DISTILLATION

IN A PACKED COLUMN

1. GENERAL

Distillation is discussed for example in McCabe et al. (1993) in section 18, pages 521-587 and packed columns and -calculations are discussed in section 22 on pages 686-737. Only continuous distillation is handled. Batch distillation, which is time dependent, does not belong to this subject of matter.

Distillation as a continuous and industrial unit operation takes usually place in one device, which is called a distillation column. Sometimes two or more columns can be used to obtain the wanted result. Distillation separates components from each other and it's based on the different vapor pressures of components. A simplified schema of distillation process is shown in figure 1.
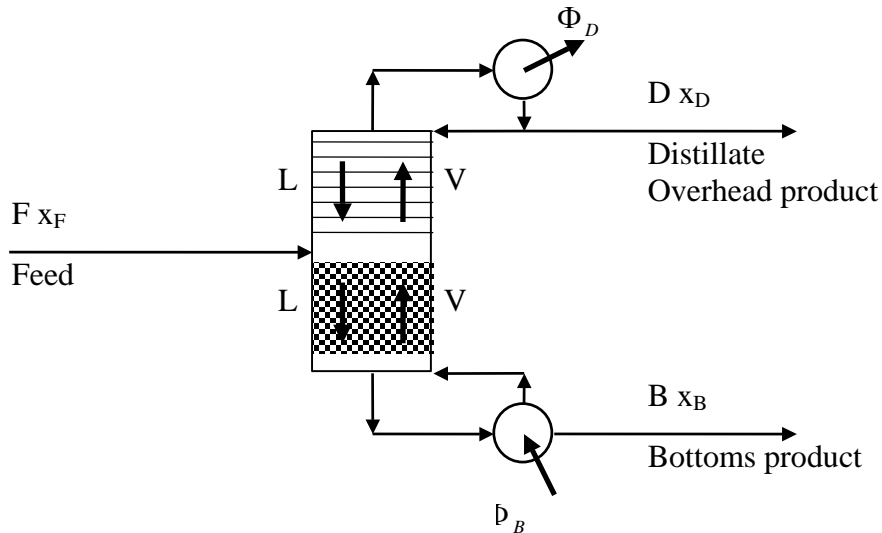
Figure 1. Simplified schema of distillation process.

In practice, continuous distillation occurs in distillation columns where vapor phase V vaporized in a reboiler by adding a heat flux Φ_B goes upwards and liquid phase L (from condenser by subtracting heat flux Φ_D) goes downwards. In order to increase the contact area between the two phases, the column is not empty but there are usually either distillation trays or packing. In figure 1 there is an unlikely case where the enriching section (the upper part) is a tray column and stripping section (the lower part) is a packed column.

Phases of the same level in the column have different concentrations, which also differ from the equilibrium concentration and therefore the mass transfer takes place between the two phases (actually due to their chemical potentials).

2. THEORY

A mixture of water and methanol is distillated in this work. The mixture is very nonideal and therefore the mass transfer is not equimolar. So, the flow rates inside the column are not constant. Also, the physical equilibrium is so nonideal, that the relative volatility varies significantly.

In order to calculate the height of the packing layer, the flow rates and concentrations of products and feeds, inner flow rates of the column and the phase equilibrium must be known. The flow rates and concentrations of the feed and product flows are obtained from the balances.

The schema of the laboratory equipment is shown in appendix 1. From appendix 1 it can be seen that the equipment consists only of one part, into which top the feed is led. So, the column corresponds to the bottom of a column shown in figure 1. The column is thus a stripping column.

Heating is done with indirect steam heating. The vaporizer is a (natural-circulation) thermosiphon, where the concentration of the outlet vapor is the same as the concentration of the inlet liquid feed, which is the same as the concentration of the bottom product of the column. In this case the vaporizer is NOT an ideal stage. The condenser of the column is a total condenser.

2.1 NOTATIONS

Behaviour of the lightest component or the component, which has the lowest boiling point and greatest vapor pressure, is usually studied in distillation, especially in binary case. This component is marked with A. So, in this case, methanol is component A. Though, this is not usually marked, so concentration without a subscript is the concentration of component A, and $x_F = x_{A,F}$.

From this follows that $x_D > x_B$ and the column is a stripping column for component A. Hence the operating line is below the equilibrium line. The concentration of component A decreases in liquid phase when moving downwards in the column.

2.2 PHASE EQUILIBRIUM

Phase equilibrium of methanol-water mixture is shown in appendix 3. Concentration data of liquid and gas phases at certain temperatures are based on measurements.

In order to keep all the calculations simple, the concentration of the gas phase should be a continuous and smooth function of the concentration of the liquid phase. A simple one is the relative volatility, which is defined as

$$\alpha \equiv \frac{y(1-x)}{x(1-y)} \tag{1}$$

So, the equilibrium line equation is

$$y^* = g(x) = \frac{\alpha x}{1+(\alpha-1)x} \qquad\qquad x^* = g^{-1}(y) = \frac{y}{\alpha+(1-\alpha)y} \tag{2}$$

This equation is advantageous because both $x \subset [0,1] \Rightarrow y^* \subset [0,1]$ and $y \subset [0,1] \Rightarrow x^* \subset [0,1]$ hold true. Therefore equations (2) are used in calculations.

As seen in Appendix 3, the relative volatility α is not constant so the equilibrium data is fitted to a second-degree polynomial with least squares method (not shown in appendixes). The result is

$$\alpha = 7.836 - 12.22x + 7.749x^2 \tag{3}$$

2.3 MASS TRANSFER RATIO

Mass transfer ratio z is a property of the process, not a physical property of the substances. Still, if assumed that

1. No heat losses.
2. All flows are saturated.
3. Enthalpy of saturated liquid and vapor is a linear function of the concentration of component A.

then the mass transfer ratio is constant. It can be calculated from equation

$$z = -\frac{I_B - i_B}{(I_A - I_B) - (i_A - i_B)} = +\frac{I_B - i_B}{(I_B - i_B) - (I_A - i_A)} = +\frac{r_B}{r_B - r_A} \tag{4}$$

Equation (4) requires that enthalpy of saturated mixture is linearized by:

$$I = (I_A - I_B)y + I_B \tag{5}$$

$$i = (i_A - i_B)x + i_B \tag{6}$$

where I_A, I_B, i_A and i_B are arbitrary parameters and they may be the enthalpies of pure compounds (which is the easists way of linearization). Notice that subscripts in equations (5) and (6) refer to components and not, for example, to bottom product. Enthalpies of methanol-water mixture are given in Appendix 4.

2.4 BALANCES OF THE REBOILER

Since all the flows at the top of the column are not saturated in this laboratory work (reflux and feed to the top are cold) it is easier to study the bottom of the column.

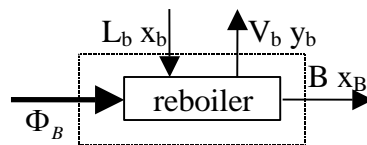**Figure 2. Bottom of the column.**

The balance area of the bottom of the column is shown in figure 2. The balances are

Total $$L_b = V_b + B \tag{7}$$

Component A(methanol) $$L_b x_b = V_b y_b + B x_B \tag{8}$$

Heat or enthalpy $$L_b i_b + \Phi_B = V_b I_b + B i_B \tag{9}$$

B, x_B, and heat Φ_B are known.

2.4.1 Siphon

The reboiler in this laboratory equipment is a siphon, in which case the concentration of vapor fed back to the column is the same as the concentration of the bottom product, so $y_b = x_B$ and material balance of component A (8) gives $x_b = y_b = x_B$.

Balance equation reduces to:

Total $$L_b = V_b + B \qquad (10)$$

Component A(methanol) $$L_b x_B = V_b y_B + B x_B \qquad (11)$$

Heat or enthalpy $$L_b i_B + \Phi_B = V_b I_B + B i_B \qquad (12)$$

Clearly, the heat balance (12) gives

$$V_b = \frac{\Phi_B}{I_b - i_B} \qquad (13)$$

Enthalpies of liquid and gaseous methanol-water mixture are given in Appendix 4. When V_b is calculated, L_b is obtained from the total balance.

2.5 OPERATING LINE

The operating line equation is generally:

$$y = f(x) = \frac{L}{V} x + \frac{\Delta C}{V} \qquad (14)$$

Notice that this equation is a straight line only if L and V or their ratio is constant. Here ΔD and ΔC are net flows upward in the column, so the balance of the reboiler gives

$$\Delta D = -B \qquad (15)$$

$$\Delta C = -B x_B \qquad (16)$$

Operating line equation then becomes to:

$$y = \frac{L}{V} x + \frac{-B x_B}{V} \qquad (17)$$

Substituting the values of the bottom of the column gives:

$$y = \frac{L_b}{V_b} x_B + \frac{-B x_B}{V_b} = \frac{L_b x_B - (L_b - V_b) x_B}{V_b} = x_B \qquad (18)$$

Hence the operating line intersects the point (x_B, x_B).

2.5.1 Calculation

If the mass transfer ratio can be assumed as a constant, the operating line can be calculated as in the following procedure:

1. Choose $\qquad x \qquad\qquad\qquad\qquad\qquad y$

2. Calculate $\quad L = \dfrac{z - x_a}{z - x} L_a = \dfrac{z - x_b}{z - x} L_b \qquad\qquad V = \dfrac{z - y_a}{z - y} V_a = \dfrac{z - y_b}{z - y} V_b \qquad$ (19)

3. Calculate $\quad V = L + \Delta D \qquad\qquad\qquad\qquad L = V - \Delta D \qquad$ (20)

4. Calculate $\quad y = \dfrac{L}{V} x + \dfrac{\Delta C}{V} \qquad\qquad\qquad x = \dfrac{V}{L} y - \dfrac{\Delta C}{L} \qquad$ (21)

2.5.2 Operating Line in the Laboratory Column

Flow chart of the laboratory column looks as in figure 3. The column consists only of stripping section, so the operating line is as in figure 4.
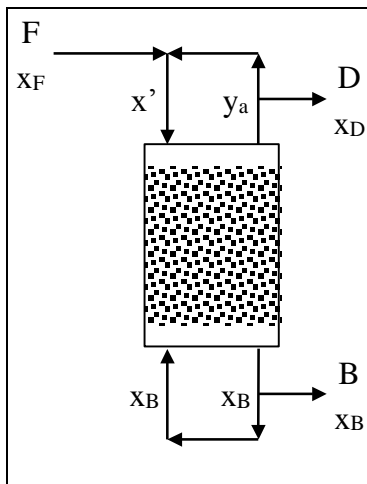
Figure 3. Flow chart of the laboratory column
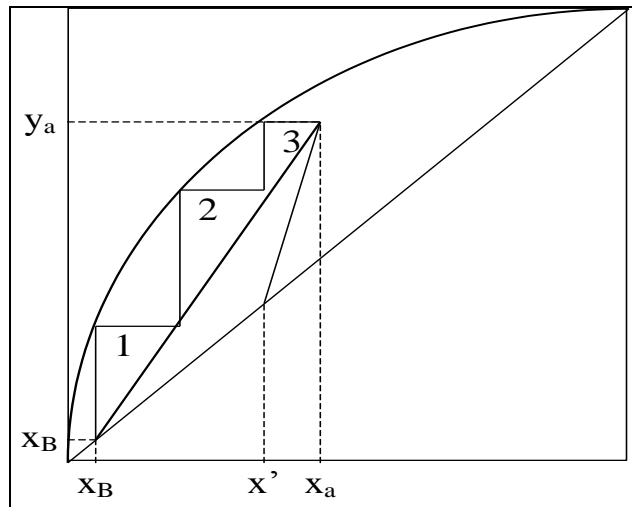
Figure 4. Operating line of the laboratory column

Notice that

- condenser is a total condenser, so $y_a = x_D$.
- feed is added to reflux, so the concenration x' of the liquid fed to the column is the weighted mean value of the concentrations of the feed and distillate.
- Reboiler is a (natural-circulation) thermosiphon, so the concentration of the vapor fed to the column is same as the concentration of the bottom product x_B.

2.6 HEIGHT OF THE PACKING LAYER

Height *h* of the packing layer can be calculated in two ways:

$$h = N \cdot HETP \qquad (22)$$

$$h = NTU \cdot \overline{HTU} \qquad (23)$$

According to equation (22) the height is a product of number of ideal stages in an *ideal stage model* and Height Equivalent to a Theoretical Plate (*HETP*).

Disadvantage of HETP method is that *HETP* has to be determined experimentally to each packing material (type and size) and to every mixture in different flow conditions separately. *HETP* varies highly as a function of flow rates and concentrations, so it is not a constant.

Equation (23) defines the height of a packing layer as a product of NTU (Number of Transfer Units) and $\overline{HTU}$ (average Height of Transfer Unit). It's possible to consider either vapor or liquid phase when using mass transfer coefficients in calculations, and notations are then y and x, respectively. Subscripts Oy and Ox refer to overall mass transfer coefficients, which can also be applied in calculations.

For practical reasons, NTU is marked with N and $\overline{HTU}$ with H from now on. As a concequence, equation (23) shortens to

$$h = N_{Oy} \cdot \overline{H}_{Oy} = N_y \cdot \overline{H}_y = N_{Ox} \cdot \overline{H}_{Ox} = N_x \cdot \overline{H}_x \qquad (24)$$

Calculations should be made at that phase where the resistant to mass transfer is greatest. The definition of the height of mass transfer unit (shown later on) includes the ratio of mass transfer coefficient and molar flow. Mass transfer coefficients vary as a function of molar flow. On the other hand, the ratio of molar flow and mass transfer coefficient fluctuates less and hence an assumption of constant H value is justifiable (compared to HETP). As marked in Equation (24), average heights of mass transfer unit can thus be used to calculate the height of final packing.

HTU method to calculate **constant contact** requires the *model of constant contact* and the calculation of mass transfer units *NTU* and average height of mass transfer unit $\overline{HTU}$. Since it's a case of a very nonideal system, partial mass transfer coefficients should be used. The following equations are commonly written for both stripper and absorber in vapor and liquid phase.

$$h = \overline{\left(\frac{V}{k_y aA}\right)} \int_{yb}^{ya} \frac{z}{(z-y)} \frac{1}{\phi_y} \frac{dy}{|y_i - y|} = \overline{H}_y \cdot N_y \qquad h = \overline{\left(\frac{L}{k_x aA}\right)} \int_{xb}^{xa} \frac{z}{(z-x)} \frac{1}{\phi_x} \frac{dx}{|x - x_i|} = \overline{H}_x \cdot N_x \qquad (25)$$

The following quantities are defined: *the (average) height of mass transfer unit in vapor phase* $\overline{H}_y$, *the (average) height of mass transfer unit in liquid phase* $\overline{H}_x$, *number of mass transfer units in vapor phase* N_y *and number of mass transfer units in liquid phase* N_x. These equations can be applied both for strippers and adsorbers, because they contain an absolute value of a difference (y-y_i) or (x-x_i).

2.7 NUMBER OF IDEAL STAGES AND HETP

The number of ideal stages is obtained by stepping off the area between operating line and equilibrium line. This can be made graphically as Figure 3b shows or numerically by a spreadsheet.

HETP depends both on the thermodynamical and physical properties of components and on the properties of process. According to McCabe et al. (1993, p. 731) HETP typically lies between 0,3m and 0,6 m. Small columns may have even smaller values. Because a small HETP means a great efficiency (a packing layer functions well) and big HETP value quite the opposite, we'll assume HETP to be 0,6 m.

2.8 HEIGHT OF MASS TRANSFER UNIT

HTU method for a **constant contact process** is based on an assumption that the used height of a mass transfer unit remains constant. They are written as ($\overline{H}_y$ as an axample)

$$\overline{H}_y = \overline{\left(\frac{V}{k_y aA}\right)} \tag{25}$$

The cross section area of equipment A and the density of mass transfer area a (which is usually assumed to be the same as the density of the area of packings) are constant in the process and equipment. We'll make an assumption that total molar flow V is not constant in the process. This makes $\overline{H}_y$ to vary, too. The mass transfer coefficient of a phase is almost directly proportional to the flow rate of the phase and therefore to the Reynolds number, too. From this follows, although V is not a constant, the ratio of total molar flow and mass transfer coefficient (L/k_x or V/k_y) is. This explains the first statement of this chapter.

The molar flow V (or L) and also the cross section area A has to be known. Notice that the quotient V/A (or L/A) means superficial velocity, not the actual velocity in a packing layer. Since the dimension of the height of mass transfer unit is m and the dimension of the density of mass transfer area is m²/m³ = 1/m, the ratio $(V/A)/k_y$ must be unitless.

- $[k'] = $ m/s $\Rightarrow [V/A] = $ m/s $\Rightarrow [V] = $ m³/s.
- $[k] = $ mol/m²/s $\Rightarrow [V/A] = $ mol/m²/s $\Rightarrow [V] = $ mol/s.

2.8.1 Mass Transfer Coefficient of a Phase

Mass transfer coefficients are *always* defined from empirical correlations based on experimental data (convection correlations). There is no theoretical way to calculate mass transfer coefficient or the density of a mass transfer area! Correlations (mass and heat transfer) usually have a form of

$$Sh = bRe^n Sc^m \qquad\qquad St_M = bRe^{n-1} Sc^{m-1} \tag{26}$$

$$Nu = bRe^n Pr^m \qquad\qquad St_H = bRe^{n-1} Pr^{m-1} \tag{27}$$

Due to the analogy between mass and heat transfer, parameters b, n and m are the same. Notice that these empirical correlations are only suitable for the same circumstances at which the measurements are carried out.

Unitless quantities can be classified as follows:
1. Describing flow mode: *Re*
2. Describing the properties of fluid: *Pr, Sc*
3. Including heat transfer coefficient, h: *Nu, St$_H$, j$_H$*
4. Including mass transfer coefficient, k: *Sh, St$_M$, j$_M$*

Expressions of them are (Here L means characteristic dimension and D is diffusivity.)

$$\mathrm{Re} \equiv \frac{Lu\rho}{\eta} \tag{28}$$

$$\text{Pr} \equiv \frac{c_p \eta}{\lambda} \quad \text{Sc} \equiv \frac{\eta}{D\rho} \tag{29}$$

$$\text{Nu} \equiv \frac{hL}{\lambda} \quad \text{St}_H \equiv \frac{Nu}{\text{Re Pr}} = \frac{h}{\rho u c_p} \qquad j_H \equiv \text{St}_H \, \text{Pr}^{2/3} \tag{30}$$

$$\text{Sh} \equiv \frac{kL}{D} \quad \text{St}_M \equiv \frac{Sh}{\text{Re Sc}} = \frac{k}{cu} \qquad j_M \equiv \text{St}_M \text{Sc}^{2/3} \tag{31}$$

As an example, an Onda correlation is presented (Taylor and Krishna, 1993, p. 355 or Perry et al. 1997, p. 5-75). It's derived for mass transfer between two fluid phases. It states that the following equation gives the mass transfer coefficient of vapor phase (k_y, m/s).

$$k_y = A \cdot \text{Re}_y^{0,7} \, Sc_y^{1/3} (a_p d_p)^{-2.0} (a_p D_y) \tag{32}$$

where

$$\text{Re}_y = \frac{\rho_y u_y}{\eta_y a_p} \tag{33}$$

where u_y = *superficial velocity* ja a_p = *density of the area of packing* (m^2/m^3). Parameter $A = 5.23$, when nominal packing size $d_p > 1/2$ in, and parameter $A = 2.0$, when $d_p < 1/2$ in.

Mass transfer coefficient k_x (m/s) is calculated from equation

$$k_x = 0.0051 \cdot \text{Re}_x^{2/3} \, Sc_x^{-0.5} (a_p d_p)^{0.4} \left(\frac{\eta_x g}{\rho_x} \right)^{1/3} \tag{34}$$

where

$$\text{Re}_x = \frac{\rho_x u_x}{\eta_x a} \tag{35}$$

where u_x = *superficial velocity* ja a = *density of the mass transfer area* (m^2/m^3). A preliminary assumption can be $a = a_p$. Notice that u_x is going to be small, because the velocity is calculated taking the free cross section area of the whole tower. It has to be taken into account that mass transfer area isn't necessarily equal to packing area; perhaps only a part of surface is wetted (then $a<a_p$). On the other hand mass transfer can occur due to the drops between the packing material (then $a>a_p$).

Surface areas of different packing types are given for example in McCabe et al. 1993 (Table 22.1, p.689). Perry et al. (1984, s. 18-34) states that accuracy of correlation is appr. $\pm$ 20 %. Appendix 6 shows an example how to calculate partial mass transfer coefficients.

2.9 NUMBER OF MASS TRANSFER UNITS

The following equations show how to calculate the number of mass transfer units.

$$N_y = \int_{yb}^{ya} \frac{z}{(z-y)} \frac{1}{\phi_y} \frac{dy}{|y_i - y|} \qquad N_x = \int_{xb}^{xa} \frac{z}{(z-x)} \frac{1}{\phi_x} \frac{dx}{|x - x_i|} \tag{36}$$

where

$$\phi_y = \frac{(z-y)-(z-y_i)}{z \ln \dfrac{z-y}{z-y_i}} \qquad\qquad \phi_x = \frac{(z-x)-(z-x_i)}{z \ln \dfrac{z-x}{z-x_i}} \tag{37}$$

where

$$z = \frac{N_A}{\sum N_i} = \frac{N_A}{N_\Sigma} \tag{38}$$

That is, the concentration difference between operating line and the interface of two phases has to be integrated. Concentrations in the function to be integrated are illustrated in Figures 1a and 1b.
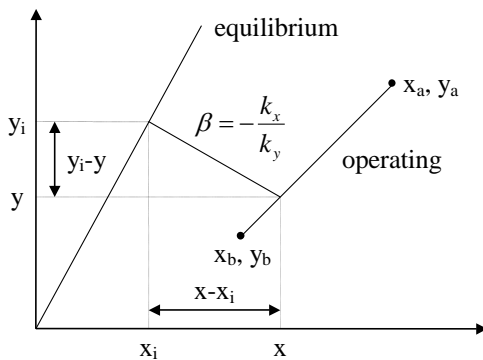
Figure 5. Stripper
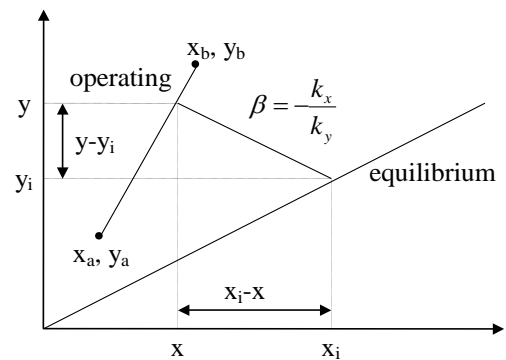
Figure 6. Absorber

Concentration at the interphase of phases can be calculated from bulk concentration and mass transfer coefficients as in equation (39).

$$\beta = \frac{y-y_i}{x-x_i} = -\frac{k_x}{k_y} \tag{39}$$

Equation (39) is based on zero volume at the interface, so neither energy nor matter can accumulate there. Material balance over the interface gives IN = OUT in all circumstances (steady state and dynamical). For an absorber it can be written:

$$\frac{1}{\phi_y} k_y (y-y_i) = \frac{1}{\phi_x} k_x (x_i - x) \tag{40}$$

The coefficient of relative velocity ϕ is not a constant in the process, but changes as a function of concentration. If we assume that the mass transfer coefficients of phases are constant in a process, it may be meaningful to ignore ϕ values, which leads to equation (39). Analogous derivation also holds for stripper. In practise numerical integration is needed. Writing a definite integral gives.

$$N_y \approx \sum_k \frac{z}{(z-y_k)} \frac{1}{\phi_{y,k}} \frac{\Delta y_k}{\left| y_k - y_{i,k} \right|} \qquad\qquad N_x \approx \sum_k \frac{z}{(z-x_k)} \frac{1}{\phi_{x,k}} \frac{\Delta x_k}{\left| x_k - x_{i,k} \right|} \tag{41}$$

where

$$\phi_{y,k} = \frac{(z - y_k) - (z - y_{i,k})}{z \ln \dfrac{z - y_k}{z - y_{i,k}}} \qquad\qquad \phi_{x,k} = \frac{(z - x_k) - (z - x_{i,k})}{z \ln \dfrac{z - x_k}{z - x_{i,k}}} \tag{42}$$

where

$$z = \frac{N_A}{\sum N_i} = \frac{N_A}{N_\Sigma} \tag{38}$$

Notice that ϕ is not constant in a process although z is. Let's consider calculations in vapor phase. In Figure 4 an arbitrary concentration y is marked on the operating line. This concentration determines unambiguous liquid concentration x: $x=f^{-1}(y)$, where $y=f(x)$ is the equation of operating line. This point of operating line (x_k, y_k) corresponds to an interfacial point $(x_{i,k}, y_{i,k})$. Point $(x_{i,k}, y_{i,k})$ must be solved iteratively: $x_{i,k}$ corresponding to point (x_k, y_k) is guessed and $y_{i,k}$ is calculated from the equation of equilibrium line. Estimation is checked by slope β (calculated from the points (x_k, y_k) and $(x_{i,k}, y_{i,k})$). Analoguos calculation can be done in liquid phase. Following guidelines are given for integration.

1. Integration interval is divided into subintervals (Δy or Δx)
2. From every subinterval $Δy_k$ and $Δx_k$ one variable value y or x is chosen → y_k or x_k.
   It can be taken from the beginning or end of the subinterval (or as an average)
3. For every subinterval the value of the function to be integrated is calculated using the chosen y or x.
3.1. Operating line point (x_k, y_k) corresponding to variable value is calculated.
   This requires calculation of flows V_k and L_k.
3.2. Interfacial point $(x_{i,k}, y_{i,k})$ corresponding to the point of operating line is calculated
   This requires iteration by means of quantity β
3.3. The value of the whole function to be integrated is calculated and then it's multiplied by $Δy_k$ or $Δx_k$.
4. Summing all up.

Spreadsheets are the best way to proceed. Appendixes include a version done by MS-EXCEL. However, no ABS function is used to make the stages easier to follow. The shorter the integration subintervals Δy or Δx are, the more accurate is the result. In the example there are 10 intervals, which results in the length of

$$\Delta y = \frac{|y_a - y_b|}{10} \qquad\qquad \Delta x = \frac{|x_a - x_b|}{10} \tag{43}$$

Of course, a more accurate result is easily obtained by setting the length of Δy or Δx shorter in the spreadsheet. Values are taken from the end points of each subinterval. It's not meaningful to calculate the average values for subintervals because shortening Δy and Δx is a faster way to improve performance.

7.10.2015

The direction of calculations has been towards higher concentration. That is, the initial point has been:

$$y_0 = MIN(y_a; y_b) \qquad x_0 = MIN(x_a; x_b) \qquad (44)$$

In order to simplify the calculations and make them more illustrative, equilibrium line is used. Constant relative volatility is a quantity to describe equilibrium and it has a form of

$$\alpha \equiv \frac{y(1-x)}{x(1-y)} \qquad (45)$$

But calculations hold for an arbitrary explicit equilibrium line. Interfacial concentrations are solved with the optimization function SOLVER in MS-EXCEL. The target has been to minimize the sum of all squares that have been taken from differences between β (b) and β' (b'). The former is calculated by mass transfer coefficients and the latter by points (x_k, y_k) and $(x_{i,k}, y_{i,k})$.

3. EQUIPMENT USED IN THE LABORATORY

3.1 COLUMN

A schematic representation is shown in Appendix 1. It illustrates, that the column consists of only one part, and feed enters the column at the top of it. Thus the column corresponds to the stripping section (the lower part of the column) in Figure 1. That makes it a stripping column.

The column is filled with Intalox-saddles of 1 in. The inner diameter reaches 0,16 m and packing height is about 1,7 m. There is a total condenser attached to the equipment. Heating is organized indirectly with steam so heating agent will not contact the fluids in the process.

The bottom part of the column has a separating wall, reaching 210 mm above it. Inlet to the reboiler is thus on the other side than the glass window. Bottoms product recovery is on the same side of the glass window. Due to this, the lines leading to the reboiler and bottoms product pump have been connected with each other.

3.2 ROTAMETERS

The following rotameters are fixed to the system:
- Feed
- Bottom
- Recycle
- Distillate

3.3 THERMOMETERS

Equipment includes several thermometers attached to it:
- Steam
- Bottom
- Top
- Feed/recycle

3.4 MEASURING EQUIPMENT

In the laboratory there should be
- Refractometer with thermostate
- Scale
- Decanter made of plastic 3000 ml   1
- Decanters 50 ml   4
- Pipettes
- Lens paper / kitchen paper

In addition
- Stop watch   1
- Safety goggles and protective gloves if needed

4. OPERATING THE DISTILLATION COLUNM

4.1 ARRANGEMENTS

A mixture of methanol and water is distillated. Methanol is of technical quality.

WARNING!!!
Methanol is toxic! Beware of skin contact! Use protective gloves!

4.2 STARTING THE WORK

- Make sure that the feed tank contains solution.
- Check that the bottom tap of recovery tank is closed and the bottom tap of feed tank is open.
- Check the lines leading to the reboiler and bottoms product pump. The valve in the connecting line should be open.
- Ensure that there's liquid in the bottom of the column. If not, pump some feed into the column.
- Open the tap of cooling water for condenser and coolers (just one). Make sure that water is circulating all over.
- The valve for direct steam heating should be closed.
- Open the valve of steam line.
- Adjust the restriction of condensation valve of heating so that a bit of steam is left in addition to condensate. During the run control the situation.

4.3 TOTAL REFLUX EXPERIMENT

- When the column has heated up (the temperature at the top is over 80 ^{0}C), turn on the distillate pump and open the reflux valve a bit.
- Adjust the reflux so that level in the bottom of the column stays constant (distillate and bottoms are not collected).
- If the level starts to rise in the accumulator, some distillate has to be collected to maintain the constant level.
- Take notes every ten minutes:
    - liquid levels of accumulator and reboiler
    - temperatures of column at the top and at the bottom
    - reading of rotameter in the reflux line.
- Steady state is obtained clear enough, when notes have stayed constant for half an hour.
- When a steady state is achieved, the following measurements are carried out:
  1. Measure the mass flow of condensate originating from heating agent. (Collect it in a container for ten minutes.)
  2. Document the pressure of heating vapor.
  3. Measure the reflection index of reflux and bottoms product.
  4. Document the reading of rotameter in the reflux line.
- Show your results to assistant before you continue.

4.4 FEED TEST

- Turn on the feed pump and open the feed tap. Adjust the flow as wanted.
- When the liquid level in the accumulator and at the bottom of the column begins to rise, turn on the distillate and bottoms product pumps.
- Keep the level constant in the accumulator and at the bottom of the column by controlling distillate and bottoms product flows.
- Mark down the levels of accumulator and reboiler, temperatures at the top and at the bottom of the column, and readings of all rotameters every ten minutes.

- After half an hour of constant readings the steady state is assumed to be reached.
- At the steady state, the following things should be done.
   1. Measure the mass flow of condensate originating from heating agent. (Collect it in a container for ten minutes.)
   2. Document the pressure of heating vapor.
   3. Measure the refraction index of feed, distillate, reflux and bottoms product.
   4. Document all rotameter readings.
- Let the assistent see your results.

4.5 FINISHING THE WORK

- Show all the data to your assistent and after a permission:
   1. Close the valve of steam line.
   2. Close the taps and turn off the pumps.
   3. Open the tap under the recovery tank.
   4. Leave the cooling on. Assistant will close the valve when the column has a temperature low enough.
   5. Clean up the surroundings.

5. REPORT

The following parts should be included in the report (each having an own chapter with a well-defined title).

1. Show the stabilization of the column graphically.

2. Change the mass flows to molar flows (all calculations are done in the molar fraction coordinates).

3. Fit the measured material balances with the MS-EXCEL program tasmays.xls.

4. Calculate the mass transfer ratio, which is assumed to be constant.

5. Calculate the end points of operating line:
- Quantities V_b ja L_b from the balances of reboiler
- Quantities V_a ja L_a.
- Another end point of the operating line (x_a, y_a).

6. Height of the packing layer h = N * HETP
- Draw diagonal, equilibrium curve and operating line (assumed to be a straight line) on a paper
- Determine the number of ideal stages N by stepping off.
- Calculate HETP for this experiment.
- Assume that HETP is 0.6 m. Calculate then h.

7. The average height of mass transfer unit $\overline{H}_y$ and $\overline{H}_x$.
- Calculate H both at the top and bottom parts of the packings and the average $\overline{H}$.
- Notice that physical and thermodynamical properties and flows change in the packed column.

8. Calculate the number of mass transfer units N

- Make the calculations at that phase where the resistance to mass transfer is greater (smaller mass transfer coefficient).
- Equilibrium line *g* is known.
- Operating line *f* is not straight, but mass transfer ratio *z* is constant.
- You can assume the relative velocity coefficient to be one, $\phi = 1$.

9. Calculate then h = N·H

10. Incorrect estimate
- No numerical estimates.
- Pay special attention to gross and systematic errors and general estimation of results.

11. Discussion

6. NOMENCLATURE

a density of mass transfer area, m^2/m^3

a_p density of surface area of packing, m^2/m^3

a_w density of wetted surface area of packing, m^2/m^3

B bottoms flow, mol/s

c molar density, mol/m^3

D distillate flow, mol/s

F feed flow, mol/s

d_p characteristic gauge, nominal packing size, m

D_{AB} binary diffusion coefficient of substances A and B, m^2/s

f operating line

g gravitational acceleration, m/s^2

g equilibrium line, $y^* = g(x)$ ja $x^* = g^{-1}(y)$

h height of a balance area, total height of a packing layer, m

i molar specific enthalpy of liquid, kJ/mol

I molar specific enthalpy of vapor, kJ/mol

$\overline{H}_x$ average height of partial mass transfer unit in liquid phase, m

$\overline{H}_y$ average height of partial mass transfer unit in vapor phase, m

k_x partial mass transfer coefficient in liquid phase, $mol/(m^2s)$

k_y partial mass transfer coefficient in vapor phase, $mol/(m^2s)$

k'_x partial mass transfer coefficient in liquid phase, m/s

k'_y partial mass transfer coefficient in vapor phase, m/s

L total molar flow of liquid, mol/s

N_x number of partial mass transfer units in liquid phase, unitless

N_y number of partial mass transfer units in vapor phase, unitless

r molar heat of evaporization, J/mol

u velocity, m/s

V total molar flow of vapor, mol/s

x molar fraction of liquid, unitless

y molar fraction of vapor, unitless

z mass transfer ratio of a component, unitless

Greek Letters

ΔC component net flow, directed towards phase V, mol/s
ΔD total net flow, directed towards phase V, mol/s
ϕ relative velocity coefficient, unitless
η viscosity, Pas $=$Ns/m^2 = kg/ms
ρ density, kg/m^3

Subscripts
A   component A
B   bottoms product, component B
D   distillate
F   feed

Superscripts
y^* equilibrium value of quantity y

7. REFERENCES

McCabe, W.L., Smith, J.C. and Harriot, P., Unit Operations of Chemical Engineering, 5th ed., McGraw-Hill, 1993.
Taylor, R. and Krishna, R., Multicomponent Mass Transfer, John Wiley & Sons, 1993

8. APPENDIXES

1. Equipment figure
2. Excel Sheet for fitting the balances
3. Equilibrium data for the mixture of methanol-water
4. Enthalpy data for the mixture of methanol-water
5. Refraction index data for mixture of methanol - water
6. H -calculation
7. N_y -calculation
8. N_x -calculation

Schematic of packed column
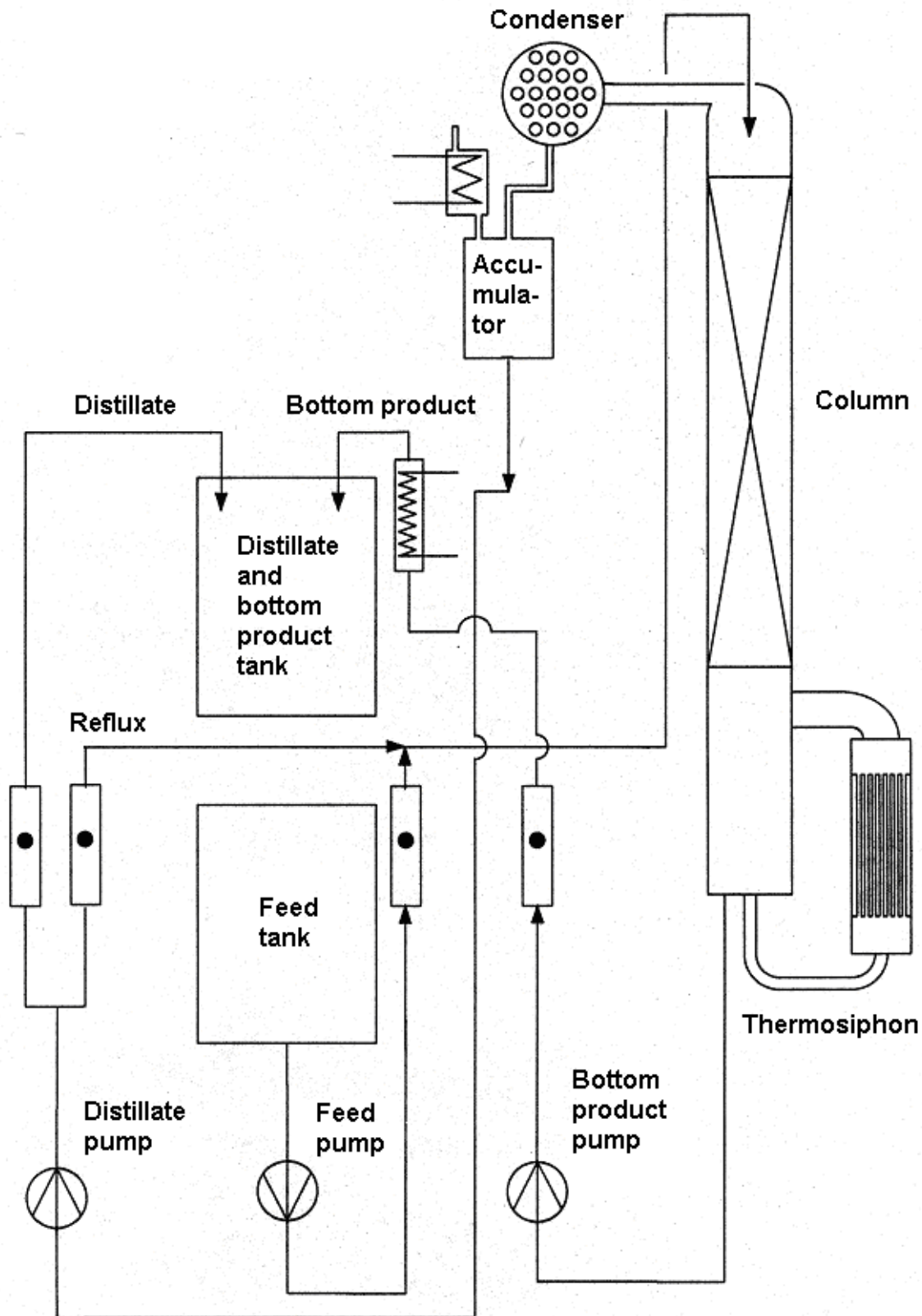
MATCHING THE BALANCES — **BINARY DISTILLATION**

F → PROCESS → D, B

WEIGHTING COEFFICIENTS

	IN	OUT	
	F	**B**	**D**
TOTAL	1.0	1.0	1.0
x1	1.0	1.0	1.0
x2	1.0	1.0	1.0

MEASURED

	IN		OUT				IN-OUT
	F		**B**		**D**		
TOTAL	2.000		3.000		3.000		-4.000
x1	0.200	0.400	0.300	0.900	0.300	0.900	-1.400
x2	0.800	1.600	0.700	2.100	0.700	2.100	-2.600
SUMx	1.000		1.000		1.000		

MATCHED

	IN		OUT				IN-OUT
	F		**B**		**D**		
TOTAL	1.100		3.300		4.400		-6.600
x1	0.110	0.121	0.330	1.089	0.440	1.936	-2.904
x2	0.890	0.979	0.670	2.211	0.560	2.464	-3.696
SUMx	1.000		1.000		1.000		

RELATIVE DIFFERENCE

	IN	OUT	
	F	**B**	**D**
TOTAL	0.450	-0.100	-0.467
x1	0.450	-0.100	-0.467
x2	-0.113	0.043	0.200

WEIGHTING COEFFICIENT * SQUARE OF RELATIVE DIFFERENCE

	IN	OUT		
	F	**B**	**D**	
TOTAL	0.203	0.010	0.218	
x1	0.203	0.010	0.218	**SUM**
x2	0.013	0.002	0.040	0.915

Equilibrium of methanol-water mixture

	Measured		Calculated	Fitted			Calculated	
T	**x**	**y**	**a**	a'	(a-a')/a %		y'=f(a',x)	(y-y')/y %
100.0	0.0000	0.0000		7.84			0.0000	0.00
95.2	0.0293	0.1831	7.43	7.48	-0.80		0.1843	-0.65
94.5	0.0346	0.2107	7.45	7.42	0.34		0.2101	0.27
93.7	0.0406	0.2363	7.31	7.35	-0.56		0.2373	-0.43
92.8	0.0478	0.2652	7.19	7.27	-1.12		0.2674	-0.82
91.8	0.0557	0.2978	7.19	7.18	0.14		0.2975	0.10
90.9	0.0644	0.3265	7.04	7.08	-0.55		0.3277	-0.37
90.0	0.0737	0.3608	7.09	6.98	1.65		0.3570	1.06
89.2	0.0838	0.3861	6.88	6.87	0.14		0.3858	0.09
89.1	0.0948	0.4142	6.75	6.75	0.06		0.4141	0.04
78.8	0.2801	0.6621	5.04	5.02	0.30		0.6614	0.10
77.6	0.3004	0.6882	5.14	4.86	5.37		0.6762	1.74
77.6	0.3212	0.6882	4.66	4.71	-0.98		0.6903	-0.30
76.9	0.3435	0.7002	4.46	4.55	-1.98		0.7043	-0.59
76.2	0.3664	0.7178	4.40	4.40	0.00		0.7178	0.00
75.7	0.3909	0.7274	4.16	4.24	-2.04		0.7314	-0.55
75.1	0.4141	0.7428	4.09	4.10	-0.43		0.7436	-0.11
74.6	0.4391	0.7597	4.04	3.96	1.85		0.7563	0.45
74.0	0.4637	0.7668	3.80	3.84	-0.84		0.7683	-0.20
67.2	0.8457	0.9360	2.67	3.04	-14.01		0.9434	-0.79
66.6	0.8867	0.9632	3.34	3.09	7.57		0.9603	0.30
65.7	0.9293	0.9771	3.25	3.17	2.34		0.9766	0.05
64.6	1.0000	1.0000		3.36			1.0000	0.00

a'=alpha'=a+b*x+c*x^2
a= 7.836
b= -12.222
c= 7.749
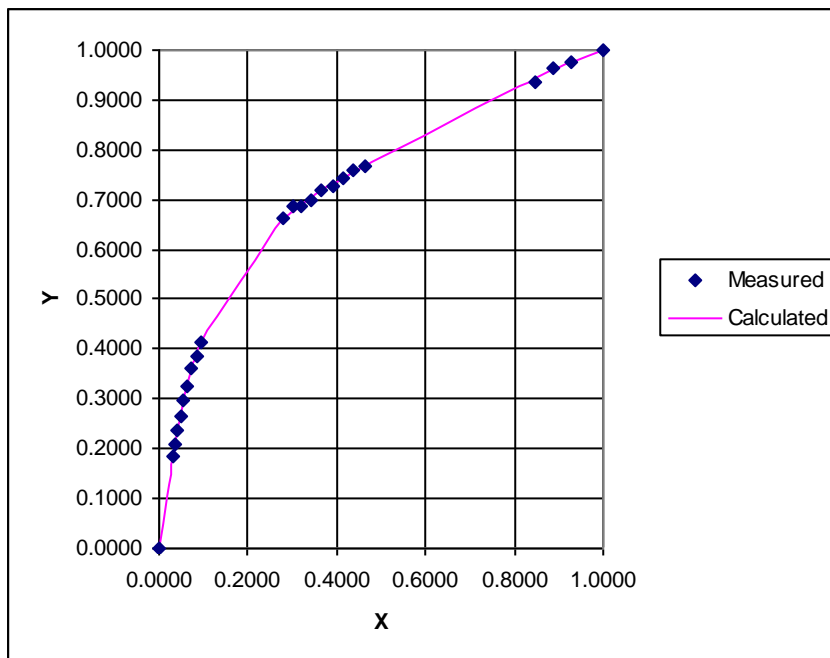
Reference: old work instructions.

Enthalpy of saturated methanol-water mixture

	Enthalpies	
x , y	i	I
mol-%	kJ/mol	kJ/mol
0	7.5	48.1
10	5.8	47.4
20	5.4	46.7
30	5.2	46.0
40	5.2	45.2
50	5.2	44.3
60	5.2	43.8
70	5.2	43.0
80	5.3	42.2
90	5.4	41.4
100	5.4	40.7

Enthalpies of pure compounds

i(A) =5.40   I(A) =40.70
i(B) =7.50   I(B) =48.10

Linearization by enthalpies of pure compounds

	Enthalpies						
x , y	i	I		i	Rel. D(i) %	I	Rel. D(I) %
mol-%	kJ/mol	kJ/mol		kJ/mol		kJ/mol	
0	7.5	48.1		7.5	0.0	48.1	0.0
10	5.8	47.4		7.3	25.7	47.4	0.1
20	5.4	46.7		7.1	31.1	46.6	0.2
30	5.2	46.0		6.9	32.1	45.9	0.3
40	5.2	45.2		6.7	28.1	45.1	0.1
50	5.2	44.3		6.5	24.0	44.4	0.2
60	5.2	43.8		6.2	20.0	43.7	0.3
70	5.2	43.0		6.0	16.0	42.9	0.2
80	5.3	42.2		5.8	9.8	42.2	0.0
90	5.4	41.4		5.6	3.9	41.4	0.1
100	5.4	40.7		5.4	0.0	40.7	0.0

Constants of linearization

Mx=-1.13   My=-7.45
Bx=6.09   By=48.16

Linearization by optimization

	Enthalpies						
x , y	i	I		i	Rel. D(i) %	I	Rel. D(I) %
mol-%	kJ/mol	kJ/mol		kJ/mol		kJ/mol	
0	7.5	48.1		6.1	33.8	48.2	0.1
10	5.8	47.4		6.0	3.1	47.4	0.0
20	5.4	46.7		5.9	8.6	46.7	0.1
30	5.2	46.0		5.8	10.6	45.9	0.2
40	5.2	45.2		5.6	8.5	45.2	0.0
50	5.2	44.3		5.5	6.3	44.4	0.3
60	5.2	43.8		5.4	4.1	43.7	0.2
70	5.2	43.0		5.3	2.0	42.9	0.1
80	5.3	42.2		5.2	2.1	42.2	0.0
90	5.4	41.4		5.1	6.0	41.5	0.1
100	5.4	40.7		5.0	8.1	40.7	0.0

Reference temperature: 0 C.
Reference: old work instructions

7.10.2015

Refraction index of methanol-water mixture in 20 ºC

MeOH x	measured t
0.000	1.3330
0.045	1.3355
0.095	1.3373
0.205	1.3412
0.275	1.3424
0.455	1.3424
0.625	1.3395
0.690	1.3382
1.000	1.3291
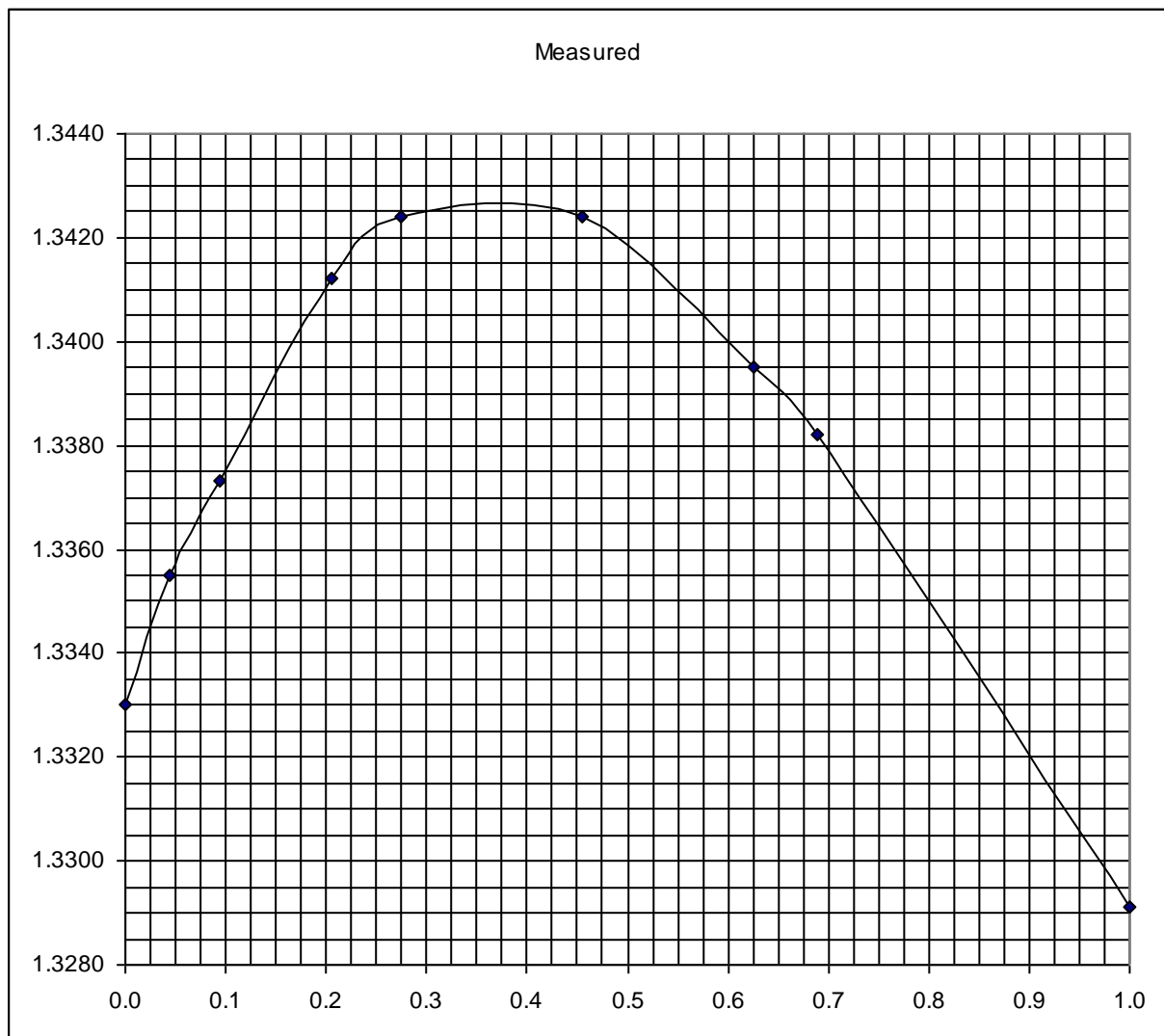
Reference: old work instructions

H

Components	A=MeOH	MA=	0.032kg/mol
	B=vesi	MB=	0.018kg/mol

Device	ap=	256.0000m2/m3	D=	0.1560m
	dp=	0.0254m	A=	0.0191m2

Transfer coefficients

	liquid		vapor				
	top	bottom	top	bottom			
T=	70.00	90.00	70.00	90.00 C	**measured**	temperature
x=	0.80	0.02	0.80	0.02	**measured**	molar fraction
M=	0.0292	0.0183	0.0292	0.0183 kg/mol	**=x*MA+(1-x)*MB**	average molar mass
eta=	0.000406	0.000317	0.000011	0.000012 kg/m/s	**table value**	viscosity
roo=	978	965	0.198	0.424 kg/m3	**table value**	density
D=	5.00E-09	5.00E-09	2.00E-05	2.00E-05 m2/s	**assumed**	diffusion coefficient
Sc=	83.0	65.6	2.7	1.4	**=eta/roo/D**	Sc-number
n=	0.350000	0.350000	0.230000	0.230000 mol/s	**measured**	molar flow
m=	0.010220	0.006398	0.006716	0.004204 kg/s	**=n*M**	mass flow
V=	0.000010	0.000007	0.033919	0.009916 m3/s	**=M/roo**	volume flow
u=	0.000547	0.000347	1.775524	0.519062 m/s	**=V/A**	flow velocity
ONDA:						
Re=	5.15	4.13	127.15	74.11	**=roo*u/eta/ap**	Re-number
k=	0.000057	0.000051	0.026288	0.014317 m/s		mass transfer coefficient
H=	0.038	0.027	0.264	0.142 m	**=u/k/ap**	height of mass transfer unit

Assumptions

liquid phase	vapor phase
properties of water	**properties of steam**
D=5.0E-9	**D=2.0E-5**
a = ap	**a = ap**

Assumptions should hold for the system since diffusion coefficients are inaccurate.

Taylor&Krishna, Multicomponent Mass Transfer, p. 358, in the example

Onda-correlation	Fair&Bravo-correlation
Hx=0.062 m	**Hx=0.071 m**
Hy=0.349 m	**Hy=0.518 m**

ky   slope of connecting lines b = - kx/ky
Not equimolar   not NA=NB; Not z=infinite; operating line not a straight line in xy-coordinates
Jako-osien lukumäärä k=10   Dy=(ya-yb)/10

Equilibrium $$\alpha \equiv \frac{y(1-x)}{x(1-y)}$$

a=alpha= $7.836-12.222x+7.749x^2$

yeq(x)=ax/(1+(a-1)x)
xeq(y)=y/(a+(1-a)y)

---

z   **rA=**   35.3 kJ/mol
          rB=   40.6 kJ/mol
          z=   7.66

Use   Stripper: Transfer liquid -> vapor (when NA > 0). Operating line below the equilibrium line.

Vaya= 0.137   **Vbyb=** 0.002   **DC=**0.00
ya=   0.630   **yb=**   0.010

Va=   0.218   **z=** 7.66   **Vb=**   0.200   **DD=**-0.15
                      NA=0.14
La=   0.368   **NB=**-0.12   **Lb=**   0.350
xa=   0.377   **xb=**   0.010
Laxa= 0.139   **Lbxb=** 0.004

Mass transfer coefficients

kx=1
ky=1   **b = - kx/ky =** -1.000

Calculation   **Dy=**0.062

k	operating				interface			Ny			
	y	V	L	x	yi	xi	b'	z/(z-y)	1/fii	DNy	Ny
0	0.010	0.200	0.350	0.010							0.00
1	0.072	0.202	0.352	0.046	0.103	0.015	-1.000	1.009	1.012	-2.06	-2.06
2	0.134	0.203	0.353	0.081	0.186	0.030	-1.000	1.018	1.021	-1.25	-3.30
3	0.196	0.205	0.355	0.117	0.266	0.047	-1.000	1.026	1.031	-0.94	-4.24
4	0.258	0.207	0.357	0.154	0.343	0.069	-1.000	1.035	1.041	-0.79	-5.03
5	0.320	0.208	0.358	0.190	0.415	0.095	-1.000	1.044	1.050	-0.71	-5.74
6	0.382	0.210	0.360	0.227	0.482	0.127	-1.000	1.052	1.060	-0.69	-6.43
7	0.444	0.212	0.362	0.264	0.543	0.165	-1.000	1.062	1.069	-0.71	-7.14
8	0.506	0.214	0.364	0.302	0.598	0.209	-1.000	1.071	1.078	-0.78	-7.92
9	0.568	0.216	0.366	0.339	0.646	0.261	-1.000	1.080	1.086	-0.93	-8.85
10	0.630	0.218	0.368	0.377	0.689	0.319	-1.000	1.090	1.094	-1.26	-10.11

Ny= **10.11**

$$N_y \approx \sum_k \frac{z}{(z-y_k)} \frac{1}{\phi_{y,k}} \frac{\Delta y_k}{|y_k - y_{i,k}|}$$

$$\phi_{y,k} = \frac{(z-y_k)-(z-y_{i,k})}{z \ln \dfrac{z-y_k}{z-y_{i,k}}}$$

$$z = \frac{N_A}{\sum N_i} = \frac{N_A}{N_\Sigma}$$

7.10.2015

kx
Not equimolar
Number of subintervals, k=10

Slope of connecting lines b = - kx/ky
not NA=NB; not z=infinite; operating line not a straight line in xy-coordinates
Dx=(xa-xb)/10

Equilibrium

$$\alpha \equiv \frac{y(1-x)}{x(1-y)}$$

a=alpha=   7.836-12.222x+7.749x^2

yeq(x)=ax/(1+(a-1)x)
xeq(y)=y/(a+(1-a)y)

---

z
rA= 35.3 kJ/mol
rB= 40.6 kJ/mol
z= 7.66

Use   Stripper: transfer liquid -> vapor (when NA > 0). Operating line below the equilibrium line.

Vaya=	0.137		**Vbyb=**	0.002	**DC=0.00**
ya=	0.630		**yb=**	0.010	
Va=	0.218	**z=** 7.66	**Vb=**	0.200	**DD=-0.15**
		NA=0.14			
La=	0.368	**NB=-0.12**	**Lb=**	0.350	
xa=	0.377		**xb=**	0.010	
Laxa=	0.139		**Lbxb=**	0.004	
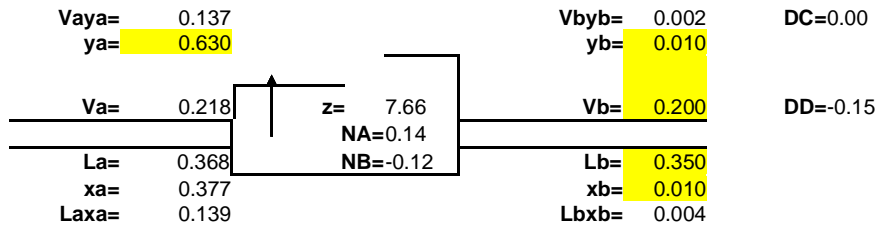
Mass transfer coefficients

kx=1
ky=1   **b = - kx/ky =** -1.000

Calculation   **Dx=0.037**

k	\(operating\)				\(interface\)			\(Nx\)			
	x	L	V	y	xi	yi	b'	z/(z-x)	1/fii	DNx	Nx
0	0.010	0.350	0.200	0.010							0.00
1	0.047	0.352	0.202	0.074	0.015	0.106	-1.000	1.006	1.004	1.18	1.18
2	0.083	0.353	0.203	0.138	0.031	0.190	-1.000	1.011	1.007	0.71	1.88
3	0.120	0.355	0.205	0.201	0.049	0.272	-1.000	1.016	1.011	0.53	2.41
4	0.157	0.357	0.207	0.263	0.071	0.349	-1.000	1.021	1.015	0.44	2.86
5	0.194	0.359	0.209	0.325	0.098	0.421	-1.000	1.026	1.019	0.40	3.26
6	0.230	0.360	0.210	0.387	0.130	0.488	-1.000	1.031	1.024	0.39	3.64
7	0.267	0.362	0.212	0.449	0.168	0.548	-1.000	1.036	1.029	0.39	4.04
8	0.304	0.364	0.214	0.509	0.212	0.601	-1.000	1.041	1.035	0.43	4.47
9	0.340	0.366	0.216	0.570	0.263	0.648	-1.000	1.046	1.041	0.51	4.99
10	0.377	0.368	0.218	0.630	0.319	0.689	-1.000	1.052	1.048	0.69	5.68
										Nx=	**5.68**
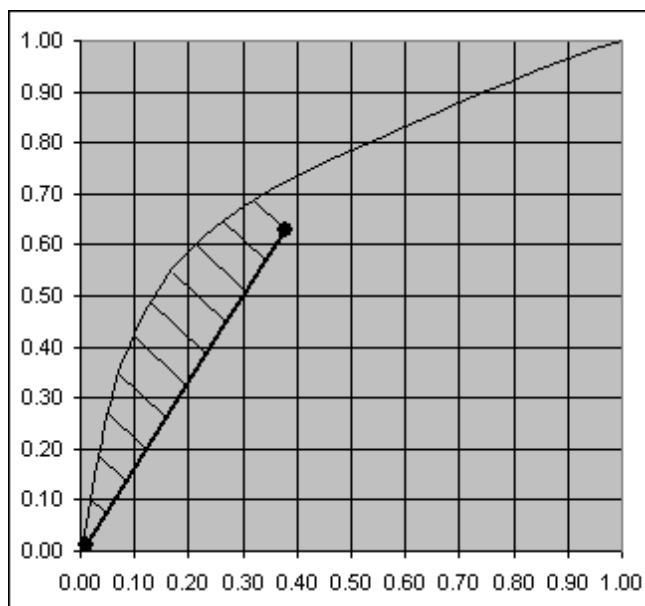
$$N_x \approx \sum_k \frac{z}{(z-x_k)} \frac{1}{\phi_{x,k}} \frac{\Delta x_k}{\left| x_k - x_{i,k} \right|}$$

$$\phi_{x,k} = \frac{(z-x_k)-(z-x_{i,k})}{z \ln \frac{z-x_k}{z-x_{i,k}}}$$

$$z = \frac{N_A}{\sum N_i} = \frac{N_A}{N_\Sigma}$$

7.10.2015