

TKP4105/TKP4110

# Oldershaw perforated plate distillation

Øyvind Eraker, Kjetil Sonerud and Ove Øyås  
Group B04

Supervisor: Margrethe Nergaard

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# 1 Introduction

This experiment has been carried out as a part of the course *Separation Technology* (TKP4105) at NTNU, Trondheim, Norway.

In this laboratory exercise an 11 volume percent mixture of ethanol and water will be distilled by Oldershaw perforated plate distillation. The objective of the experiment is to determine the time necessary to reach steady-state conditions and to find the variation in column efficiency, i.e. the number of theoretical trays needed in the column, as a function of vapor velocity using the McCabe-Thiele method. Theory concerning the phenomena flooding and weeping will be discussed, and weeping will be investigated experimentally. The goal is to achieve a fundamental understanding of distillation as a mean for physical separation of components in a liquid mixture, an understanding that is essential for the many industrial applications of the method.

## 2 Theory

### 2.1 Flooding point

All countercurrent-flow separation devices have a limited capacity. Flooding is by far the most common upper capacity limit of a distillation tray [1]. Flooding occurs when an excessive amount of liquid accumulates inside the column as a result of high vapor velocity. At the flooding point, the force exerted by the vapor pressure becomes so great that it eventually balances the gravitational force acting on the liquid, resulting in a build-up of liquid inside the column [2]. The vapor velocity over which flooding occurs is referred to as the flooding point.

Flooding has a number of observable effects: The pressure rapidly drops as the liquid accumulates on the trays; the liquid carryover from the top of the column increases quickly; the bottom flow rate is decreased as the accumulating liquid fails to reach the bottom of the column; and the system is made unstable as accumulation disturbs the equilibrium and steady-state conditions.

### 2.2 Weeping point

At steady-state conditions, the fluid in the column sieve trays is prevented from dropping down through the perforations by the vapor pressure of the boiling mixture being distilled. However, if the vapor flow gets too low, the liquid can no longer be held out of the perforations and will “weep” through them instead of overflowing over the downcomer [2]. This results in loss of contact between the vapor and the liquid, thereby lowering the efficiency of the column. The weep point is the gas velocity at which weeping first becomes noticeable [1].

## 2.3 McCabe-Thiele method

### 2.3.1 Introduction and assumptions

The McCabe-Thiele method is a widely used mathematical-graphical method for analysis of binary distillation by determination of the number of theoretical trays (or equilibrium stages) needed for a given separation [3]. It was presented by Warren L. McCabe and Ernest W. Thiele in 1925 [4]. The fundamental assumption of the method is *constant molar overflow* through the distillation column, eliminating the need for an energy balance [1, 3].

The composition at each theoretical tray is completely determined by the mole fraction of one of the two components. The number of theoretical trays may therefore be found from mass balances and vapor-liquid equilibrium (VLE) data for the lighter component alone.

### 2.3.2 Construction of the McCabe-Thiele diagram

Mass balances and VLE data for the lower boiling component allow the construction of a McCabe-Thiele diagram as exemplified in Fig. 12 on page 21. Naming the lower boiling feed component A, the horizontal axis represents  $x$ , the mole fraction of A in the liquid phase, and the vertical axis represents  $y$ , the mole fraction of A in the vapor phase. Plotting the VLE data gives the equilibrium line, relating the equilibrium vapor phase compositions to their corresponding liquid phase compositions.

The operating lines for the enriching and stripping sections are representations of the material balance equations and are straight as a consequence of the constant molar overflow assumption [1]. For the enriching section, the operating line is described by the equation

$$y_{n+1} = \frac{R}{R+1}x_n + \frac{x_D}{R+1} \quad (2.1)$$

where  $y_{n+1}$ ,  $x_n$  and  $x_D$  are the mole fractions of A in the vapor phase in tray  $n+1$ , in the liquid phase in tray  $n$  and in the liquid phase in the distillate respectively.  $R$  is the reflux ratio defined as  $R = L_n/D$  where  $L_n$  and  $D$  represent the reflux stream from tray  $n$  and the distillate stream respectively.

The stripping section operating line is given by

$$y_{m+1} = \frac{L_m}{V_{m+1}}x_m - \frac{Wx_W}{V_{m+1}} \quad (2.2)$$

where  $y_{m+1}$  is the mole fraction of A in the vapor phase in tray  $m+1$ ;  $V_{m+1}$  is the vapor flow from tray  $m+1$ ;  $L_m$  is the liquid flow from tray  $m$ ;  $W$  is the bottoms flow; and  $x_m$  and  $x_W$  are the mole fractions of A in the liquid phase in tray  $m$  and in the bottoms flow respectively.

The condition of the feed is represented by the quantity  $q$  which is defined as follows:

$$q = \frac{\text{heat needed to vaporize 1 mol of feed at entering conditions}}{\text{molar latent heat of vaporization of feed}} \quad (2.3)$$

If the feed enters at its boiling point,  $q = 1$ ; if the feed enters as vapor at the dew point,  $q = 0$  [3]. The  $q$ -line in a McCabe-Thiele diagram is the line with slope  $q/(q - 1)$  going through the intersection of the enriching and stripping operating lines as well as the  $x = y$  line at the feed composition  $x = x_F$ . It is described by the equation

$$y = \frac{q}{q - 1}x - \frac{x_F}{q - 1} \quad (2.4)$$

It can be seen that the  $q$ -line is horizontal at  $q = 0$  and vertical at  $q = 1$ . Examples of  $q$ -line slopes at various feed compositions are shown in Fig. 1.

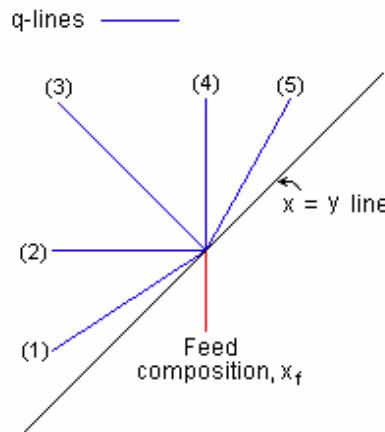


Figure 1:  $q$ -line slopes at various feed compositions: (1) superheated vapor feed, (2) saturated vapor feed, (3) partially vaporized feed, (4) saturated liquid feed and (5) subcooled liquid feed.

### 2.3.3 Determining the column efficiency at total reflux

To determine the number of theoretical trays needed in a column (and thereby the column efficiency), “steps” are made in the McCabe-Thiele diagram, starting at the top tray (distillate) where  $x = x_D$ . The steps consist of straight lines, alternately horizontal and vertical, going between the equilibrium line and the operating lines. Each horizontal line connects the liquid composition at the operating line of a tray to the equilibrium liquid composition of the tray below. Each vertical line connects the equilibrium vapor composition of a tray to the operating line vapor composition of the same tray. These steps represent the theoretical plates (or equilibrium stages) necessary to achieve a specific degree of separation by distillation for a binary mixture.

It is now obvious that the number of theoretical trays needed in a binary distillation depends upon the operating lines, and, as can be seen from Eq. (2.1) and (2.2) (knowing that  $V_{n+1} = L_n + D$ ), the operating lines in turn depend on the reflux ratio,  $R = L_n/D$ . One of the limiting values of  $R$  is that of total reflux, or  $R = \infty$ , in which case the slope of the enriching operating line in Eq. (2.1) becomes 1 and the operating lines of both sections coincide with the  $45^\circ$  diagonal line  $x = y$  [3]. Thus, at total reflux, the steps in the McCabe-Thiele diagram can be constructed between the equilibrium line and the  $x = y$  line.

## 2.4 Gas chromatography

Gas chromatography (GC), is a common type of chromatography used in analytical chemistry for separating and analysing compounds that can be vaporized without decomposition. Typical uses of GC include testing the purity of a particular substance, or separating the different components of a mixture. In some situations, GC may help in identifying a compound [5].

In GC, the mobile phase (or “moving phase”) is a carrier gas, usually an unreactive or inert gas such as helium or nitrogen. The stationary phase is a microscopic layer of liquid or polymer on an inert solid support, inside a piece of glass or metal tubing called a column. The instrument used to perform gas chromatography is called a gas chromatograph. Upon analysis, the gaseous compounds being analyzed interact with the column walls, which are coated with different stationary phases. This causes each compound to elute at a different time, known as the retention time of the compound, comparison of which makes GC useful for analysis.

A GC can also be used for quantitative analysis through the use of a detector. In this experiment (with the Thermo Scientific FOCUS GC Gas Chromatograph) a FID-detector is used, as shown in Fig. 2. The basis of such a flame ionization detector is that the electrical resistance of the flame is very much affected by traces of organic compounds, although the precise mechanism is quite complex [6]. By computer-aided calculations, the amount of the compound involved can be found - in this experiment given as a volumetric fraction of ethanol.

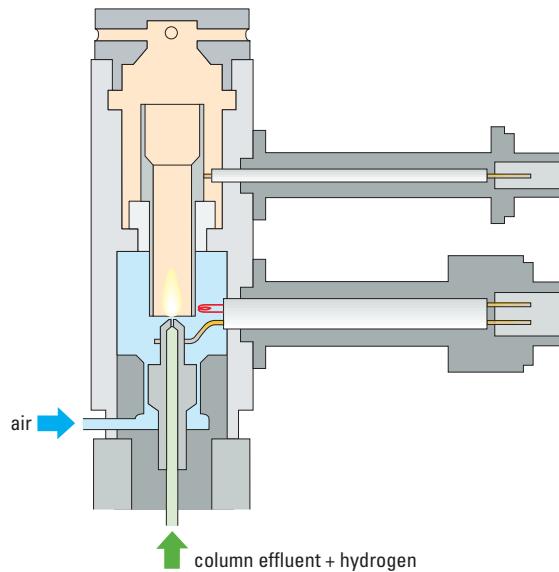


Figure 2: The figure shows the FID-unit of a Thermo Scientific FOCUS GC Gas Chromatograph [7]

## 3 Experimental

### 3.1 Preparing the equipment

- The PC and cooling water were turned on with the bottom and distillate tap lines closed.
- The oldershaw perforated plate column was filled with 5800 ml 11% ethanol (by volume).
- The main power cables were plugged in and the column was set to total reflux.
- The control box of the heater was turned on. The power was first set to 90% and the temperature to 105 °C.
- When the temperature or the pressure changed substantially, the power was set to 50%.
- The timing started when the vapour stream started condensing.
- The laboratory exercise was divided into two exercises, part 1 and 2.

### 3.2 Exercise 1: Time required to reach steady state condition

- Samples were taken from the top of the column every 5 minutes for an hour, so that 12 samples were taken in total. After the 12th sample was taken, a last sample was taken for part two.
- All the samples were analyzed in the gas chromatograph. The system had reached steady state when the composition of the samples was approximately constant.

### 3.3 Excercise 2: Efficiency vs. vapor velocity

- The power was switched from 50% to an arbitrary power, and the column was left alone until it had reached steady-state (approximately the same time as in part 1).
- Samples were taken from the top and the bottom, each sample containing 20 ml.
- The samples were analyzed, and the efficiency of the column and the theoretical number of stages were calculated, the latter using a McCabe-Thiele diagram.
- The vapor velocity was calculated.
- Part 2 was performed several times, until 5 different velocities for 5 different powers were obtained.
- At the end, the flooding and weeping points were investigated. The approximative power level at the weeping point was recorded.
- The equipment was turned off after the last sampling, and the column was emptied.

## 4 Results

Fig. 3 shows the volume fraction of ethanol in the distillate plotted against time at a power level of 50%. Fig. 4 - 8 show the McCabe-Thiele diagrams for the different power levels used in the experiment, starting with a power level of 25%.

Fig. 9 shows how the vapor velocity changes as the power level is increased from 25% to 80%. Furthermore, Fig. 10 shows how the column efficiency is related to the vapor velocity.

A sensitivity analysis of the McCabe-Thiele method is shown in Fig. 11. Here, the number of theoretical trays is plotted against mole fraction of ethanol in the distillate in order to investigate how sensitive the method is to changes in the distillate composition.

The approximative power level at the observed weeping point was 18%.

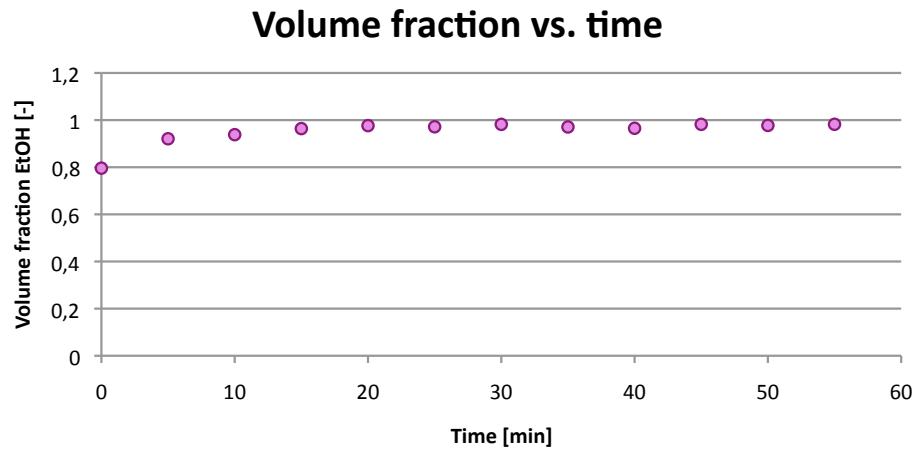


Figure 3: Volume fraction of ethanol in the distillate plotted against time. The power duty is 50%.

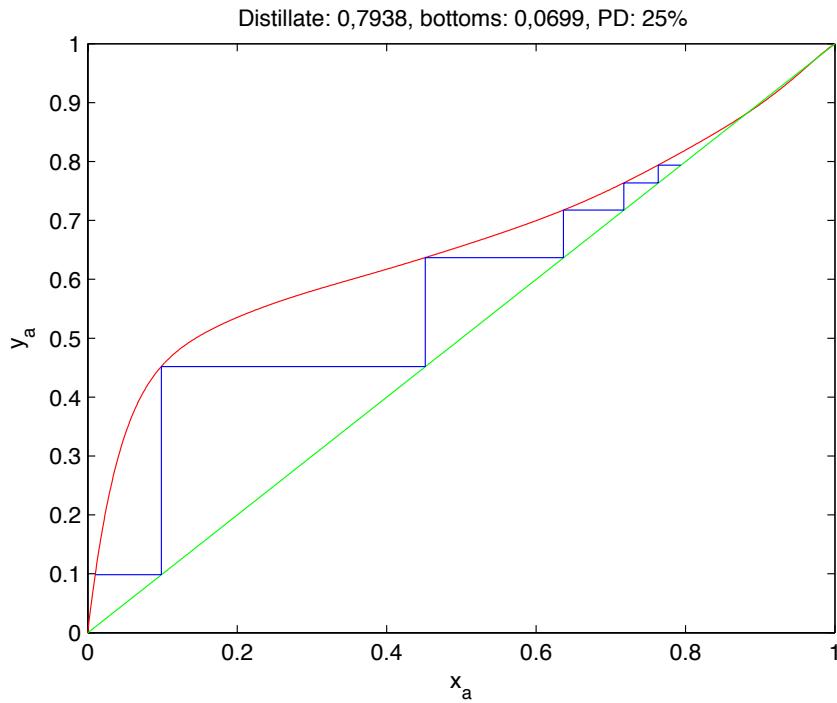


Figure 4: McCabe-Thiele diagram for a power duty of 25%. Here,  $x_a$  is the mole fraction of ethanol in the liquid phase and  $y_a$  is the mole fraction of ethanol in the gas phase. The theoretical number of steps is 6.

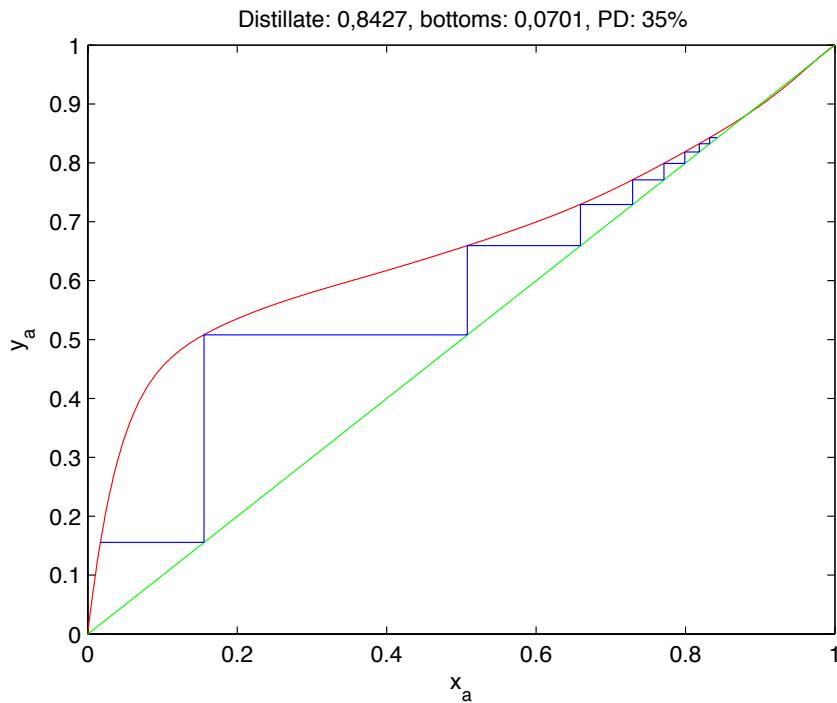


Figure 5: McCabe-Thiele diagram for a power duty of 35%. Here,  $x_a$  is the mole fraction of ethanol in the liquid phase and  $y_a$  is the mole fraction of ethanol in the gas phase. The theoretical number of steps is 9.

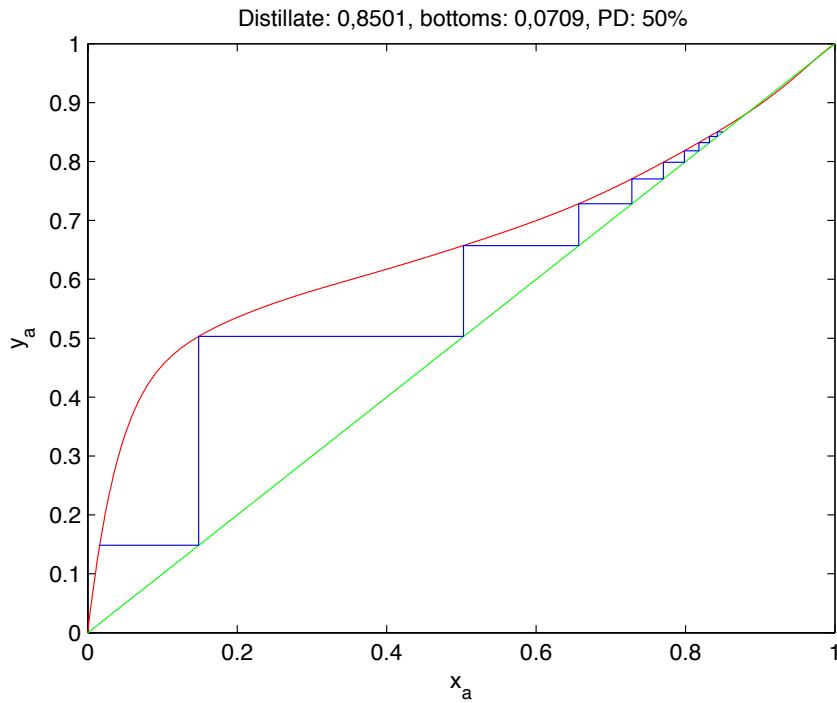


Figure 6: McCabe-Thiele diagram for a power duty of 50%. Here,  $x_a$  is the mole fraction of ethanol in the liquid phase and  $y_a$  is the mole fraction of ethanol in the gas phase. The theoretical number of steps is 10.

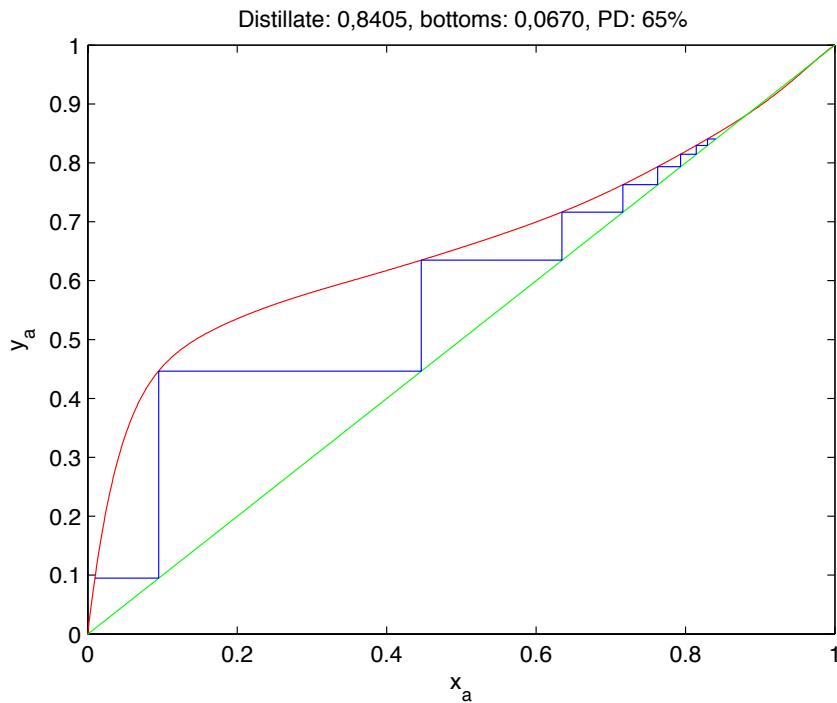


Figure 7: McCabe-Thiele diagram for a power duty of 65%. Here,  $x_a$  is the mole fraction of ethanol in the liquid phase and  $y_a$  is the mole fraction of ethanol in the gas phase. The theoretical number of steps is 9.

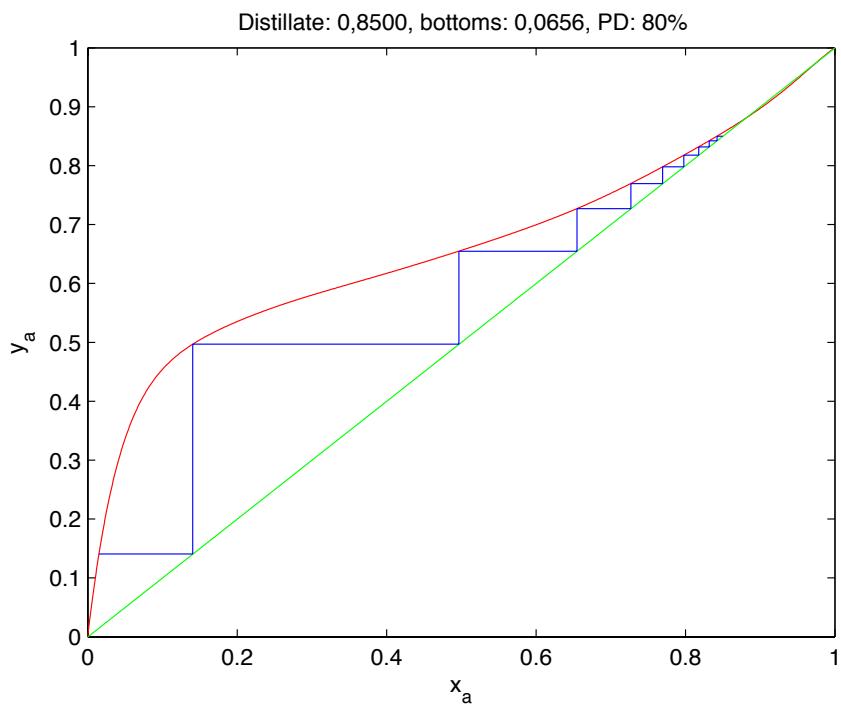


Figure 8: McCabe-Thiele diagram for a power duty of 80%. Here,  $x_a$  is the mole fraction of ethanol in the liquid phase and  $y_a$  is the mole fraction of ethanol in the gas phase. The theoretical number of steps is 10.

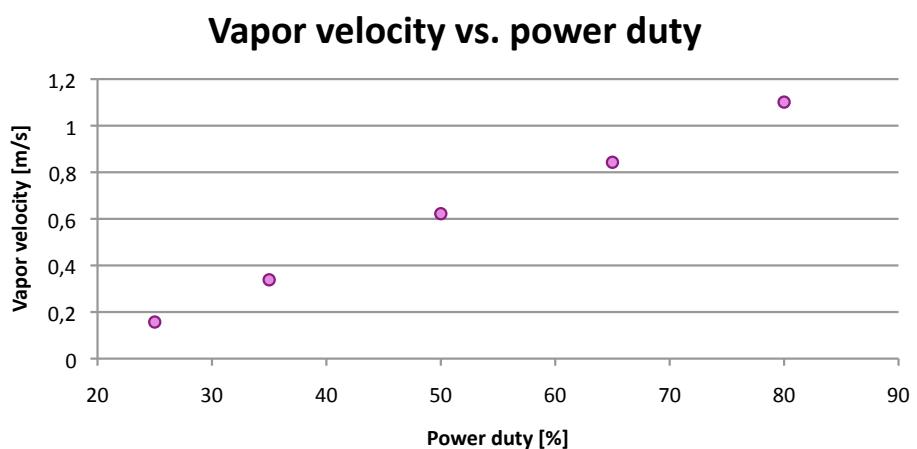


Figure 9: Vapor velocity plotted against power duty.

### Column efficiency vs. vapor velocity

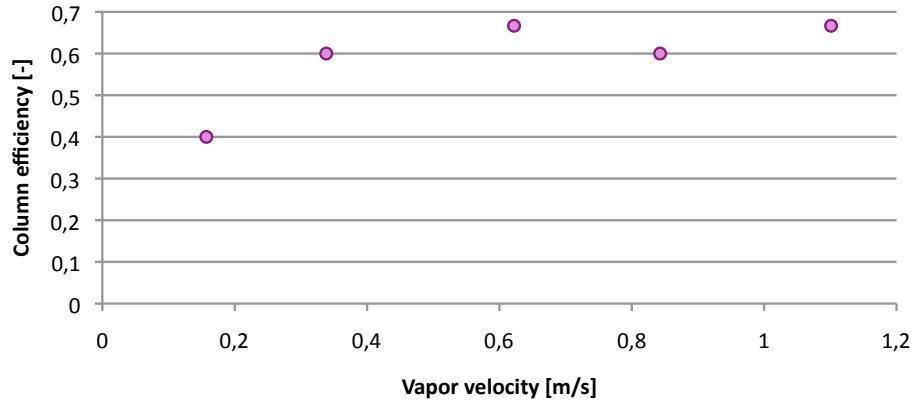


Figure 10: Vapor velocity plotted against column efficiency.

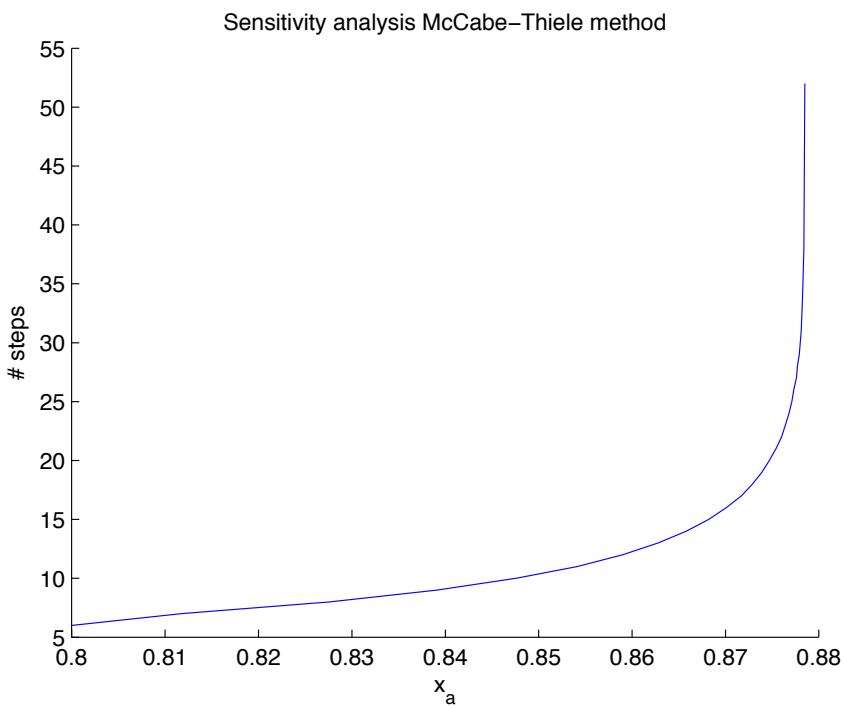


Figure 11: A sensitivity analysis of the McCabe-Thiele method. The number of theoretical steps is plotted against  $x_a$ , the mole fraction of ethanol in the distillate.

## 5 Discussion

The time the column needs to achieve an approximately steady-state condition can be seen from Fig. 3. The concentration stabilizes after about 20 minutes. For the last 35 minutes the concentration of the distillate is approximately constant, which means that equilibrium has been reached in each tray. As long as the system is run with total reflux at the same power duty, the equilibria will not change, so the concentration of the distillate will remain constant.

According to the results gained by the gas chromatograph, most of the volume fractions of the distillates are above 96 vol%. This is not possible, since the azeotrope of a mixture of ethanol and water is at 96 vol%, so the results are obviously deviating from what is theoretically possible. Reasons for this may be errors in the measurement of the solution when preparing samples for the gas chromatograph, as well as errors in the actual gas chromatograph that may be a result of bad calibration of the equipment. When calculating the column efficiency at different power duties of the reboiler, the theoretical number of trays needed for the distillation was calculated by using a McCabe-Thiele diagram. When using the McCabe-Thiele diagram the concentrations of the distillate gained from the gas chromatograph could not be used, since these concentrations were above the azeotropic point in the diagram. In agreement with the supervisor, the McCabe-Thiele diagrams were made with an upper molar fraction of 0,85 mol % ethanol for the distillate. This was accomplished by scaling down all of the results by 10 %. In this way, all the results are still relatively correct to each other, so it is possible to compare them in a plot. However, the actual numbers for the column efficiency for each power duty is not correct, as the results are scaled. From Fig. 11 it is obvious that it is a big difference if the upper molar fraction is scaled to 0,850 mol%, which is equivalent to 10 theoretical steps, or to 0,879 mol%, which is equivalent to 52 theoretical steps. This affects all of the results, even the relative. If the results are scaled to an area where the sensitivity is lower than it is supposed to be, the number of steps for each power duty will be too close to each other since the number of steps is an integer (and not a decimal). It is impossible to know exactly what the right scaling factor is, but from Fig. 11 it seems that the choice of a 10% scaling is reasonable.

During the first 60 minutes, 24 samples of the distillate were taken. Because these samples were of high concentration of ethanol, this may have altered the total concentration of ethanol in the column. Another source of error is that the liquid in the bottom of the column weren't totally homogeneous when the samples were taken. This may have affected the samples.

Weeping was first observed at an approximate power duty of 18%. There was no observed loss of column efficiency at this point, which is in agreement with the theory [1]. Flooding was not observed during the experiment, probably because the power duty was too low (the maximum power duty used was 80%).

A trend can be seen from Fig. 9. When the power duty is increased, the vapor velocity increases. This makes sense from a physical point of view, because when more heat is added, more of the liquid will be evaporated. This results in more vapor, and in order to maintain the steady state there is an increase in vapor velocity.

From Fig. 10 it appears that the results are unambiguous, and there is no certain trend. Therefore, it is difficult to draw any conclusions regarding whether the vapor velocity has any influence on the column efficiency or not based on these results.

## 6 Conclusion

The time needed to reach steady-state at 50% power duty was approximately 20 minutes. The vapor velocity was observed to increase with increased power duty, as expected. The plot of column efficiency versus vapor velocity yields unambiguous results, and no conclusion about the relationship between the two variables can be drawn from it.

Trondheim, November 11, 2011.

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Øyvind Eraker

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Kjetil Sonerud

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Ove Øyås

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## A Calculations

### A.1 Calculating the required amount of ethanol

For the Oldershaw column, it is given [2] that the amount of ethanol/water mixture should be 5800 mL in total. This mixture should contain 11 mol% of ethanol. The ethanol that is provided is a 96 volume% mixture of ethanol and water.

The mole fraction of ethanol,  $x_{EtOH}$ , is given by

$$x_{EtOH} = \frac{n_{EtOH}}{n_{tot}} = \frac{n_{EtOH}}{n_{EtOH} + n_{H_2O}} \quad (\text{A.1})$$

The number of moles of ethanol is given by

$$n_{EtOH} = \frac{V_{EtOH}\rho_{EtOH}}{M_{EtOH}} \quad (\text{A.2})$$

where  $\rho_{EtOH} = 0,78934$  g/mol is the density of pure ethanol [1]. Likewise, the number of moles of water is given by

$$n_{H_2O} = \frac{V_{H_2O}\rho_{H_2O}}{M_{H_2O}} = \frac{(V_{tot} - V_{EtOH})\rho_{H_2O}}{M_{H_2O}} \quad (\text{A.3})$$

where  $\rho_{H_2O} = 0,9970$  g/mol is the density of pure water [10]

Combining Eq. A.1, A.2 and A.3 yield

$$x_{EtOH} = 0.11 = \frac{\frac{V_{EtOH}\rho_{EtOH}}{M_{EtOH}}}{\frac{V_{EtOH}\rho_{EtOH}}{M_{EtOH}} + \frac{(V_{tot} - V_{EtOH})\rho_{H_2O}}{M_{H_2O}}} \quad (\text{A.4})$$

which can be solved for  $V_{EtOH}$  using WolframAlpha. With numbers inserted, the result is

$$V_{EtOH} = 1655 \text{ mL} \quad (\text{A.5})$$

which is the amount of pure ethanol needed. This is equal to

$$V_{EtOH,96\%} = \frac{1655}{0.96} = 1724 \text{ mL} \quad (\text{A.6})$$

The volume of water needed is therefore

$$V_{H_2O} = (V_{tot} - V_{EtOH}) = (5800 - 1724) \text{ mL} = 4076 \text{ mL} \quad (\text{A.7})$$

## A.2 Calculation the vapor velocity from the GC results

From the GC results the composition of the samples is known - more specific, the volume fraction of ethanol,  $x_{v,EtOH}$ , is known. The total volume flow in mL/min,  $q_{tot}$ , is also known from the pump level.

The mass flow of ethanol,  $\dot{m}_{EtOH}$ , can be calculated from the volume flow of ethanol,  $q_{EtOH}$ .

$$x_{v,EtOH} q_{tot} = q_{EtOH} \implies \dot{m}_{EtOH} = x_{v,EtOH} q_{tot} \rho_{EtOH} \quad (\text{A.8})$$

where  $\rho_{EtOH}$  is the density of pure ethanol. The mole flow of ethanol,  $\dot{n}_{EtOH}$ , can then be found

$$\dot{n}_{EtOH} = \frac{\dot{m}_{EtOH}}{M_{EtOH}} \quad (\text{A.9})$$

where  $M_{EtOH} = 46,07$  g/mol is the molar mass of ethanol. The same approach can be used to find the mole flow of water,  $\dot{n}_{H_2O}$ . Here, the volume fraction of water is given as  $x_{v,H_2O} = (1 - x_{v,EtOH})$ . The total mole flow,  $\dot{n}_{tot}$ , can then be found from the mass balance

$$\dot{n}_{tot} = \dot{n}_{EtOH} + \dot{n}_{H_2O} \quad (\text{A.10})$$

By assuming that the ideal gas law is a valid approximation, inserting the total mole flow gives the total gas flow,  $\dot{V}_g$

$$\dot{V}_g = \frac{\dot{n}_{tot} RT}{P} \quad (\text{A.11})$$

where  $T$  is the absolute temperature and  $R$  is the universal gas constant. Dividing by the cross section area of the column gives the vapor velocity in m/s

$$v_g = \frac{V_g}{60 \cdot 1000 \cdot A} = \frac{V_g}{60 \cdot 1000 \cdot \frac{\pi}{4} 0,07^2} \quad (\text{A.12})$$

For the purpose of plotting the McCabe-Thiele diagram, the mole fraction of ethanol,  $x_{EtOH} = \frac{\dot{n}_{EtOH}}{\dot{n}_{tot}}$ , can be found by combining Eq. (A.10) and Eq. (A.9).

### A.3 Assignment calculations

Table 1: The given data for the assignment.

Data	Symbol	Value
Weight of empty pycnometer 1	$W_1$	11.98170 g
Weight of empty pycnometer 2	$W_2$	11.77385 g
Weight of pycnometer 1 with water	$W_{1,H_2O}$	16.95955 g
Weight of pycnometer 2 with water	$W_{2,H_2O}$	16.75745 g
Weight of pycnometer 1 with distillate	$W_{1,dist}$	16.0903 g
Weight of pycnometer 2 with bottom product	$W_{2,bp}$	16.6962 g
Amount of distillate	$V_{dist}$	20 mL
Time taken to collect the distillate	$t$	45 s
Reflux ratio	R	50%
Density of water at 25°C [10]	$\rho_{H_2O}$	0,9970 g/cm <sup>3</sup>

Data for the assignment is given in Table 1. From these data the volume of the pycnometers can be calculated from

$$V_i = \frac{W_{i,H_2O} - W_i}{\rho_{H_2O}} \quad (\text{A.13})$$

With numbers from Table 1 inserted, the results are

$$V_1 = 4,993 \text{ cm}^3 , \quad V_2 = 4,999 \text{ cm}^3 \quad (\text{A.14})$$

The densities of the samples can then be calculated from

$$\rho_{sample} = \frac{W_{i,sample} - W_i}{V_i} \quad (\text{A.15})$$

With numbers from Table 1 inserted, the results are

$$\rho_1 = 0,8229 \text{ g/cm}^3 , \quad \rho_2 = 0,9847 \text{ g/cm}^3 \quad (\text{A.16})$$

Using data from Green and Perry [1], the weight percent of ethanol in the two samples,  $w\%_{dist}$  for distillate and  $w\%_{bp}$  for bottom product, can be found. The results are

$$w\%_{dist} = 88,1\% \text{ ethanol} \quad , \quad w\%_{bp} = 8,0\% \text{ ethanol} \quad (\text{A.17})$$

The mole fraction for a binary mixture is defined as

$$x_a = \frac{n_a}{n_{tot}} = \frac{n_a}{n_a + n_b} \quad (\text{A.18})$$

This can be rewritten as

$$x_a = \frac{\frac{m_a}{M_a}}{\frac{m_a}{M_a} + \frac{m_b}{M_b}} = \frac{\frac{w\%m_{tot}}{M_a}}{\frac{w\%m_{tot}}{M_a} + \frac{(100-w\%)m_{tot}}{M_b}} \quad (\text{A.19})$$

The total mass,  $m_{tot}$  can be eliminated and the result is

$$x_a = \frac{\frac{w\%}{M_a}}{\frac{w\%}{M_a} + \frac{(100-w\%)}{M_b}} \quad (\text{A.20})$$

The mole fraction of ethanol in the samples can now easily be calculated from Eq. A.20. Inserting numbers from Table 1, and the values  $M_{H_2O} = 18,02 \text{ g/mol}$  and  $M_{EtOH} = 46,07 \text{ g/mol}$ , it is found that

$$x_{EtOH,dist} = 0,7433 \quad , \quad x_{EtOH,bp} = 0,0329 \quad (\text{A.21})$$

The vapor velocity,  $v$ , can be calculated from Eq. A.22

$$v = \frac{V_{gas}}{A \cdot t} \quad (\text{A.22})$$

where  $A$  is the cross section area of column,  $t$  is the time taken to gather the sample and  $V_{gas}$  is the volume that the liquid sample would have in the gas state. This volume can be calculated, assuming that the ideal gas law can be applied, from

$$V_{gas} = \frac{n_{gas}RT}{P} \quad (\text{A.23})$$

where  $R$  is the universal gas constant,  $T$  is the absolute temperature and  $P$  is the pressure. Assuming that the pressure in the column is atmospheric and that the average temperature is  $T = 80^\circ\text{C} = 253,15\text{K}$ , the approximative gas volume can be calculated from Eq. A.23.

In general, the total mass balance for a mixture of two components,  $a$  and  $b$ , gives

$$m_a + m_b = m_{tot} \quad (\text{A.24})$$

Introducing mole fractions, the number of moles of each component and the molar masses of each component, the mass balance can be rewritten as

$$x_a n_{tot} M_a + x_b n_{tot} M_b = m_{tot} \quad (\text{A.25})$$

where  $n_{tot}$  is the total number of moles in the sample,  $x_a$  and  $x_b$  are the mole fraction of components  $a$  and  $b$  and  $M_a$  and  $M_b$  are the molar masses of component  $a$  and  $b$ . Inserting  $x_b = 1 - x_a$  for a binary mixture, the equation can be written as

$$x_a n_{tot} M_a + (1 - x_a) n_{tot} M_b = m_{tot} \quad (\text{A.26})$$

This leads to a general equation for the total number of moles in a binary mixture

$$n_{tot} = \frac{m_{tot}}{x_a M_a + (1 - x_a) M_b} \quad (\text{A.27})$$

The total number of moles in the sample is found from combining Eq. A.27 with  $m_{tot} = \frac{\rho_{tot} V_{tot}}{f_R}$ , so that

$$n_{tot} = \frac{\frac{V_{tot}}{f_R} \rho_{tot}}{x_a M_a + (1 - x_a) M_b} \quad (\text{A.28})$$

where  $f_R = 0, 5$  is the reflux ratio. With numbers inserted, this yields

$$n_{tot} = 0, 8468 \text{ mol} \quad (\text{A.29})$$

From Eq. A.23 it is then found that

$$V_{gas} = 0, 02454 \text{ m}^3 \quad (\text{A.30})$$

The numbers are inserted into Eq. A.22, yielding

$$v = \frac{V_{gas}}{\frac{\pi}{4} D^2 \cdot t} = \frac{0, 02454}{\frac{\pi}{4} 0, 07^2 \cdot 45} \approx 0, 14 \text{ m/s} \quad (\text{A.31})$$

Using the McCabe-Thiele plotting tool written in MATLAB (see Appendix B), the plot seen in Fig. 12 was generated for the composition given in Eq. A.21. From this figure it is observed that the estimated number of ideal stages is 5.

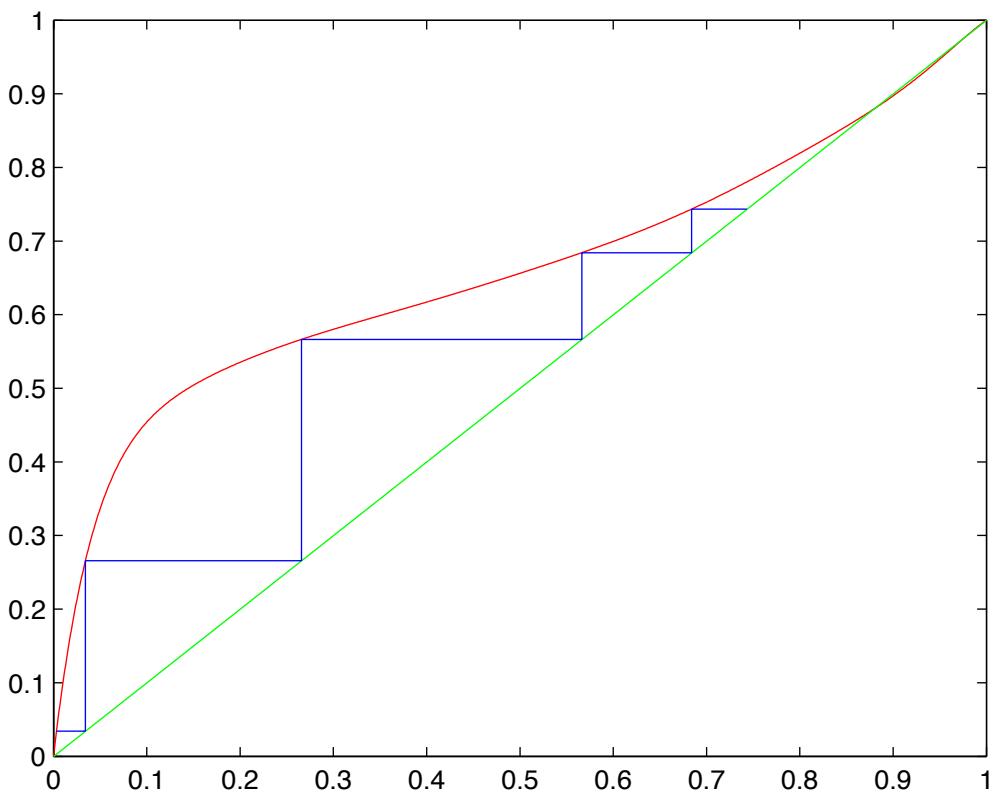


Figure 12: McCabe-Thiele plot for the system where  $x_{EtOH,dist} = 0,7433$  and  $x_{EtOH,bp} = 0,0329$ .

## B Laboratory safety

Chemical cards for the compounds used in this experiment as well as apparatus cards for the equipment are included below. The information in the chemical cards was found using the NTNU EcoOnline library. A risk evaluation form for the experiment is also included.

## Kjemikaliekort

	STOFFNAVN Etanol					FORMEL <chem>C2H5OH</chem>	
							HELSEFARE Ingen utpreget helsefare.
FYSIKALSKEDATA	Sm.p./fr.p.	Kokep.	Fl. p.	Molar masse	Ekspl.gr.		BRANNFARE Meget brannfarlig
	-117 °C	79 °C	425 °C	46,07 g/mol	3-19%		REAKSJONS FARE
VERNETILTAK	Alt arbeide med farlige kjemikalier skal utføres i avtrekkskap eller i godt ventilert og godkjent rom. Nøddusj og mulighet for øyeskylling skal finnes på arbeidsplassen.  Skift forurensede klær. Vask hender og ansikt etter arbeid med produktet. Åndedrettsvern (type A) skal anvendes ved forekomst av damper/aerosoler. Ved risiko for direkte kontakt eller sprut skal øyebeskyttelse benyttes. Bruk ikke kontaktlinser ved håndtering. Vernehansker skal benyttes ved fare for direkte kontakt og sprut.					ADMINISTRATIV NORM	
HELSEFARE	Innånding	Lett irriterende på slimhinnene i nese og øvre luftveierRisiko for absorpsjon.					BRANNSLUKKING Slukkes med pulver, skum, karbondioksid eller vann.
	Svelging	Brekninger. Kvalme. Absorpsjon av toksiske kvantiteter kan gi: Symptom som ved alkoholpåvirkning. Eufori.					MERKNADER
	Hud	Svimmelhet. Bevisstløshet. Åndedrettstopp.					
	Øyne	Lett irritasjon. Langvarig hudkontakt kan avfette huden og fremkalte hudbetennelse.Hudsprekker.					
FØRSTEHJELP	<b>ØYNE</b> Skyll godt med rennende vann.Hvis øyeirritasjonen vedvarer skal en gå til spesialist.				<b>SVELGING</b> Drikk mye vann. Kontakt lege. Gi aktivt kull (20-40g 10% slurry) om mulig. Fremkall ikke brekning!		
	<b>HUD</b> Ta av forurenset toy. Skyll huden med mye vann.				<b>INNÅNDING</b> Frisk luft og hvile. Gi kunstig åndedrett ved behov. Kontakt lege ved vedvarende symptomer.		

SPESIELLE MERKNADER	
SPILL/ RESTER	Destrueres etter lokale forskrifter. Håndter forurenset forpakning på samme måte som substansen. Klassifisert som farlig avfall. Forhindre utslipp til kloakk: Eksplosjonsrisiko!
LAGRING	Forpakningen oppbevares lukket og tørt ved romtemperatur (+15 °C til +25 °C). Holdes vekk fra antennelseskilder.



## APPARATURKORT

Fakultet for naturvitenskap og teknologi

### FYLLES UT MED BLOKKBOKSTAVER

**FORSØK/APPARATUR:** Oldershaw perforated plate distillation column.....

**STOFF:** Etanol..... **MENGDE:** 4076 mL.....

.....  
.....

**FAREMOMENTER:** Meget brannfarlig.....

.....

**MAKS. TID FOR DRIFT UTEN TILSYN:** .....

**BEGRENSINGER I ARBEIDSTID:** .....

**PÅBUDT VERNEUTSTYR:** Vernebriller og -hansker.....

**ANDRE VERNETILTAK:** .....

**ANDRE OPPLYSNINGER:** .....

.....

**HANDLEMÅTE VED NØDSTOPP:** .....

.....

.....

**VEILEDER:** Margrethe Nergaard..... **TLF.NR.:** ..... **SIGN.:** .....

**APP. ANSVARLIG:** ..... **TLF.NR.:** ..... **SIGN.:** .....

**OPERATØR:** ..... **TLF.NR.:** ..... **SIGN.:** .....

**GYLDIG FRA DATO:** ..... **TIL DATO:** .....

NTNU	Risikovurdering	utarbeidet av	Nummer	Dato	
		HMS-avd.	HMSRV2603	04.02.2011	
HMS/KS		godkjent av	side	Erstatter	
		Rektor	1 av 2	9.2.2010	

**Enhet:** Institutt for kjemisk prosessteknologi

**Dato:** 19.10.11

**Linjeleder:** Øyvind Gregersen

**Deltakere ved risikovurderingen (m/ funksjon):** Øyvind Eraker, Kjetil Sonerud og Ove Øyås (Felleslab, gr. B04)

ID nr	Aktivitet fra kartleggings-skjemaet	Mulig uønsket hendelse/ belastning	Vurdering av sannsynlighet (1-5)	Vurdering av konsekvens:				Risiko-verdi	Kommentarer/status Forslag til tiltak
				Menneske (A-E)	Ytre miljø (A-E)	Øk/materiell (A-E)	Omdømme (A-E)		
1	Destillasjon av etanol	Brann	2	C	A	C	C	C2	Ha brannslukningsutstyr lett tilgjengelig. Unngå åpne flammer.
2	Destillasjon av etanol	Eksplosjon	1	C	A	C	C	C1	Sjekke at utstyret er tett. Unngå åpne flammer.
3	Destillasjon av etanol	Forgiftning	1	A	A	A	A	A1	Unngå innånding av damp og annen eksponering for etanol.
4	Gasskromatografi	Gasslekkasje	3	C	B	B	B	C3	Sjekke at utstyret er tett.

**Sannsynlighet**

1. Svært liten
2. Liten
3. Middels
4. Stor
5. Svært stor

**Konsekvens**

- A. Svært liten
- B. Liten
- C. Moderat
- D. Alvorlig
- E. Svært alvorlig

**Risikoverdi (beregnes hver for seg):**

**Menneske = Sannsynlighet x Konsekvens Menneske**

**Ytre miljø = Sannsynlighet x Konsekvens Ytre miljø**

**Økonomi/materiell = Sannsynlighet x Konsekvens Øk/matriell**

**Omdømme = Sannsynlighet x Konsekvens Omdømme**

# Oldershaw distillation

[min]	t	pump level [mL/min]
0		-
5		68,8
10		81,4
15		74,6
20		76,4
25		78,4
30		83,0
35		74,7
40		71,6
45		80,3
50		81,1
55		82,5

heat	[mL/min]
	pump
35%	44,6
25%	19,9
65%	110,9
80%	146,0

Order of samples for GC : 1 - 12, B1, 35B, 35D, 25B, 25D,  
 18B, 18D, 65B, 65D, 80B, 80D

# McCabe-Thiele Plotting Tool

Use this plotting tool to make a McCabe-Thiele plot for a binary mixture with total reflux

By: Øyvind Eraker & Kjetil Sonerud - 20.10.2011

## Contents

- [Without empirical data](#)
- [Getting the empirical data and fitting the curve](#)
- [Plotting the curves](#)
- [Making the steps](#)

### Without empirical data

The Sigurd equation is used for the equilibrium line

```
clear all;
close all;

% Equilibrium curve computation using function equilib
% and constant relative volatility alpha

% Comment out if empirical data exist

% steps = 20;
%
% for i=1:steps
%     y=((1/(steps-1))*(i-1));
%     ye(i)=(1/(steps-1))*(i-1);
%     xe(i)=fzero('equilib',0.5,[],y);
% end
```

### Getting the empirical data and fitting the curve

Empirical data from:

Gmehling, J. and Onken, U., 1977. Vapor-Liquid Equilibrium Data Collection: Aqueous-Organic Systems. Vol. 1, Part 1. Germany: Dechema.

We use the MATLAB function `polyfit` to find a polynom of size n that matches the data, and the function `polyval` to plot these values over the range defined by `x_poly`

```
xe_e = [0:0.05:1];
ye_e = [0 0.3372 0.4521 0.5056 0.5359 0.5589 0.5794 0.5987 0.6177 0.6371 0.6558 0.6765 0
.6986 0.725 0.755 0.784 0.8167 0.8591 0.8959 0.9474 1];

x_poly = 0:0.001:1;
n=10;

p = polyfit(xe_e,ye_e,n);
f = polyval(p,x_poly);
g =@(x,y)(p(1)*x^10+p(2)*x^9+p(3)*x^8+p(4)*x^7+p(5)*x^6+p(6)*x^5+p(7)*x^4+p(8)*x^3+p(9)*
x^2+p(10)*x^1 - y);
```

### Plotting the curves

Plotting the equilibrium line and the operating line

```

% equilibrium line
plot(x_poly,f,'r');
hold on;

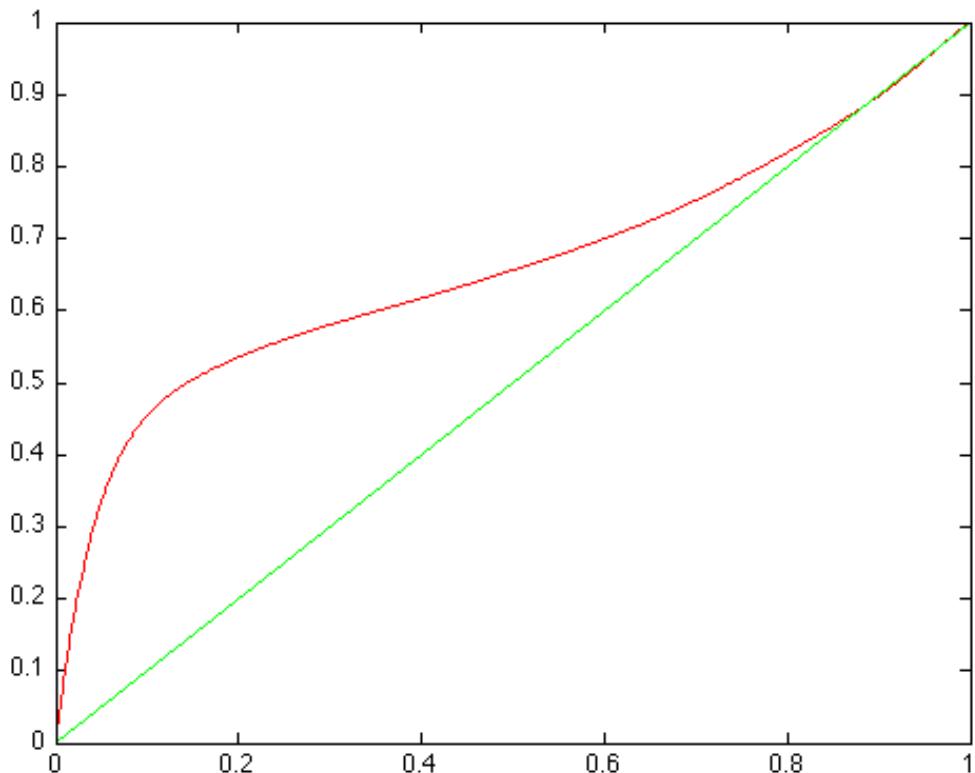
% operating line
set(line([0 1],[0 1]),'Color',[0 1 0]);

% distillate (|xd|) and bottom (|xb|) mole fractions are defined from
% experimental data

xd=0.7433;
xb=0.0329;

figure(1);
hold on;
axis([0 1 0 1]);

```



## Making the steps

The MATLAB function `set(line([x1 x2], [y1 y2]))` is used to make the steps.

```

i=1;
xp(1)=xd;
yp(1)=xd;
y=xd;
while (xp(i)>xb),
    xp(i+1)=fzero(g,0.5,[],y);
    set(line([xp(i) xp(i+1)],[yp(i) yp(i)]),'Color',[0 0 1]);

    yp(i+1)=xp(i+1);
    y=yp(i+1);

if (xp(i+1)>xb) set(line([xp(i+1) xp(i+1)],[yp(i) yp(i+1)]),'Color',[0 0 1]);
end

```

```

i=i+1;
end

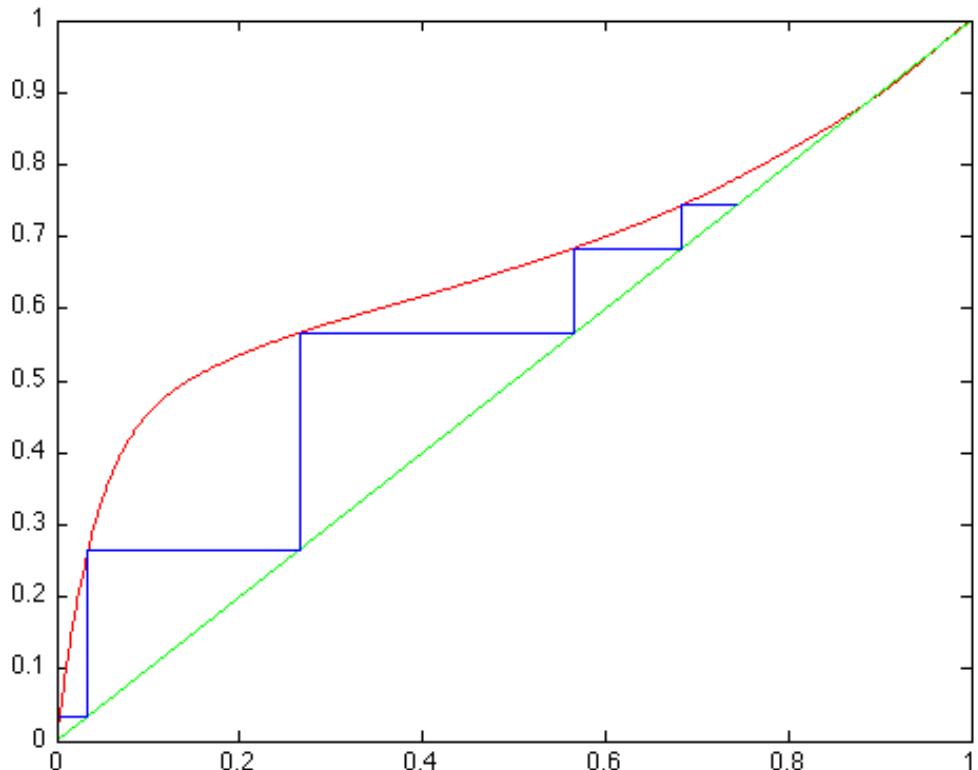
% Displaying the (integer) number of necessary steps.

disp('Number of steps:');
disp(i-1);

hold off;

```

Number of steps:  
5



# A sensitivity test for the McCabe-Thiele method

This script is used to get a graphical view of how the number of theoretical steps is affected when the concentration approaches the azeotropic point.

By: Kjetil Sonerud and Øyvind Eraker - 09.11.2011

## Contents

- [Making the operating line for McCabe-Thiele](#)
- [Making the steps](#)
- [The coordinate where the step number changes from the last step number are saved in a vector](#)
- [Plotting the sensitivity](#)

## Making the operating line for McCabe-Thiele

```
clear all;
close all;

% Empirical data are used to create a polynomial fit, used as an operating
% line

xe_e = [0:0.05:1];
ye_e = [0 0.3372 0.4521 0.5056 0.5359 0.5589 0.5794 0.5987 0.6177 0.6371 0.6558 0.6765 0
.6986 0.725 0.755 0.784 0.8167 0.8591 0.8959 0.9474 1];

x_poly = 0:0.001:1;
n=10;
p = polyfit(xe_e,ye_e,n);
f = polyval(p,x_poly);
g =@(x,y)(p(1)*x^10+p(2)*x^9+p(3)*x^8+p(4)*x^7+p(5)*x^6+p(6)*x^5+p(7)*x^4+p(8)*x^3+p(9)*
x^2+p(10)*x^1 - y);
```

## Making the steps

Here, the theoretical amount of steps are calculated.

```
j = 0;
steps_c = 0;
start = 0.80;
slutt = 0.8785;
for xd = start:0.0001:slutt

xb=0.07;
i=1;
xp(1)=xd;
yp(1)=xd;
y=xd;
while (xp(i)>xb),
    xp(i+1)=fzero(g,0.5,[],y);
    yp(i+1)=xp(i+1);
    y=yp(i+1);
    i=i+1;
end
```

**The coordinate where the step number changes from the last step number are saved in a vector**

```
if steps_c <(i-1)
    j = j + 1;
    frac(j)=xd;
    steps(j)=i-1;
end
steps_c = i - 1;
```

## Plotting the sensitivity

```
hold on
plot(frac,steps);
```

