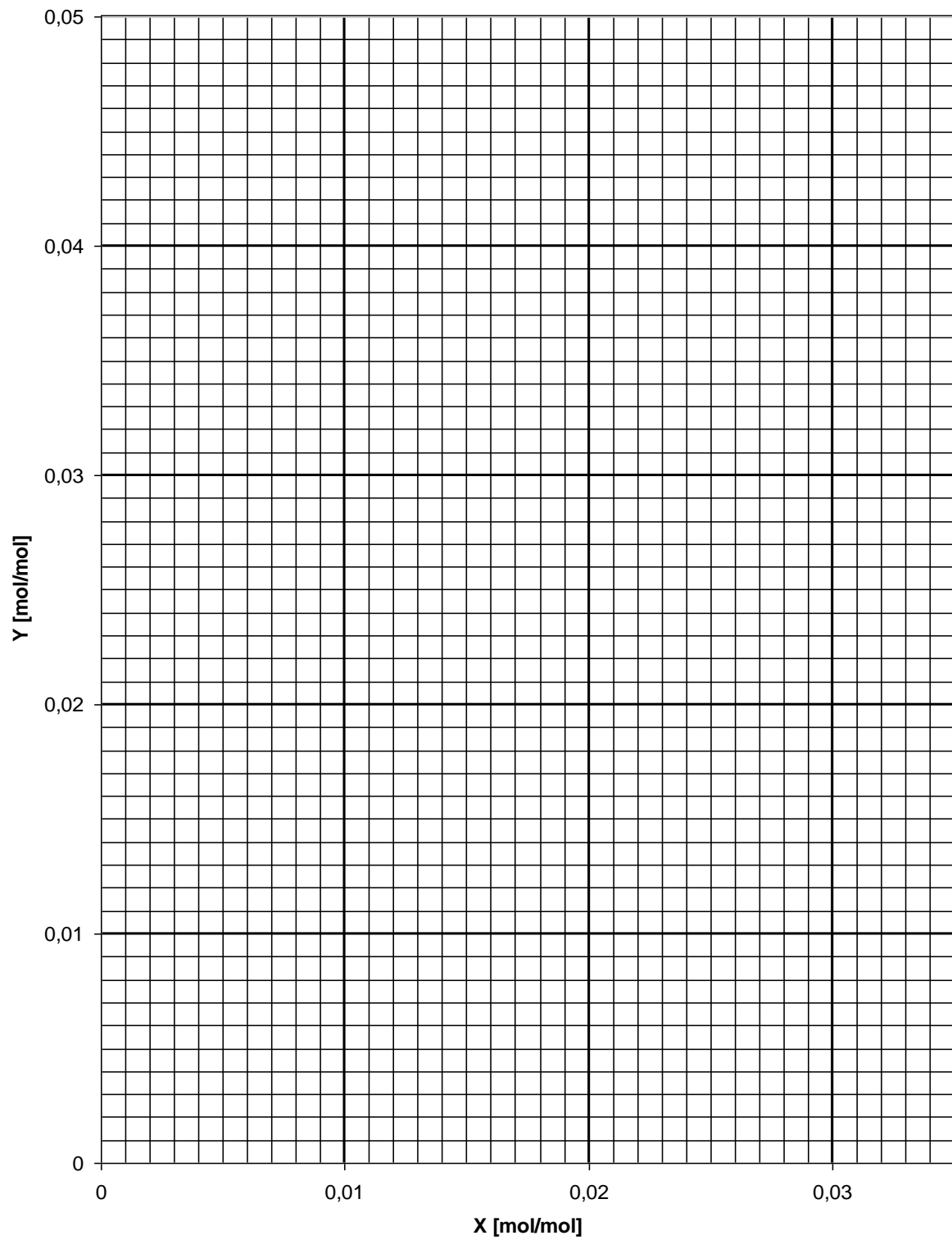
### Equilibrium data:

Temperature [°C]	Henry coefficient [bar]
20	0.73
25	0.96
30	1.23
35	1.55

### Further assumptions:

- Heat absorption by gas phase can be neglected.
- Gas phase behaves ideal.
- Non-volatile absorbent.
- The amount of ammonia dissolved in water at the column inlet can be neglected.
- Mass transfer resistance gas side  $\gg$  liquid side
- The liquid phase is ideal mixed
- Absorbed stream can be neglected in heat balance.



*Figure 5-2: X,Y-diagram*

### 5.3 HTU-NTU concept

In a first purification step a packed column is used to separate benzene from a gas stream using oil as absorbent. The column is operated in countercurrent mode, has a diameter of  $d = 470$  mm and is filled with Berl-Saddles-38mm.

- 5.3.1 Draw the operating line and the equilibrium line into the enclosed diagram. Determine the equation describing the operating line.
- 5.3.2 Determine the gas-sided number of transfer units  $NTU^{OG}$  using the method of Baker & White.
- 5.3.3 Determine the height of the packing needed for this separation task using the HTU-NTU concept.
- 5.3.4 Assume the mass transfer can be described by the equilibrium stage-model. Calculate the HETP-value of one theoretical stage for this case

#### Process data/property data:

Gas stream:	Inlet	$y_1 = 0.02$ ,	$Y_1 = 0.0204$ mol/(mol gas)
	Outlet	$y_2 = 0.00498$ ,	$Y_2 = 0.005$ mol/(mol gas)
	Average molar mass		$M_{\text{inert}} = 11$ kg/kmol $\frac{\text{kg}}{\text{kmol}}$
	Gas stream		$\dot{n}_{G,\text{inert}} = 0,01051$ $\frac{\text{kmol}}{\text{s}}$
	Operating conditions		$T = 26^\circ\text{C}$ , $p = 1.07 \cdot 10^5$ Pa
Liquid stream:	Inlet	$x_2 = 0.0272$ ,	$X_2 = 0.0280$ mol/(mol oil)
	Outlet	$x_1 = 0.1063$ ,	$X_1 = 0.1190$ mol/(mol oil)
	Average molar mass		$M = 260$ kg/kmol $\frac{\text{kg}}{\text{kmol}}$
	Liquid stream		$\dot{n}_{L,\text{inert}} = 1.787 \cdot 10^{-3}$ kmol/s
	Operating conditions:		$T = 26^\circ\text{C}$
	Density		$\rho = 840$ $\frac{\text{kg}}{\text{m}^3}$
Packaging data:	Specific interfacial area		$a_{\text{eff}} = 37.4$ m <sup>2</sup> /m <sup>3</sup>
Equilibrium line:	$Y = 0.125 * X$		
Mass transfer coefficients:	$k^{OG} * c^{OG} = 0.00183$ kmol/(m <sup>2</sup> s)		

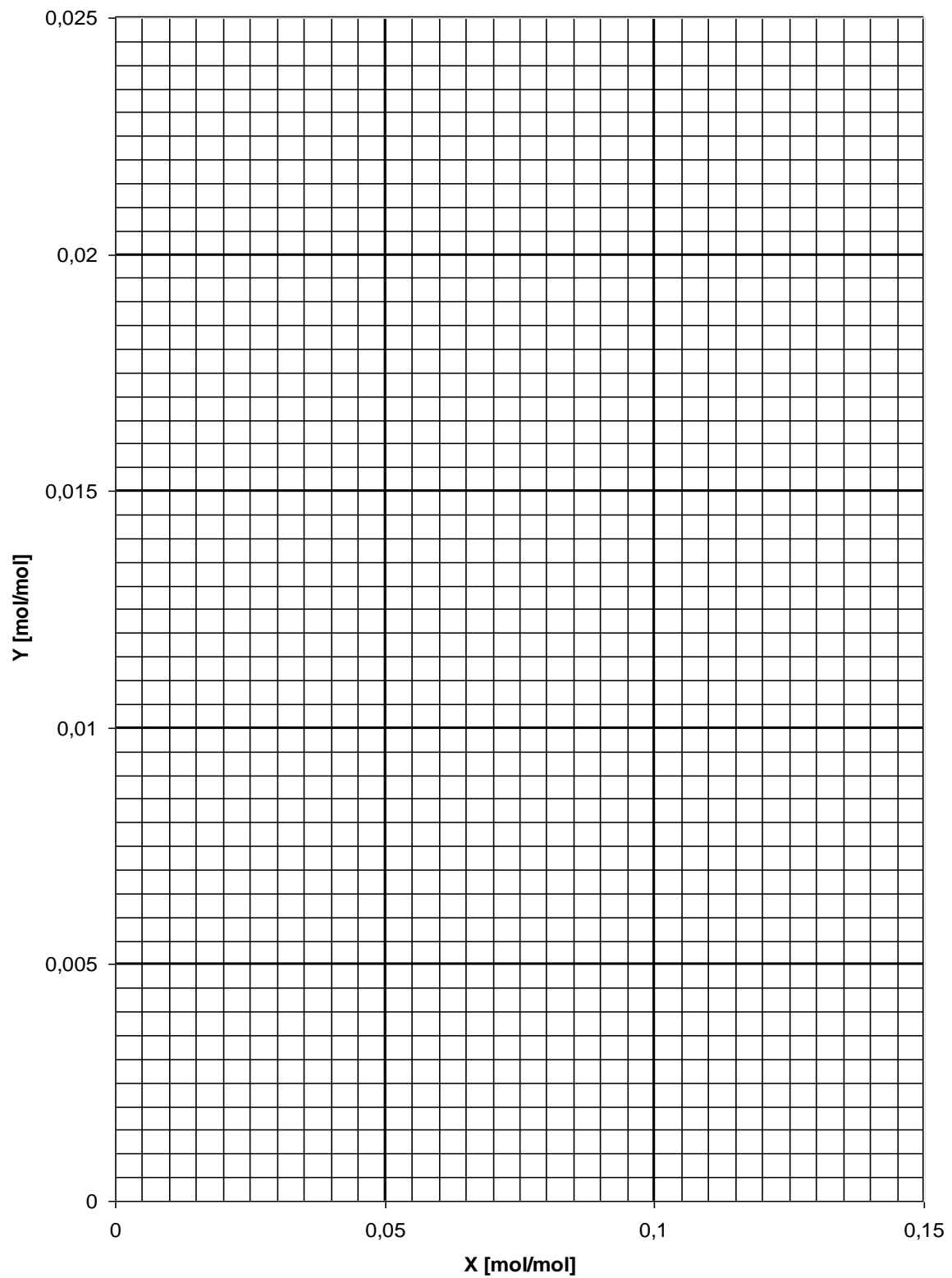


Figure 5-3: X,Y diagram